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Structural and Residual Stress Analysis by Nondestructive Methods

Viktor Hauk

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Structural and Residual Stress Analysis by Nondestructive Methods

Evaluation – Application – Assessment

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Preface

The field of stress analysis has gained its momentum from the widespread applications in industry and technology and has now become an important part of materials science. Various destructive as well as nondestructive methods have been developed for the determination of stresses. The author of this book aimed at a comprehensive review of the nondestructive techniques for strain measurement and stress evaluation. Experts of the respective fields were recruited to contribute to this book.

For all the experimental methods described in this book, i.e. X-ray and neutron diffraction, ultrasonic and micromagnetic techniques, an explanation is given of the underlying physical principles, the instruments used, the data acquisition strategies and the data evaluation procedures. Representative results are given and compared with the results of mechanical testing methods, of theoretical investigations, and of finite-element calculations, although the calculation methods themselves are not dealt with. The bulk of the results refer to metallic materials; but ceramic, polymeric and composite materials are dealt with as well.

The largest part of this book is devoted to X-ray stress analysis (XSA). A special effort was made to deal with all those cases where the simple $\sin^2\psi$ -method is inapplicable or leads to questionable results, i.e. where the lattice parameter vs. $\sin^2\psi$ -distributions are nonlinear because of texture, strong plastic deformation or very steep stress gradients. The main content of this book will therefore be the measuring and evaluation methods, which can help to solve the problems of today, the numerous applications of metallic, polymeric and ceramic materials as well as of thin-film-substrate composites and of advanced microcomponents. The big fields of origin and calculation of residual stresses are not dealt with. This book contains data, results, hints, and recommendations that are valuable to the laboratories for the certification and accreditation of their stress analysis. Some of the experimental results shown here were deliberately chosen from rather early investigations using film techniques, and that for two reasons: on the one hand to demonstrate the value of the early investigations, and on the other hand to stimulate similar studies using modern equipment to confirm and to supplement the early results.

Stress analysis is an active field in which many questions remain unsettled. Accordingly, unsolved problems and conflicting results are discussed in this book as well. Last but not least, the assessment of the experimentally determined residual and structural stress states on the static and dynamic behavior of materials and components will be discussed in a special part.

The total number of publications dealing with the topic of this book is so large that it was not possible to list them all. Nevertheless, we think that the references given here will be very useful for any newcomer in the field. For the purpose of documenting the development of a special method from its very beginnings to the present state-of-the-art, some papers published already a long time ago were included into the list of references. Of course, such a selection of "historically important" publications will inevitably be subjective. Some enlightening ideas on this matter can be found in the booklet "The History of Physics" by Max von Laue (Universitäts-Verlag, Bonn, 1946), from which we quote a paragraph (p. 15 - 16, translation by L. Pintschovius).

"For all epochs the following statement is valid: when an important finding has been published by a particular scientist, it will not take long to hear that the finding is not really new. There will be persons claiming either for themselves or for a third party to have had

"essentially" the same idea. Sometimes these complaints are not totally wrong. There are cases where a certain discovery is "in the air", and indeed is made independently by several persons as a result of the general development. According to Lord Rutherford it is even rarely the case that a scientific discovery is made without substantial preparatory work by the scientific community. Nevertheless, complaints of the sort in question should be met with scepticism. Very often they are based on vaguely worded conjectures that are interpreted with hindsight as equivalent to the clear view achieved by others. Sometimes a person makes an observation or gets an idea, but the importance and consequences of this observation or this idea is grasped only by somebody else. A discovery should be dated only to that point of time when it was described with sufficient clarity and definiteness to have an impact on further progress. If it is true that it was published this way, then the original publication should not be scrutinized punctiliously, if really every detail is correct. Perfection cannot be achieved by men."

Concerning the references the author tried to follow the spirit of Max von Laue: Cite the first conjecture and the most complete handling of the new effect. We hope that this procedure to make reference older literature of premium papers will stimulate other scientists to imitate this model.

The author is pleased to present this book to students and engineers of materials science and scientists working in laboratories at universities or in industries. We hope that the book will find interested readers.

The author gratefully thanks the named experts for their valuable contributions which helped to shape the book into an integral whole. Special thanks are granted to Prof. Dr. W. Reimers and to Dr. H. Behnken for their engagement adding or improving some sections. The English-American wording was kindly revised and improved by Dr. H. Ledbetter, National Institute of Standards and Technology, Boulder, Colorado, USA and by Dr. L. Pintschovius, Institut für Nukleare Festkörperphysik, Kernforschungszentrum Karlsruhe, Karlsruhe. The help preparing the manuscript and the secretary work with special care for figures, tables and formulae of Dipl.-Phys. J. Birkhölzer, S. Charisis, Dipl.-Phys. D. Chauhan and Dipl.-Phys. B. Krüger of the authors group in Aachen and of Mrs. B. Luhm, Hahn-Meitner-Institut, Berlin will be appreciated. The Publisher brought up a lot of patience till the finishing of the book. Thank you.

1 Introduction

1.1 Existing literature

The interest and significance of a scientific branch is documented by the published literature, papers in journals and books. The number of papers which appeared in journals each year is plotted in Figure 1. Three periods of growing efforts can be distinguished: up to 1960, from 1960 to 1980, and later. At present, more than 100 papers per year are published. Not all are of genuine nature, part of the sum is secondary literature. But Conferences in Europe, International Conferences on Residual Stresses (1. ICRS in Garmisch Partenkirchen 1986, 2. ICRS in Nancy 1988, 3. ICRS in Tokushima 1991, 4. ICRS in Baltimore 1994, 5. ICRS in Linköping 1997) and the yearly Denver X-Ray Conferences are of big importance; they elicit great response and give impetus to the worldwide expansion and development of this scientific section.

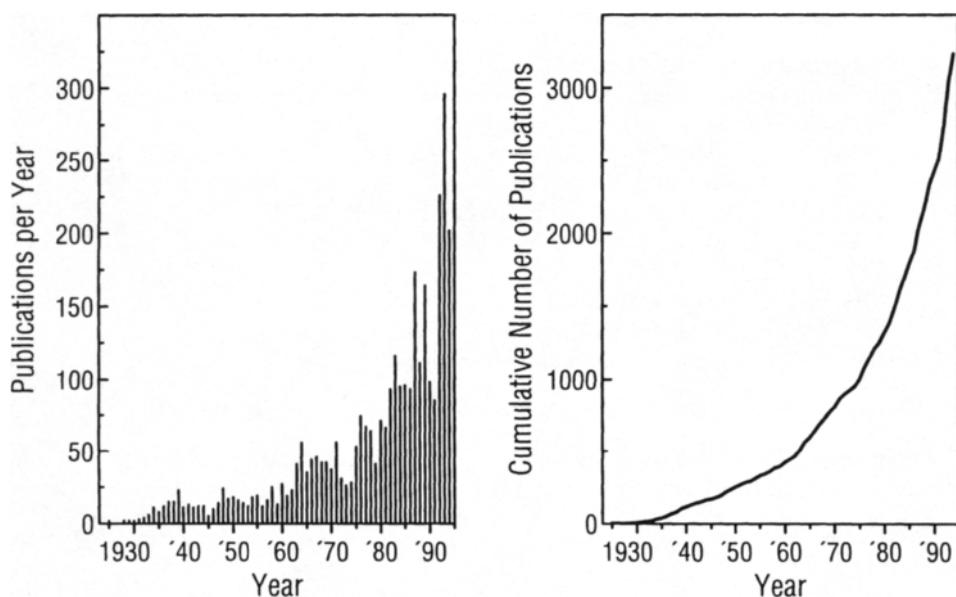


Figure 1. The publications per year and the cumulative number on X-ray and neutron-ray stress analysis. (Literature survey by J. Hauk), plot [1] updated.

The following books, major parts of books and Conference proceedings treat the origin, the measurement and the assessment of structural and residual stresses.

F. Halla, H. Mark: Röntgenographische Untersuchung von Kristallen, J.A. Barth, Leipzig 1937.

H.P. Klug, L.E. Alexander: X-Ray Diffraction Procedures, I. Wiley, New York 1954.

G. Clark: Applied X-Rays, Mc Graw-Hill Book Comp. New York, Toronto, London, 4th edition 1955.

Internal Stresses and Fatigue in Metals, eds: G.M. Rassweiler, W.L. Grube, Elsevier Publishing Company, Amsterdam, London, New York, Princeton 1959.

M. v. Laue: Röntgenstrahleninterferenzen, Akad. Verlagsges., Leipzig, 3rd edition 1960.

A. Peiter: Eigenspannungen I. Art - Ermittlung und Bewertung, Michael Tritsch Verlag, Düsseldorf 1966.

R. Glocker: Materialprüfung mit Röntgenstrahlen unter besonderer Berücksichtigung der Röntgenmetallkunde, Springer-Verlag, Berlin, Heidelberg, New York, 5th edition 1971.

Residual Stress Measurement by X-Ray Diffraction, SAE J 784a (1971).

Mechanische Anisotropie, ed.: H.P. Stüwe, Springer-Verlag, Wien, New York 1974.

X-Ray Studies on Mechanical Behaviour of Materials, ed.: S. Taira, The Society of Materials Science, Japan 1974.

Spannungsermittlungen mit Röntgenstrahlen. In: Härtere Tech. Mitt. 31, 1+2/76, 1976.

J. Hauk, Literature on residual-stress analysis with X- and neutron-rays.

(Schriftum über Spannungsermittlung mit Röntgen- und Neutronenstrahlen)

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1.2 Significance of structural and residual stress analysis for materials science and technology

The next three blocks are taken from a previous paper [2].

It is well established that virtually no material, no component and no structure of technical importance exists free of residual stresses (RS). RS are always produced if regions of material are inhomogeneously elastically or plastically deformed in such a permanent manner that incompatibilities of the state of deformation occur. Especially in structural parts, a great variety of RS-states can exist as a consequence of various technological treatments and manufacturing processes.

As a matter of fact, RS can have both detrimental and favourable consequences for the behavior of materials, components and structures under certain conditions. The experiences reach from the explosion of heat-treated shafts lying unloaded on stock up to the dramatic increase of fatigue strength of components resulting from mechanical or thermomechanical surface treatments. Now as before, however, in actual cases the knowledge of the really existing RS is mostly unsatisfactory, and a large lack of RS-data exists in this respect. Consequently, reliable experimental methods for RS-determination are of great practical importance.

All technical components and structures have to be designed and fabricated with sufficient reliability. In the correct dimensioning of structural parts, the relation between local stresses and strength response of the materials used must be considered. Consequently, RS have to be taken into account in the design to make the component safe and reliable.

The stress state is a characteristic parameter of the material state; together with the microstructure and the texture, the stress situation defines the material properties. A summary of the origins of RS is given in Figure 2. It includes the RS that arise from various mechanical treatments.

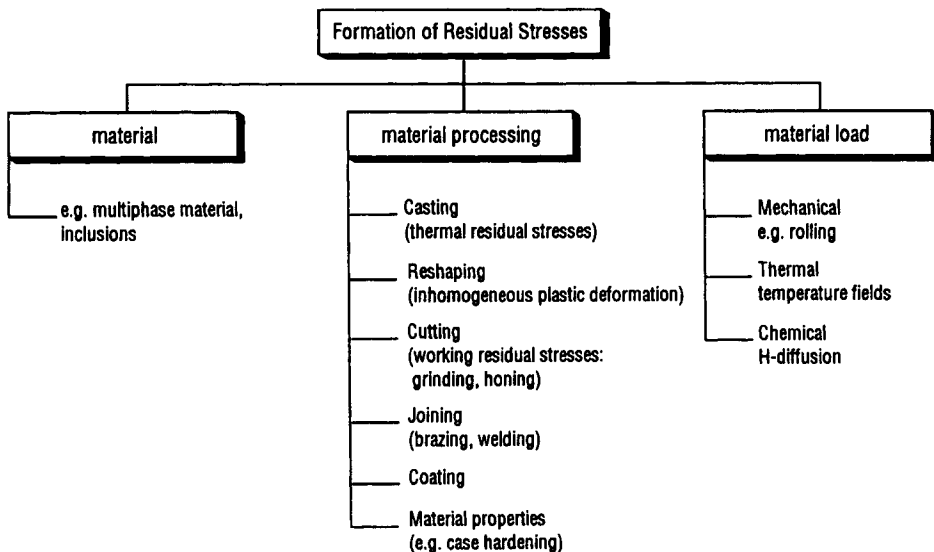


Figure 2. Origin of residual-stress formation [3].

The importance and the wide scope of residual-stress analysis is also demonstrated by the applications that E. Macherauch listed in his introduction to the session "Residual Stresses and the Distortion due to the Influence of Heat" within the 3rd ECRS held 1992 in Frankfurt/Main [4]:

- Analysis and evaluation of residual-stress states occurring as a consequence of distinct heat treatments or combinations of thermal and non-thermal treatments.
- Improvement of measuring techniques for the analysis of residual-stress systems.
- Modeling of distinct processes causing residual stresses and quantitative calculations of residual-stress distributions.
- Determination and evaluation of micro-residual stresses.
- Elucidation of the influence of grain size and grain anisotropy on the development of residual-stress states.
- Residual-stress analysis of thermally treated multiphase material states.
- Evaluation of shape- and size-effects on the development of residual stresses.
- Determination of unknown material data for the calculation and evaluation of residual-stress distributions.

The term stress state means the knowledge of the triaxial stress tensor over the entire cross section of the component, the determination of all kinds of RS in all phases of the material, and a complete study of the outermost surface region. In recent years, progress has been achieved concerning the knowledge about the different kinds of micro-RS and about the influences, that different parameters - like external mechanical load, temperature or environment - exert over their stability or relaxation. Here, diffraction methods, which allow to separate micro- and macro-RS, are an indispensable tool. A better understanding of the processes in the material science was also achieved with the help of the Eshelby/Kröner [5,6] theory. These papers and the many applications of the theory are basic tools of all scientists in the strain-stress area.

Progress in materials science and technology has lead to new challenges for residual-stress analysis. As examples, advanced multiphase materials, thin-film-substrate composites, ceramics, and polymers must be mentioned. Studying the RS-state of these materials, especially after various mechanical surface treatments, resulted in advances of measuring techniques and of evaluation methods. Here, problems accumulate due to depth gradients of macro- and micro-RS, of microstructure and of texture. Although theoretical and experimental studies already dealt with this complex situation, further progress must be achieved.

Stress analysis by nondestructive methods has brought new conceptions of stress values into the discussion. Earlier, values exceeding some hundreds of MPa were commonly handled. Measurements with X-rays on film-substrate composites yielded large values of many thousands of MPa. Metallurgical studies of the influence of solute atoms in thin films by Oettel and others [7] gave values of ten thousand MPa. Values of this order are hard to believe, but they are realistic and must be considered.

Stress analysis is important for components and constructions of every size. The stress state of joints in platforms in rough sea environment, of large diameter pipelines, of essential parts of aircrafts, or of machines has to be determined, as well as that of small components in micro electrical devices and single crystals within polycrystalline metals. The areas that are to be checked for RS are of different size, they range from large areas in the neighborhood of welding zones to small parts of microchips. The extensions of stress fields are demonstrated by a plot of linear dimensions of microstructures and defects [8] in Figure 3.

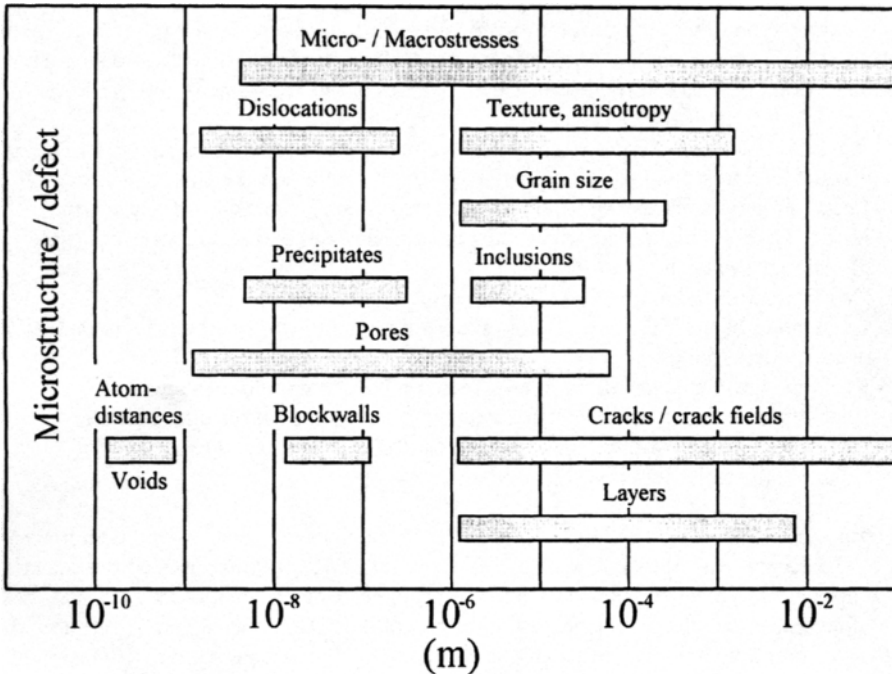


Figure 3. Linear dimensions of microstructure and defects [8].

The strain- stress- analysis is of particular utility for elucidating causes of failure. Even the stresses are basically relaxed due to the formation of cracks and the occurrence of fractures, measurements offer a lot of information on the damaged spot itself, on the region nearby, or on another appropriate spot of the component.

Residual stresses are of special interest in view of the improvement of material properties and of the increase of the lifetime of components. It has to be the aim to induce RS of the proper sign and magnitude for the choice of an appropriate manufacturing process and of the fabrication parameters. The RS should be distributed in such a way over the component that the external load is counterbalanced as far as possible, especially in the most critical part of the component [9].

The performance of stress analysis regarding time and location is related with the manufacturing and the lifetime of the component. Of specific interest are in situ installations to control the origin, the propagation, the stability, the increase or the relaxation of RS. In many production facilities, the RS-state analysis is a quality control instrument. Despite much progress in stress analysis with regard to scientific knowledge its use in industrial laboratories and on site, there will ever be need for further studies and new applications. The following chapters will show the present state of the art of the nondestructive methods, the problems that remain unsolved, and the accommodations to use the techniques in science and practice. The goals are to elaborate new ideas, new methods of measurement and evaluation, to increase their use in manufacturing processes and to insert the effect of RS into standards and customers specifications.

1.3 Characteristics of different methods of evaluating structural-load stresses (LS) and residual stresses (RS)

The methods to determine LS and RS increased in recent years, and also the variety of applications. This has one main consequence: the selection of the proper method has become more complex. A lot of characteristics have to be considered to find the optimal technique. Figure 4 shows the linear dimensions of different nondestructive methods for RSA. In cases where information about the stress states over several magnitudes of length is needed, at least two complementary methods must be used.

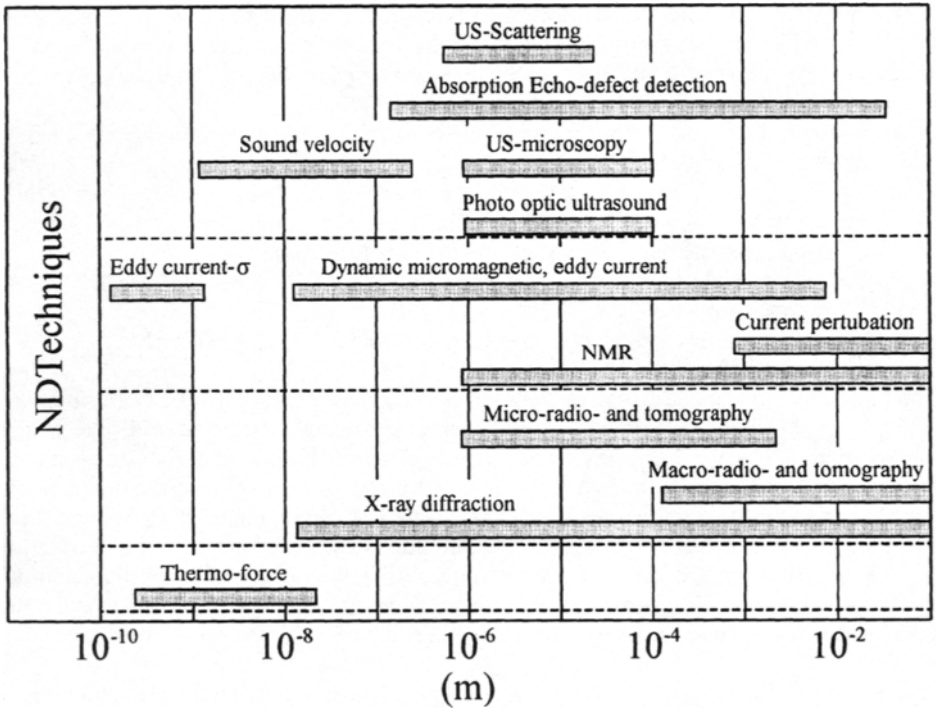


Figure 4. Nondestructive testing techniques versus linear dimensions [8].

The different nondestructive methods will be dealt with in the respective sections and chapters. Here, a comparison of the different methods will be given without going into detail, more or less as a summary. The first comparative description was published by [2]; we present it in an updated and revised form. The important features are:

- Physical principle, measurement of strain or other material parameter
- Relative or absolute method
- Required data for evaluation
- Determination of a single stress component or a combination thereof (σ_{11} - σ_{33} , $\sigma_{11} \pm \sigma_{22}$)
- LS and/or RS

Macro- and/or micro-RS, phase specific measurement
 Penetration depth
 Accuracy of measurement
 Surface condition
 Minimum gauge volume
 Possibility of determining gradients
 Examples of equipment, time, objects
 Required environment conditions

Table 1 contains the characteristics of various techniques, some remarks will follow [2 updated].

LS and RS cannot be determined directly. Distinct physical quantities have to be measured, from which the kinds of RS causing them can be derived. In this respect, a distinction has to be made between destructive and nondestructive measuring techniques. According to their nature RS can be derived

- from macroscopic strains that are released while material is removed from parts loaded by RS. This is the basis of all mechanical methods, thus exclusively investigating RS of the I. kind
- from homogeneous residual lattice-strain distributions. This is the basis of X-ray and neutron diffraction methods, which determine RS of the I. and II. kind
- from propagation velocities and birefringence of ultrasonic waves influenced by RS I. kind. A survey is given in Table 2 [10].
- from magnetic properties and phenomena that are influenced by all kinds of RS.

The mechanical techniques, well established in materials technology, have been improved in recent years by the development of new semi-nondestructive methods, with which also strains in relatively small areas in surface near regions of distinct material states can be measured. The X-ray techniques have achieved a wider application because the equipment has become easier to handle, and the measurements are less time consuming. The neutron-diffraction techniques offer unique advantages in determining RS within the cross section of structural parts. In recent years, nondestructive techniques using ultrasonic and magnetic quantities have also been rapidly developed. However, further investigations have to be made and more practical applications must be developed, if reliable results are to be obtained with these new methods.

Figure 5 shows the range of the analyze methods [2]. The trend of stress-state studies is the use of more than one method to solve complicated stress states or to calibrate one method by the other. The final results should be complemented by an error analysis and a modeling study.

Table 1. Characteristic data of the various techniques to measure strains and related quantities to evaluate LS and RS [2 updated].

	Techniques	Measuring quantities	Required data	Surface condition	Smallest analyzed area/diameter
Mechanic	Boring out, turning off, sectioning	Strains caused by releasing of stresses	Young's modulus, Poisson's ratio, calibration coefficients/ functions	Suitable for applica- tions of strain gages	1.8 mm 14 mm ring diamet.
	Hole drilling, Ringcore				
Diffraction	X-rays	Bragg's angles, lattice strains in different directions of stress states	X-ray elastic constants, lattice parameter of stress- free material state in special cases	Roughness (arithmetical mean) < 2× average penetration depth	0.2 mm diamet. times penetration depth 0.5×0.5×10 mm ³ volume
	Synchrotron radiation				
	Neutrons				
Ultrasonic	Acousto-elasticity	Velocities or times-of-flight of longitudinal and/or shear and/or SH and/or Rayleigh waves propagating and vibrating in principal stress di- rections and, in case of texture, also in principal directions of texture	Ultrasonic path length, Young's, shear moduli and III. order-elastic constants or acoustic-elastic con- stants of material under investigation	Roughness < 0.1 × ultra- sonic wavelength with $0.3 \leq \lambda \leq 5$ mm Brushed surfaces sufficient	30 mm ² probe area
Micro- magnetic	Magnetinductive	Incremental permeability vs. H_t (tangential magnetic field strength), eddy current parameters	The measured ferromag- netic quantities have to be calibrated by X-ray or by strain-gage measurements	Roughness $\leq 0.1 \times$ probe diameter, surface preparations are not necessary	1mm ²
	Magnetoelastic	Amplitude of the magnetostrictively excited ultrasonic waves vs. H_t , amplitude of the acoustic Bark- hausen noise vs. H_t			10 mm ²
	Ferromagnetic	Amplitude of the magnetic Bark- hausen noise vs. H_t , distortion factor			0.5 mm ²

Table 1. continued.

Evaluated Stress	Accuracy of evaluated stress	Evaluation of stress gradient	Equipment Measuring time	Applications
I. kind	± 10 MPa	Possible Variation of hole drilling depth Variation of ring notch depth	Strain gage (rosette) measuring device, depending on the extent of the measurements	All kinds of components (dimension ≥ 10 mm) if geometrical conditions are fulfilled
triaxial stress states, I.+II. kind, separation possible in specific cases, applicable to each phase of multiphase-materials with sufficient volume content	Depending on materials state and measuring effort ± 10 MPa.	Variation of wavelength and/or etching depth Shifting of specimen in steps of 0.5 mm	X-ray source, diffractometer, minutes to hours per stress component. Synchrotron-ray source, diffr., min. to hours per stress component. Neutron-ray source, diffr., hours per stress component	All kinds of components and materials with at least one crystalline phase of sufficient volume content
I. kind (macrostresses), surface stresses, bulk stresses. The result is a mean value of the principal stress or of the difference of two principal stresses acting in the part of the component, propagated by ultrasonic wave(s)	± 3.5 % of stress value $\pm (5-10)$ MPa	If microstructure is known alteration of frequency of surface waves for analyzing depths between 0.3 and 5 mm	Set-ups to measure ultrasonic times-of-flight, set-ups for automated evaluation of stress states, 1 measurement per minute up to 200 measurements per second	Components of metallic and ceramic materials, increasing efforts are necessary in case of pronounced textures
I.+II.+III. kind, multiparameter testing procedures are necessary to suppress disturbances and to get quantitative results after calibration	Reproducibility about 10 %	Variation of analyzed frequency with particular consideration of microstructure and texture	Multiparameter micromagnetic device, 1 sec. per measuring quantity	Components of ferromagnetic material, except regions which can not be excited magnetically

Table 2. Determination of the macrostress states by means of ultrasonic techniques [10].

stress state	technique	result	remarks
surface stresses uniaxial or biaxial	shear horizontal longitudinal wave (SL)	$\sigma_1; \sigma_2$	penetration depth ≤ 6 mm length ≥ 30 mm
	Rayleigh wave (R)	(σ_1, σ_2)	penetration depth ≤ 3 mm ($\sim 0.3 - 0.5$ mm steps) length ≥ 50 mm
	SH-wave (SH)	(σ_1, σ_2)	penetration depth ≤ 3 mm length ≥ 50 mm
volume stresses uniaxial	longitudinal and trans- verse wave (L+T)	σ_1	gage area ≥ 3 mm diam. length = thickness
biaxial	double refraction (2T) longitudinal wave (L) 2T+L	$\sigma_1 - \sigma_2$ $\sigma_1 + \sigma_2$ σ_1, σ_2	gage area ≥ 3 mm diam. length = thickness
triaxial	longitudinal wave (L) transverse wave (T)	$\sigma_1, \sigma_2, \sigma_3$ $\sigma_1, \sigma_2, \sigma_3$	test area ≥ 3 mm diam. length = thickness propagation along mainstress direction
	double refraction (2T)	$\sigma_1 - \sigma_3$ $\sigma_3 - \sigma_2$ $\sigma_2 - \sigma_1$	along σ_1 along σ_2 along σ_3

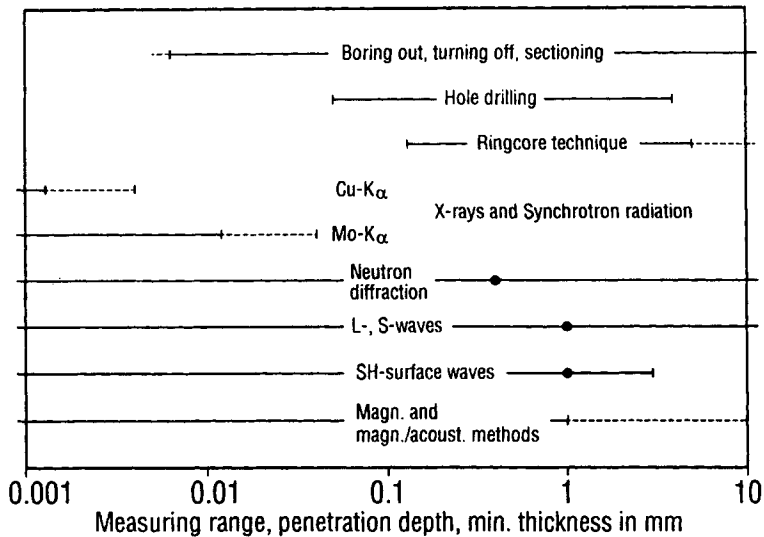


Figure 5. Measuring range and penetration depth of the different techniques, minimal thickness of the material to be tested [2].

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2 X-ray diffraction

2.01 Highlights in the history of diffraction methods - first notice, entire treatment

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2.02 Symbols and abbreviations

$\underline{C}^i, \underline{S}^i, \underline{L}^i$	unity vectors of the crystal-, specimen- and laboratory-system; $i=1,2,3$
ξ, ω, ζ	transformation matrices between the laboratory- and the specimen-system, crystal- and specimen-system, crystal- and laboratory-system
$\underline{m}$	unity vector in measuring direction
φ, ψ	azimuth and pole angle of the measuring direction $\underline{m}$ within the specimen-system
η, ρ	azimuth and pole angle of the measuring direction $\underline{m}$ within the crystal-system
ψ^*, ψ'	stress-free, stress-independent direction within the specimen-system
Ω	orientation of a crystal within the specimen-system
$f(\Omega)$	orientation-distribution-function (ODF)
λ	rotation-angle of a crystallite around the lattice-plane normal; also wave length; also Lamé-constant
Θ	Bragg's angle
τ	penetration depth
$(hkl), \{hkl\}$	lattice plane, all equivalent lattice planes, Miller' indices
$[uvw], \langle uvw \rangle$	lattice-plane normal direction, all equivalent normal directions
RD, TD, ND	rolling-, transverse-, normal-direction
$\langle \rangle$	average value
$D_{\{hkl\}}$	lattice distance of the plane $\{hkl\}$
D_0	stress-free lattice distance
ϵ	strain
σ	stress
$D_{\varphi, \psi}, \epsilon_{\varphi, \psi}$	average lattice distance, average strain in the (φ, ψ) direction of all crystallites which contribute to the interference line/peak

$D_{\psi+}, D_{\psi-}$	lattice distance for $\psi > 0^\circ$, $\psi < 0^\circ$
$\varepsilon_{\varphi, \psi}(\Omega)$	average strain of the crystallites of the orientation Ω in the measuring direction (φ, ψ)
$\sigma(\Omega), \varepsilon(\Omega)$	stress- and strain-tensor and their coordinates averaged over $\sigma_{ij}(\Omega), \varepsilon_{ij}(\Omega)$ the volume of a crystal or of a crystallite-group with the orientation Ω within the specimen-system
$\bar{\sigma}, \bar{\sigma}^\alpha$	average stress tensor, -of a phase
$\langle \sigma \rangle^\alpha$	average phase stress determined
σ_{ii}, σ_{ik}	components of stress
σ_i	principal stress
α, β, γ	phases
δ	amorphous polymeric phase
$\bar{\sigma}_0$	part of the average phase stresses which is independent of the macrostresses
$\varepsilon_0(\Omega), \sigma_0(\Omega)$	part of $\varepsilon(\Omega)$ and $\sigma(\Omega)$ which is independent of average phase stresses
ε^m, σ^m	macro-strain- and stress-tensor
σ^L	load stress (LS)
$\sigma^I, \sigma^{II}, \sigma^{III}$	residual stress (RS) I., II., III. kind; macro-, micro-RS
c, C, s, S	tensors of stiffnesses and of compliances of the mono- (small) and polycrystal (capital)
$c_{\alpha\beta}, s_{\alpha\beta}$	monocrystal stiffnesses and -compliances in the crystal-system, Voigt's notation
$c_{ijkl}(\Omega), s_{ijkl}(\Omega)$	coordinates of the stiffness- and compliance-tensor of the crystallites of the orientation Ω in the specimen-system
t	elastic polarization tensor
$f_{ijkl}^\alpha, f_{mn}^\alpha$	stress transfer factors, coordinates of stress transfer tensor of the α phase, tensor-, Voigt's notation
f^α	$f_{1111}^\alpha - f_{3311}^\alpha$

E, K, G, ν	Young's-, compression-, shear-modulus, Poisson's ratio
λ, μ	Lamé's constants of the elastic isotropic material
$R_{p0,2}, R_m, A$	yield-, tension-strength, elongation
$s_1, \frac{1}{2}s_2$	X-ray elastic constants (XEC) of quasiisotropic materials
$F_{ij}(\varphi, \psi, hkl)$	X-ray stress factors of textured materials related to the specimen-system
$\langle \sigma \rangle_\alpha$	measured average on α phase
$\langle \sigma^{II} \rangle_\alpha$	average micro-RS of a phase α
$\langle \sigma^{III} \rangle_\alpha$	average micro-RS of those regions of a phase α which contribute to the interference line

nomenclature	material
macrostructure	homogeneous, heterogeneous
microstructure	single-phase (monophase), dual-/multiphase
elastic anisotropy	isotropic, anisotropic
texture	mechanically isotropic, textured (preferred orientation)
strain	compatibility
stress	equilibrium, compensation
example of a homogeneous material:	two phases, isotropic or textured, (each volume element is representative of the bulk)
example of heterogeneous material:	layer-, fiber-composite

Abbreviations

FWHM	Full Width at Half Maximum
GIXD	Grazing Incidence X-ray Diffraction
GXR	Grazing X-ray
LS	Load Stress
NSA	Neutron Stress Analysis
RS	Residual Stress
XEC	X-ray Elastic Constant(s)
XEF	X-ray Elastic Factor
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
XSA	X-ray Stress Analysis

2.03 Some basic relations to the stress analysis using diffraction methods

H. Behnken

2.031 Introduction

In this chapter some basic relations and agreements will be described which are necessary for the stress analysis using X- and neutron rays, for the interpretation of the measured data, and for the calculation of averaged elastic properties and X-ray elastic constants (XEC). In particular the following subjects will be considered:

- Representation of strains, of stresses, and of the data of elastic properties by tensors of 2nd and 4th rank
- The reference systems of coordinates and the transformation of tensors
- Orientations of crystals within a polycrystalline material
- Calculation of elastic properties by averaging the single-crystal elasticity data
- Relations between the stresses within a material and the results of strain measurements using diffraction methods
- Kinds of residual stresses and their mutual relations

In the following, tensors of 1st rank (vectors), 2nd and 4th rank are used to describe forces, stresses and strains, and the elastic material properties, respectively. The definition of tensors, their transformation behavior, and their representation is comprehensively outlined by Nye [1]. Here, only the results that are necessary in later chapters are presented.

For the writing of products of vectors, 2nd- and 4th-rank tensors, and of matrices, the summation convention will be applied, i.e. if an index occurs twice in a term, the summation is taken from 1 to 3.

Tensors of 2nd rank as well as their components are noted by greek letters (e.g. stress tensor σ , strain ϵ , with the components σ_{ij} and ϵ_{ij}), those of 4th rank by latin letters (S , s , c , c_{ijkl}). The capital letters stand for 4th rank tensors describing macroscopic behavior or the behavior of a material phase (e.g. S_{ijkl} the elastic compliances of the specimen); the respective small letters appertain to single (mono) crystallites or to crystal orientations (e.g. s_{ijkl} , c_{ijkl}). Vectors are underlined. Unity vectors in direction of axes X^1 , X^2 , X^3 are noted according to the appertaining axes: $\underline{X}^1$, $\underline{X}^2$, and $\underline{X}^3$.

2.032 Stresses, strains, and elastic material properties

To illustrate and define the stress components we consider a small cube with face areas A within a homogeneously stressed body. The cube is aligned to the axes of the coordinate system X^1, X^2, X^3 (Fig. 1). Each face is characterized by one of the unit vectors $\underline{X}^1, \underline{X}^2, \underline{X}^3$ or $-\underline{X}^1, -\underline{X}^2, -\underline{X}^3$. Firstly we regard the faces towards the positive directions. The surrounding material will produce forces $\underline{f}^i$ on the face i of the cube. They are proportional to the area A . Each of the forces $\underline{f}^i$ can be resolved in the components parallel to the three axes: f^i_1, f^i_2, f^i_3 . Now, the stress component σ_{ij} is defined to be the force per area acting on the face i in direction j :

$$\sigma_{ij} = \underline{f}^i \cdot \underline{X}^j / A \quad (1)$$

For instance, σ_{11} is acting on the 1-face in 1-direction, σ_{23} acts on the 2-face in 3-direction. σ_{ii} are called normal components, because the respective forces act normally to a face, the σ_{ij} ($i \neq j$) are called shear components, they act parallel to a face. Since the small cube is assumed to be homogeneously stressed, the forces on the three opposite sides are of the same amount but of opposite direction. The faces' normals are opposite too, Equ.1 therefore yields the same stress. The equilibrium consideration [1] of the stressed volume element reveals the symmetry: $\sigma_{ij} = \sigma_{ji}$. The stress components σ_{ij} form a symmetrical tensor of 2nd rank.

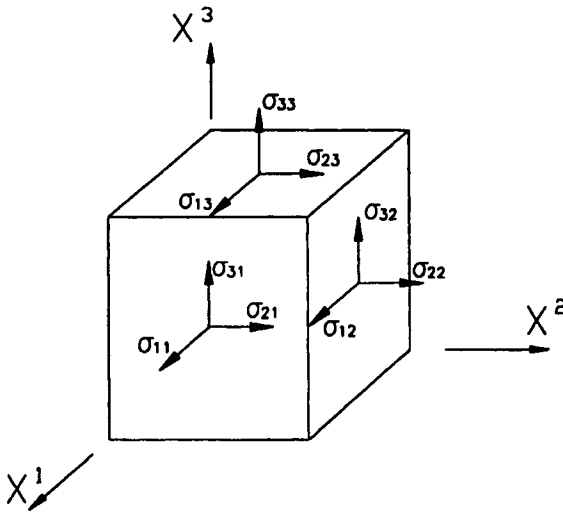


Figure 1. To the definition of the stress components.

If a body is stressed, each point x within the body may undergo a displacement u , which in the following is assumed to be small. The strain of a one dimensional string with the initial length L_0 is known to be $\epsilon(x) = du/dx$. If ϵ is homogeneous, i.e. it does not depend on x , it holds:

$$\epsilon = (L - L_0)/L_0 = \Delta L/L_0 \quad ; \quad u(x) = \epsilon x. \quad (2)$$

In three-dimensional bodies the displacement of a point $\underline{x}$ is described by the vector $\underline{u}(\underline{x}) = (u_1, u_2, u_3)$, Fig.2. In the vicinity of a fixed point $\underline{x}_0$ the displacements can be developed up to first order, which is sufficient for small distances $\Delta \underline{x} = \underline{x} - \underline{x}_0$.

$$u_i(\underline{x}_0 + \Delta \underline{x}) = u_i(\underline{x}_0) + (\partial u_i / \partial x_1) \Delta x_1 + (\partial u_i / \partial x_2) \Delta x_2 + (\partial u_i / \partial x_3) \Delta x_3; \quad i = 1, 2, 3 \quad (3)$$

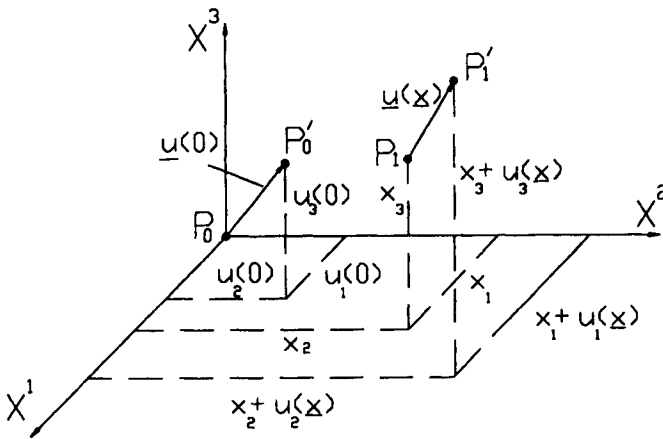


Figure 2. To the definition of the strain components.

The spatial variations of displacements $e_{ij} = \partial u_i / \partial x_j$ form a tensor of 2nd rank. Equ.3 can be rewritten:

$$\begin{aligned} u_i(\underline{x}_0 + \Delta \underline{x}) &= u_i(\underline{x}_0) + e_{ij} \Delta x_j & (= u_i(\underline{x}_0) + \sum_{j=1}^3 e_{ij} \Delta x_j; \text{ summation convention!}) \\ &= u_i(\underline{x}_0) + \frac{1}{2}(e_{ij} - e_{ji}) \Delta x_j + \frac{1}{2}(e_{ij} + e_{ji}) \Delta x_j & i = 1, 2, 3 \\ &= u_i^t + u_i^r + u_i^s \end{aligned} \quad (4)$$

The antisymmetrical part of the tensor e

$$\frac{1}{2}(e_{ij} - e_{ji}) = \frac{1}{2}(\partial u_i / \partial x_j - \partial u_j / \partial x_i) \quad (5)$$

describes the rotation of the volume around $\underline{x}_0$. The term u_i^r as well as the translational part $u_i^t = u_i(\underline{x}_0)$ is of no interest in regard of material behavior. The term u_i^s describes the contribution of the strain to the displacements. The strain alters the distances and the relative positions between the points within the body. The strain tensor is defined as the symmetrical part of the tensor e :

$$\varepsilon_{ij} = \frac{1}{2}(e_{ij} + e_{ji}) = \frac{1}{2}(\partial u_i / \partial x_j + \partial u_j / \partial x_i) \quad (6)$$

The diagonal components ε_{11} , ε_{22} , and ε_{33} , i.e. the normal strains, signify the stretches in the respective directions. For instance $\varepsilon_{22} = \partial u_2 / \partial x_2$, as it would be in the one-dimensional case. If

only normal strains are present, a cube of the initial dimensions d_1, d_2, d_3 will be strained to the dimensions $d_1 (1+\epsilon_{11}), d_2 (1+\epsilon_{22}), d_3 (1+\epsilon_{33})$. The non-diagonal components stand for the shear strains. The geometrical meaning of ϵ_{13} , as example, is the alteration of the angle between two lines that were initially parallel to the coordinate axis X^1 and X^3 , respectively. By applying the shear strain ϵ_{13} the angle between these lines changes from $\frac{1}{2}\pi$ to $(\frac{1}{2}\pi - 2\epsilon_{13})$.

Strains and stresses both form symmetrical 2nd rank tensors. They each have $3^2=9$ components $\sigma_{ij}, \epsilon_{ij}$, which can be written in matrix notation

$$\sigma = \begin{pmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{21} & \sigma_{22} & \sigma_{23} \\ \sigma_{31} & \sigma_{32} & \sigma_{33} \end{pmatrix} \quad \epsilon = \begin{pmatrix} \epsilon_{11} & \epsilon_{12} & \epsilon_{13} \\ \epsilon_{21} & \epsilon_{22} & \epsilon_{23} \\ \epsilon_{31} & \epsilon_{32} & \epsilon_{33} \end{pmatrix} \quad (7)$$

Because of the symmetry $\sigma_{ij} = \sigma_{ji}$ and $\epsilon_{ij} = \epsilon_{ji}$, only 6 of the 9 components are independent and are sufficient to describe the stress and the strain at a point in the material.

If a coordinate system has been fixed and the stress tensor is known, we may ask for the stress on a plane located normally to a special direction with unit vector $\underline{n}$. This question corresponds to the calculation of the projection of a vector $\underline{\sigma}$ on the direction $\underline{n}$ which is given by the scalar product $\underline{\sigma} \cdot \underline{n} = v_i \cdot n_i$. Correspondingly, the stress in direction $\underline{n}$ is the projection of the stress tensor σ on $\underline{n}$:

$$\begin{aligned} \sigma_{\underline{n}} &= \sigma_{ij} n_i n_j \quad \left(= \sum_{i=1}^3 \sum_{j=1}^3 \sigma_{ij} n_i n_j \right) \\ &= \sigma_{11} n_1^2 + \sigma_{22} n_2^2 + \sigma_{33} n_3^2 + 2 \sigma_{12} n_1 n_2 + 2 \sigma_{13} n_1 n_3 + 2 \sigma_{23} n_2 n_3 \end{aligned} \quad (8)$$

Similarly the strain in the direction $\underline{n}$ is given by

$$\begin{aligned} \epsilon_{\underline{n}} &= \epsilon_{ij} n_i n_j \\ &= \epsilon_{11} n_1^2 + \epsilon_{22} n_2^2 + \epsilon_{33} n_3^2 + 2 \epsilon_{12} n_1 n_2 + 2 \epsilon_{13} n_1 n_3 + 2 \epsilon_{23} n_2 n_3 \end{aligned} \quad (9)$$

Introducing the representation of the vector $\underline{n}$ by its azimuth φ and polar angle ψ :

$$\underline{n} = (\cos\varphi \sin\psi, \sin\varphi \sin\psi, \cos\psi)$$

and using the geometrical identity $2 \sin\alpha \cos\alpha = \sin 2\alpha$, one obtains:

$$\begin{aligned} \epsilon_{\underline{n}} &= \epsilon_{11} \cos^2\varphi \sin^2\psi + \epsilon_{22} \sin^2\varphi \sin^2\psi + \epsilon_{33} \cos^2\psi \\ &\quad + \epsilon_{12} \sin 2\varphi \sin^2\psi + \epsilon_{13} \cos\varphi \sin 2\psi + \epsilon_{23} \sin\varphi \sin 2\psi \end{aligned} \quad (10)$$

When a body underlies certain stresses, the strain response depends on the elastic properties of the material. The strain can be of elastic and of plastic kind.

$$\epsilon = \epsilon^{el.} + \epsilon^{pl.} \quad (11)$$

When stresses are released, the elastic strain will vanish and the plastic part will remain. For stresses not exceeding the yield limit of the material, the strain response is of elastic kind

only. If the elastic strains are sufficiently small, they depend linearly on the applied stresses. This is actually valid in most practical cases.

$$\varepsilon^{el.} \sim \sigma \quad (12)$$

In the one-dimensional case the Young's modulus E connects the stress and the strain (Hooke's law):

$$\sigma = E \varepsilon \quad (13)$$

The most general linear relation between the stress and the strain tensor is given, when each stress component depends linearly on all the 9 strain components. That are 9 equations with 9 independents.

$$\sigma_{ij} = c_{ijkl} \varepsilon_{kl} \quad (14)$$

This relation defines the 4th rank tensor of elastic stiffnesses c , it has $3^4 = 81$ components c_{ijkl} .

Before the relations between the strains and stresses are further discussed, the definition of tensor products have to be introduced.

The product ($b \cdot c$) of two fourth rank tensors b and c with the components b_{ijkl} and c_{ijkl} is defined [1,2] as:

$$(b \cdot c)_{ijkl} = b_{ijmn} c_{mnkl} \quad \left(= \sum_{m=1}^3 \sum_{n=1}^3 b_{ijmn} c_{mnkl} \right) \quad (15)$$

and the product ($c \cdot \varepsilon$) of a tensor of 4th rank (c) with a tensor of 2nd rank (ε):

$$(c \cdot \varepsilon)_{ij} = c_{ijmn} \varepsilon_{mn} \quad (16)$$

The inverse tensor of 4th rank is defined by

$$c \cdot c^{-1} = I \quad (17)$$

$$\text{with } I \text{ the unit tensor of rank 4:} \quad I_{ijkl} = \frac{1}{2} (\delta_{ik} \delta_{jl} + \delta_{il} \delta_{jk}) \quad (18)$$

$$\delta_{ij} \text{ Kronecker delta:} \quad \delta_{ij} = \begin{cases} 1 & \text{if } i = j \\ 0 & \text{if } i \neq j \end{cases}$$

With Equ.14 the stress tensor results as the product of the tensor of elastic stiffnesses c and the strain tensor ε .

$$\sigma = c \cdot \varepsilon \quad (19)$$

Relation (14) and (19) are called the generalized Hooke's law.

The stress as well as the strain tensor is symmetric, and so the tensor c is written symmetrical too:

$$c_{ijkl} = c_{jikl} = c_{ijlk} \quad (20)$$

Therefore, the maximum amount of independent components reduces to 36. It is now convenient to introduce the abbreviations of the Voigt's notation [3,4]. Each pair of indices ij will be abbreviated by one Voigt-index m , according to the following scheme:

$$\begin{aligned} 11 &\rightarrow 1; \quad 22 \rightarrow 2; \quad 33 \rightarrow 3; \\ 23 &\rightarrow 4; \quad 13 \rightarrow 5; \quad 12 \rightarrow 6; \\ 32 &\rightarrow 4; \quad 31 \rightarrow 5; \quad 21 \rightarrow 6; \end{aligned} \quad (21)$$

Relation (14) can now be expressed by the 6 equations

$$\begin{aligned} \sigma_1 &= c_{11} \varepsilon_1 + c_{12} \varepsilon_2 + c_{13} \varepsilon_3 + c_{14} \varepsilon_4 + c_{15} \varepsilon_5 + c_{16} \varepsilon_6 \\ \sigma_2 &= c_{21} \varepsilon_1 + c_{22} \varepsilon_2 + c_{23} \varepsilon_3 + c_{24} \varepsilon_4 + c_{25} \varepsilon_5 + c_{26} \varepsilon_6 \\ \sigma_3 &= c_{31} \varepsilon_1 + c_{32} \varepsilon_2 + c_{33} \varepsilon_3 + c_{34} \varepsilon_4 + c_{35} \varepsilon_5 + c_{36} \varepsilon_6 \\ \sigma_4 &= c_{41} \varepsilon_1 + c_{42} \varepsilon_2 + c_{43} \varepsilon_3 + c_{44} \varepsilon_4 + c_{45} \varepsilon_5 + c_{46} \varepsilon_6 \\ \sigma_5 &= c_{51} \varepsilon_1 + c_{52} \varepsilon_2 + c_{53} \varepsilon_3 + c_{54} \varepsilon_4 + c_{55} \varepsilon_5 + c_{56} \varepsilon_6 \\ \sigma_6 &= c_{61} \varepsilon_1 + c_{62} \varepsilon_2 + c_{63} \varepsilon_3 + c_{64} \varepsilon_4 + c_{65} \varepsilon_5 + c_{66} \varepsilon_6 \end{aligned} \quad (22)$$

and the components c_{mn} can be arranged in a 6×6 matrix. But it has to be noted at this point, that the c_{mn} are not the components of a tensor. If calculations are performed containing tensor multiplications or tensor transformations, the components have to be treated using the 4-indices tensor notation. The Voigt's notation with only 2 indices should be regarded as a convenient abbreviation.

The consideration of the elastic energy reveals, that, additionally to the symmetry relations of Equ.20, it holds: $c_{ijkl} = c_{klij}$, i.e. $c_{mn} = c_{nm}$ in Voigt's notation [1,3]. That makes the matrix of c_{mn} symmetric, and the number of independent components reduces to 21.

The inversion of Equ.19 yields the generalized Hooke's law in the form

$$\varepsilon = s \sigma \quad \text{and} \quad \varepsilon_{ij} = s_{ijmn} \sigma_{mn} \quad ; \quad \text{with } s = c^{-1} \quad (23)$$

The tensor s of the elastic compliances s_{ijmn} is the inverse tensor of the stiffness tensor c . The Voigt's notation applies for s too, but the following additional agreements are usual.

$$\begin{aligned} s_{mn} &= s_{ijkl} \quad \text{for } (m \leq 3 \text{ and } n \leq 3) \\ s_{mn} &= 2 \cdot s_{ijkl} \quad \text{for } (m \leq 3 \text{ and } n > 3) \text{ or vice versa} \\ s_{mn} &= 4 \cdot s_{ijkl} \quad \text{for } (m > 3 \text{ and } n > 3) \end{aligned} \quad (24)$$

These agreements hold for the tensor s only. The Voigt's notation of all other 4th rank tensors in this book will follow the previously described scheme:

$$c_{mn} = c_{ijkl} \quad (25)$$

The number of independent components is further reduced by the symmetry elements of the crystal lattice [1]. For instance, the s_{mn} and c_{mn} of orthorhombic crystals show the following arrangement with 9 independent components:

$$c = \begin{pmatrix} c_{11} & c_{12} & c_{13} & 0 & 0 & 0 \\ \cdot & c_{22} & c_{23} & 0 & 0 & 0 \\ \cdot & \cdot & c_{33} & 0 & 0 & 0 \\ \cdot & \cdot & \cdot & c_{44} & 0 & 0 \\ \cdot & \cdot & \cdot & \cdot & c_{55} & 0 \\ \cdot & \cdot & \cdot & \cdot & \cdot & c_{66} \end{pmatrix} \quad (26)$$

The matrix is symmetric, therefore, as usual, only the upper triangle is written. With increasing symmetry of the crystal lattice one obtains further simplifications, i.e. for hexagonal symmetry:

$$\begin{aligned} s_{11} = s_{22}, \quad s_{13} = s_{23}, \quad ; \quad c_{11} = c_{22}, \quad c_{13} = c_{23}, \\ s_{44} = s_{55}, \quad s_{66} = 2(s_{11} - s_{12}) \quad ; \quad c_{44} = c_{55}, \quad c_{66} = \frac{1}{2}(c_{11} - c_{12}) \end{aligned} \quad (27)$$

and for cubic symmetry:

$$\begin{aligned} s_{11} = s_{22} = s_{33} \quad ; \quad c_{11} = c_{22} = c_{33} \\ s_{44} = s_{55} = s_{66} \quad ; \quad c_{44} = c_{55} = c_{66} \\ s_{12} = s_{13} = s_{23} \quad ; \quad c_{12} = c_{13} = c_{23} \end{aligned} \quad (28)$$

For elastically isotropic bodies it holds additionally to Equ.28:

$$s_{44} = 2(s_{11} - s_{12}) \quad ; \quad c_{44} = \frac{1}{2}(c_{11} - c_{12}) \quad (29)$$

In the last case only two independent components are left: $S_{11} = 1/E$, $S_{12} = -\nu/E$. The elastic behavior of an isotropic body is fully described by the Young's modulus E and the Poisson's ratio ν . Several other descriptions are in use too, for instance noting the modulus of compressibility K and the shear modulus G . But, in any case, two values are sufficient to describe isotropic behaviour, they all can be transferred each to another combination according to Table 1 [2].

The arrangements of tensor components c_{mn} and s_{mn} of different crystal systems are disposed in Table 2 [5]. A comprehensive collection of the known single-crystal data s_{mn} and c_{mn} is given in [6]. In accordance to [1], the components c_{ijkl} are called stiffnesses, and the s_{ijkl} compliances, as it is the American usage. The c_{ijkl} are written in units of MPa or N/mm², the s_{ijkl} in MPa⁻¹ or mm²/N. Both c and s describe the elastic properties of a crystal. Not specifying whether c or s is meant, we will speak of elastic data. (Some English authors use the notation "elastic modulus" for the s_{ijkl} , and "elastic constants" for the c_{ijkl} . This can be confusing, because the macroscopic Young's modulus (unit MPa) would be an elastic constant, not an elastic modulus)

Table 1. Relations between the macroscopic elastic data of quasiisotropic materials

	E, ν	E, G	λ, μ	K, G	μ, ν	c_{11}, c_{12}	s_{11}, s_{12}
E	E	E	$\frac{(3\lambda+2\mu)\mu}{\lambda+\mu}$	$\frac{9KG}{3K+G}$	$2(1+\nu)\mu$	$\frac{(c_{11}-c_{12})(c_{11}+2c_{12})}{c_{11}+c_{12}}$	$\frac{1}{s_{11}}$
ν	ν	$\frac{E-2G}{2G}$	$\frac{\lambda}{2(\lambda+\mu)}$	$\frac{3K-2G}{6K+2G}$	ν	$\frac{c_{12}}{c_{11}+c_{12}}$	$-\frac{s_{12}}{s_{11}}$
K	$\frac{E}{3(1-2\nu)}$	$\frac{EG}{3(3G-E)}$	$\frac{3\lambda+2\mu}{3}$	K	$\frac{2\mu(1+\nu)}{3(1-2\nu)}$	$\frac{c_{11}+2c_{12}}{3}$	$\frac{1}{3(s_{11}+2s_{12})}$
$G = \mu$	$\frac{E}{2(1+\nu)}$	G	μ	G	μ	$\frac{c_{11}-c_{12}}{2}$	$\frac{1}{2(s_{11}-s_{12})}$
λ	$\frac{\nu E}{(1-2\nu)(1+\nu)}$	$\frac{G(E-2G)}{3G-E}$	λ	$\frac{3K-2G}{3}$	$\frac{2\mu\nu}{1-2\nu}$	c_{12}	$-\frac{s_{12}}{(s_{11}-s_{12})(s_{11}+2s_{12})}$
c_{11}	$\frac{(1-\nu)E}{(1-2\nu)(1+\nu)}$	$\frac{G(4G-E)}{3G-E}$	$\lambda+2\mu$	$\frac{3K+4G}{3}$	$\frac{2\mu(1-\nu)}{1-2\nu}$	c_{11}	$\frac{s_{11}+s_{12}}{(s_{11}-s_{12})(s_{11}+2s_{12})}$
c_{12}	$\frac{\nu E}{(1-2\nu)(1+\nu)}$	$\frac{G(E-2G)}{3G-E}$	λ	$\frac{3K-2G}{3}$	$\frac{2\mu\nu}{1-2\nu}$	c_{12}	$-\frac{s_{12}}{(s_{11}-s_{12})(s_{11}+2s_{12})}$
s_{11}	$\frac{1}{E}$	$\frac{1}{E}$	$\frac{\lambda+\mu}{\mu(3\lambda+2\mu)}$	$\frac{2G+6K}{18KG}$	$\frac{1}{2\mu(1+\nu)}$	$\frac{(c_{11}+c_{12})}{(c_{11}-c_{12})(c_{11}+2c_{12})}$	s_{11}
s_{12}	$-\frac{\nu}{E}$	$\frac{2G-E}{2EG}$	$-\frac{\lambda}{2\mu(3\lambda+2\mu)}$	$\frac{2G-3K}{18KG}$	$-\frac{\nu}{2\mu(1+\nu)}$	$-\frac{c_{12}}{(c_{11}-c_{12})(c_{11}+2c_{12})}$	s_{12}
s_{11}^m	$-\frac{\nu}{E}$	$\frac{2G-E}{2EG}$	$-\frac{\lambda}{2\mu(3\lambda+2\mu)}$	$\frac{2G-3K}{18KG}$	$-\frac{\nu}{2\mu(1+\nu)}$	$-\frac{c_{12}}{(c_{11}-c_{12})(c_{11}+2c_{12})}$	s_{12}
$\frac{1}{2}s_{12}^m$	$\frac{1+\nu}{E}$	$\frac{1}{2G}$	$\frac{1}{2\mu}$	$\frac{1}{2G}$	$\frac{1+\nu}{2\mu(1+\nu)}$	$\frac{1}{c_{11}-c_{12}}$	$s_{11}-s_{12}$

Table 2. Forms of the matrices of elastic stiffnesses and elastic compliances for the different crystal classes. The components beneath the diagonals are not potted. The matrices are symmetric with respect to the diagonals. International as well as Schoenflies notation of the crystal classes. [5].

crystal system	crystal class	arrangement of stiffnesses and compliances in matrix notation; number of independent components		crystal system	crystal class	arrangement of stiffnesses and compliances in matrix notation; number of independent components	
triclinic	all classes		21	tetragonal	4 (C ₄)		7
					$\bar{4}$ (S ₄)		
monoclinic	all classes		13		4/m (C _{4h})		7
					4mm (C _{4v})		
orthorhombic	all classes		9	hexagonal	$\bar{4}2m$ (D _{2d})		6
					422 (D ₄)		
trigonal	3 (C ₃)		7	cubic	$\frac{4}{m}mm$ (D _{4h})		5
	$\bar{3}$ (C _{3i})						
	32 (D ₃)		6	isotropic			3
	3m (C _{3v})						
	$\bar{3}m$ (D _{3d})						2

$\cdot$	$\bullet$	$\bullet\bullet$	$\circ\circ$	Θ	$\blacklozenge$
$s_{ij} = 0$	$s_{ij} \neq 0$	$s_{ij} = s_{kl}$	$s_{kl} = -s_{ij}$	$s_{ij} = 2(s_{11} - s_{22})$	$s_{kl} = 2s_{ij}$
$c_{ij} = 0$	$c_{ij} \neq 0$	$c_{ij} = c_{kl}$	$c_{kl} = -c_{ij}$	$c_{ij} = \frac{1}{2}(c_{11} - c_{22})$	$c_{kl} = c_{ij}$

2.033 Reference systems and transformations of tensors

The components of the stress and strain tensor are connected with the coordinate system. The same tensor will show other components, if one chooses another system and describes the components with respect to the new coordinate axes. That holds for vectors as well as for 2nd and 4th rank tensors. A vector $\underline{m}$ is known to be transformed from a system X^1, X^2, X^3 to new axes X^1', X^2', X^3' by applying the transformation matrix a_{ij} :

$$m'_i = a_{ij} m_j \quad (30)$$

The components a_{ij} of the transformation matrix are given by the cosines between the new axes X^i and the old axes X^j . It can be derived from the following scheme [1]:

		old			
		X_1	X_2	X_3	
new	X_1'	a_{11}	a_{12}	a_{13}	; $a_{ij} = \cos(X^i, X^j)$
	X_2'	a_{21}	a_{22}	a_{23}	
	X_3'	a_{31}	a_{32}	a_{33}	

$$(31)$$

Each transformation matrix has the following properties: The row vectors are unit vectors and are perpendicular to each other, the same holds for the column vectors. The inverse matrix is equal to the transposed matrix

$$(a_{ij})^{-1} = (a_{ij})^T = (a_{ji}). \quad (32)$$

Therefore we can transform the stress components from the X' system to the X system by applying the transposed matrix $(a_{ji}) = (a_{ij})^T$:

$$m_i = a_{ji} m'_j \quad (33)$$

The transformation of 2nd and 4th rank tensors are performed analogously:

$$\sigma'_{ij} = a_{im} a_{jn} \sigma_{mn} \quad ; \quad \sigma_{ij} = a_{mi} a_{nj} \sigma'_{mn} \quad (34)$$

and

$$c'_{ijkl} = a_{im} a_{jn} a_{ko} a_{lp} c_{mnop} \quad ; \quad c_{ijkl} = a_{mi} a_{nj} a_{ok} a_{pl} c'_{mnop} \quad (35)$$

On the other side, Equ.34 can be used to define a tensor of 2nd rank: The 9 components σ_{ij} form a tensor of rank 2, if they transform from one set of coordinate axes to a new set according to Equ.34. The same holds for 4th rank tensors and Equ.35

Some combinations of tensor components, the scalar invariants of the tensor, do not alter, if the tensor is transformed to another coordinate system:

$$\text{vector } \underline{m}: \quad i = m_1^2 + m_2^2 + m_3^2 \quad (36)$$

tensor 2nd rank σ : $i_1 = \sigma_{11} + \sigma_{22} + \sigma_{33}$

$$i_2 = -(\sigma_{11} \sigma_{22} + \sigma_{33} \sigma_{11} + \sigma_{22} \sigma_{33}) + \sigma_{12}^2 + \sigma_{13}^2 + \sigma_{23}^2 \quad (37)$$

$$i_3 = \sigma_{11} \sigma_{22} \sigma_{33} + 2 \sigma_{12} \sigma_{13} \sigma_{23} - \sigma_{11} \sigma_{23}^2 - \sigma_{22} \sigma_{13}^2 - \sigma_{33} \sigma_{12}^2$$

tensor 4th rank c : $i_1 = c_{ijkl} \delta_{ij} \delta_{kl}$

$$i_2 = c_{ijkl} \delta_{ik} \delta_{jl} \quad (38)$$

There are some special coordinate systems, that are used frequently:

crystallite system C:

(unity vectors $\underline{C}^i$)

The axes are parallel to the symmetry axes of the considered crystal, and are built up by the three unity vectors $\underline{C}^i$ ($i=1,2,3$). The components of tensors of elastic stiffnesses show up with the simple arrangement according to Table 2.

system of

principal stresses P:

With respect to these reference axes, all non-diagonal components of the stress tensor σ are zero. For each symmetric 2nd rank tensor one set of principal axes exists. The respective normal components are called principal stresses.

specimen system S:

(unity vectors $\underline{S}^i$)

The 3-axis is the specimen's normal (ND), the 1- and the 2-axis correlate with the symmetry directions in the surface, e.g. the rolling direction (RD) and the transverse direction (TD) in case of rolled specimens.

laboratory system L:

(unity vectors $\underline{L}^i$)

The laboratory (measuring) system is connected with the direction of measurement. If a physical property is measured in the direction $\underline{m}$, the $\underline{L}^3$ -axis is parallel to $\underline{m}$. The 2-axis lies parallel to the specimen's surface. Herewith the direction of the vector $\underline{L}^1$ is fixed too as the product $\underline{L}^2 \times \underline{L}^3$.

Strains, stresses, and the elasticity tensors c and s can be described with respect to each of the coordinate systems. Parameters described with respect to the laboratory (measuring) system are marked by an apostrophe (example ϵ'_{33}), for the specimen system no indication will be added. If necessary parameters in the crystal system will be marked by a $^\circ$ (example c°_{13})

The matrices for the transformation from one to another system are given by the cosines between the axes of the respective old and the new set of axes. Fig. 3 illustrates the notation of the different matrices [7].

For performing multiplications of tensors according to Equ.15 and 16 it is important to express the components of both tensors with respect to the same set of coordinate axes.

With respect to the specimen system the direction of the measurement, $\underline{m}$ ($= \underline{L}^3$), is given by its polar coordinates, the azimuth φ and the polar angle ψ . It is usual to denote directions $(\varphi+180^\circ, \psi)$ as $(\varphi, -\psi)$, Fig. 4. The angle λ describes the rotation of the crystallites around the measuring direction $\underline{m}$, see section 2.036a.

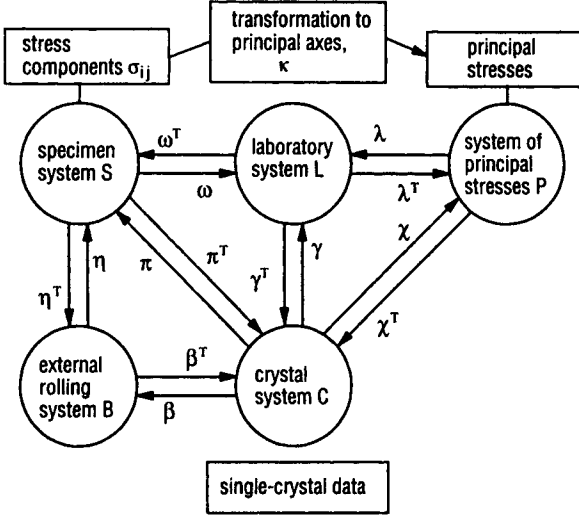


Figure 3. Notation of the different transformation matrices.

Fig. 5 shows the relative orientation between the specimen system and the laboratory system. The transformation matrix ω_{ij} between these systems can be expressed with φ and ψ :

$$(\omega_{ij}) = \begin{pmatrix} \cos\varphi \cos\psi & \sin\varphi \cos\psi & -\sin\varphi \\ -\sin\varphi & \cos\varphi & 0 \\ \cos\varphi \sin\psi & \sin\varphi \sin\psi & \cos\psi \end{pmatrix} \quad (39)$$

The third column vector is the direction of measurement $\underline{m}$, that coincides with the 3-direction of the laboratory system.

The value ϵ'_{33} in the laboratory system corresponds to the projection of the strain tensor ϵ in direction $\underline{m}$, Equ.10. ϵ'_{33} is as well obtained by transforming the ϵ tensor from the specimen- to the laboratory system.

$$\epsilon'_{ij} = \omega_{ik} \omega_{jl} \epsilon_{kl} \quad (40)$$

$$\begin{aligned} \epsilon_{\underline{m}} &= \epsilon_{ij} m_i m_j \\ &= \epsilon'_{33} = \omega_{3k} \omega_{3l} \epsilon_{kl} \\ &= \epsilon_{11} \cos^2\varphi \sin^2\psi + \epsilon_{22} \sin^2\varphi \sin^2\psi + \epsilon_{33} \cos^2\psi \\ &\quad + \epsilon_{12} \sin 2\varphi \sin^2\psi + \epsilon_{13} \cos\varphi \sin 2\psi + \epsilon_{23} \sin\varphi \sin 2\psi \end{aligned} \quad (41)$$

which corresponds to Equ.10, again using the relation $2 \sin\alpha \cos\alpha = \sin 2\alpha$.

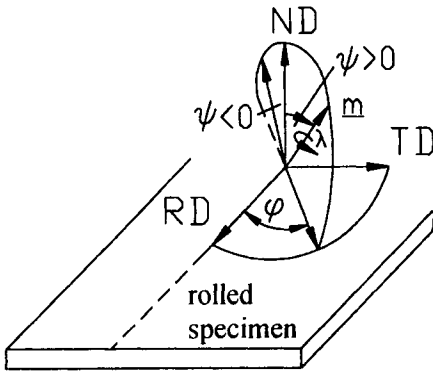


Figure 4. Description of the measuring direction by the angles ϕ and ψ .

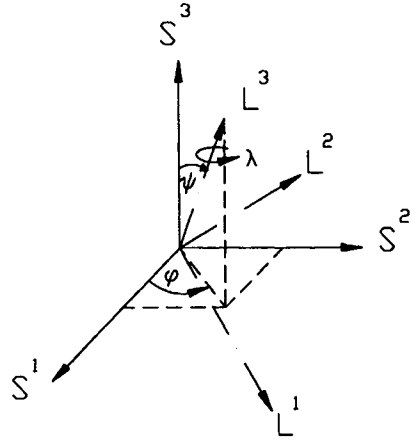


Figure 5. Orientation of the laboratory system relative to the specimen system.

2.034 Orientation of crystals within a polycrystalline material

Most materials are polycrystalline compounds of one or more material phases, they are built up by numerous monocrystals of different sizes and shapes. Each phase consists of crystals with the identical crystal lattice and physical properties. The crystals of one phase may differ to each other by their size and shape, and by the orientation of their lattice with respect to the specimen system.

One of the parameters determining the behaviour of a polycrystal is the distribution of the crystal orientations within the material. If the orientations are randomly distributed the material is quasiisotropic, i.e. the properties of the macroscopic material are the same in each direction, although, on a microscopic scale, the constituent crystals show - in general - an anisotropic behaviour. If the material is textured, i.e. the orientations are not randomly distributed, also the macroscopic body can be anisotropic.

The orientation of a crystallite in a polycrystalline composite will be described in respect to the specimen system and is given by the transformation matrix π that connects the crystal- and the specimen system. The matrix elements π_{ij} are the cosines between the vectors $\underline{S}_j$ and $\underline{C}_i$.

It is often useful to express the orientation between the specimen-system and the crystal system by the three Euler angles (ϕ_1, ϕ, ϕ_2) or by the Miller indices $\{hkl\} \langle uvw \rangle$.

$\{hkl\}$ represents the lattice plane that lies parallel to the specimen's surface and $\langle uvw \rangle$ the lattice direction parallel to the specimen's 1-axis.

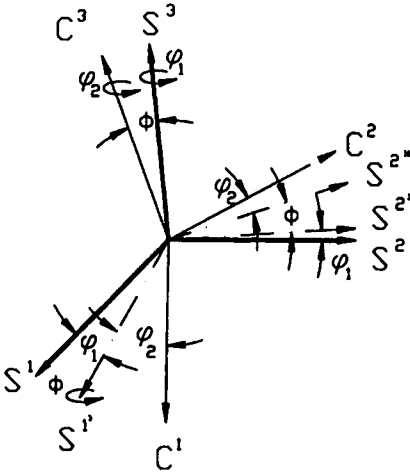


Figure 6. To the definition of the Euler angles.

The specimen system can be aligned to the crystal system by applying the following consecutive rotations of Euler angles $\varphi_1, \phi, \varphi_2$ [8], Fig.6.

The first rotation φ_1 around S^3 transforms the S^1 and the S^2 axis to the new axes $S^{1'}$ and $S^{2'}$. $S^{1'}$ has to be perpendicular to S^3 and to C^3 .

The second rotation ϕ around the $S^{1'}$ axis has to align the S^3 to the C^3 axis and transforms $S^{2'}$ to $S^{2''}$.

The third rotation φ_2 around C^3 aligns $S^{1'}$ to C^1 as well as $S^{2''}$ to C^2 .

$$0 \leq \varphi_1 \leq 2\pi; 0 \leq \varphi_2 \leq 2\pi; 0 \leq \phi \leq \pi$$

The transformation matrix π^T from the specimen to the crystallographic system can now be expressed using one of these descriptions [8]:

$$\pi^T_{ij}(\varphi_1, \phi, \varphi_2) = \begin{pmatrix} \cos\varphi_1 \cos\varphi_2 - \sin\varphi_1 \sin\varphi_2 \cos\phi & \sin\varphi_1 \cos\varphi_2 + \cos\varphi_1 \sin\varphi_2 \cos\phi & \sin\varphi_2 \sin\phi \\ -\cos\varphi_1 \sin\varphi_2 - \sin\varphi_1 \cos\varphi_2 \cos\phi & -\sin\varphi_1 \sin\varphi_2 + \cos\varphi_1 \cos\varphi_2 \cos\phi & \cos\varphi_2 \sin\phi \\ \sin\varphi_1 \sin\phi & -\cos\varphi_1 \sin\phi & \cos\phi \end{pmatrix} \quad (42)$$

$$\pi^T_{ij}((hkl)\langle uvw \rangle) = \begin{pmatrix} \frac{u}{N} & \frac{kw-lv}{MN} & \frac{h}{M} \\ \frac{v}{N} & \frac{lu-hw}{MN} & \frac{k}{M} \\ \frac{w}{N} & \frac{hv-ku}{MN} & \frac{l}{M} \end{pmatrix} \quad \text{with} \quad \begin{aligned} M &= (h^2 + k^2 + l^2) \\ N &= (u^2 + v^2 + w^2) \end{aligned} \quad (43)$$

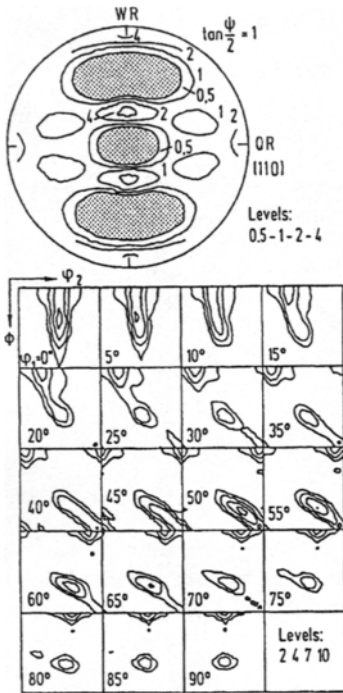


Figure 7. Intensity pole figure of the $\{110\}$ of a cold-rolled ferritic steel 1.0370 and the ODF in cuts of the Euler angle $\phi_1 = \text{const.}$

by means of X- or neutron-rays. The evaluation, calculation and assessment are among others outlined in [8,9,10].

In textured materials some orientations are highly occupied with crystals whereas other orientations are less represented. Besides the ODF, the orientations of the mainly present crystallite groups can serve to describe the texture state of a material, using the $\{hkl\}\langle uvw \rangle$ notation. For example, in cold rolled ferritic steels, the crystallite group $\{211\}\langle 011 \rangle$ is often the main texture component. Crystallite group Ω means the ensemble of all those crystallites having the same orientation Ω . Although spatially separated, for calculations of the average elastic behavior or for special stress evaluation methods, they can be treated as one crystal.

2.035 Averages of elastic data

Strain measurements by diffraction methods or by mechanical methods yield the mean value of all those crystallites that contribute to the measurement. To get the respective connection between the strains and the appertaining stresses, it is necessary to know the averaged elastic behaviour of the crystals, which results from averaging the elastic properties

The orientation distribution function (ODF) describes the relative frequency of the different crystal orientations in the polycrystal, [8,9,10]. It will be noted as $f(\Omega)$, Ω stands for the orientation expressed by the Euler angles: $f(\phi_1, \phi, \phi_2)$. It is normalized:

$$\frac{1}{8\pi^2} \int f(\Omega) d\Omega = 1 \quad (44)$$

Random distribution means $f(\Omega) \equiv 1$. Usually the ODF is graphically plotted as contour lines in the Euler space (ϕ_1, ϕ, ϕ_2) for $\phi_1 = \text{constant}$ or $\phi_2 = \text{constant}$. The orientations belonging to different combinations of angles may be physically identical due to the symmetry of the crystal lattice. It is therefore only necessary to consider the ODF in a part of the total range of angles of the Euler space. The necessary range is further determined by the specimen's symmetry. For cubic materials with orthorhombic specimen symmetry (e.g. rolled steel sheets) the range 0° to 90° of all Euler angles is sufficient to represent the ODF [8,9,10]. An example is shown in Fig.7. In fact this part of the space can be further divided in three parts, each containing all physically different crystal orientations [8].

The ODF can be evaluated from a number of intensity pole figures of different peaks measured

over the volume under study. Averaged tensors will be noted by acute parenthesis, if necessary, showing the kind of averaging, for example an averaging per volume

$$C = \langle c \rangle^V = \frac{1}{V} \int c \, dV \quad (45)$$

The average of a physical parameter taken over the volume of all crystallites of a phase will now be replaced by the average over all crystallite orientations. By doing this, the following possible influences are neglected: relations of orientations of neighbouring crystallites, the shape of the crystallites and the correlation between crystallite shape and orientation.

$$\langle c(\Omega) \rangle^\alpha = \frac{1}{8\pi^2} \int_{\Omega} c(\Omega) f(\Omega) \, d\Omega \quad (46)$$

with the ODF $f(\Omega)$ as weight function. If one uses the Euler angles, $d\Omega$ can be replaced:

$$d\Omega = \sin\phi \, d\phi_1 \, d\phi \, d\phi_2 \quad (47)$$

In Equ.46 the components of the tensors $c(\Omega)$ of the different orientations have to be described in the same system, usually the specimen system, and therefore previously transformed, according to Equ.35, from the crystal system, where they are known (c_{mnop}^o), to the specimen system:

$$\langle c \rangle_{ijkl}^\alpha = \frac{1}{8\pi^2} \iiint \pi_{im} \pi_{jn} \pi_{ko} \pi_{lp} c_{mnop}^o f(\phi_1, \phi, \phi_2) \sin\phi \, d\phi_1 \, d\phi \, d\phi_2 \quad (48)$$

A simplification of the averaging of a tensor c over all orientations is possible if the material is not textured, therefore $f(\Omega) \equiv 1$. The averaged tensor is isotropic and only two linear independent components are present. For every tensor of fourth rank the two scalar invariants [11,12]

$$c_{ijkl} \delta_{ij} \delta_{kl} \quad \text{and} \quad c_{ijkl} \delta_{ik} \delta_{jl}$$

exist (Equ.38), therefore it holds [11]:

$$\begin{aligned} \langle c \rangle_{ijkl} \delta_{ij} \delta_{kl} &= c(\Omega)_{ijkl} \delta_{ij} \delta_{kl} \\ \langle c \rangle_{ijkl} \delta_{ik} \delta_{jl} &= c(\Omega)_{ijkl} \delta_{ik} \delta_{jl} \end{aligned} \quad (49)$$

Equ.49 is valid for each orientation Ω . Therefore the components of c^o with respect to the crystal system may be inserted. These are known and the same for all crystallites of a phase. The both independent components of the averaged tensor $\langle c \rangle$ can be calculated using Equ.28, 29. All macroscopic parameters as Young's modulus, shear- and compression modulus, Poisson's ratio as well as the Lamé constants λ and μ are therefore fixed. Two of them can be taken as independent, the relations to the others are collected in Table 1 [2].

2.036 Relations between the stress state and the results of strain determinations using diffraction methods

2.036a Elastically isotropic material

The X-ray stress evaluation is based on the diffraction of the X-rays on the lattice of the crystals. The same is valid for the procedure with neutron-rays.

For every wavelength λ one can observe interferences in those directions the scattering vector of which coincides with a vector of the reciprocal lattice [5]. One formulation of the condition for the occurrence of interferences is the Bragg's law:

$$2D(hkl) \sin\Theta = n\lambda \quad (50)$$

therein $D(hkl)$ is the lattice plane distance (interplanar spacing) of the plane collective with the Miller's indices $\{hkl\}$, Θ the Bragg's angle, and n the order of the interference, which usually will be combined with the Miller's indices; example $\{222\}$ is the second order ($n=2$) of the $\{111\}$ interference. 2Θ is the angle between the primary and the diffracted X-ray beam, both beams are inclined by Θ to the lattice plane. For a fixed wavelength λ the 2Θ positions of the interferences are directly connected with the interplanar spacings $D\{hkl\}$. The precise measurement of the 2Θ angles, therefore, yields the D -spacings and, if the stress-free spacing D_0 is known, also the strain $\epsilon = (D-D_0)/D_0$.

All those crystallites of a polycrystalline material contribute to the $\{hkl\}$ -interference line, the lattice plane (hkl) or physically equivalent planes of which are oriented perpendicular to the measuring direction which is given by the direction of the scattering vector or by the halved angle between in- and outgoing X-ray, Fig.7. The orientation of the crystallites contributing to the interference can be transformed to each other by rotation λ around the measuring direction $\underline{m}$, see Fig.4 and 5.

Crystallites remote from the surface contribute with less weight to the interference line because of the exponential attenuation of the X-rays by the material.

$$I(d) = I_0 \exp(-\mu d) \quad (51)$$

with μ the attenuation coefficient of the material and d the penetrated distance.

The positions of the diffracted interference line therefore yield the average D -values, taken over the volume V^c of all crystallites contributing to the interference, weighted with the exponential decrease:

$$D = \frac{\int_{V^c} D(z) \exp(-z/\tau) dz}{\int_{V^c} \exp(-z/\tau) dz} \quad (52)$$

with z the depth and τ the penetration depth, i.e. the thickness of the surface region from which the part $(1-1/e) \approx 63\%$ of the information is obtained. It depends on the wavelength, the material, and of the direction of measurement. These averages, that are taken over the contributing crystallites considering the attenuation of the X-rays will be meant, if we speak about "X-ray averaging", which sometimes will be marked by an index "x". The intensity of the

interference line supplies a measure of the mean volume part of the participating crystallite orientations. The respective averaging of neutron measurements are in detail discussed in part 3.

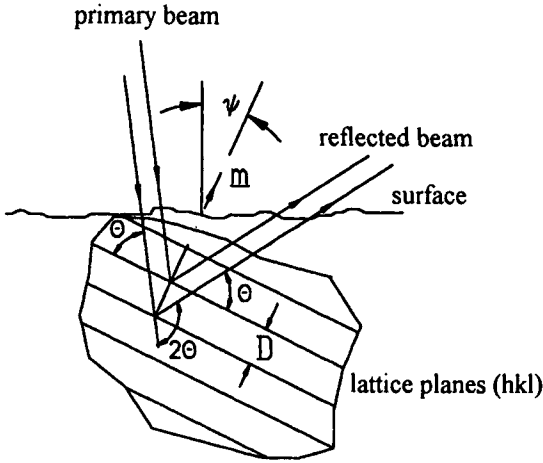


Figure 8. Reflection of X- or neutron-rays at the lattice planes of a crystallite.

The principle of the stress analysis by X- and neutron rays is the determination of the stress state in the material by the measurement of the interplanar spacings in different directions. The strain measured in the direction $\underline{m}$, that in the specimen system is given by the angles φ, ψ , corresponds to the ϵ'_{33} -component in the laboratory system, Fig.5.

$$\epsilon_{\underline{m}} = \epsilon_{\varphi\psi} = \langle \epsilon_{33} \rangle^x = \frac{D_{\varphi\psi} - D_0}{D_0} \quad (53)$$

If the elastic properties of the material are homogeneous on a microscopic scale, the X-ray results and the macroscopic strain values are identical. $\epsilon_{\varphi\psi}$ can be expressed as projection of the strain tensor in the measuring direction (φ, ψ) within the specimen system [13], Equ.9, or as the ϵ'_{33} component in the laboratory system, according to Equ.41.

$$\begin{aligned} \epsilon_{\varphi\psi} &= \langle \epsilon_{ij} \rangle^x m_i m_j \\ &= \langle \epsilon_{11} \rangle^x \cos^2\varphi \sin^2\psi + \langle \epsilon_{22} \rangle^x \sin^2\varphi \sin^2\psi + \langle \epsilon_{33} \rangle^x \cos^2\psi \\ &\quad + \langle \epsilon_{12} \rangle^x \sin 2\varphi \sin^2\psi + \langle \epsilon_{13} \rangle^x \cos\varphi \sin 2\psi + \langle \epsilon_{23} \rangle^x \sin\varphi \sin 2\psi \end{aligned} \quad (54)$$

Assuming the ϵ_{ij} to be homogeneous within the penetration depth of the X-rays and the material to be elastically isotropic, and applying the Hooke's law of isotropic materials:

$$E \epsilon_{ij} = (1+\nu) \sigma_{ij} - \nu \delta_{ij} (\sigma_{11} + \sigma_{22} + \sigma_{33}) \quad (55)$$

we obtain the respective relation between $\epsilon_{\varphi\psi}$ and the σ_{ij} :

$$\begin{aligned}
\varepsilon_{\varphi\psi} = & \frac{1}{2}s_2^m [\sigma_{11} \cos^2\varphi \sin^2\psi + \sigma_{22} \sin^2\varphi \sin^2\psi + \sigma_{33} \cos^2\psi] \\
& + \frac{1}{2}s_2^m [\sigma_{12} \sin 2\varphi \sin^2\psi + \sigma_{13} \cos\varphi \sin 2\psi + \sigma_{23} \sin\varphi \sin 2\psi] \\
& + s_1^m [\sigma_{11} + \sigma_{22} + \sigma_{33}]
\end{aligned} \tag{56}$$

with the macroscopic elastic data

$$\frac{1}{2}s_2^m = \frac{1+\nu}{E}, \quad s_1^m = \frac{-\nu}{E} \tag{57}$$

Equ.56 represents the strain-stress relation of an elastically isotropic body. It is valid only, if the measuring method of $\varepsilon_{\varphi\psi}$ averages over all phases and all crystallite orientations, that means over a representative volume of the material. Examples are the mechanical and the non-destructive method using ultrasonic waves. But in general, it is not valid in this form for the results of diffraction measurements.

Strain measurements using X- or neutron-rays samples for each direction (φ, ψ) only those crystallites the planes $\{hkl\}$ of which are perpendicular oriented to the direction (φ, ψ) . This part of all crystallites is different for each measuring direction. The equation corresponding to Equ.54 is therefore not the projection of the same averaged strain tensor on each measuring direction, but for each direction a different strain tensor must be considered. The $\langle \varepsilon \rangle_{ij}^*$ on the right side of Equ.54 should be replaced by $[\langle \varepsilon \rangle^*(\varphi, \psi)]_{ij}$ which is dependent on (φ, ψ) , i.e. the measuring direction m. The result corresponds to Equ.54 if the crystals are elastically isotropic, but it will differ for anisotropic crystals, as it is discussed in the next section.

2.036b Quasiisotropic polycrystalline material, definition of the X-ray elastic constants (XEC)

Measurements using diffraction methods always pick up only a small part of all crystallites, depending on the lattice plane under study. The elastic behavior of this collective may differ from the macroscopic one due to the elastic anisotropy of the crystals. To take the elastic anisotropy into consideration the constants $\frac{1}{2}s_2^m$ and s_1^m in Equ.56 were formally replaced by the $\{hkl\}$ -dependent "X-ray elastic constants (XEC)" $s_1(hkl)$ and $\frac{1}{2}s_2(hkl)$. The validity of this procedure was experimentally often proved. The theoretical proof was done by Stickforth [14]. He showed that for macroscopic stresses the macroscopic isotropy and homogeneity are sufficient presuppositions. Independently on the model assumption about the crystallite coupling, the averaged stresses $\bar{\sigma}^\alpha$ of a phase result in lattice strain distributions according to Equ.58 [2,14,15], which was firstly introduced by [13].

$$\begin{aligned}
\varepsilon_{\varphi\psi} = & \frac{1}{2}s_2(hkl) [\sigma_{11} \cos^2\varphi \sin^2\psi + \sigma_{22} \sin^2\varphi \sin^2\psi + \sigma_{33} \cos^2\psi] \\
& + \frac{1}{2}s_2(hkl) [\sigma_{12} \sin 2\varphi \sin^2\psi + \sigma_{13} \cos\varphi \sin 2\psi + \sigma_{23} \sin\varphi \sin 2\psi] \\
& + s_1(hkl) [\sigma_{11} + \sigma_{22} + \sigma_{33}]
\end{aligned} \tag{58}$$

Equ.58 is the fundamental strain-stress relation of the X-ray stress analysis. Triaxial stress states can easily be evaluated using the measured lattice-strain distributions in azimuths $\varphi = 0^\circ, 45^\circ$ and 90° [16].

The XEC can be determined experimentally in uniaxial tension or bending tests [17,18], or they can be calculated using the single-crystal data and a model about the elastic coupling of the crystallites within the material [15,19,20,21,22,23]. Averaging the XEC of all planes (hkl) have to result in the macroscopic value:

$$\langle s_1(hkl) \rangle^{hkl} = s_1^m = \frac{-\nu}{E} \quad (59)$$

$$\langle \frac{1}{2}s_2(hkl) \rangle^{hkl} = \frac{1}{2}s_2^m = \frac{1+\nu}{E} \quad (60)$$

X-ray measurements will be done predominantly on a certain azimuth. Equ.58 simplifies for $\varphi=0^\circ$:

$$\begin{aligned} \epsilon_{\varphi=0,\psi} &= s_1(hkl) [\sigma_{11} + \sigma_{22} + \sigma_{33}] + \frac{1}{2}s_2(hkl) \sigma_{33} \\ &+ \frac{1}{2}s_2(hkl) [\sigma_{11}-\sigma_{33}] \sin^2\psi \\ &+ \frac{1}{2}s_2(hkl) \sigma_{13} \sin 2\psi \end{aligned} \quad (61)$$

The diagrams D or ϵ versus $\sin^2\psi$ show linearity if only principal stresses are present. Shear components lead to elliptical splitting between the values $D_{\psi<0}$ and $D_{\psi>0}$ (ψ -splitting).

However, Equ.58 and all consequently connected relations regard only the parts of strains, that are originated by averaged stresses $\bar{\sigma}^\alpha$. The different kinds of residual stresses are the subject of the next paragraph 2.037.

2.036c Textured polycrystalline material, definition of the X-ray stress factors

In case of textured materials, the relation between the measurable lattice strains and the averaged stresses within the respective phase cannot be presented in the simple form of Equ.58. D -vs.- $\sin^2\psi$ plots regularly show not up not with linear dependences but with more or less oscillations, depending on the lattice plane. Exceptions are the $\{h00\}$ and $\{hhh\}$ of cubic materials. They are principally linear [24] as long as influences of texture in connection with average phase stresses are present only. Also the D -vs.- $\sin^2\psi$ dependences of textured materials built up by elastically isotropic crystallites are linear, so they are created by averaged stresses $\bar{\sigma}^\alpha$. The dependence of the X-ray-averaged strains on the mean stresses will be described by the X-ray stress factors F_{ij} , [7,25,26]:

$$\epsilon_{\varphi\psi} = F_{ij}(\varphi, \psi, hkl) \bar{\sigma}_{ij} \quad (62)$$

They are dependent on the measuring direction and the respective lattice plane $\{hkl\}$. But they do not form a tensor and, therefore, must not be transformed to other coordinate systems. Due to the elastic anisotropy, the strain value $\epsilon_{\varphi\psi}$, and herewith the F_{ij} , are dependent on the

frequency of the different orientations, i.e. the texture present. The transition to the quasi-isotropic material follows by comparison with Equ.58 and 61. For quasiisotropic materials the F_{ij} stand for a combination of the s_1 and $\frac{1}{2}s_2$. For example $F_{11}(0^\circ, \psi, hkl)$ becomes:

$$\begin{aligned} \text{textured} &\rightarrow \text{quasiisotropic} \\ F(0^\circ, \psi, hkl) &\rightarrow s_1(hkl) + \frac{1}{2}s_2(hkl) \sin^2\psi \end{aligned} \quad (63)$$

The respective combinations ($\varphi=0^\circ$) for the other F_{ij} are:

$$F_{ij}(0^\circ, \psi, hkl)_{\text{quasiisotropic material}} = \begin{pmatrix} s_1(hkl) + \frac{1}{2}s_2(hkl)\sin^2\psi & 0 & (1/2) \frac{1}{2}s_2(hkl)\sin^2\psi \\ 0 & s_1(hkl) & 0 \\ (1/2) \frac{1}{2}s_2(hkl)\sin^2\psi & 0 & s_1(hkl) + \frac{1}{2}s_2(hkl)\cos^2\psi \end{pmatrix} \quad (64)$$

For a given stress state the profiles of F_{ij} versus $\sin^2\psi$ determine the form of the D-vs.- $\sin^2\psi$ distributions. As well as the XEC they can be determined experimentally by tension- or bending tests, or they can be calculated from the single crystal data considering the respective weight function ODF [25,27,28,29]. Details will be discussed in chapter 2.13

As it is the fact for Equ.58 in case of quasiisotropic materials, also Equ.62 considers only that part of stresses that is caused by the averaged stresses $\bar{\sigma}$ within the phase.

2.037 Kinds of stresses, and their mutual relations

All stress states within a material which are independent of outside forces are called residual stresses (RS). Load- or applied stresses (LS) are caused by outside forces. The average of the residual stresses taken over each cross section of the body has to be zero.

A classification of the different kinds of RS was made in [30] on the basis of the literature published up to that date. The distinction of RS of kind I (macro-RS), and of kind II and III (micro-RS) was fixed. Up to now, this is the basis of discussion about stresses determinable by diffraction methods.

The RS of kind I, σ^I , is the volume average of the position-dependent residual stresses $\sigma(x)$, taken over all crystallites and phases within the considered volume, e.g. the volume exposed to the X-ray beam or the volume studied by strain gauges. It has to be chosen large enough to represent the macroscopic material.

$$\sigma^I = \frac{1}{V} \int_V \sigma(x) dV \quad (65)$$

A shift or a release of σ^I can cause macroscopic alterations of the body's shape. This is the basis of mechanical methods of stress determination.

The σ^{II} within a crystal are defined as the mean deviation from the macrostress level, and σ^{III} as the position-dependent deviations from the average stress of the crystal:

$$\sigma^{\text{II}} = \frac{1}{V} \int_{\substack{\text{crystal} \\ \text{volume } V}} [\sigma(x) - \sigma^{\text{I}}] dV \quad (66)$$

$$\sigma^{\text{III}}(x) = \sigma(x) - \sigma^{\text{I}} - \sigma^{\text{II}} \quad (67)$$

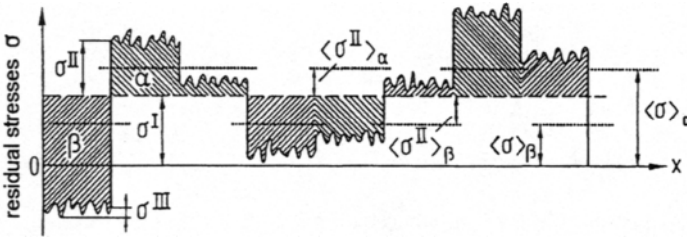


Figure 9. To the definition of residual stresses [31].

To accommodate the conception of the micro-RS to the results of X-ray and neutron-ray methods, we will comprehend as RS II the average $\langle \sigma^{\text{II}} \rangle_\alpha$ taken over the crystallites of the phase or over the crystallites contributing to the measurement, Fig.8, [31]

$$\text{RS II} = \langle \sigma^{\text{II}} \rangle_\alpha = \frac{1}{V} \int_{\substack{\text{phase} \\ \text{volume } V}} [\sigma(x) - \sigma^{\text{I}}] dV \quad (68)$$

The respective average of σ^{III} is equal to zero, by definition. According to the definition, the average value $\langle \sigma^{\text{II}} \rangle_{\Delta V}$ taken over all crystallites gets zero; ΔV is a volume which contains sufficient crystallites to be representative. But diffraction methods study the different phases separately, and these may be stressed against each other due to differences of their elastic and plastic properties. Furthermore, even in single-phase materials only a part of all crystallites, depending on the measuring direction, contributes to the interference line. The strain of this part may differ from the strain of another crystal collective that diffracts at a second measuring direction. As a consequence there may be $\langle \sigma^{\text{II}} \rangle_x$ measurable even in single-phase materials.

Similar reasons hold for the existence of $\langle \sigma^{\text{III}} \rangle_x$. The RS σ^{III} average to zero over one crystallite, so it is defined. Therefore, it should influence the widths of the peaks only and not the line shifts. This holds, if the whole volume of the crystallites contribute to the measurement with the same weight. But especially in plastically deformed materials this is not the case. Within the crystallites dislocation cell structures may have been created. The volume inside the cells and the cell walls are stressed against each other. The regions of low dislocation density (cell interiors) show higher diffraction capability than the cell walls with their high dislocation density. Therefore, the cell interiors predominate the observed peak shifts and stresses $\langle \sigma^{\text{III}} \rangle_x$ can be determined [32,33,34].

The X-ray method determines the stress $\bar{\sigma}$ of the considered phase of the material. $\bar{\sigma}$ is the average value taken over those crystallites of a phase α that contribute to the result, if necessary, it will be marked with an index, $\bar{\sigma}^\alpha$. According to this definition $\bar{\sigma}^\alpha$ contains LS, RS of kind I, the average RS-value of kind II, and the X-ray RS-average of kind III in the phase.

$$\bar{\sigma}^\alpha = \sigma^L + \sigma^I + \langle \sigma^{II} \rangle^\alpha + \langle \sigma^{III} \rangle^\alpha = \sigma^m + \langle \sigma^{II} \rangle^\alpha + \langle \sigma^{III} \rangle^\alpha \quad (69)$$

The terms σ^L and σ^I are constant within the exposed volume. The sum $\sigma^L + \sigma^I$ is the stress average over all crystallites and phases. It will be signified as macrostresses (index m), and can be determined also by mechanical methods.

The microstresses $\langle \sigma^{II} \rangle^\alpha$ represent the average mutual constraints between the phases which are compensating each other.

$$\sum_{\alpha=1}^n c^\alpha \langle \sigma^{II} \rangle^\alpha = 0 \quad (70)$$

with c^α the volume portions and n the number of phases.

In case of absence of $\langle \sigma^{III} \rangle^\alpha$, the respective averaging of the stresses $\bar{\sigma}$ over all the n different phases present in the volume ΔV will result in the macrostress σ^m :

$$\sum_{\alpha=1}^n c^\alpha \bar{\sigma}^\alpha = \sigma^L + \sigma^I = \sigma^m \quad (71)$$

Equations 70 and 71 are used to separate macro- and microstresses.

In the following, a subdivision of the microstresses will be introduced. This is of advantage for the handling of the formulae to determine the XEC, chapter 2.13, of the separation of the micro-RS of different origins, chapter 2.06, and of the interpretation of lattice strain distributions of plastically deformed materials, chapter 2.16. The detailed consideration is necessary since the averaged $\langle \sigma^{II} \rangle^\alpha$ contain parts which depend on load stresses and macro-RS.

All crystallites of a phase in the volume ΔV which have the same orientation Ω will be collected to a crystallite group, abbreviation Ω . The averaged stress state of a crystallite group is $\sigma(\Omega)$. The crystallites of one group have all the same elastic data expressed with respect to a space-fixed coordinate system. The crystallite group Ω will therefore be treated as one single crystal with the orientation Ω . In the absence of texture, the phase-averaged value $\bar{\sigma}^\alpha$ is therefore

$$\bar{\sigma}^\alpha = \langle \sigma(\Omega) \rangle^{\Omega, \alpha} \quad (72)$$

The stress $\sigma(\Omega)$ consists of a part which is originated by the averaged stresses $\bar{\sigma}$ (linear dependent) and of an independent part which is also present when $\bar{\sigma}^\alpha=0$, called $\sigma_0^\alpha(\Omega)$, and in the same way $\varepsilon_0^\alpha(\Omega)$.

$$\sigma^\alpha(\Omega) = \sigma_0^\alpha(\Omega) + B(\Omega) \bar{\sigma}^\alpha \quad (73)$$

$$\varepsilon^\alpha(\Omega) = \varepsilon_0^\alpha(\Omega) + A(\Omega) \bar{\sigma}^\alpha \quad (74)$$

$$\text{with } \varepsilon_0^\alpha(\Omega) = s(\Omega) \sigma_0^\alpha(\Omega)$$

$$A(\Omega) = s(\Omega) B(\Omega)$$

The elastic coupling of the crystallites within a phase is expressed by the 4th rank tensors A and B. They determine the distribution of the averaged stresses $\bar{\sigma}$ on the different orientations or the crystallite groups. Different assumptions of models are in use to calculate the tensors A and B. They will be treated in chapter 2.13.

It can be proved [2,14], that the second term on the right side of Equ.74, $A(\Omega) \bar{\sigma}$, will lead to a lattice-strain distribution according to Equ.58.

The micro-RS $\sigma_0(\Omega)$ are compensating each other between the crystallites of different orientations and they will deliver no contribution to the stress average value. But they can essentially influence the $D_{\varphi\psi}$ -or $\varepsilon_{\varphi\psi}$ -distributions. The reason is merely the selective character of diffraction methods. The usual evaluation of measured results by diffraction methods on the basis of Equ.58 presumes, that this part can be neglected.

The microstresses $\sigma_0(\Omega)$ are correlated with the orientation of the crystallite groups. They may be originated by anisotropic plastic behavior or by anisotropic thermic expansion of the crystallites. They have to compensate between all crystallite orientations, i.e. between the crystallite groups of a phase; it holds:

$$\langle \sigma_0(\Omega) \rangle^\alpha = 0 \quad ; \quad \langle \varepsilon_0(\Omega) \rangle^\alpha = 0 \quad (75)$$

Analogous to the definition of the stress $\sigma_0(\Omega)$ as $\sigma(\Omega, \bar{\sigma}^\alpha=0)$, there will be a part of the phase stress $\bar{\sigma}^\alpha$ which is independent from load stresses or macroscopic residual stresses (load- and macro-RS)

$$\bar{\sigma}_0^\alpha = \bar{\sigma}^\alpha (\sigma^I + \sigma^L = 0) \quad (76)$$

$$\text{with } \sum_{\alpha=1}^n c^\alpha \bar{\sigma}_0^\alpha = 0 \quad (77)$$

These stresses $\bar{\sigma}_0^\alpha$ are originated by differences in the macroscopic plastic behavior and by the macroscopic thermal expansion coefficients of the phases within the material, whereas the $\sigma_0(\Omega)$ were originated by microscopic or orientation-dependent differences within a phase.

To get the correlation with the usual classification of the stresses, the distribution of the load stresses and the RS of kind I on the different phases has still to be regarded. These both macroscopic kinds of stresses are defined to be constant over many crystallites and their effect on the strain of the crystallites is supposed to be the same in macroscopically homogeneous materials. This is the reason why they are called macrostresses.

Every macrostress causes an alteration of the averaged phase stresses $\bar{\sigma}^\alpha$ which depends on the space arrangement and the macroscopic elastic characteristics of all phases. An averaged stress state within the volume of the material $\sigma^m = \sigma^L + \sigma^I$ may not be equally distributed to all phases. Deviations of the phase stresses $\bar{\sigma}^\alpha$ from the macrostress level $\sigma^L + \sigma^I$ are

defined as microstresses $\langle \sigma^{II} \rangle^\alpha + \langle \sigma^{III} \rangle^\alpha$. These parts of microstresses, that are originated by macro-stresses, are proportional to the latter as long as no relaxation by plastic deformation, creep or diffusion exists.

With defining the tensor f of the stress-transfer factors f_{ijkl} :

$$(f^\alpha)_{ijkl} = \frac{\partial(\bar{\sigma}^\alpha)_{ij}}{\partial(\sigma^L + \sigma^I)_{kl}} \quad (78)$$

the averaged stress of the phase α can be written as

$$\bar{\sigma}^\alpha = \bar{\sigma}_0^\alpha + f^\alpha [\sigma^L + \sigma^I] = \bar{\sigma}_0^\alpha + \sigma^L + \sigma^I + [f^{\alpha-I}] [\sigma^L + \sigma^I] \quad (79)$$

with

$$\sum_{\alpha=1}^n c^\alpha f^\alpha = I \quad (80)$$

With Equ.77 and 80, the averaging of Equ.79 yields again Equ.71. The definition of f in Equ.78 can simpler be written using Voigt's notation.

$$(f^\alpha)_{mn} = \frac{\partial(\bar{\sigma}^\alpha)_m}{\partial(\sigma^L + \sigma^I)_n} ; \quad m, n = 1..6 \quad (81)$$

In macroscopically homogeneous materials, f is of isotropic structure as long as surface effects can be neglected.

After introducing the subdivision of stresses, the following relations hold, in accordance with Fig. 9 (CG: crystallite group)

$$\begin{aligned} \langle \sigma^{II} \rangle^{CG} &= \sigma(\Omega) - \sigma^L - \sigma^I \\ &= \sigma_0(\Omega) + B(\Omega) \bar{\sigma}_0^\alpha + [B(\Omega) f^{\alpha-I}] [\sigma^L + \sigma^I] \end{aligned} \quad (82)$$

$$\begin{aligned} \langle \sigma^{II} \rangle^\alpha &= \bar{\sigma}^\alpha - \sigma^L - \sigma^I \\ &= \bar{\sigma}_0^\alpha + [f-I] \cdot [\sigma^L + \sigma^I] \end{aligned} \quad (83)$$

The last equation illustrates, that the RS of kind II, defined as the averaged value $\langle \sigma^{II} \rangle^\alpha$, beside the constant term $\bar{\sigma}_0$, contains a part $[f^{\alpha-I}] [\sigma^L + \sigma^I]$ which is dependent on the macro-stress. The tensor $[f^{\alpha-I}]$ is a measure of the differences between the elastic properties of the phases within the material. This tensor can experimentally be evaluated by determining the compound-XEC, or it can be theoretically calculated. In the calculations, the mutual elastic constraints between the phases are considered by respective models about the crystal coupling. This will be discussed in chapter 2.13. The above mentioned considerations are valid for macroscopically homogeneous materials. The tensor f can be position-dependent if the distribution of the phases is macroscopically heterogeneous, e.g. in layer- and fiber-composite materials. In these cases distinction has to be made between the stress-transfer factors of the load stress and of the RS of kind I.

2.038 References

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2.04 Lattice strain measuring techniques

2.041 Physical fundamentals

X-rays are electromagnetic waves with wavelengths ranging from approximately 1 to 0.01 nm (10 to 0.1 Å). X-rays are generated when electrons with a high velocity are decelerated upon penetrating matter. In principle, X-rays can be produced by two different mechanisms. When a fast electron is decelerated on passing through the strong magnetic field near a nucleus, it can give off a part of its energy as X-rays with the frequency ν , which is given by the following relation:

$$\Delta E = h\nu \quad (1)$$

As this equation clearly shows, the maximum energy of the X-rays cannot be greater than that of the accelerated electron, which is given by the acceleration voltage. Thus, the shortest possible wavelength that can be produced with a given acceleration voltage can be calculated using Equation 1. This is only valid if the electron gives off all its energy in one step.

$$\Delta E_{\max} = eU = h \cdot \nu_{\max} = \frac{hc}{\lambda_{\min}} \quad (2)$$

e charge of the electron: $1.602 \cdot 10^{-19} \text{ C}$

U acceleration voltage in volt

h Planck's constant: $6.626 \cdot 10^{-34} \text{ Js}$

c light velocity: $2.998 \cdot 10^8 \text{ ms}^{-1}$

After inserting the constants into Equation 2, the following equation is obtained:

$$\lambda_{\min} = \frac{12400}{U} \text{ Å} \quad (3)$$

For example, the shortest X-ray wavelength that can be obtained using an acceleration voltage of 25 kV is $\lambda = 0.496 \text{ Å}$.

But deceleration takes place in steps of any size between zero and $\Delta E_{\max}$; thus the radiation consists of a spectrum of energies, forming a white spectrum.

The wavelength at the highest intensity of the radiation is found at about $1.5\lambda_{\min}$. Towards high energies, the profile ends abruptly at the wavelength $\lambda_{\min}$, corresponding to Equation 3. Towards lower energies, the relative intensity approaches zero very smoothly. If the acceleration voltage suffices to eject an inner shell electron from the target material, the continuous X-ray spectrum is superposed by characteristic radiation lines. The wavelength of this radiation is characteristic for each element (characteristic spectrum).

The origin of the characteristic X-ray radiation can be explained according to Bohr's model of the atom.

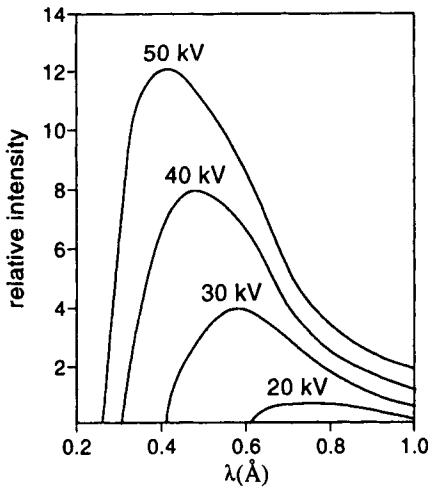


Figure 1. The maximum of the intensity, as well as the short-wavelength limit of the continuous X-ray spectrum, move towards shorter wavelengths as the acceleration voltage is increased (tungsten anode) [1].

When an electron near the nucleus is hit by a highly energetic electron, it can be ejected from the atom. Thus, the atom gains energy, and reaches an excited state. Having lost an electron from the K shell, the atom reaches a K state; when an electron is missing from the L shell, the atomic state is called an L state, and so on. Because the bonding energy of K-electrons exceeds that of L-electrons, the K state possesses more energy than the L state. X-ray radiation is produced, when the excited atom decays to the ground state by filling the vacancy

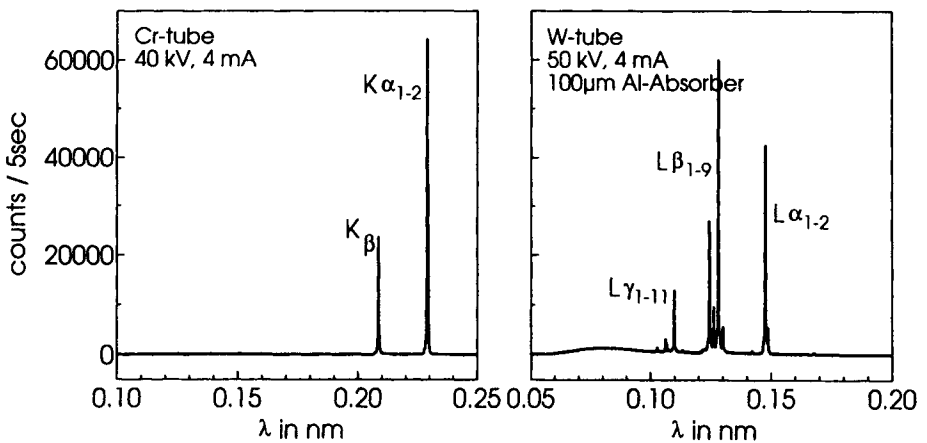


Figure 2. The characteristic radiation: K series of a Cr tube and L series of a W tube [2].

with an electron from a higher energy shell. The energy that this electron is losing in going from a higher to a lower energy shell is emitted in form of X-rays.

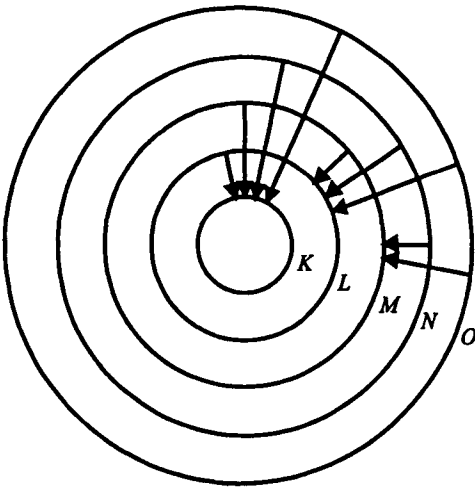
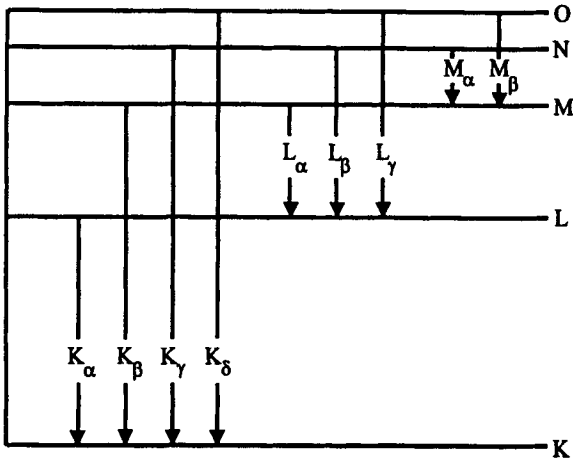


Figure 3. Energy level diagram (above) and simplified shell model of the atom (below) [1].

For X-ray diffraction studies, mainly the K-series is used. According to Moseley's law, the atomic number Z is proportional to the square root of the frequency ν of the X-ray radiation lines.

$$Z = k_1 \cdot \sqrt{\nu_{K\alpha}} + k_2 \quad (4)$$

k_1, k_2 - constants

$\nu_{K\alpha}$ - frequency of K_α -radiation

When passing through matter, X-rays are attenuated. The attenuation in the frequency range used for X-ray diffraction is caused by photoabsorption and scattering.

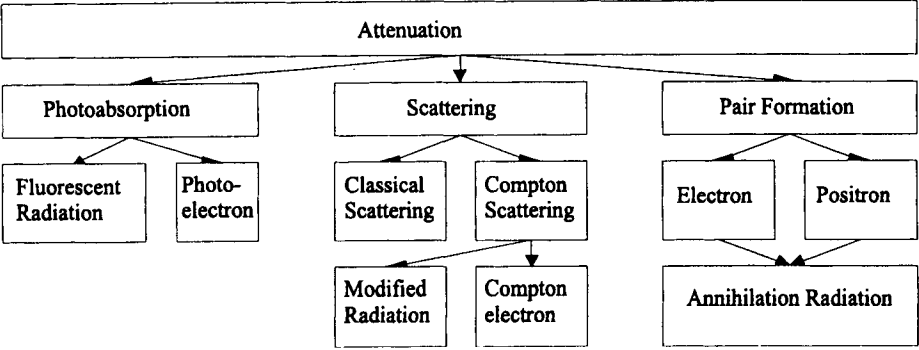


Figure 4. Relation between the physical processes in X-ray attenuation (for radiation energies less than 1.02 MeV, only the left and middle column apply; for radiation energies higher than 1.02 MeV, all columns are valid).

When a thin, monochromatic X-ray, possessing the intensity I_0 , passes through a homogeneous plate, t cm thick and oriented perpendicular to the ray, the intensity of the emerging X-ray is given by Equation 5:

$$I = I_0 \exp(-\mu t) \tag{5}$$

μ is called the linear attenuation coefficient in cm^{-1} . Because the attenuation of the intensity depends on the amount of the material that is penetrated, μ is often divided by the material density, yielding the mass-attenuation coefficient, a value independent of the chemical and physical state of the absorber. μ/ρ is given in cm^2/g . In mixtures and compounds, the mass-attenuation coefficient is the sum of the component mass-attenuation coefficients. The μ/ρ values depend on the wavelength. When the μ/ρ values are plotted as a function of the X-ray wavelength for any element, the curve is not smooth, but it has pronounced drops, which are called absorption edges. Figure 5 shows this function for tungsten. For wavelengths between 2.0 and 1.2 Å the value of μ/ρ decreases continuously towards shorter wavelengths (higher energies), and then it increases abruptly. This happens when the energy of the radiation is just high enough to eject an electron from the L-III shell of the atom, causing a rise in the attenuation coefficient. When the energy is increased further, at first, the mass-attenuation coefficient decreases; upon reaching certain energies; it rises again.

When dealing with X-ray diffraction, it is, for a number of reasons, of interest to pay attention to the absorption edges. For example, there are absorption filters and monochromators, which are distinguished by possessing absorption edges within the range of K_β -radiation, while they attenuate K_α -radiation significantly less. Also, an adverse composition of the photomaterial may lead to an attenuation of the radiation by means of

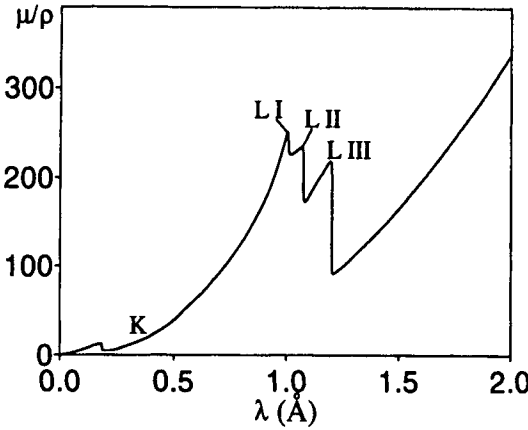


Figure 5. Mass-attenuation coefficient and in cm^2/g absorption edges of tungsten [1].

photoabsorption. As a consequence, fluorescent radiation is produced, which increases the X-ray background radiation level.

When dealing with low incidence angles α , refraction and total reflection are of interest [3, 4]. Here, formulae will be used, which were presented by R.W. James and used by P. K. Predecki et al [5].

Figure 6 and also Figure 26 in paragraph 2.045 show the principal arrangement for grazing incidence measurements.

The deviation of the measured value $2\Theta^l$ from the true value $2\Theta_0$ is given by

$$2\Theta^l - 2\Theta_0 = \frac{\delta}{\sin 2\Theta_0} \left[2 + \frac{\sin \alpha}{\sin(2\Theta_0 - \alpha)} + \frac{\sin(2\Theta_0 - \alpha)}{\sin \alpha} \right] = \delta \left(\frac{2}{\sin 2\Theta_0} + \frac{1}{\alpha} \right) \quad (6)$$

$$\delta = \frac{Ne^2 \lambda^2}{2\pi mc^2} = 1 - n \approx 10^{-6} \quad (7)$$

The refraction coefficient is denoted as n . According to [6], the index of refraction for X-rays is

$$n = 1 - \delta - i\beta \quad (8)$$

where δ is the refractive, and β the absorption-index decrement. The critical angle for total reflection is $\Theta_{\text{total}} \approx \sqrt{2\delta}$. Figure 7 shows the reflectivity curves normalized at Θ_{total} . The fraction of reflection depends on the ratio of absorption over refraction.

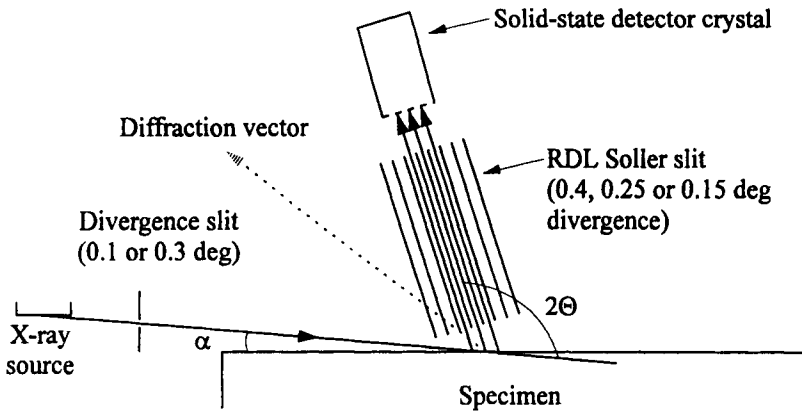


Figure 6. Principal arrangement for grazing incidence measurements on an Ω -diffractometer, asymmetric pseudo parallel beam optics [5].

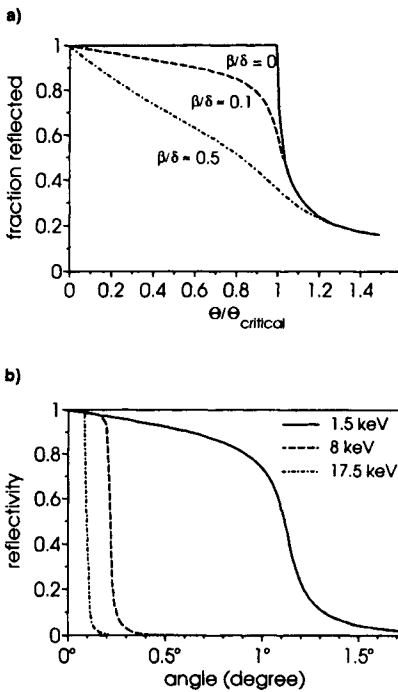


Figure 7. a) Reflectivity curves based on the parameters β and δ , in the index of refraction. The curves are normalized at the critical angle. b) Examples of the reflectivity of SiO_2 as a function of the angle for 1.5, 8 and 17.5 keV photons [6].

2.042 Radiation sources; choice of X-ray tube

The X-ray sources are either tubes with static or with rotating anodes and the synchrotron radiation. The characteristics that are important for the selection of the suitable tube are the following: wavelength of the target material, focal size, maximum rating and intensity on the specimen to be tested. In the following, typical features will be demonstrated. Detailed information on the tubes may be obtained from the leaflets of the manufacturers. Table 1 contains the wavelengths of the usually produced tubes. The maximum voltage usually is 50 or 60 kV.

Table 1. Common target elements with appertaining wavelengths of eigenradiations in nm and β -filters [7, 8].

Target element	$K_{\alpha 1}$	$K_{\alpha 2}$	$K_{\beta 1}$	β -filter
Al	0.833899	0.834144	0.79811	–
Ti	0.274841	0.275205	0.251382	–
V	0.250340	0.250718	0.228430	Ti
Cr	0.2289649	0.2293531	0.2084789	V
Mn	0.210175	0.210574	0.191005	Cr
Fe	0.1935979	0.1939923	0.1756554	Mn
Co	0.1788893	0.1792801	0.1620703	Fe
Cu	0.1540501	0.1544345	0.139216	Ni
W*	0.147634	0.148738	0.128175	Cu
Mo	0.0709261	0.0713543	0.0632253	Zr
Ag	0.0559363	0.0563775	0.049701	Pd,Rh,Ru
Au	0.0180185	0.0185064	0.0158971	Ir,Os,Re,W

* values for $L_{\alpha 1}$, $L_{\alpha 2}$, $L_{\beta 1}$

Table 2 shows the focal spot sizes and the maximum rating of the different types. Effective line- and point focuses are usually available. The high intensity beam of the rotating anodes allows the use of a strong parallel beam to measure lattice strains up to high values ψ .

Table 2. Some examples of data of X-ray tubes. Focal spot sizes are in mm times mm, the maximum rating is given in watts [9].

target material	static anode			rotating anode			
	normal focus 1 x 10	fine focus 0.4 x 8	long fine focus 0.4 x 12	0.5 x 10	0.3 x 3	0.2 x 2	0.1 x 1
Cr	1800	1300	1900	12000	3600	2000	800
Fe	1500	900	1000	9000	2700	1500	600
Co	1800	1200	1800	12000	3600	2000	800
Cu	2000	1500	2200	18000	5400	3000	1200
Mo	2400	2000	3000	18000	5400	3000	1200
W	2400	2000	3000				

All different designs use beryllium as the material for the windows. The attenuation coefficients μ for beryllium are listed in Table 3 for different radiations [10].

Table 3. Attenuation coefficients in cm^{-1} of beryllium and air for different radiations [10, 11].

radiation	Ti- K_{α}	Cr- K_{α}	Fe- K_{α}	Co- K_{α}	Cu- K_{α}	Mo- K_{α}	W- L_{α}	W- L_{β}
μ_{Be}	10.841	6.2345	3.7555	2.9415	1.8315	0.481	1.6465	1.221
μ_{air}	0.06793	0.03922	0.02338	0.01868	0.01193	0.00124	0.010445	0.006855

Although the attenuation of the emitted radiation varies for different tube constructions, the relative absorption shows a similar behaviour for different wavelengths. As an example, Figure 8 shows the absorption of the window of a tube.

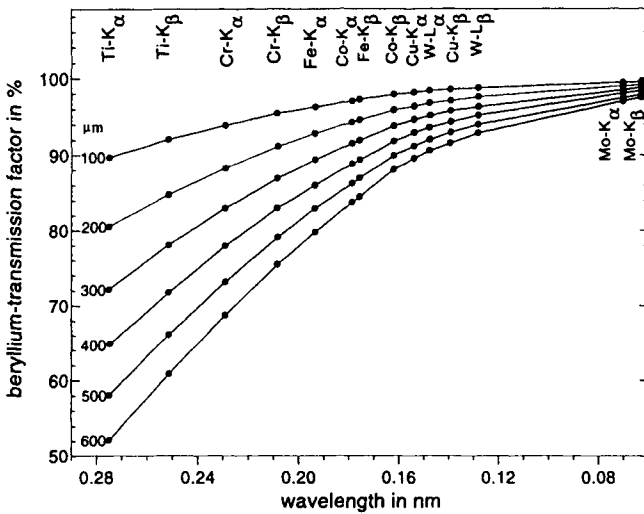


Figure 8. Beryllium-transmission factor versus wavelength, thickness of window of X-ray tube as parameter [11].

Additional attenuation of the radiation occurs on the way from the tube to the specimen and to the counter. The attenuation in air can be calculated approximately according to the following formula.

$$\left(\frac{\mu}{\rho}\right)_{\text{air}} = 0.23_0 \left(\frac{\mu}{\rho}\right)_{\text{O}_2} + 0.76_7 \left(\frac{\mu}{\rho}\right)_{\text{N}_2} + 0.01_3 \left(\frac{\mu}{\rho}\right)_{\text{Ar}} \quad (9)$$

The mass-attenuation coefficient is a function of the wavelength. Figure 9 shows the air-transmission-factor versus the wavelength for different path lengths in air.

Figure 9 shows that the attenuation of the X-ray intensity in the window and in the air is significant especially for those X-rays with relatively high wavelengths.

One should monitor the proper performance of the tubes. The following checks should be done periodically: homogeneity of the radiation at the focus, loss of intensity and tests on the presence of foreign radiation. In the following, some results collected by the members of the German Task Group „Eigenspannungen“ will be presented. Figure 10 shows the decrease of the intensity of the peaks of a dual-phase steel versus its lifetime.

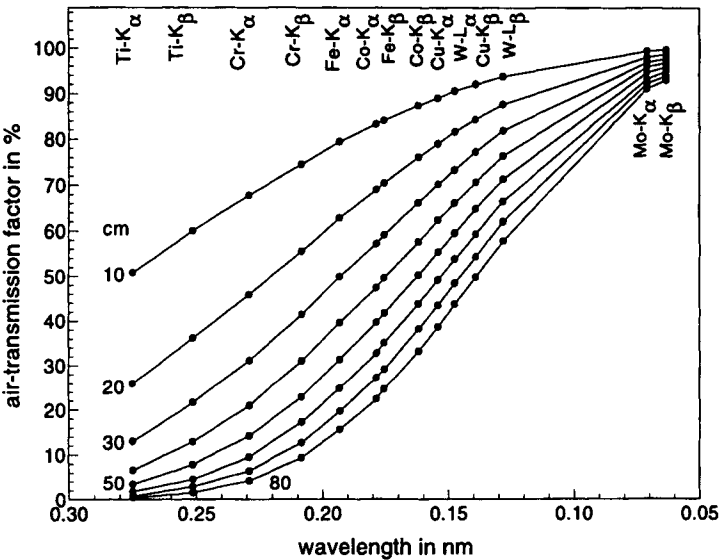


Figure 9. Air-transmission factor versus wavelength, path length of X-rays as parameter [11].

The loss of intensity of the two tubes is enormous, but there is the third tube, which still has the original intensity. From the tungsten cathode, a foreign radiation, W-L_α radiation, can be observed after relatively short burning time, Figure 11.

In Figure 12 the intensity loss and the increased W-L_α radiation of some tubes are plotted.

Automated X-ray equipments with diffractometer are in operation for 24 hours a day in many laboratories. Especially tubes with Chromium-anodes are used for many years in tests with steel components. In often-used tubes, the decrease of intensity and the presence of foreign radiation, W-L radiation, might be noticed. It is necessary to test the X-ray tubes regularly in regard to W-L radiation, otherwise overlapping of interference lines in the front reflection region will occur. The control tests should be made without β-filter and with constant load parameters of the tube, e.g. 40 kV and 10 mA. As a reference sample, a Si-gauge is recommended. Here, the {111}-peak should be measured, and its average intensity should be taken for a ψ-range over - 45° ≤ ψ ≤ 45°.

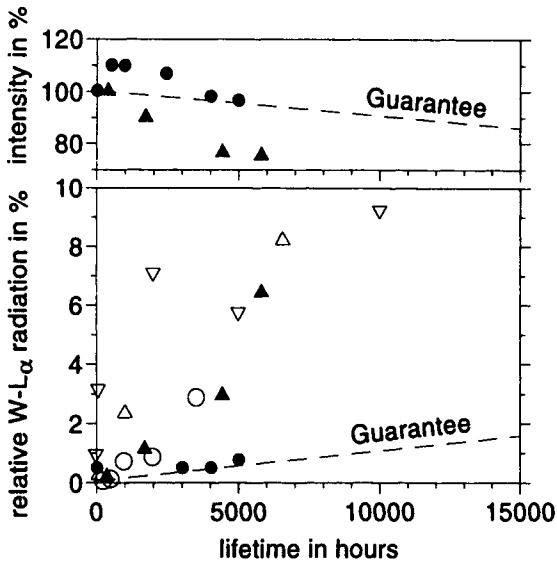


Figure 12. Intensity of the characteristic (above) and of the foreign radiation (below) versus burning time of Cr tubes, measurements by B. Eigenmann, University Karlsruhe (TH), S. Hartmann, H. Ruppersberg, University Saarbrücken and V. Hauk, B. Krüger, RWTH Aachen [11].

The above mentioned German Task Group has suggested a guarantee declaration saying the following, see Table 4.

Table 4. Proposed guarantee for X-ray tubes. Courtesy AWT-Fachausschuß „Eigenspannungen“ [11].

- Spectral purity
 - to be tested on the {111} peak of Si test plate or of Au-powder at 40 kV without any β -filter
 - as delivered: peaks of foreign radiations < 0.1 % relative to the respective radiation
 - increase of the W-L-radiation peak < 0.1% / 1000h
- Loss of intensity < 1% / 1000h of an appropriate peak
- The rating of the tube during the lifetime should be registered as well as the maximum value stated by the manufacturer

There are some methods for suppressing or at least diminishing the foreign radiation. Table 5 lists some methods to do so and their results.

Table 5. Intensity in counts per 5 seconds of tubes with a Cu anode [12].

Arrangement, tube			$K_{\alpha 1}$	K_{β}	$L_{\alpha 1}$
No filter	new		29000	7000	112
	used		20000	5000	600
Ni-filter	new		15000	200	—
	used		11000	145	15
Mono-chromator	quartz prim.	new	17000	—	—
	graphite sec.	used	9000	8	6
	multilayer prim.	used	11000	20	5

Synchrotron radiation

This X-ray source is known since about 1960, but in recent years more facilities have been installed for the use in XSA studies. The physical and technical aspects are collected in [13, 14]. Stress studies were performed by [15-20]. The synchrotron radiation is emitted in the neighborhood of electron or positron storage rings. It is an electromagnetic radiation with a wide spectrum ranging from ultraviolet to X-ray wavelengths. Further characteristics include: polarized continuous radiation, small divergence and high intensity. The main advantages for the use in stress analysis are the following: according to the installed monochromator, the wavelength can be continuously varied so that, with a fixed 2θ Bragg's angle, the different $\{hkl\}$ peaks vs. ψ rotation can be measured [19]. The great variety of wavelengths is the main advantage in contrast to the individual ones of the X-ray tube. Figure 13 graphically shows the peaks of Ni and Fe in the range $140^\circ \leq 2\theta \leq 170^\circ$ versus the wavelength.

With the radiation of X-ray tubes, only a restricted variety of peaks can be tested, whereas the use of synchrotron radiation allows access to many more peaks. Consequently, an important range of penetration depths can be realized, which is useful for the analysis of stress gradients. Also, it is possible to perform measurements at high- ψ polar angles, which is especially helpful for carrying out measurements in the Ω -mode. Figure 14 shows results of D-measurements on cold rolled Ni in the Ω - and the Ψ -mode up to high ψ -tilts [17].

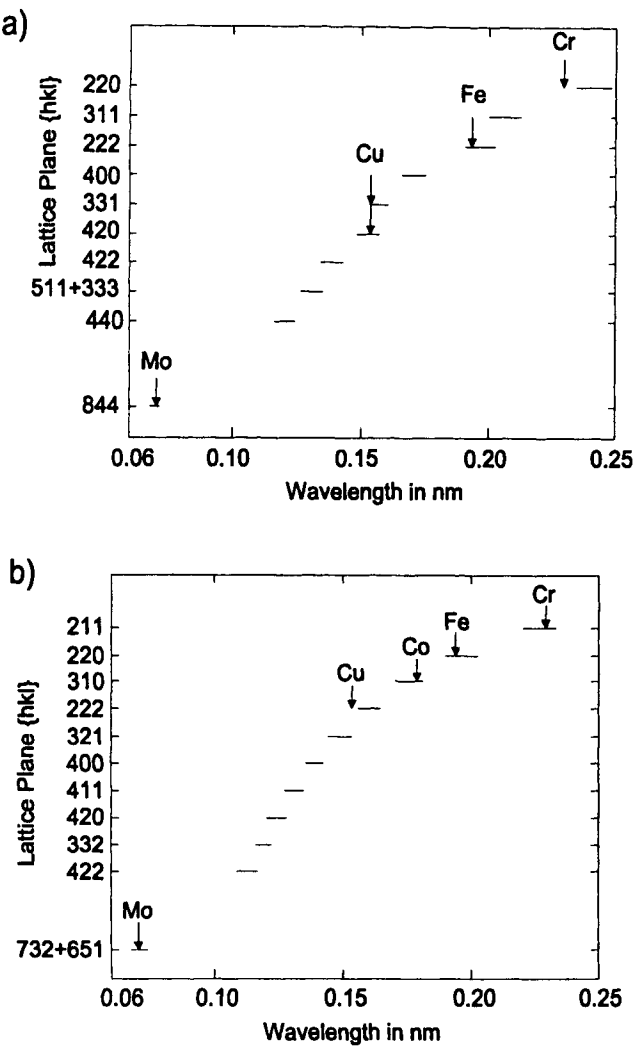


Figure 13. a) Peaks of fcc nickel in the region of $140^{\circ} \leq 2\theta \leq 170^{\circ}$ [20],
b) Peaks of bcc iron in the region of $140^{\circ} \leq 2\theta \leq 170^{\circ}$ [21].

Energy dispersive method

With the use of very hard radiation of 150 keV, the RS profile of steel components up to 10 mm thick can be tested.

A further advantage of this method is the possibility to gain results for many peaks in a short time and up to $\psi \leq 88^{\circ}$, and thus to measure the strain profile well below the surface.

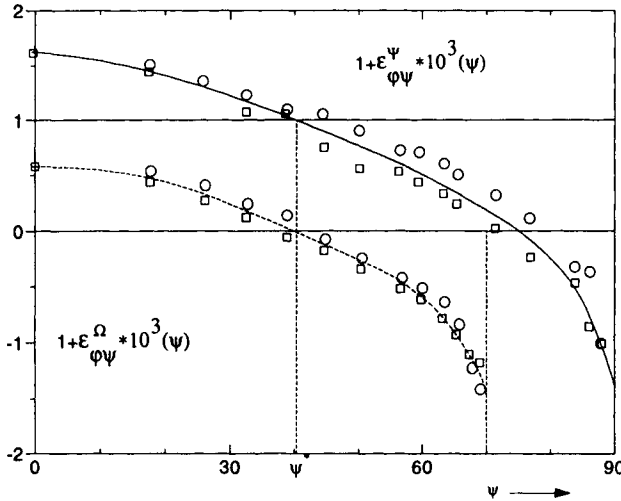


Figure 14. Data obtained from the (531) reflections of a cold-rolled nickel plate: $1 + \epsilon_{0/90\psi}^{\Psi} \cdot 10^3$ obtained with Ψ -goniometry: full curve. $\epsilon_{0/90\psi}^{\Omega} \cdot 10^3$ obtained with Ω -goniometry for $2\Theta = 140^\circ$: dashed curve. Experimental points corresponding to $\epsilon_{0\psi} \cdot 10^3$ and $\epsilon_{90\psi} \cdot 10^3$: circles and squares, respectively [17].

Since the introduction of this method by [22, 23], two papers [24, 25] have been published on this subject, both report on the RS-state of a ground steel plate that was studied by X-rays [26]. Detailed results are presented below.

As the X-ray source, the bremsradiation of a W-anode is used. Using high energies up to 150 keV, the transmission technique is applied so that it is possible to determine the stress state throughout the cross section of the sample. The diffracted X-ray beam is then analyzed by an energy-dispersive germanium detector. The main formulae are

$$E\{hkl\} = \frac{hc}{2D\{hkl\}\sin\Theta} = \frac{12.4}{2D\{hkl\}\sin\Theta} \quad ; \quad \epsilon\{hkl\} = \left(\frac{\Delta D}{D}\right)_{\{hkl\}} = \left(\frac{\Delta E}{E}\right)_{\{hkl\}} \quad (10)$$

Due to the short wavelengths at an acceleration voltage of 150 keV, the intensity of the reflections rapidly decreases with increasing 2Θ -position of the detector. So the 2Θ -values are limited to $\leq 10^\circ$, which results in a parallelepiped shape of the gauge volume element. Limiting the acceleration voltage to approximately 50 kV, then higher 2Θ -values for the detector are possible. Of course the penetration depth of the radiation is then reduced, so that in this case the measurements are performed in reflection-mode geometry. In [24], the diffracted radiation was measured at a fixed angle of $2\Theta = 33.5^\circ$. A wide spectrum of peaks of iron from {110} up to {530+433}, at $\psi = 0^\circ$ up to $\psi = 88^\circ$ and on different azimuths was measured, Figure 15.

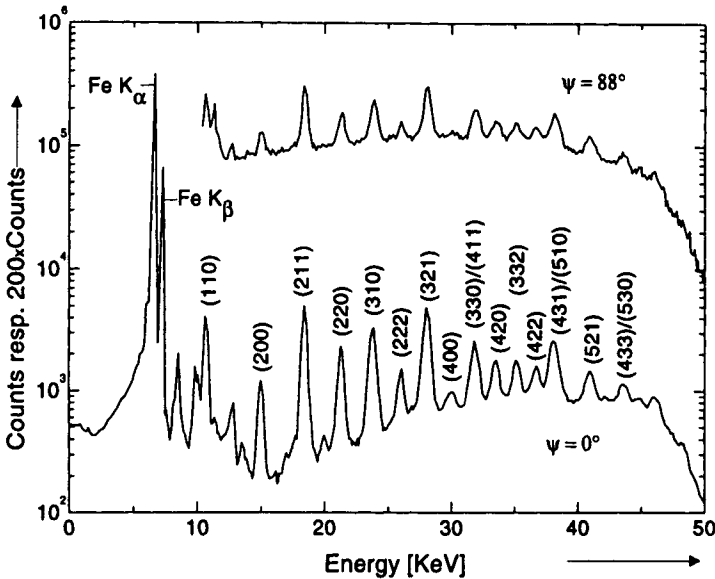


Figure 15. Energy dispersive X-ray diffraction spectra of a ground steel plate, $\psi = 0^\circ$ and 88° [25].

From fitted curves, the ϵ vs. $\sin^2\psi$ dependencies were evaluated.

New impulses to the art of the diffraction techniques and the use of XSA in materials science can be expected from this new radiation source.

2.043 Measuring schemes

The principal arrangement of all XSA and NSA methods is the Debye-Scherrer technique, which, in some countries, is also called Hall technique. Figure 16 illustrates the geometrical arrangement for performing measurements with monochromatic radiation on polycrystalline specimens. If the wavelength of the radiation used and the interplanar lattice distances of the polycrystalline sample material are of the same order of magnitude, then the interference lines $\{hkl\}$ of the specimens arise at discrete 2Θ -values. Shortly after the introduction of this technique, the question of focusing divergent rays was solved. The focusing principles of Bragg-Brentano and of Seemann-Bohlin were introduced (Figure 17). The focusing condition says that the focus of the X-ray tube, the irradiated area of the specimen and the interference line have to lie on a circle, the focusing circle. In this way, the reflected intensity from all individual crystallites fulfilling Bragg's law is focused best, so yielding sharp interference lines. So the position of the interference line can be precisely measured, hence allowing the precise determination of the interplanar lattice spacings. Then, the strain values can be obtained on an absolute scale from the shift of the interference line.

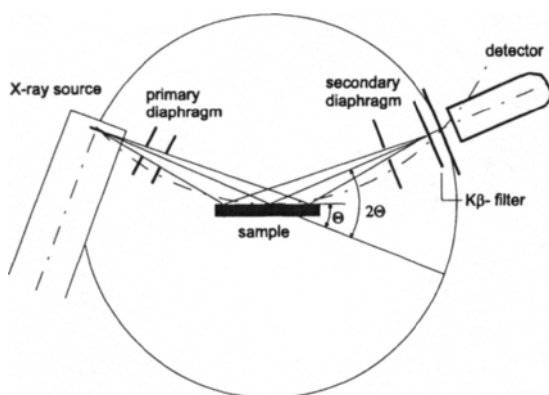


Figure 16. X-ray optics in a powder diffractometer.

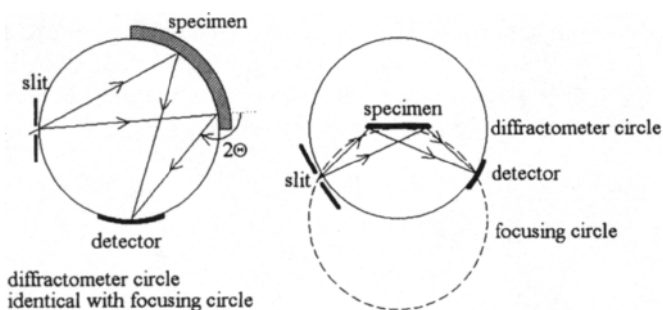


Figure 17. Focusing conditions according to Seemann-Bohlin (left) and Bragg-Brentano (right).

Preponderantly used are the K_{α} -doublets of different radiations and rarely the single K_{β} -line of different radiations. In recent years, continuous polychromatic W-radiation was chosen for the energy dispersive X-ray method. Also, continuous polychromatic neutron radiation and different monochromators are being used. The distribution of the diffracted intensity is registered by a single proportional or scintillation counter or by a position sensitive counter (PSC) in the front-reflection $2\theta \leq 90^\circ$ (θ the Bragg's angle) or in the back-reflection-area $2\theta > 90^\circ$, especially in the range from 150° to 165° . Bragg's equation yields the resolving power of the determination of the lattice-plane distance.

$$\lambda = 2D \cdot \sin \Theta \quad (11)$$

λ : wavelength,

D : lattice-plane distance

$$\Delta 2\Theta = 2 \left(\frac{\Delta \lambda}{\lambda_0} - \frac{\Delta D}{D_0} \right) \tan \Theta \quad \text{for } \lambda = \text{const.} \quad (12)$$

$$\Delta 2\Theta = -2 \frac{\Delta D}{D_0} \cdot \tan \Theta$$

The lattice-plane distance is calculated by well known formulae from the lattice constants and angles according to the crystal system. For materials with a cubic structure, the formula is given by

$$D_{\{100\}} = D_{\{hkl\}} \cdot (h^2 + k^2 + l^2) \quad (13)$$

D_0 is the lattice-plane distance or the lattice constant of the strain-, stress-free state. For a given lattice strain, the peak shift increases with $\tan \Theta$. This is the reason for measuring lattice strains in the back-reflection area. But with high-performance diffractometers, using long counting times and small steps in $\Delta 2\Theta$, precise measurements on polymeric materials, as well as on metallic and ceramic materials, are also possible in the front region.

The strain can be calculated very accurately if D_0 is known.

$$\epsilon = \frac{D - D_0}{D_0} = \frac{\sin \Theta_0 - \sin \Theta}{\sin \Theta} \quad (14)$$

The accuracy of a measurement on different lattice planes using different radiations should be checked by plotting the $D_{\{100\}}$ values versus an appropriate test parameter. The discussion and assessment of the test results should take the evaluated strain into account.

Diffraction methods are phase selective (Figure 18).

The pole angle ψ (angle between the normals of the specimen and the selected lattice plane) $\psi \leq 0$, the azimuth ϕ and the angle ξ of the specimen cut in regard to the rolling, grinding, or other special directions are displayed in Figure 19. λ is the angle around the normal to the lattice plane (not shown).

Although the present methods to evaluate strain and stresses require diffractometers of different designs, depending on the purpose, see chapter 2.05, the basic film method is still in use.

Figure 20 shows the schematic arrangement of a flat-film camera with the film positioned in the back-reflection region. To determine the distance between the X-ray illuminated spot on the specimen and the film, a calibration powder in the form of a thin layer on the specimen is usually utilized. If the material is not fine grained, spotty rings or single reflection spots will be noticed. The shape of the spots, the number of their appearances and their variances

can be analysed easily. The film technique is revitalized by the increasing use of now available imaging plates.

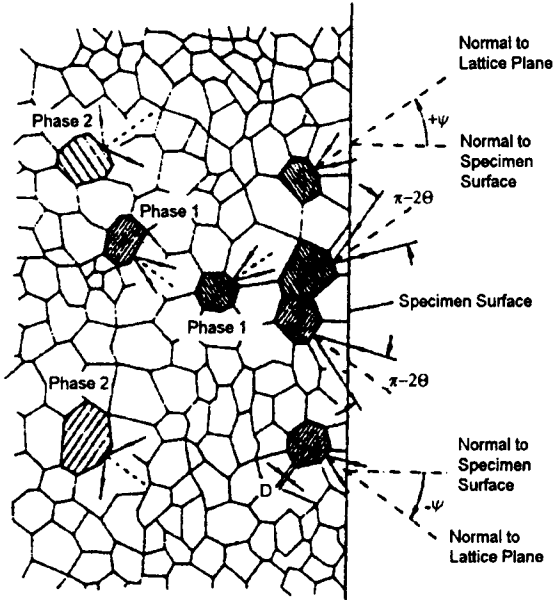


Figure 18. Reflecting grains in a polycrystalline biphasic material.

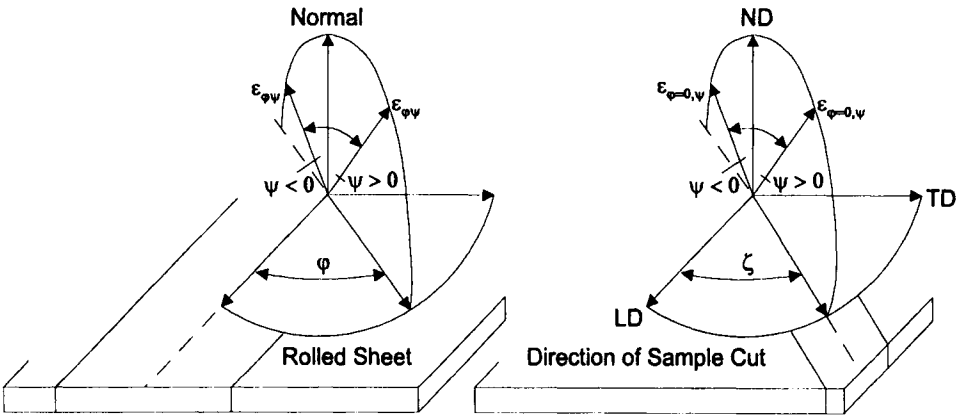


Figure 19. Definitions and notations of angles and directions.

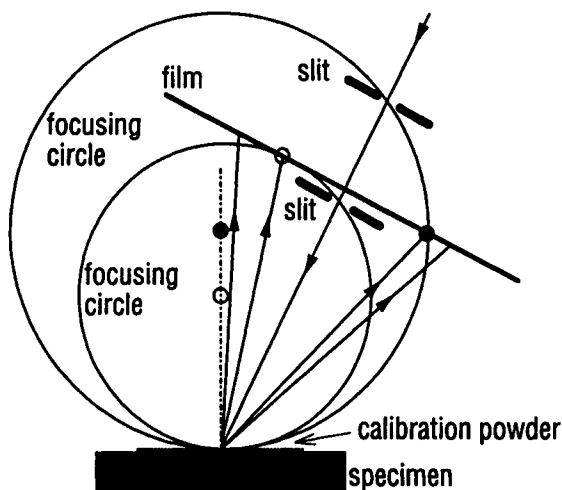


Figure 20. Principle of a flat-film camera in back reflection position.

Ω - and Ψ -diffractometers are in use. Both arrangements are usually based on the Bragg-Brentano focusing principle. This means that the angular velocity of the detector is twice that of the sample around the same axis. The definition of the pole angle $\psi \leq 0$ directions of both diffractometers can be found in Figure 21.

The azimuth $\varphi = 0$ in the specimen should be defined by the direction of drawing, rolling, mechanical surface treatments, or injection molding. In Ω -diffractometer, the rotation of the specimen occurs around the diffractometer axis, and vertical to that in the Ψ -diffractometer. In an Ω -diffractometer the specimen is oriented in such a way, that the azimuth $\varphi = 0$ and the reflected X-ray at $\psi = 0$ form an acute angle. $\psi < 0$ means a rotation of the specimen-normal direction towards smaller 2θ -angles and vice versa. In Figure 21 a tilt ω' is also marked within the Ψ -arrangement. This tilt is necessary for measurements in near-surface regions. The maximum tilt ω' is $\omega'_{\max} = \Theta$. This could be called the combined Ψ - Ω -method.

The characteristics of both types of diffractometers are listed in Table 6. The Ψ -mode is used increasingly in modern installations. But designs are known, that allow one to measure both in the Ω - and the Ψ -mode. More information is given in chapter 2.05.

The choice of the anode material of the X-ray tube depends on the material to be tested, the interference line and the maximum rating of the tube. Modern tubes have two windows with a point focus and two with a line focus. Table 7 shows the different effective sizes of the focus used in Ω - and in Ψ -diffractometers. Usually, fine focus should be used in tests with a monochromator and on curved areas. β -filters are in use on the tube- and/or detector-side.

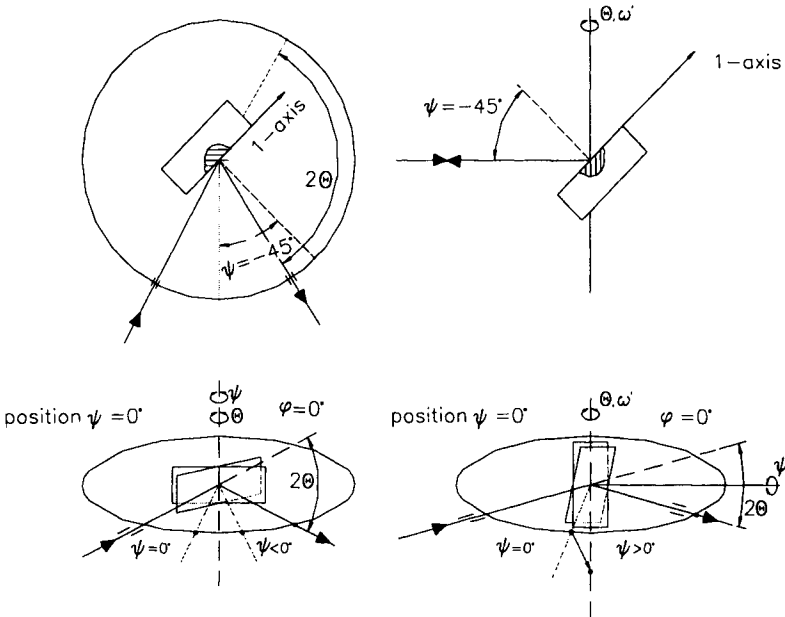


Figure 21. Schemes of Ω (left) and Ψ (right) diffractometers.

Table 6. Characteristics of diffractometer types.

characteristics	Ω -diffractometer	Ψ -diffractometer
axis of $-\psi$ -tilt	perpendicular to the diffractometer plane	parallel to the diffractometer plane
beam geometry with respect to specimen	asymmetrical for $\psi < 0$ and $\psi > 0$	symmetrical for $\psi > 0$ and $\psi < 0$
aperture to limit defocusing errors	line, perpendicular to the diffractometer plane, uncritical but has to be limited	point, critical
irradiated specimen surface	line shaped, decreasing steadily for $\psi < 0$ and $\psi > 0$	point-shaped, slightly increasing with $ \psi $
positioning of specimen	sensitive in regard to excentricity	less critical
PLA correction	required for broad interference lines	not required if only slopes of D vs $\sin^2\psi$ are evaluated
maximum velocity	high for routine measurements because of high intensity	low for large diffractometer radius
lattice-strain measurement	practically restricted to range of back reflection, $\sin^2\psi \leq 0.8$, $\psi \leq \theta$	no restriction in 2θ , $\sin^2\psi \leq 0.9$ for back reflection
texture analysis	not appropriate	state-of-the-art for modern devices

Figure 22 shows the positions of tube, detector and sample in the Ω -mode.

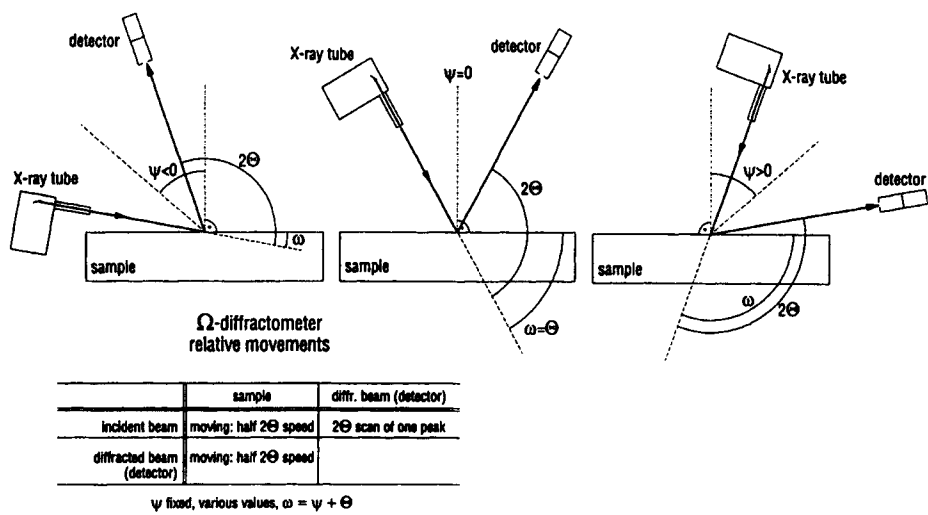


Figure 22. Different positions in the Ω -mode.

Table 7. Effective sizes of focus used in Ω - and Ψ -diffractometers.

Diffractometer	Normal focus	Fine focus	Long-Fine-focus
Ω	$0.1 \times 10 \text{ mm}^2$	$0.04 \times 8 \text{ mm}^2$	$0.04 \times 12 \text{ mm}^2$
Ψ	$1 \times 1 \text{ mm}^2$	$0.4 \times 0.8 \text{ mm}^2$	$0.4 \times 1.2 \text{ mm}^2$

For defining small gauge areas, e.g. notches or certain parts of the cross section, masks are applied on the surface. Care must be taken that no foreign interference line disturbs the peak that is to be measured.

Regarding the size of the slits, the following should be observed: They are smaller for the Ω - than for the Ψ -mode, smaller in the back than in the front reflection zone, and smaller for $\sin^2\psi \geq 0.5$ than for lower $\sin^2\psi$ -values. The angular ranges are from 0.1° to 1° . Soller slits with divergence $\leq 0.15^\circ$ are used on the tube- and/or detector-side. In Japan, also two Soller slits are used to achieve a maximum insensitivity against misalignment, Figure 23.

Monochromators are also used on the tube- and/or detector-side; they are curved or plane, Figure 24. Depending on the X-ray diffractometer arrangement and the required resolution, different monochromator materials can be chosen: Quartz, Germanium, Graphite.

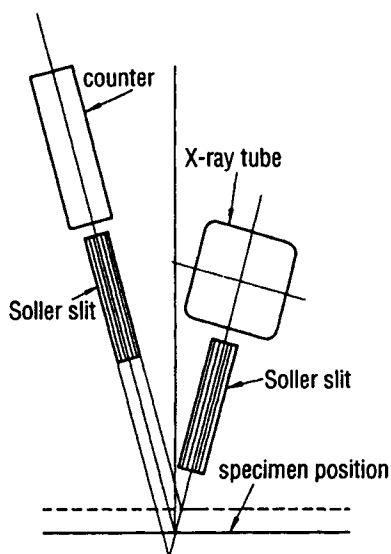


Figure 23. The parallel beam method uses two Soller slits [27].

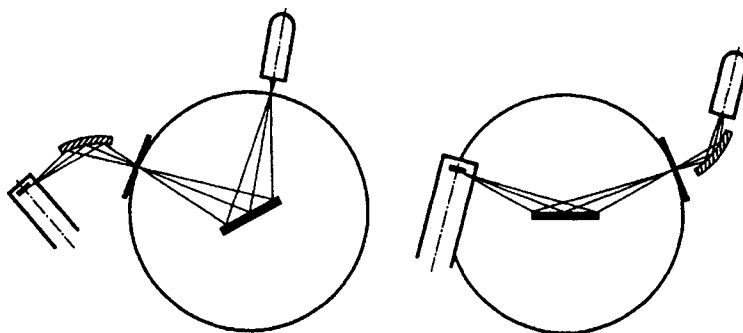


Figure 24. Possible positions of monochromators [28].

Polymeric semicrystalline materials have interference lines from the crystalline phase only in the front reflection zone. Therefore, Ψ -diffractometers are used to extend the accessible ψ -range. In the case of amorphous polymeric materials, a fine crystalline powder must be added to the polymer and the peaks of this powder have to be measured to evaluate the RS-state. Figure 25 illustrates the possible arrangements of both the amorphous and the semicrystalline polymeric material.

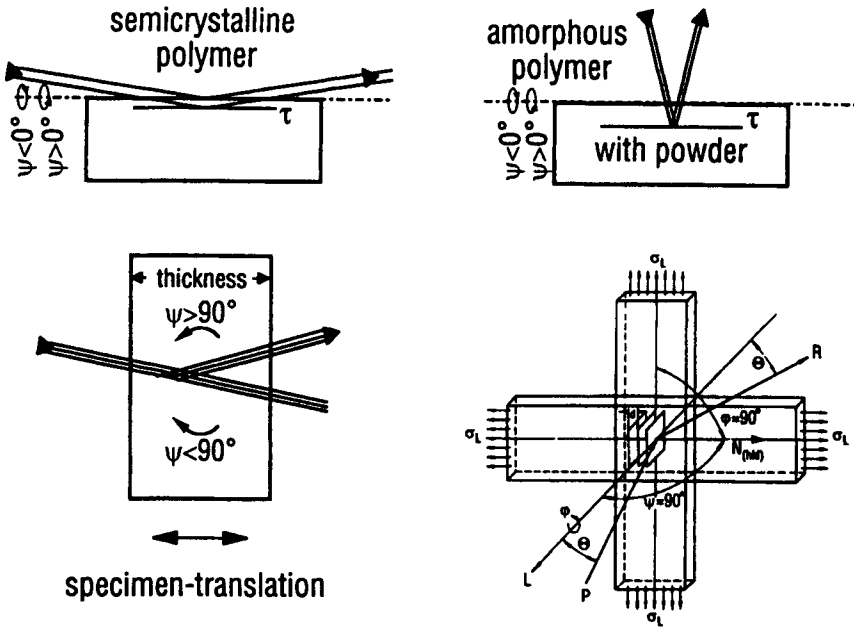


Figure 25. Measuring geometry for the analysis in amorphous and semicrystalline polymeric materials.

If the rays are parallel (long Soller slit) the specimen can be irradiated at a very low angle α of only a few degrees. In this way, very thin surface layers can be tested.

A low-incidence angle Ω -arrangement has been used frequently in recent years. The aim is to measure the lattice strain in near-surface regions. The Seemann-Bohlin-focusing principle is applied. Figure 26 shows the details.

Several interference lines are evaluated. Hereby, different ψ -positions are obtained. For the measurements at $\psi \leq 0$, the specimen must be rotated through 180° . The following connection holds: $\psi = -(\Theta - \alpha)$, α being the incidence angle of a few degrees. The reverse-rays way was proposed by [29]. A very low incidence angle, smaller than 1° , was used by [30, 31].

For an optimum definition of the gage volume element to be measured, a Soller slit with a small divergence of $\leq 0.15^\circ$ is mounted on the detector side.

The geometry arrangement used for high energy X-ray diffraction is shown in Figure 27.

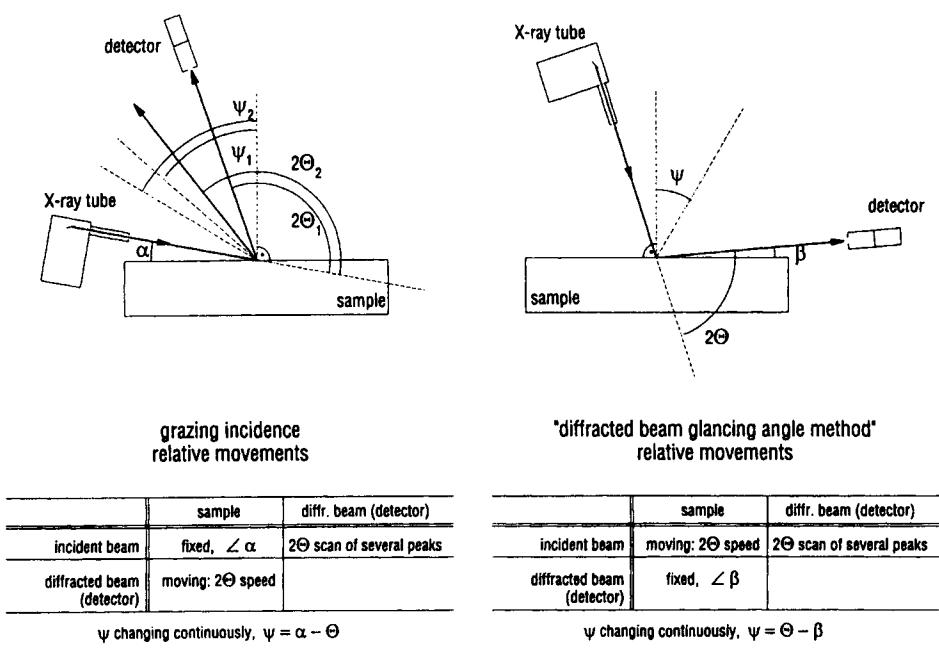


Figure 26. Ω arrangement with low incidence angle using the Seemann-Bohlin-focusing principle.

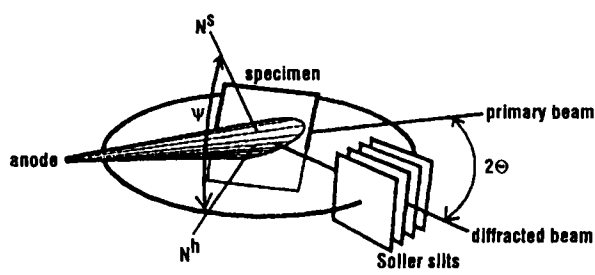


Figure 27. Principal setup for energy dispersive experiments [32].

2.044 Alignment, calibration

The producers of diffractometer arrangements deliver instructions for the basic alignment. The calibration of the equipment is performed with fine crystalline powders. An evenly thin layer of this powder should be placed on a glass plate or on a sticking strip directly on the component. The following powders are used as calibration substances: especially Au but also Ag, Cr, Fe, Ni, Si. $K_{\alpha 1}$ - and $K_{\alpha 2}$ -peaks must be clearly separated. The distance $\Delta 2\theta$ between the calibration peak and the peak used for the later measurements should be smaller than 3° . In the case of precise, absolute measurements, the maximum displacement $\Delta 2\theta$ of the calibration peaks should not exceed ± 0.01 in 2θ throughout the total ψ -range $-\psi_{\max} \leq \psi \leq +\psi_{\max}$. It is important not to use coarse-grained powder because it might easily lead to a miscalibration.

In the case of materials with a relatively low attenuation factor (polymers, ceramics), the larger penetration depth must be corrected for eccentricity with a stress-free sample.

Some papers and studies on the alignment and calibration of diffractometer devices can be found in [33-37].

2.045 Interference-peak determination

W. Reimers

The exact determination of the interference-peak position is a prerequisite for the evaluation of residual stresses. In [38-40], the fundamental peak location methods are demonstrated. The increasing requirements of the determination of residual stress states, in particular of triaxial residual-stress states, and the development of data processing result in improvements of determining the peak positions. There are worked-out processes, which correct the diffraction-angle-dependent part of the intensity, taking into consideration the K_{α} -doublet splitting. The asymmetrical influences of steep gradients on the peak shape are investigated, and will be discussed in chapter 2.15. Because of progress in measuring equipment and the automation of the measuring procedure, the measured data for the interference profile are available as discrete values of I vs. 2θ in most cases. From this data set, the evaluation of the peak position can be performed in a straightforward way. But for each step of the evaluation, the validity of the physical conditions implied has to be checked carefully beforehand. In a first step, the measured intensity values are corrected for the absorption of the X-ray beam, which depends on the material and on the beam path of the incident and the reflected beam. The absorption correction factor A , for specimens with a thickness much larger than the penetration depth of the X-rays, is given by:

$$A = \frac{1}{2\mu} (1 - \tan \psi \cot \Theta \cos \eta) \quad (15)$$

μ - attenuation coefficient

η describes the rotation of the sample around the measuring direction given by φ and ψ [41].

For the determination of the peak position, not absolute but relative intensities are necessary. In this case the attenuation coefficient is canceled out.

For fixed values of ψ and η , data processing yields:

$$I_A(2\Theta) = \frac{I(2\Theta)}{A(2\Theta)} \quad (16)$$

When measurements are performed in the Ω -mode ($\eta = 0^\circ$), Equation 15 simplifies, and for series measurements in the Ψ -mode ($\eta = 90^\circ$), A becomes a constant.

In the next step, a background correction is recommended. In XSA, interference lines are often broad, caused by microstresses, small grain size effects, or the back-scattering measuring procedure. Furthermore, comparatively small intensity to background ratios are often found for different reasons. Thus, an asymmetry of the background may seriously influence the determination of the peak-position. The intensity data $I_A(2\Theta)$ are corrected for the background on both sides of the interference line by

$$I_{A,B}(2\Theta) = I_A(2\Theta) - f_B(2\Theta) \quad (17)$$

The background-correction function $f_B(2\Theta)$ is obtained by fitting a polynomial of low order (usually first or second) to the background intensities b_l respectively b_r on the left hand side respectively right hand side of the interference line. This procedure requires a rather precise determination of the background on both sides of the interference line. Consequently, in cases where the background on one side of the interference line cannot be measured, e.g. for interference-peak positions at very high 2Θ values, XSA should be performed on a different interference line.

The polarization factor $P(2\Theta)$ and the Lorentz factor $L(2\Theta)$ [42] are further correction terms. Whereas $L(2\Theta)$ is only applied to the integral-reflection intensity, a stepwise correction $P(2\Theta)$ is of importance especially for broad interference lines:

$$L(2\Theta) = \frac{1}{\sin 2\Theta} \quad (18)$$

$$P(2\Theta) = \frac{1 + \cos^2 2\Theta}{2} \quad (19)$$

$L(2\Theta)$ is a geometrical correction factor valid for all cases, where the rotation axis for the profile measurement in the Θ - 2Θ - mode is perpendicular to the diffraction plane. The polarization factor corresponding to Equation 19 refers to the partial polarization of the X-radiation of a standard X-ray fine-structure tube. A geometrical correction that depends on the measurement techniques has to be completed [43]. The entire correction factor of the integral intensity for the Debye-Scherrer method is $1 + \cos^2 2\Theta / \sin^2 \Theta \cos \Theta$. In the case of the stepwise, discrete intensity measurement $\cos \Theta$ will become = 1. If a monochromator is used, an additional polarization of the X-radiation is provided, so that in principle the polarization correction has to be applied twice, where the P -factor that is caused by the first diffraction by the monochromator is given by:

$$P_M = (\cos^2 2\Theta_M + 1)/2 \quad (20)$$

Θ_M is the take-off angle of the monochromator. If the monochromator position is kept fixed during the interference profile measurement, P_M is a constant and may be dropped. Special polarization factors have to be calculated when using synchrotron radiation, which is usually highly polarized.

For an evaluation of the absolute peak position, the background and the PLA correction factors have to be taken into account. If only relative values are needed for the assessment, the interference lines should be corrected for background and PLA for high values of $\psi(\sin^2\psi > 0.5)$.

In XSA, mostly X-radiation, conditioned only by K_β -filters, is used for reasons of intensity. Therefore, the $K_{\alpha 1}$ - $K_{\alpha 2}$ splitting and its intensity ratio of approximately 2:1 leads to an asymmetry or even splitting of the observed interference lines. The spacing $\Delta 2\Theta$ of the $K_{\alpha 1}$ - $K_{\alpha 2}$ doublet interference line of the stress-free material with constant D_0 is

$$\Delta 2\Theta = 2 \frac{\Delta \lambda}{\lambda} \tan \Theta_0 \quad (21)$$

The doublet splitting increases with the tangent of Bragg's angles and with smaller wavelengths. The line doublet broadens with increasing inhomogeneous strains and/or decreasing sizes of the crystallites.

A doublet splitting of about 1° in 2Θ of a stress-free material is blurred even by relatively small inhomogeneous strains of the grains measured. In materials with strongly inhomogeneous strains, doublet splittings only occur at high Bragg's angles if $\Delta 2\Theta$ ($K_{\alpha 1}$, $K_{\alpha 2}$) exceeds 2° .

Neglecting the doublet structure of the profiles may lead to uncertainties in the further evaluation. This situation may be avoided either by experimental or by subsequent data treatment methods (Figure 28).

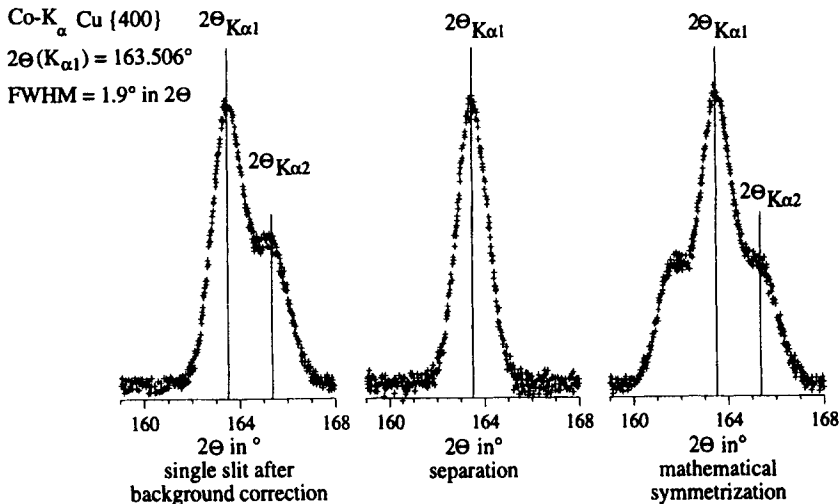


Figure 28. Corrections for the $K_{\alpha 1}$ / $K_{\alpha 2}$ -splitting.

The $K_{\alpha 2}$ line may be suppressed by a secondary monochromator, which, however, leads to a loss of intensity. A further experimental solution of the $K_{\alpha 1}$ - $K_{\alpha 2}$ doublet problem is offered by the use of symmetrizing slits [44], which renormalize the intensity ratio of $K_{\alpha 1}$ - $K_{\alpha 2}$ to 2:1 because of their shape. But attention has to be paid to the peak shift caused by the slit geometry. Depending on the $K_{\alpha 1}$ - $K_{\alpha 2}$ splitting, the evaluated peak position has to be corrected:

$$\begin{aligned} 2\Theta &= 2\Theta_{\text{sym}} - (2\Theta_{\alpha 2} - 2\Theta_{\alpha 1})/2 \\ \Theta_{\alpha i} &= \arcsin(\lambda_{\alpha i}/2D) \end{aligned} \quad (22)$$

However, several drawbacks of the use of symmetrizing slits are evident: the peaks are broadened because of the doubled aperture of the slits in comparison with the single-slit geometry. Furthermore, the symmetrizing double slits have to be adjusted for each anode tube material and for the 2Θ -region under study. Moreover, no slit systems can be installed in the diffracted beam path when using a position-sensitive detector. So parallel to the progressing automation of the measurements, the mathematical methods for taking care of the doublet of the interference structure lines are advancing. With a mathematical procedure, the effect of symmetrizing slits without any peak shift can be simulated on single-slit measurements [45]:

$$I_{\text{sym}}(2\Theta) = I(2\Theta) + \frac{1}{2}I(2\Theta + \delta) \quad (23)$$

δ : doublet separation

Besides an increase in the intensity, the advantage of this creates a working procedure of general availability, even if measuring with a position-sensitive detector or monochromator. Two methods for the α_2 -elimination are commonly used: the substitution correction (usually referred to as the Rachinger method [46,47]) and the Fourier-correction [48]. Both methods are based on the assumptions that (I) the two components have the same shape, (II) the intensity ratio is known, and (III) the doublet separation is also known. According to [49] one obtains:

$$I_{\alpha 1}(2\Theta) = I_{\text{total}}(2\Theta) - RI_{\alpha 1}(2\Theta - \delta) \quad (24)$$

$$R = I_{\alpha 1, \text{max}} / I_{\alpha 2, \text{max}}$$

$$2\Theta_{\alpha 2} - 2\Theta_{\alpha 1} = 2 \arcsin\left(\frac{\lambda_{\alpha 2} - \lambda_{\alpha 1}}{2D} + \sin \Theta_{\alpha 1}\right) - 2\Theta_{\alpha 1}$$

However, $\delta = \Delta 2\Theta$ is known to increase with increasing values of 2Θ . Therefore, it is more efficient to use a doublet separation that is constant on such a scale. Thus, transforming the profile on the reciprocal $\sin\Theta$ -scale, the corresponding doublet separation becomes

$$\delta = (k-1)\sin\Theta \quad \text{with } k = \lambda_{K\alpha 2} / \lambda_{K\alpha 1} \quad (25)$$

So, after replacing 2Θ by $\sin\Theta$ in Equation 23 and denoting the first position on the left side of the profile by $x_s = \sin\Theta_s$, the subsequent positions required for the separation are obtained by $x_m = x_s k^m$. The use of Equation 23 then yields a recursive formula for the Rachinger separation that is given by:

$$\begin{aligned} I_{\alpha 1}(x_s) &= I_{\text{total}}(X_s) \\ I_{\alpha 1}(x_s k^m) &= I_{\text{total}}(x_s k^m) - R I_{\alpha 1}(x_s k^{m-1}) \quad m \geq 1 \end{aligned} \quad (26)$$

After having applied the α_2 separation to the experimental data, the profile should be transformed to the D-scale, where symmetrical intensity distributions should be obtained. So, asymmetries are easily detected either by optical inspection or by calculating the derivation to symmetrical functions e.g. Gaussian distributions. The observation of asymmetric Rachinger corrected profiles necessitates a careful inspection of the material state under study:

- insufficient measuring time may give rise to the occurrence of erroneous reflection profiles.

Statistical considerations for the fit quality are given below.

- insufficient grain statistics may give rise to Bragg reflections of individual grains for accidental diffractometer positions, thus e.g. enhancing the $K_{\alpha 1}$ or $K_{\alpha 2}$ reflection subprofile. Effects caused by coarse grains are also indicated by intensity variations at different diffractometer positions ϕ , ψ . Accurate lattice-strain determinations can only be achieved if a sufficient number of grains of the material phase under study is randomly oriented in the specimen area irradiated by the incident X-ray beam. This presupposition is not fulfilled in coarse-grained materials or in materials with preferred orientations of the grains. In such cases, the interference lines show different intensities when varying the measuring directions $\pm \psi$. With a ψ -scan at a constant Bragg's angle Θ_0 , one can settle whether there is a coarse-grained material or a material with strong preferred orientations. Figure 29 illustrates the method showing three material states: fine crystalline, textured and coarse grained. With a film tilted between X-ray and surface of the specimen the material state can also be checked.

In coarse-grained materials, the number of crystallites measured can be increased by a motion of the sample's surface relative to the incident X-ray beam, by a slight pendulous motion of the object surface around an axis through the center of gravity of the irradiated area, by an enlargement of the irradiated surface area, or by using X-rays with deeper penetration. The attenuation of X-rays depends on the wavelength, the measuring direction and the material under investigation.

- The observed reflection profile may be due to a superposition of crystallographically different interference lines. For steel, the influence of the C-content on the reflection line splitting is demonstrated schematically in Figure 30. Measured reflection profiles are shown in Figure 31.

Another peak determination problem exists for broadened martensitic interference lines $\{211\}$ measured with Cr-K_{α} radiation on hardened steels. Steel components are prevalingly tested with Cr-K_{α} radiation. The $\{211\}$ peak of the ferritic phase lies at $2\Theta = 156.07^\circ$. The FWHM reaches values of 10° or more than 20° in 2Θ .

When the broad $\{211\}$ peak of martensite can not be measured with the usual Cr-K_{α} radiation, other peaks can be chosen.

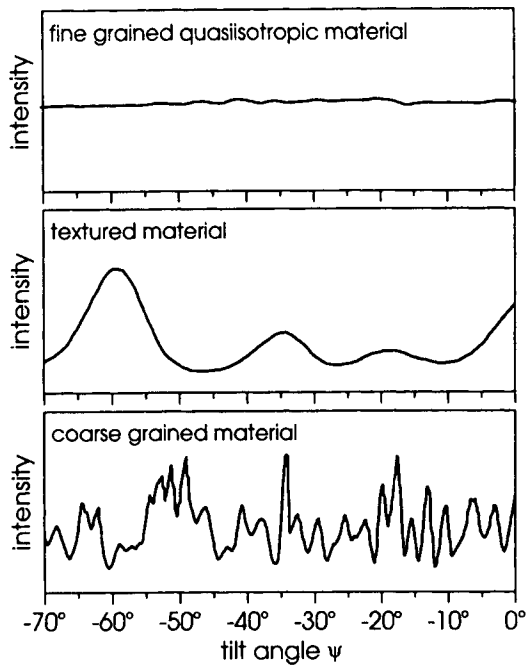


Figure 29. ψ -scan of fine-crystalline quasisotropic, textured and coarse grained materials.

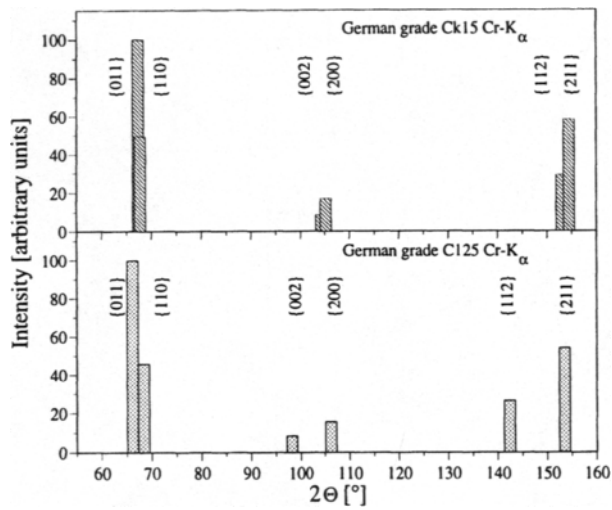


Figure 30. Reflection splitting in steels with different carbon contents.

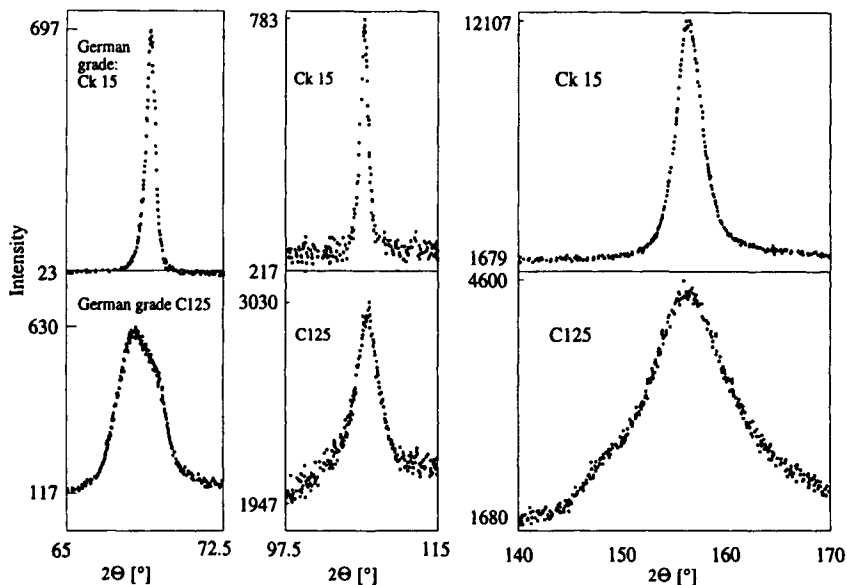


Figure 31. Reflection profiles for steels with different carbon contents.

Evidently, superposition of reflections is often observed in two-phase or multiphase materials (Figure 32). In such a case it should be examined, whether it is possible to investigate another, undisturbed interference line.

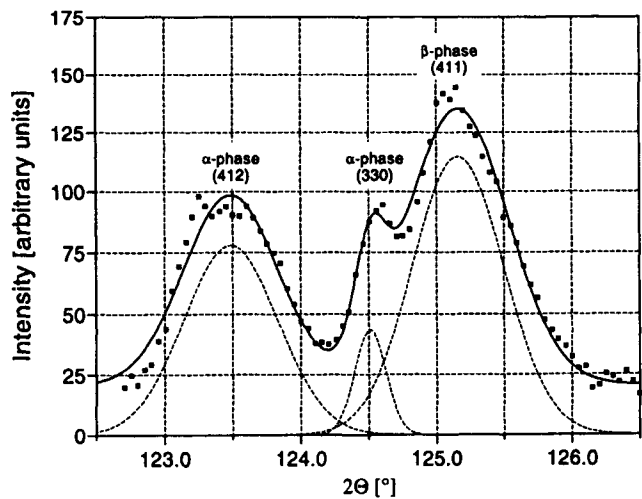


Figure 32. Superposition of reflections in two-phase HPSN.

A correct determination of the interference peak position may be achieved, if it is possible to perform a double or multiple profile analysis (Figure 33).

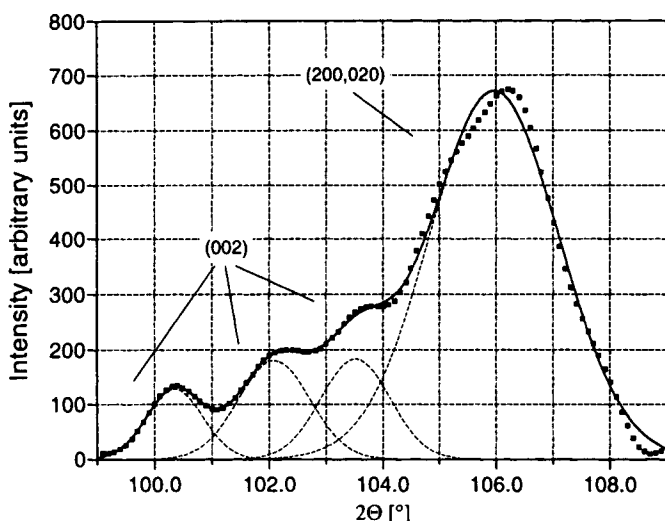


Figure 33. Splitting of the {200}-martensite interference line caused by the tetragonal distortion of the unit cell.

- Gradients of stresses or of the D_0 -value within the penetration depth of the X-rays influence the reflection profiles. Adequate evaluation procedures are discussed in chapter 2.15.
- Stacking faults may give rise to asymmetric D-distributions. Details are discussed in chapter 2.09.

For the interference-peak determination on the basis of corrected and inspected data, several methods are available, which may be divided into two categories: - Interference-peak determination methods which are based on the analysis of selected parts of the profile (e.g. $H/2$ - method, parabolafit).

These peak evaluation methods may be applied e.g. to study polymeric materials, where only superponed interference profiles at very low 2Θ -values are available (Figure 34).

However, if the full interference profile is available, the disadvantage of these methods is evident, because most part of the measured information is not used. So these methods should only be used exceptionally. Problems that may arise from the suppression of the background correction or from ignoring possible profile asymmetries have been discussed above.

- Interference peak determination methods based on the analysis of the full profile. Here the center-of-gravity method is still widespread. This method is often performed on the 2Θ -scale of the data, because $K_{\alpha 1}$ - $K_{\alpha 2}$ -splitting is quasiautomatically averaged out in the result. The interference peak position is calculated by

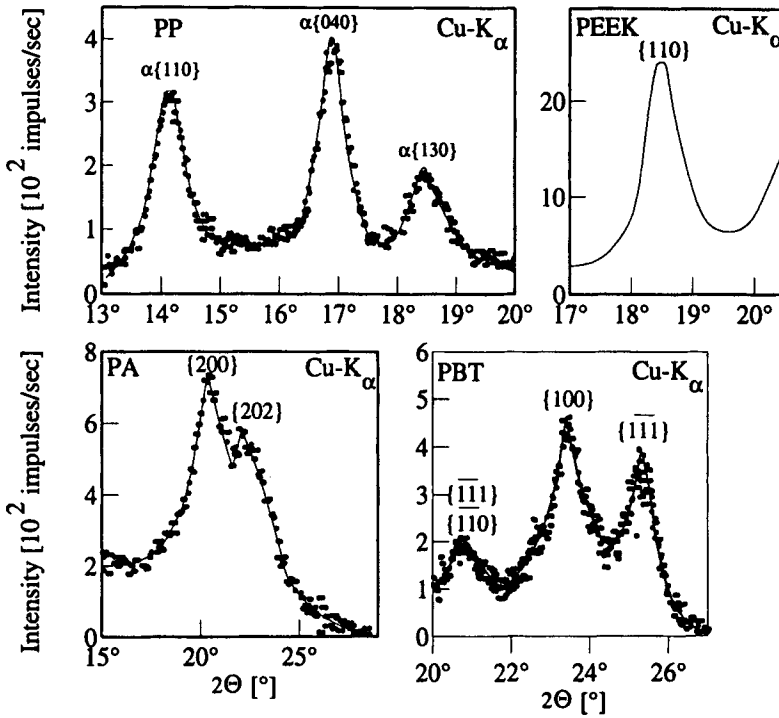


Figure 34. Reflection profiles for polymeric materials.

$$\langle 2\theta_c \rangle = \frac{\int I(2\theta) 2\theta d2\theta}{\int I(2\theta) d2\theta} \quad (27)$$

Asymmetries in the reflection profiles are indicated by a shift in the $\langle 2\theta_c \rangle$ values, when the integration limits in Equation 27 are varied systematically. In this case the center-of-gravity of the entire K_α -separated line will give the correct 2θ -position. Usually the integration limits should be varied from 0.55 to 0.80 of the maximum peak-intensity in steps of 0.05 in case of the symmetrized line and from 0.20 to 0.70 in steps of 0.10 in case of the separated line. Averaging these values of peak positions validates the result in a statistical manner. Investigations on computed interference lines, symmetrized or K_α -separated with and without PL-correction, were made, to show the errors in determining the peak location by using the center-of-gravity method for several peak positions in 2θ (Figure 35) [50].

The disadvantage of the center-of-gravity method is its limitation to only one profile. In contrast to the center-of-gravity method the Gauss-algorithm offers the advantage that it may be easily extended to two or more subprofiles:

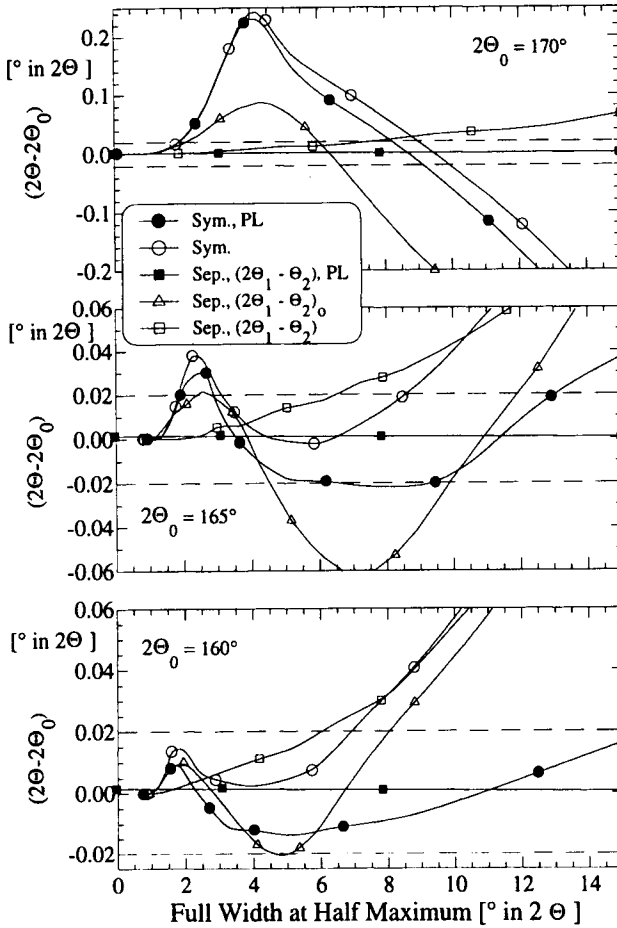


Figure 35. Errors $(2\Theta - 2\Theta_0)$ of the peak positions from different evaluations. The errors are displayed as a function of the FWHM (Cu- $K_{\alpha 1}$ -interferences, $2\Theta_0 = 160^\circ, 165^\circ, 170^\circ$) [50].

$$I_{\text{total}}(D) = \sum_{i=1}^n I_{i,\text{max}} \exp\left(-\frac{(D - D_{i,\text{max}})^2}{2\sigma_i^2}\right)$$

$I_{i,\text{max}}$ – maximum intensity of subprofile i

$D_{i,\text{max}}$ – D -value at the maximum intensity of the subprofile i

$\sigma_i = \frac{w_i}{\sqrt{2 \ln 2}}$ with w_i = full width at half maximum of the subprofile i

(28)

It should be mentioned that Rachinger corrected profiles should be used preferably, where the $I(2\Theta)$ values are converted to $I(D)$ values.

Figure 36 gives an example for a multi-Gauss evaluation.

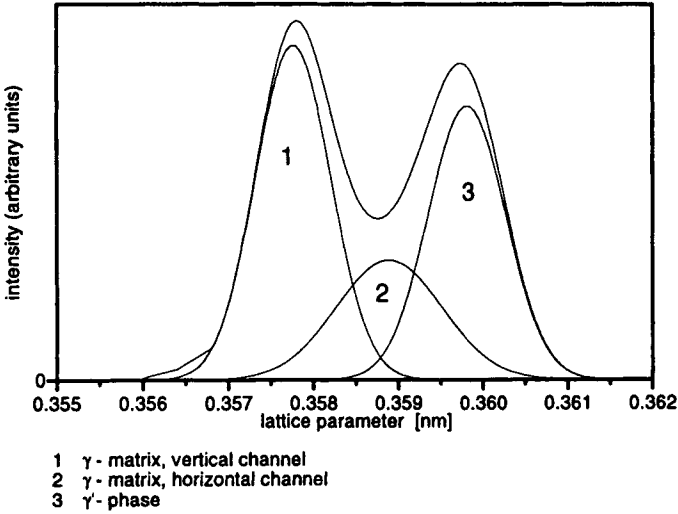


Figure 36. Superposition of reflection profiles in the nickelbase superalloy SC 16 after uniaxial creep deformation in [001].

In special material conditions or in the case of high resolution experiments (e.g. parallel-beam methods, synchrotron radiation), the experimental profiles are no more represented by Gaussian distributions. Here, it is recommended to use Pearson VII-functions, or the more physically based Voigt-functions, which are more flexible in describing profiles over the whole range from Lorentzian to Gaussian distributions. The Pearson VII-function [51] is given by:

$$I(x) = I_0 \left[1 + \frac{(x - x_0)^2}{m\sigma^2} \right]^{-m} \quad (29)$$

The parameter m varies with the peak shape. For narrow peaks $m = 1$ is appropriate, in which case (29) is a Lorentzian. When m approaches infinity, it can be shown that $I(x)$ Equation 29 becomes a Gaussian function.

A different access to the analysis of D-vs.- $\sin^2\psi$ distributions is possible by the cross-correlation method [52,53], where the interference line positions are calculated relative to a reference interference peak position, e.g. measured at $\psi = 0^\circ$.

$$\phi(\Delta 2\Theta) = \sum_{i=1}^n I_1(2\Theta_i) \cdot I_2(2\Theta_i + \Delta 2\Theta) \quad (30)$$

$I_1(2\Theta_i)$ - intensity of the reference reflection at $2\Theta_i$

$I_2(2\Theta_i + \Delta 2\Theta)$ - intensity of the second reflection at $2\Theta_i + \Delta 2\Theta$

The maximum of the function $\varnothing (\Delta 2\Theta)$ yields the shift $\Delta 2\Theta$ of the reflection line to be evaluated relative to the reference-peak position.

In Figure 37 the different methods for peak position determination are graphically displayed. In Table 8 the methods are listed and assessed.

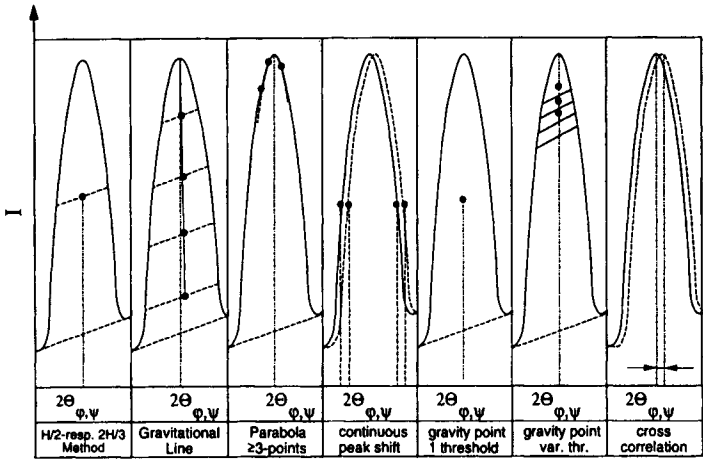


Figure 37. Comparison of methods used for peak position determination.

Table 8. Assessment of methods for peak position determination.

shape of interference determination of peak position	$K_{\alpha 1} + K_{\alpha 2}$ doublet	doublet symmetrized	$K_{\alpha 1}$ separated, K_{β}	multi peak interference	$2\Theta(z)$ -gradient
gravitational line	O	+	+	-	O
H/2 method	-	-	O	-	-
continuous peak shift	-	-	O	-	-
2/3 H method	O	O	O	-	-
parabola, ≥ 3 points	-	O	+	-	-
fitting (Gauss, Pearson)	O	-	+	+	-
gravity point with fixed low threshold	+	-	+	-	+
gravity point with varying threshold	+	+	+	-	O
$\frac{\partial I(2\Theta)}{\partial (2\Theta)}$	-	-	+	-	-
cross correlation	+	-	+	-	-

For judging the quality of a profile, using e.g. Gauss or Pearson VII-functions, the χ^2 -test is often used:

$$\chi^2 = \sum_{i=1}^n \left(\frac{y_i - y(x_i, a_1, \dots, a_m)}{\Delta_i^2} \right) = \text{Min!} \quad (31)$$

$a_1, \dots, a_m$ - fitting parameters

Δ_i - standard deviations of the individual intensities

Since the value of χ^2 depends on the number of fitting parameters applied, it is useful to proceed to a more general description of the fit quality by calculating the goodness-of-fit probability Q [54]:

$$Q = Q\left(\frac{1}{2}v, \frac{1}{2}\chi^2\right) \quad (32)$$

$v = n - m$

n - number of measured intensity values

m - number of fitting parameters

$Q(a, x)$ is the uncomplete Gamma-function:

$$Q(a, x) = \frac{1}{\Gamma(a)} \int_0^x e^{-t} t^{a-1} dt. \quad (33)$$

Experience shows that the model used for the interference-profile fitting should be basically correct when $\chi^2 \approx v = n - m$ is achieved. In this case, Q -values of $Q > 10^{-3}$ are calculated, whereas for Q -values $\leq 10^{-18}$ the fitting model used has to be revised.

The statistical error of the fitting parameters Δm_j , e.g. interference-peak position, full width at half maximum, are calculated by:

$$\Delta m_j^2 = \frac{(K_{jj})^{-1}}{n - m} \sum_{i=1}^n \frac{(I_{i,obs} - I_{i,calc})^2}{(\Delta_{i,obs})^2} \quad (34)$$

K_{jj} - diagonal element j of the inverse coefficient matrix K^{-1} of the normal equation system.

2.046 Basic data

The following figures and tables should help to perform lattice-strain measurements and evaluations of stresses. The wavelengths of industrially produced sealed X-ray tubes with the given anode-metals are listed in Table 1. Also, filter materials are named, which serve to absorb the K_β -radiation.

The lattice constants of different materials and calibration powders are listed in Tables 9, 10. These data were used to calculate the different parameters noted here. For some metals, getting pure powders is difficult, so the lattice constant may be different and a calibration versus Au-powder must be made. When samples for calibration purposes are not fine-grained, and procedures used to enhance the number of reflecting crystallites are not sufficient to counteract the coarse-grained effects, erroneous results may be obtained.

Figures 38-40 show the Debyeograms of the calibration powders Au, Cr and Fe for the different K_α radiations of Ti, Cr, Fe, Co, Cu, Mo. For the often-used Si calibration sample, the 2Θ values are given in Table 11.

The penetration depths are defined as those distances from the surface, out of which 63% or $1-1/e$ of the intensity of the interference lines originate. Corresponding values for Ω - and Ψ -diffractometers are given by the formulae in the first line of Table 12. For some materials in Table 13 the attenuation coefficients are listed and Figure 41 shows these penetration depths vs. $\sin^2\psi$, when distinct $\{hkl\}$ -planes are measured with specified K_α -radiations. Mean penetration depths can be calculated for $\sin^2\psi = 0.3$ i.e. $\psi = 33.21^\circ$. Also values are used corresponding to depths that contribute about 95% or 99% to the total intensities. Table 14 contains $\tau_{0,3}$ (Ψ -diffractometer) and τ_0 (Ω - and Ψ - diffractometer) for iron-base materials versus different radiations. The dependence of the penetration depths versus $\sin^2\psi$ is different for Ω - and Ψ -diffractometers in the region of high ψ -angles. For clarification only, some curved lines are drawn in Figure 41 for the Ω -mode. For $\psi = \Theta$, the penetration depth of the Ω -arrangement equals zero. In some gradient studies, the fact is used that the strains of different crystallites in the same depth can be measured with one radiation on the same interference line using the Ω - and the Ψ -mode of the diffractometer. The penetration depth decreases if the specimen thickness is finite and approximates the thickness t itself. The formula used in thin film studies holds:

$$\tau = \tau_0 \cos \psi \left[-\ln \left(e^{-1} + e^{-t/\tau_0} - e^{(-t/\tau_0 - 1)} \right) \right] \quad (35)$$

Figure 42 shows an example [55].

Table 9. Lattice constant at room temperature and crystal system of distinct base materials [7, 8].

material	lattice constant in nm	crystal system
α -Fe	0.28665	b.c.c.
γ -Fe	0.359 ± 0.001	f.c.c
Al	0.40491	f.c.c
Cu	0.36141	f.c.c
Ni	0.35238	f.c.c
α -Ti	$0.29505, \frac{c}{a} = 1.5873$	hex.

Table 10. Lattice constant of the most often used calibration powders at room temperature [7, 8] and crystal system.

calibration powder	lattice constant in nm	crystal system
Ag	0.40865	f.c.c.
Au	0.40786	f.c.c.
Cr	0.28844	f.c.c.
Fe	0.28665	b.c.c.
Ge	0.56575	2f.c.c.
Si	0.54319	2f.c.c.
W	0.31650	b.c.c.
Nb	0.32986	b.c.c.
CdO	0.46951	2f.c.c.

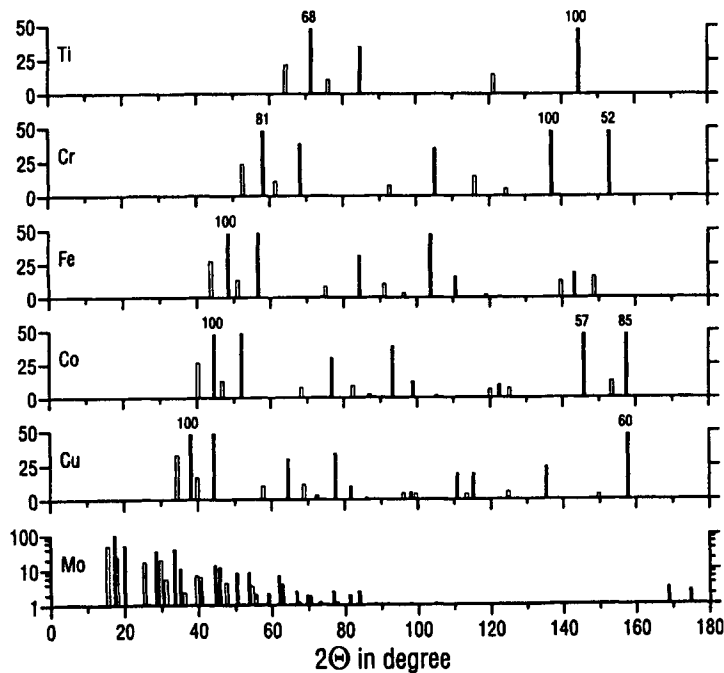


Figure 38. Interference lines of Au-powder obtained with different $K_{\alpha 1}$ - (closed column) and $K_{\beta 1}$ - (open column) radiations.

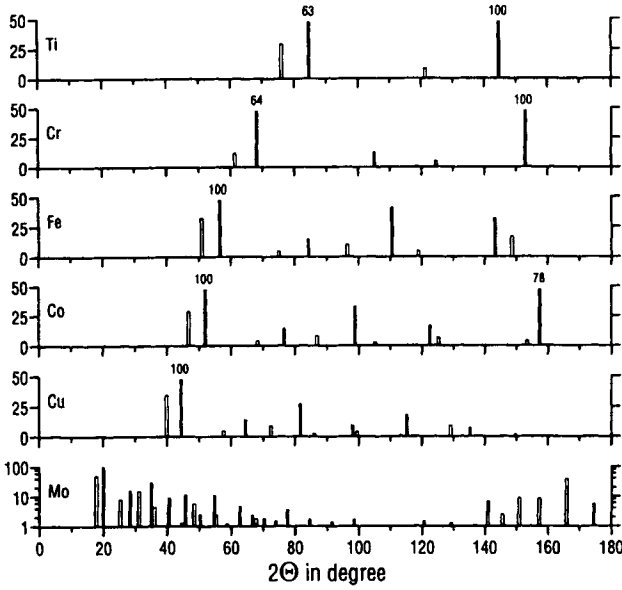


Figure 39. Interference lines of Cr obtained with different $K_{\alpha 1}$ - (closed column) and $K_{\beta 1}$ (open column) radiations.

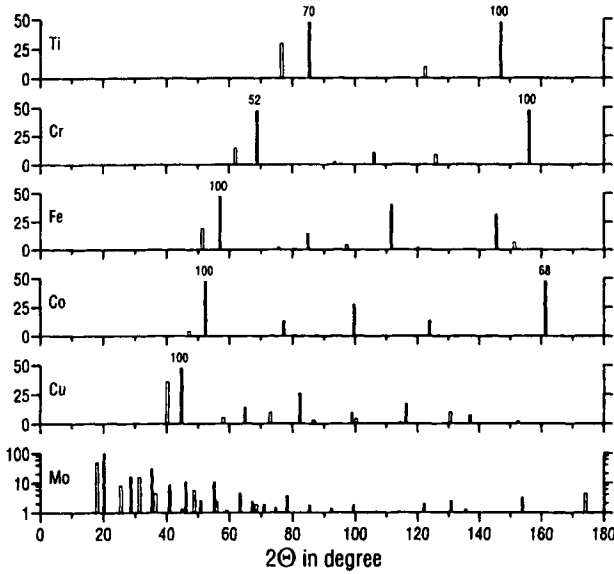


Figure 40. Interference lines of Fe obtained with different $K_{\alpha 1}$ - (closed column) and $K_{\beta 1}$ (open column) radiations.

Table 11. Two-theta-angles for the reflection lines of silicon [56].

hkl	D in nm	Mo K _{α1}	Cu K _{α1}	Co K _{α1}	Fe K _{α1}	Cr K _{α1}
111	0.313537	12.988	28.443	33.150	35.966	42.830
220	0.192001	21.288	47.303	55.530	60.550	73.204
311	0.163739	25.016	56.123	66.221	72.480	88.720
400	0.135766	30.284	69.131	82.419	90.956	114.964
331	0.124587	33.076	76.377	91.767	101.966	133.526
422	0.110852	37.316	88.032	107.584	121.670	
511-						
333	0.104512	39.670	94.954	117.702		
440	0.096001	43.358	106.711	137.604		
531	0.091794	45.452	114.094	154.011		
620	0.085866	48.788	127.547			
533	0.082816	50.708	136.897			
444	0.078384	53.798	158.638			
711-						
551	0.076044	55.596				
642	0.072570	58.508				
731-						
553	0.070701	60.212				
800	0.067883	62.988				
733	0.066346	64.622				
822-						
660	0.064001	67.298				
751-						
555	0.062707	68.878				
840	0.060716	71.476				
911-						
753	0.05609	73.016				

Table 12. Formulae for the penetration depth and the PLA factor.

	Ω-diffractometer	Ψ-diffractometer
penetration depth	$\frac{1}{2\mu} \frac{\sin^2 \Theta - \sin^2 \Psi}{\sin \Theta \cdot \cos \Psi}$	$\frac{1}{2\mu} \sin \Theta \cdot \cos \Psi$
PLA	$\frac{1}{2\mu} \frac{1 + \cos^2 2\Theta}{\sin^2 \Theta} (1 - \tan \Psi \cdot \cot \Theta)$	$\frac{1}{2\mu} \frac{1 + \cos^2 2\Theta}{\sin^2 \Theta}$

Table 13. Attenuation coefficients μ in cm^{-1} of different metals for distinct eigenradiations[10].

Metal	Fe	Al	Cu	Ni	Ti
density in g/cm^3	7.87	2.70	8.93	8.90	4.51
Ti- K_{α}	1437.6	693.8	2212.2	1997.8	513.0
Cr- K_{α}	873.3	421.1	1343.9	1213.6	2739.3
Mn- K_{α}	691.4	333.3	1064.0	960.8	2167.9
Fe- K_{α}	552.6	255.2	850.3	767.2	1731.9
Co- K_{α}	445.3	202.1	685.3	618.8	1395.3
Cu- K_{α}	2490.6	130.0	455.9	411.6	927.5
Mo- K_{α}	288.5	13.2	432.7	394.3	107.9

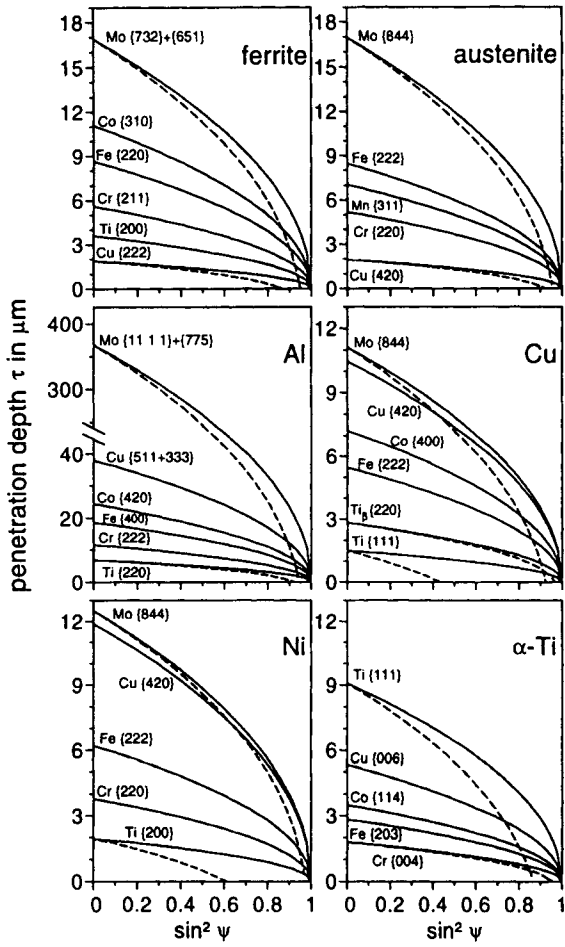


Figure 41. 63% penetration depth versus $\sin^2 \psi$; material noted, different radiations and peaks, Ψ -diffractometer, Ω -mode dashed line.

Table 14. Mean penetration depth of different radiations in iron base materials.

radiation	hkl	2Θ	μ [μm ⁻¹]	τ _{0,3} [μm]		τ ₀ [μm]
				Ψ	Ω	
Cr-K _α	211	156.072	0.08733	4.69	4.60	5.60
Fe-K _α	211	111.618	0.05526	6.26	5.02	7.48
Co-K _α	211	99.694	0.04453	7.18	4.99	8.58
Cr-K _α	200	106.024	0.08733	3.83	2.90	4.57
Ti-K _α	200	146.993	0.14376	2.79	2.69	3.33
Cu-K _α	222	137.129	0.24906	1.56	1.46	1.87
+monochromator	211	82.325	0.24906	1.11	0.49	1.32

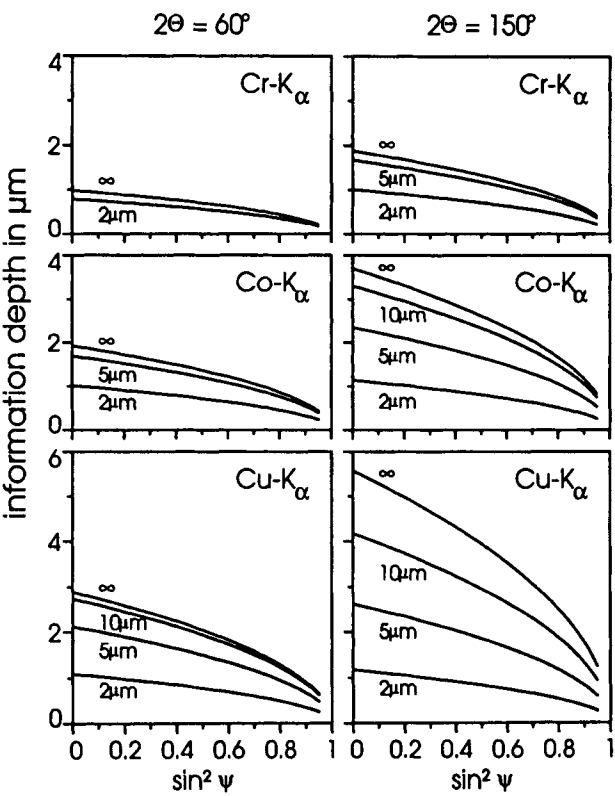


Figure 42. Thickness τ of those surface regions, where 1 - 1/e of the reflected intensity comes from, depending on the layer thickness t. The calculations were made for a Ψ-diffractometer and a TiN layer material [55].

The peak intensity I of an interference line is determined by

$$I \sim H S^2 P L A \quad (36)$$

where H is the multiplicity factor, S the structural factor, P the polarization factor, L the Lorentz factor, and A the absorption factor. For measurements with Ω - or Ψ -diffractometers, the PLA product is given by the equations in the second line of Table 12. The PLA values increase with increasing 2Θ . Using Ω -diffractometers, PL depends on the measuring directions ψ according to the data collected in Table 15. For measurements with Ψ -diffractometers, PLA is independent of ψ .

The data needed to evaluate strain measurements on the base materials of both iron, Al, Cu, Ni, Ti with different radiations on various peaks are listed in the following Tables 16-21 [58, 59 supplemented]. Data for appropriate calibration powders are also given. The peaks of calibration powders which may overlap with the peaks of the material to be studied are marked by a *. Values $2\Theta_c > 2\Theta_0$ are identified by **.

Table 15. PL-data for measurements with Ω -diffractometer; for the integral intensity (integral width) the data must be multiplied with the factor $1/\cos\Theta$ [57].

$2\Theta / \psi$	0°	$\pm 15^\circ$	$\pm 30^\circ$	$\pm 45^\circ$	$\pm 60^\circ$
140°	1.7970	1.6218	1.4194	1.1430	0.6642
		1.9723	2.1747	2.4511	2.9299
142°	1.8131	1.6459	1.4527	1.1888	0.7318
		1.9804	2.1736	2.4375	2.8945
144°	1.8292	1.6699	1.4860	1.2348	0.7998
		1.9884	2.1723	2.4235	2.8586
146°	1.8450	1.6939	1.5193	1.2809	0.8680
		1.9962	2.1707	2.4091	2.8220
148°	1.8605	1.7176	1.5525	1.3270	0.9365
		2.0035	2.1686	2.3940	2.7846
150°	1.8756	1.7410	1.5855	1.3731	1.0052
		2.0103	2.1658	2.3782	2.7461
152°	1.8902	1.7639	1.6181	1.4189	1.0739
		2.0165	2.1623	2.3615	2.7065
154°	1.9042	1.7864	1.6504	1.4646	1.1427
		2.0220	2.1580	2.3438	2.6656
156°	1.9175	1.8082	1.6821	1.5099	1.2115
		2.0267	2.1528	2.3250	2.6234
158°	1.9299	1.8294	1.7133	1.5548	1.2802
		2.0305	2.1465	2.3051	2.5797
160°	1.9416	1.8498	1.7439	1.5992	1.3486
		2.0333	2.1392	2.2839	2.5345
162°	1.9523	1.8694	1.7738	1.6431	1.4167
		2.0351	2.1308	2.2615	2.4879
164°	1.9620	1.8881	1.8028	1.6863	1.4844
		2.0359	2.1212	2.2378	2.4396
166°	1.9707	1.9059	1.8310	1.7288	1.5516
		2.0356	2.1104	2.2127	2.3899

Table 16. Data needed to perform lattice strain measurements on b.c.c. iron, ferrite and martensite of iron base materials.

radiation	wave-length nm	filter	peak plane {hkl}	peak angle $2\theta_0$	$2\theta_{\alpha 2}$ - $2\theta_{\alpha 1}$	penetration depth $\mu\text{m}, \sin^2\psi=0.3$		calibra- tion powder	peak plane {hkl} _c	peak angle $2\theta_c$
						Ω	Ψ			
Ti-K α	0.274841	-	{200}	146.99	0.52	2.7	2.8	Al*	{220}	147.45**
Cr-K α	0.2289649	V	{211}	156.07	0.93	4.6	4.7	Cr	{211}	152.92
Fe-K α	0.1935979	Mn	{220}	145.54	0.76	6.9	7.2	Au	{400}	143.37
Co-K α	0.1788893	Fe	{211}	99.69	0.30	5.0	7.2	Au	{222}	98.87
			{310}	161.32	1.59	9.2	9.3	Au	{420}	157.48
Cu-K α	0.1540501	Mono- chrom.	{222}	137.13	0.73	1.5	1.6	Au	{422}	135.39
								Si*	{533}	136.89
Mo-K α	0.0709261	Zr	{732} +{651}	153.88	3.17	13.8	14.1	Cr*	{732} +{651}	150.97

Table 17. Data needed to perform lattice strain measurements on f.c.c. iron, retained austenite and austenitic base materials.

radiation	wave-length nm	filter	peak plane {hkl}	peak angle $2\theta_0$	$2\theta_{\alpha 2}$ - $2\theta_{\alpha 1}$	penetration depth $\mu\text{m}, \sin^2\psi=0.3$		calibra- tion powder	peak plane {hkl} _c	peak angle $2\theta_c$
						Ω	Ψ			
Ti-K α	0.274841	-	{111}	83.06	0.13	0.9	1.9	Cr	{110}	84.70**
			{200}	99.92	0.18	1.6	2.2	Si*	{311} _{β}	100.28**
Cr -K β -K α	0.2084789	-	{311}	148.74	-	5.8	7.1	Au	{222} _{α}	152.99**
	0.2289649		{220}	128.84	0.41	3.9	4.3	Au	{222} _{β}	124.59
Mn-K α	0.2101747	Cr	{311}	152.26	0.89	5.7	5.9	W	{220}	139.81
								Si	{422}	142.88
Fe-K α	0.1935979	Mn	{311}	126.83	0.47	6.0	6.8	Ge	{333} +{511}	125.51
			{222}	138.15	0.61	6.6	7.1	Au	{400}	143.37**
								Cr	{220}	143.32**
								Si	{333} +{511}	135.69
Cu-K α	0.1540501	Mono- chromator	{420}	147.28	0.99	1.6	1.9	Ge	{711} +{551}	152.96**
			{331}	138.53	0.76	1.5	1.9	Ge	{444}	141.21**
								Si	{533}	136.89
Mo-K α	0.0709261	Zr	{844}	150.87	2.79	13.6	14.0	W*	{831} +{750} +{743}	149.10

Table 20. Data needed to perform lattice strain measurements on nickel and nickel base materials.

radiation	wave-length nm	filter	peak plane {hkl}	peak angle $2\theta_0$	$2\theta_{\alpha 2}$ - $2\theta_{\alpha 1}$	penetration depth $\mu\text{m}, \sin^2\psi=0.3$ $\Omega \quad \Psi$		calibra- tion powder	peak plane {hkl} _c	peak angle $2\theta_c$
Ti-K α	0.274841	-	{200}	102.51	0.19	1.2	1.6	Si	{311}	100.28
Cr-K α	0.2289649	V	{220}	133.53	0.45	2.9	3.2	Au	{311}	137.17**
								Si*	{331}	133.53
Fe-K α	0.1935979	Mn	{222}	144.20	0.73	5.0	5.2	W	{310}	150.55**
								Si	{531} β	146.19**
Cu-K α	0.1540501	Ni	{420}	155.67	1.36	9.7	9.9	Au	{511}	157.81**
			{331}	144.65	0.91	9.3	9.7	Ge	{711}	152.96
									+{551}	
Mo-K α	0.0709261	Zr	{844}	160.84	4.66	10.3	10.5	Au	{422}	135.39
								W*	{662}	155.27

Table 21. Data needed to perform lattice strain measurements on titanium and titanium base materials.

radiation	wave-length nm	filter	peak plane {hkl}	peak angle $2\theta_0$	$2\theta_{\alpha 2}$ - $2\theta_{\alpha 1}$	penetration depth $\mu\text{m}, \sin^2\psi=0.3$ $\Omega \quad \Psi$		calibra- tion powder	peak plane {hkl} _c	peak angle $2\theta_c$
Ti-K α	0.274841	-	{110}	137.34	0.39	7.1	7.6	Si	{400} β	135.57
Cr-K α	0.2289649	V	{201}	136.50	0.49	1.3	1.4	Au*	{311}	137.17**
			{004}	155.80	0.92	1.5	1.5	Au	{222}	152.99
Fe-K α	0.1935979	Mn	{203}	156.50	1.15	2.3	2.4	W	{310}	150.55
Co-K α	0.1788893	Fe	{114}	154.48	1.13	2.9	2.9	W	{222}	156.46**
								Au	{420}	157.48**
								Si*	{531}	154.02
Cu-K α	0.1540501	Ni	{213}	139.38	0.78	4.0	4.2	Si	{533}	136.89
			{006}	161.36	7.06	4.4	4.5	Au*	{511}	157.81
									+{333}	
			{302}	148.43	1.31	4.2	4.3	Si*	{444}	158.63
								W	{400}	153.54**
Mo-K α	0.0709261	Zr						Si*	{642} β	147.15
the many peaks are not listed										

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2.05 Stationary and mobile X-ray equipment

W. Pfeiffer

2.051 Historical review

During the past 15 years X-ray diffraction techniques have been greatly improved and are now a reliable tool for measuring residual stresses. This development was not least promoted by the general availability of powerful and easy to use X-ray equipment, computers and software. Previously commercial equipment had only allowed measurements on samples that were not too demanding concerning the size, geometry and complexity of the stress state. However, easy to handle, flat and 'well-sized' samples, being the rule in X-ray phase analysis, are rather the exception in stress analysis. The requirement to analyze bigger samples or real parts of complicated geometry using high diffraction angles and the side inclination method [1] lead to the development of numerous unique measuring devices. Figure 1, for example, shows a stationary diffractometer with 13 axes for measurements on large components. A remarkable feature of this equipment is the capability of a completely independent positioning and rotation of the X-ray tube and the detector. This results in an exceptional flexibility.

The specific demands of X-ray residual stress analysis have also been taken into consideration where commercial equipment was developed, since the interest in residual stress measurements has increased. Within the wide field of X-ray diffraction techniques residual stress analysis is a minor application, which is usually ordered only as part of an application package. Therefore, only mobile equipment is designed especially for stress measurement. Stationary diffractometers are mostly powder-diffractometers, equipped with special devices that may be used both for stress and texture analysis.

The development of mobile equipment started with the design of stationary equipment applicable to large components (e.g. [3]). Stress determinations on large components not fitting into the circle of conventional diffractometers made it necessary to develop so called 'centreless' diffractometers which, of course, imply serious restrictions concerning small diffraction angles used for powder diffraction methods. Next the availability of position sensitive detectors (PSD) reduced the total measurement times to minutes. Additionally the PSD's allowed the use of miniaturized X-ray tubes, small sized X-ray generators and autonomous cooling systems. Thus, the essential components needed for really mobile equipment have become available. During the last 20 years an increasing number of manufacturers have been offering mobile equipment (see [4, 5]). Today (1995) five companies are on the market (American Stress Technologies Inc. (AST), PROTO Manuf. Ltd., Rigaku, Siemens and TEC).

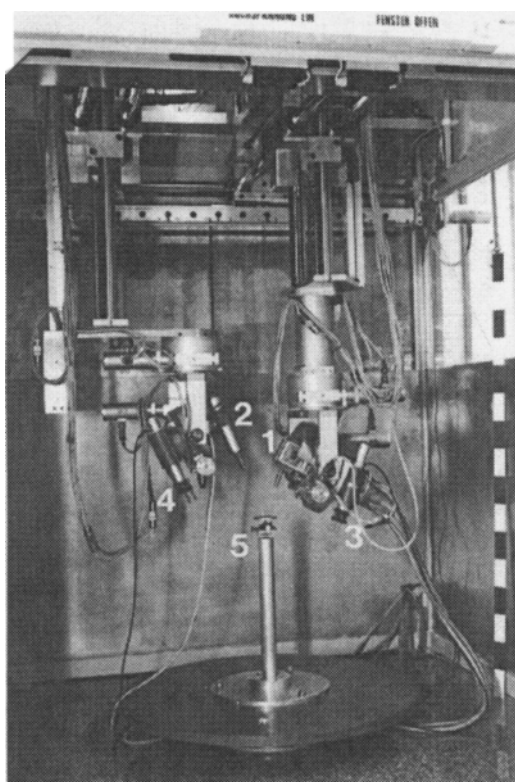


Figure 1. 13-axes diffractometer designed for stress measurements on large components [2].

- 1 = X-ray tube,
- 2 = detector,
- 3, 4 = alignment laser,
- 5 = specimen carrier.

In the following an overview is given about the state of *commercial equipment* and some new developments suitable for stress analysis.

2.052 Stationary equipment

Most residual stress measurements are performed with stationary diffractometers using the *bragg-brentano parafofocusing* technique (see Fig. 2). Monochromators may be added to the beam path when e.g. problems arise due to overlayed peaks (focusing primary monochromatores) or in case of high fluorescence radiation produced by the sample investigated (focusing or non-focusing secondary monochromators). The problem of the extreme loss of intensity when using focusing primary monochromators may be overcome by the new X-ray 'mirrors'. Using the diffraction of the primary beam at a 'multilayer' single crystal with the layers slightly tilted a monochromized beam of excellent intensity and high parallelism can be produced [e.g. 6]. The diffractometers are usually located inside a radiation protective box protecting the operator completely from radiation during usual operation of the equipment. But one should realize that the alignment of the equipment or the inspection of the

size and location of the measurement spot often can be performed only by direct observation with the box opened.

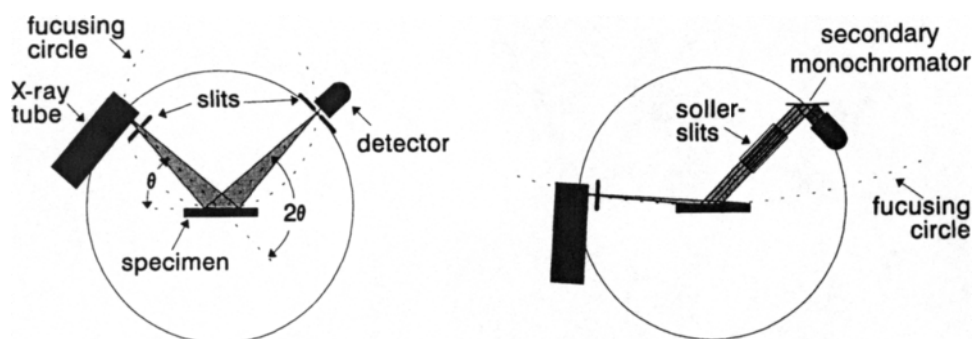


Figure 2. Basic set-up of diffractometers using the bragg-brentano parafocusing technique (left) and some optional devices for collimating and monochomazating of the X-ray beam (right).

Precision and speed of the angular positioning of today's diffractometers fulfill highest demands as a rule. Using diffractometer diameters in the range of 400 mm to 600 mm, numbers like $\pm 0.005^\circ$ for the absolute accuracy of angular positioning or $\pm 0.0025^\circ$ in the case of built-in optical encoder disks are stated. At first glance such accuracy seems to be unnecessary for stress determination but becomes important, if measurements must be performed at unfavorable small diffraction angles or if results derived from different lattice planes must be combined. The advantage of high positioning speeds up to 1000° per minute is obvious if one recognizes the sum of angular movements of sample and detector during a typical stress measurement.

Manufacturers of X-ray diffraction equipment place increasing importance on *computer control of operating functions*. Positioning of measurement spots and directions and collimating of the beam path by primary and secondary slit systems by stepping motors or control of the X-ray generator and the energy discrimination of the detector can often be performed comfortably and precisely with the help of the computer and software. Extensive software packages are available for all common diffraction methods. Especially AT-compatible PC's using the WINDOWS® operating system have been gaining acceptance against workstations. The DEC-VAX®-computers, widely spread in former times have become the exceptional case.

The special demands of X-ray stress analysis on measuring techniques are met by the manufacturers using different strategies. Thanks to slim X-ray tubes and shielding housings *high diffraction angles* up to $2\theta = 165^\circ$ can be reached by nearly every equipment. In order to allow measurement using the *side inclination method* so called Ψ -diffractometers [1] are used or different attachments are available which all may be used also for texture analysis using the Schulz method [7].

The Seifert-version of a Ψ -diffractometer, the 'TS-Attachment', is based on the concept of Krause and Demirci [8], see Fig. 3. A remarkable feature of this attachment is the parallelogram system used for the ψ -tilting which does not restrict the lateral sample extension and allows for θ -angles down to 0° . Additionally the samples can be rotated by the angle φ for determination of different stress components or for texture measurements.

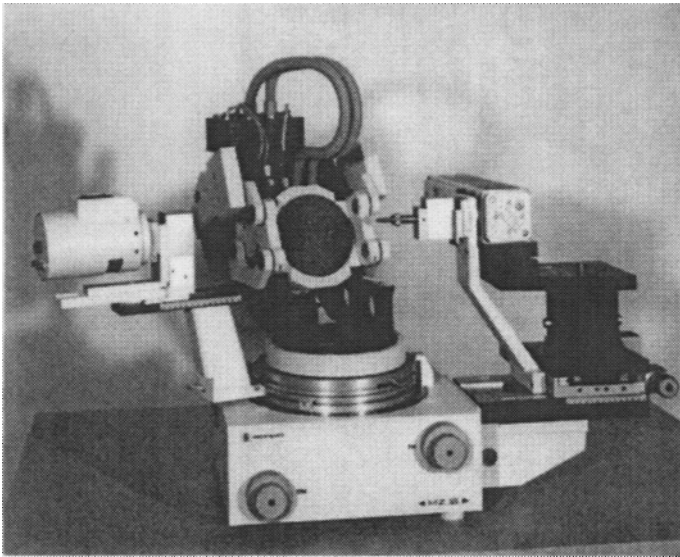


Figure 3. Special attachment allowing for both residual stress and texture measurement (Seifert 'TS-Attachment')

The Huber diffractometer '4030' also does not suffer from limitations concerning the lateral sample extension since some drawbacks of the predecessor (which was based on the concept of [1]) have been eliminated. Additionally the new design allows for an increase of the accessible sample volume by a simple enlargement of the mechanical dimensions of the attachment.

The possibility of, both stress and texture measurements, is also facilitated by acentric open Eulerian cradles, which are available through all major manufacturers of diffractometers (see example in Fig. 4). The gap in the χ -circle eliminates shading of the high diffraction angles, which are important for stress measurements. The displacement of the χ -circle with respect to the φ -circle increases the accessible sample volume. Some types of Eulerian cradles may also be equipped with linear stages for positioning purposes or to reduce coarse grain effects by linear oscillation of the sample.

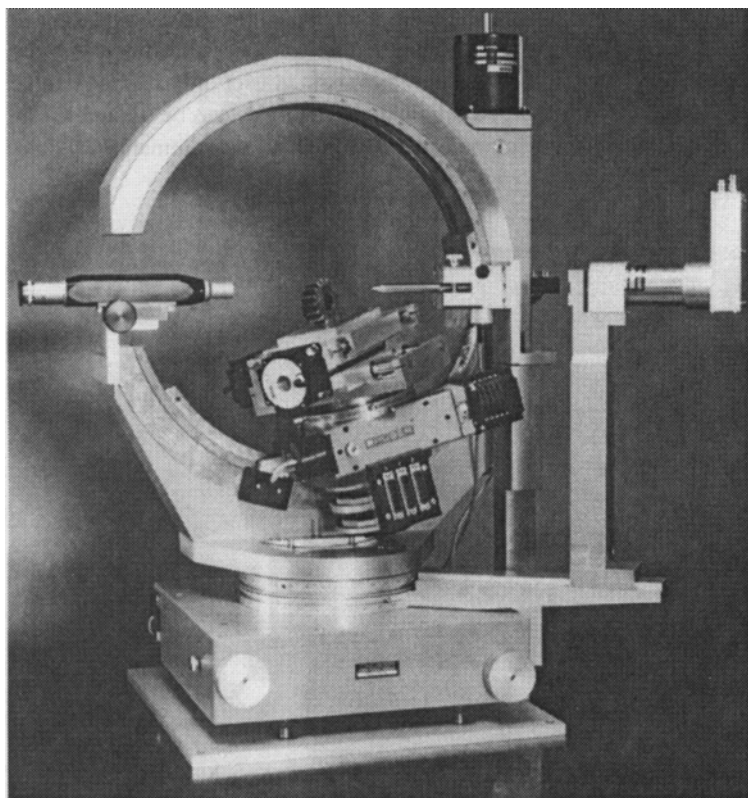


Figure 4. Acentric open Eulerian cradle allowing for both residual stress and texture measurements (Huber 424 - 512.51)

Of course the *size and weight of samples* fitting into all these attachments are limited and depend additionally on the size of the diffractometer circle and on additionally mounted accessories. In general feasible sample sizes up to $100 \times 100 \times 100 \text{ mm}^3$ and weights up to 3 kg are specified by the manufacturers. Larger and heavier samples may be possibly handled by stationary equipment using a θ - θ configuration, if one abstains from the preferable side inclination method: By keeping the sample in a fixed position and moving the X-ray tube and the detector, large and heavy components may simply be placed in front of the diffractometer and positioned by special linear stages (see example in Fig. 5). But possible locations of measurement spots are still restricted by the height of the X-ray focus above the basis of the diffractometer and by the needed freedom of movement of tube and detector.

Measurements on extremely small areas may be preferably performed using specially designed *micro-diffractometers* (see example in Fig. 6). They are equipped with collimator systems allowing for beam diameters of, e.g., $20 \mu\text{m}$, focused laser pointers and video microscopes for adjustment of the measurement spot.

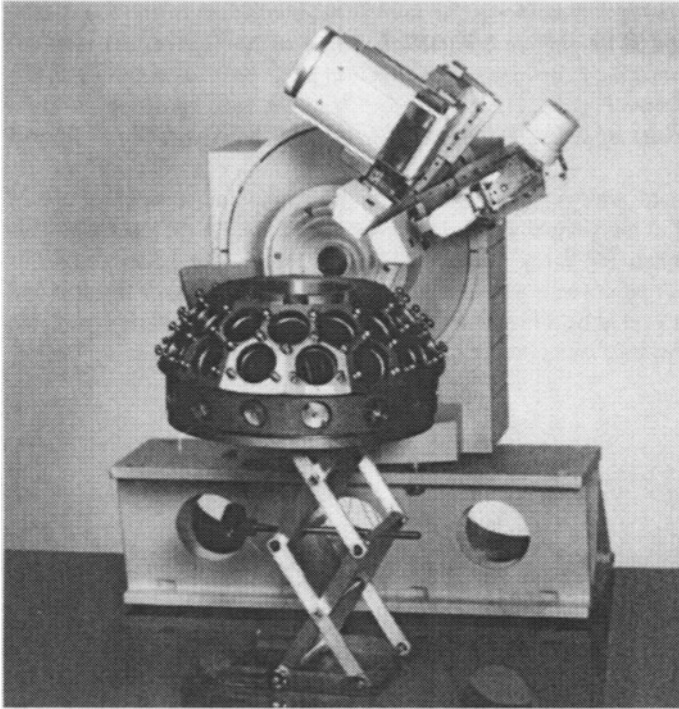


Figure 5. Typical experimental arrangement of residual stress measurements on large components using a θ - θ -diffractometer (Philips 'PW 3050/10' diffractometer with ceramic tube and automated slit system).

Accessories for an easy *mounting and alignment* of such samples, which do not have the size and geometry of flat 'standard' samples are rarely available for conventional powder diffractometers.

Open Eulerian cradles and the other attachments mentioned above may be equipped with linear stages and micrometer gauges in order to align curved samples by simply adjusting the measurement spot to maximum (or minimum) height during lateral displacement of the sample. A flexible *collimating* of the primary beam, which may be needed for measurements of exactly defined areas or curved samples, can be done effectively only with the help of self built masks or collimators, as a rule. The loss of intensity when using small sized pinhole collimators may be compensated by the above mentioned 'X-ray mirrors' or by using the effect of total reflection in collimators based on glass capillaries [9].

In the case of diffractometers operating in iso-inclination technique and using the line focus of the X-ray tube automatic divergence slits may be applied. These slit systems allow to keep the width of the irradiated area constant during ψ -tilting in the case of the iso-inclination method. But due to the length of the line focus the length of the irradiated area cannot be reduced effectively by slits without loss of most of the intensity. A more universal

arrangement for stress measurements therefore is the use of the point focus of the X-ray tube with the primary beam collimated by pinhole collimators. The disadvantage of less intensity and angular resolution when recording conventional diffraction patterns may be solved by the new 'ceramic-tube' of Philips (see Fig. 5). The high accuracy of the focus position allows a change from point to line focus by a simple rotation of the tube head without additional alignment.

Localization of the irradiated area in general needs the laborious and inaccurate use of fluorescence material applied to the sample and a dangerous inspection by the operator. This disadvantage may be eliminated by X-ray collimator carriers with laser beam tracer, as supplied by Huber for use with point focus arrangements. This equipment makes the size and location of the irradiated area visible by a bright semiconductor laser beam, which is directed collinearly with the X-ray beam by a small mirror turned into the collimator axis.

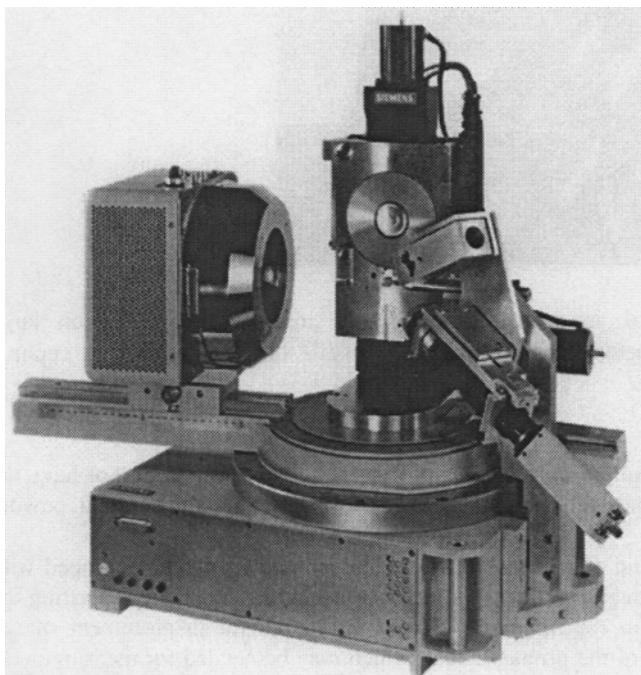


Figure 6. Microdiffractometer with area detector, laser pointers and video microscope for adjustment of the measurement spot (Siemens 'PLATTFORM'-diffractometer with 'GADDS'-detector system)

2.053 Mobile equipment

Deciding criteria for reliable and economic stress determinations using mobile equipment are the *size and weight* of the equipment, the possibility of a *stiff connection* between diffractometer and component to be measured and, above all, a *simple alignment* of the measurement spot and direction of measurement. The weight of the today's mobile diffractometers is in the range of 10 kg to 25 kg, the weight of the complete equipment in the range of 75 kg up to 125 kg with an overall size fitting into a usual passenger car. In particular, the AST-diffractometer 'G2' stands out due to its small size (especially with the ϕ -rotation dismounted) being the result of the use of solid state photo detectors with a distance to the specimen of only 50 mm (Fig. 7). Such an ultra compact construction is advantageous in the case of measurements in cramped conditions (e.g. measurements inside tubes). On the other hand, a small distance between diffractometer and component may be of disadvantage, if the measurement spot is located on a concave surface with the surroundings being close to parts of the diffractometer.

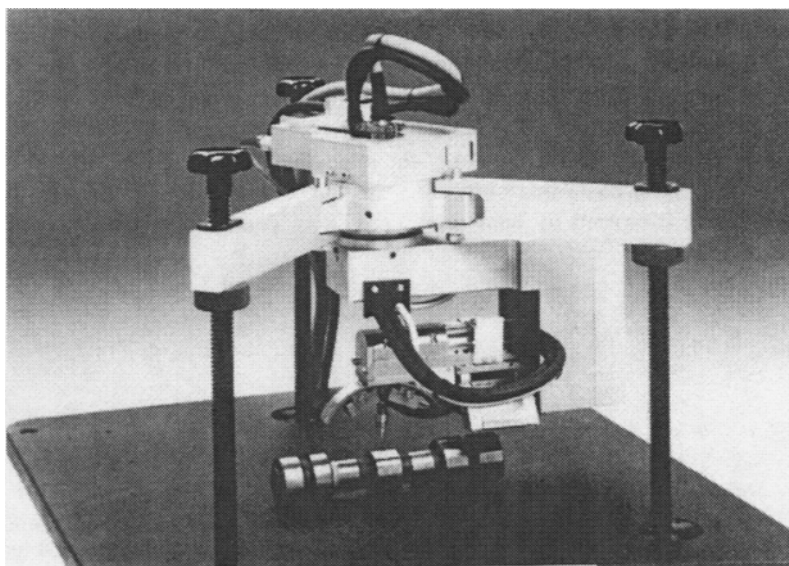


Figure 7. Compact mobile X-ray stress analyzer with position sensitive solid state photo detectors (AST).

The coupling between component and diffractometer normally is done by tripods equipped with magnetic mountings or suckers. Some diffractometers may be mounted on a wheel stand as an option allowing additional linear and angular movements for precise adjustment of measurement spots. Without such wheel stands the precise adjustment of the location of the

measurement spot and of the ψ_0 -angle is a tricky and time-consuming procedure, because the complete diffractometer has to be moved stepwise. For the alignment of the distance between diffractometer and sample the manufacturers usually provide micrometer gauges and linear stages. In the case of the AST-diffractometer the alignment of the height of the diffractometer is completely automated. The quality of the adjustment of height and the maintenance during operation of course cannot be as precise as in stationary diffractometers. Since most mobile diffractometers may be operated in the side inclination mode this does not affect the accuracy of stress determinations significantly when performed at high diffraction angles.

The effects of misalignment are reduced by the scintillation counter version of the Rigaku-diffractometer as this diffractometer uses parallel beam optics [10]. The direction of the stress measurement can be selected by a stepping motor operated rotation of the diffractometers (optionally in some cases) which enables one to determine the complete stress tensor without a time-consuming manual moving of the equipment.

The 2θ -range simultaneously obtained by the PSD's is about 20° - 25° . Thus the total diffraction lines can be recorded simultaneously in most cases. The 2θ -position of the centre of the PSD in some cases can be changed in a continuous way by a simple movement of the detector (AST, Rigaku) or, which is a time-consuming operation, by exchange of the detector mounting (Siemens). The highest diffraction angle to be measured is in the range of 164° - 170° with a value of 164° being not very satisfactory in the case of the most frequent application, the measurement of broadened $\{211\}$ -lines of steel. Due to geometrical restrictions and an increasing influence of misalignments on the accuracy with increasing ψ -angles the ψ -range is limited to about $\pm 45^\circ$ in most cases.

Conduction of the measurement and treatment of the stored data are usually performed by AT-compatible PC's and menu-controlled software. This gives a better flexibility in the treatment of 'unusual' data compared to 'hardwired' calculation parameters, that are better applicable to not too demanding routine measurements.

2.054 Detectors

The *scintillation counter* is a basic accessory of a diffractometer. This type of detector permits uncomplicated handling, as its counting characteristic is nearly independent of operating time and temperature fluctuations. Due to small dead time effects high intensities can be measured. Thus, measurement times comparable to those of PSD's (which show higher dead time effects as a rule) may be possible when strong diffraction lines, large irradiated areas and/or high tube power are available. As resolution and reproducibility of angular measurements are fixed by size and position of slits, a high reproducibility of measurements can be attained over long periods of time due to the fixed mechanical alignment of the equipment. Therefore the scintillation counter is also the best suited detector for the alignment of an X-ray diffractometer.

In the field of (the more expensive) *position sensitive detectors* a whole lot of different types of linear detectors have been developed (for an overview see [11]). For residual stress measurements especially proportional counters have been proved, which use 50 mm up to 100 mm long anode wires [12] or fan-shaped wound cathode wires [13] operating in gas filled

ionization chambers. The gas mostly consists of an Ar/CH₄- or Ar/CO₂-mixture. For Cr-radiation a higher quantum gain can be expected when a (more expensive) Xe/CH₄-mixture is used. The anode-wires are 20 µm glass-wires with a high-resistance coating or metal-wires (Pt). Two different principles are used to locate the position of the ionization event caused by the X-ray photons. In the case of high-resistance anode wires the current ratio between the left- and right-hand end of the wire is determined. The second principle determines the difference between rise times of the signal at the ends of the anode wire. Although problems of the high resistance coatings have been solved and the current ratio systems provide a better local resolution, the rise time systems have been proved for stress measurements since their electronic is simpler (and cheaper) and their wires are more robust. Common to all systems are some problems with 'blind' sections of the wire due to an increasing deposit of cracked gas molecules during operation. Additionally a significant influence of the temperature and pressure of the gas on the pulse height discrimination and the position calibration has to be considered. These effects necessitate an increased effort in controlling the counting characteristics of the PSD's. Thus, the profit of decreased measurement time compared to scintillation counters is partly lost. An interesting alternative may be therefore the use of PSD's on the basis of solid state photo detectors. Up to now the application of these detectors has been the exception, but the development is in progress (see the mobile equipment of AST).

For special applications of stress analysis, e.g. measurement of very small areas, strongly textured materials, coarse grained materials, or single crystals, *area detectors* may become of increasing interest. These two-dimensional PSD's are able to detect part of or complete diffraction cones of one or more diffraction planes at once. Since the diffraction geometry is not compatible to the classical $\sin^2\psi$ -method, modified evaluation methods like the ϕ -integral [14] or ϕ -rotation method [15] or a conversion to traditional diffraction data have to be applied. Three different types of area detectors are of special interest for X-ray diffraction: One system, being a two-dimensional version of the proportional detectors with anode wires, uses a multi-wire grid, see e.g. [16]. The second type uses the photoluminescence effect, see Fig. 8 and e.g. [15, 17]: First, a latent image of the diffraction pattern is stored on a specially coated flat plate. Then the imaging plate is scanned by a 150 µm laser beam with the photoluminescence light being detected by a coupled photo multiplier. The third type of an area detector is the CCD-based solid state photo detector [18]. A one-dimensional version is already used in mobile equipment. Due to the possibility of in-situ observation of the measurement, it is expected that the solid state detector will win through against the imaging plate.

2.055 Software

An essential part of today's X-ray diffraction equipment is an extensive software package supporting all common diffraction methods. The packages in general have a modular structure. Basic programs manage, e.g., the system parameters or the adjustment of the diffractometer and electronics. Special stress software packages allow comfortable and, thanks to graphics support, also clear treatment of measured data.

Besides the basic routines for PLA-, $K\alpha_2$ - and background-correction different methods for the determination of the peak position are available: Common methods are the parabolic fit to the top part of the peak, the centroid methods and the mid of the cord method. Sometimes routines for fitting the peak using analytical functions or the cross correlation method are available. In addition to the calculation of single normal stress some programs allow the calculation of shear components due to ψ -splitting or the evaluation of the complete stress tensor from measurements performed at different φ -angles.

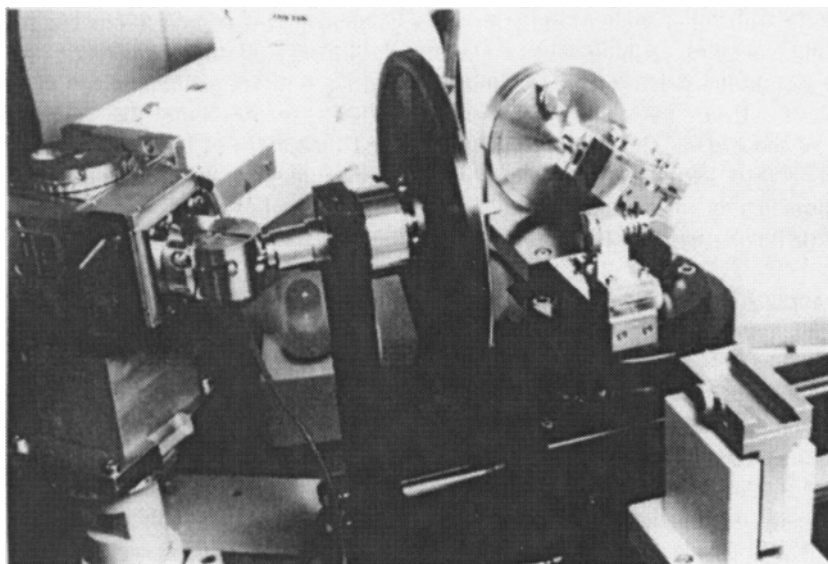


Figure 8. Experimental arrangement for residual stress measurements using an imaging plate detector [15].

Single or double exposure techniques are rarely used due to the extreme uncertainties related to stress determinations using these techniques. Other evaluation methods like the φ -rotation method [15] or the integral method [19] are rarely used.

Thanks to commercially available software based on well established methods of stress analysis there is usually no need for self made software anymore. On the other hand especially due to the large number of available parameters for measurement and evaluation the risk of using inadequate conditions has increased. Therefore the evaluation of a PC-based expert system [20] may be useful for the assessment of X-ray residual stress determinations.

2.056 Recommendations

It is obvious, that the choice of a diffractometer to be used for stress measurements strongly depends on the type of samples to be investigated, on the type of additional diffraction experiments to be performed and on the budget available. However, some basic requirements should be fulfilled in most cases and therefore the following requirements can be given.

High diffraction angles up to $2\theta = 165^\circ$ should be possible and the diffractometer should allow the side inclination method at least by using optional attachments. In order to be able to analyze also bigger samples or real parts of complicated geometry accessories for an easy mounting and alignment of such samples should be available. The possibility of the localization of the irradiated area by laser pointers or large-distance microscopes is often of great advantage.

Deciding criteria for reliable and economic stress determinations using mobile equipment are the size and weight of the equipment, the possibility of a stiff connection between diffractometer and component to be measured and, above all, a simple alignment of the measurement spot and direction.

The scintillation counter is a basic accessory of a stationary diffractometer. One-dimensional position sensitive detectors are meanwhile reliable instruments that reduce measurement times dramatically in case of weak diffraction lines and small irradiated areas. But there is also an increased need for maintenance and calibration procedures. Area detectors are still very expensive and have some drawbacks if the diffractometer has to be used for general diffraction experiments. They are therefore mainly of interest for some special applications concerning coarse grained materials, single crystals or small spot sizes.

An essential part of today's X-ray diffraction equipment is a software package supporting all common diffraction methods. The stress software must be able to calculate normal and shear stress components as well as the complete stress tensor using the established methods. The software should be clearly structured and easy to be handled. Measurement parameters, conditions of evaluation, and results have to be clearly documented and should be stored in some standard data format allowing for further use by e.g. standard word processing and desktop publishing programs. Standard measurements should be possible with a minimum of interaction. For more complicated applications a larger flexibility in selection of measurement and evaluation parameters is needed and it is very helpful if intermediate results are available for further evaluations by user designed software.

2.057 References

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2.06 Definition of macro- and microstresses and their separation

The definition of residual stresses (RS) besides the structural or load stresses (LS) was settled in /1,2,3/. The bases were the numerous proposals in the literature. RS I are called in the US-literature macro-RS and meanwhile this is a common designation. Of course, load stresses are macrostresses. RS II and RS III are called micro-RS. Dealing with micro-RS has led to an extended definition of RS II. They are not related to a single crystallite but to the average value of several crystallites /4/. Also RS III have been studied on plastically deformed crystals and polycrystalline metals /5,6/. The dislocation structure, cells and walls, were early considered to explain the origin of compression-RS in plastically deformed quasi-homogeneous metals. Micro-RS appear as peak shift and peak broadening. In paragraph 2.037 the kinds of stresses, their definitions and relations are presented and discussed. The main formulae will be repeated here:

$$\bar{\sigma}^\alpha = \sigma^L + \sigma^I + \langle \sigma^{II} \rangle^\alpha + \langle \sigma^{III} \rangle^\alpha \quad (1)$$

$$\sum_{\alpha=1}^n c^\alpha \langle \sigma^{II} \rangle^\alpha = 0 \quad (2)$$

and with

$$\langle \sigma^{III} \rangle^\alpha = 0 \quad (3)$$

$$\sum_{\alpha=1}^n c^\alpha \sigma^\alpha = \sigma^L + \sigma^I = \sigma_{macro} \quad (4)$$

These formulae enable one to separate macro- $(\sigma^L + \sigma^I)$ - and micro- $(\langle \sigma^{II} \rangle^\alpha)$ -RS if strain measurements on all phases of the material in question exist. The separation of $\langle \sigma^{II} \rangle^\alpha$ and $\langle \sigma^{III} \rangle^{\alpha'}$ is not known at the present. α' is a part of α , see paragraph 2.037.

The problem of separating macro- and micro-RS was raised in early times and was related to the question of stress compensation within the cross section and within the phases of the material. It was pointed out in 1964 that in hardened martensitic-austenitic steels micro-RS besides macro-RS are present and the way to determine RS I was shown based on measurements on both phases /7/. During the discussion about shear components in steels after grinding, in 1981 the following formulae were introduced /8/ with t the thickness of the flat specimen and c the volume content of iron:

$$\int_0^t \sigma^I dz = 0 \quad \sigma^I \text{ dependent on } z \text{ only} \quad (5)$$

Table 1. Designation and characteristics of micro residual stresses and the observed microstresses in monophasic materials (open symbols), multiphase materials (closed symbols), cubic ($\square, \blacksquare$), non cubic phases ($\Delta, \blacktriangle$) /21,22/.

Designation			Origin	X- ray analysis of ϵ, D			Compensation of microstress				
				FWHM	$\epsilon, D - \sin^2\psi$	lattice-plane dependence	between crystal orientations	dislocation-cell walls	between phases	homogeneous region	orientation dependence
random stress			plastic strain, transformation	>		possible	within and between crystals	within and between crystals	not concerned	RS II: one crystal RS III: -	
phase averages	elastic deformation	mean stress	different E and ν of the phases	=	isotropic materials: linear dependences	due to elastic anisotropy	not concerned	not concerned	$\blacktriangle \blacksquare$	phase	no
	plastic deformation		plastic strain		$\square$						
	thermal		different expansion coefficients		not concerned						
	phase transformation		change of cell dimension								
orientation averages	elastic deformation	intergranular stress	elastic anisotropy	$\approx$		due to elastic anisotropy	corresponds to the different models of XEC calculation $\Delta \square, \blacktriangle \blacksquare$		not concerned	crystallite group	yes
	plastic deformation		plastic strain	$\approx$	oscillation around horizontal line	strong	$\square \blacksquare$	$\square \blacksquare$			
	thermal		thermal, non cubic systems	=	isotropic materials: linear dependences textured materials: oscillations		Δ				
	phase transformation		change of non cubic cell								

$\langle \sigma^{II} \rangle_{Fe}$ and $\langle \sigma^{II} \rangle_{Fe_3C}$ should be constant over the cross section

$$c \int_0^t \sigma_{Fe}^{I+II} dz + (1 - c) \int_0^t \sigma_{Fe_3C}^{I+II} dz = 0 \tag{6}$$

and in each depth

$$c \langle \sigma_{13} \rangle_{Fe}^{II} + (1 - c) \langle \sigma_{13} \rangle_{Fe_3C}^{II} = 0 \tag{7}$$

$$\sigma_{33}^I = 0$$

In the following years the separation of macro- and micro-RS and their compensation were often applied to different material problems /4,9,10,11,12,13,14/. In the following papers there is a writing mistake in the formulae cited: σ_{α}^I should be replaced by σ_{α}^{I+II} in /15,16,17,18,19/.

Table 1 describes the different RS II and RS III, their origins, and their manifestations /20,21,22/. In general RS II are today defined as the average RS of a phase or of a group of crystallites minus the macro-RS I. Lot of attempts have been made to alter the definitions of the RS or to add new notations. But after thorough thoughts the above mentioned definitions are well established. The very important effect in X-ray and neutron ray diffraction is the fact that macro- and micro-RS are both contributing to the lattice strain, the peak shift. Furthermore, the micro-RS can be partly dependent from the macro-RS, but partly non dependent, section 2.122d. The experimental methods to determine macro- and micro-RS are listed in Table 2 /23/. In most cases, the weighted-averaged value over the measuring volume will be evaluated. This does not matter if gradients of any kind are not present. But it has to be taken into account especially if steep gradients in thickness direction are present. Attention should be paid to the explanations of chapter 2.15. Influences of texture have to be considered prior to the separation of macro- and micro-RS.

Table 2. Methods to determine macro- and micro-RS /23/

method	RS I RS (II + III)		
mechanical, for example bending arrow	×		
ultrasonic	×		
micromagnetic	×	+	×
diffraction by X- or neutron rays	×	+	×
diffraction on all phases of the material	×		×
diffraction by X-ray on very thin plates			×
diffraction by neutron rays as average over total cross section			×

2.07 Evaluation of LS and RS

2.071 Formulae and data

2.071a Nontextured, mechanically isotropic material

Reference is made to chapter 2.03. But in the following the consequences for practical use of the basics are drawn. According to the theory of elasticity the strain $\varepsilon_{\varphi,\psi}$ is given as a function of the strain components ε_{ij} ($i, j = 1, 2, 3$) by

$$\varepsilon_{\varphi,\psi} = \frac{D_{\varphi,\psi} - D_0}{D_0} = \varepsilon_{11} \cos^2 \varphi \sin^2 \psi + \varepsilon_{12} \sin 2\varphi \sin^2 \psi + \varepsilon_{13} \cos \varphi \sin 2\psi \\ + \varepsilon_{22} \sin^2 \varphi \sin^2 \psi + \varepsilon_{23} \sin \varphi \sin 2\psi + \varepsilon_{33} \cos^2 \psi \quad (8)$$

This formula was used for the first time in the XSA by /24/. Using the generalized Hooke's law

$$\sigma_{ij} = \frac{1}{\frac{1}{2}s_2} \left[\varepsilon_{ij} - \delta_{ij} \frac{s_1}{\frac{1}{2}s_2 + 3s_1} (\varepsilon_{11} + \varepsilon_{22} + \varepsilon_{33}) \right] \quad (9)$$

or

$$\varepsilon_{ij} = \frac{1}{2}s_2 \sigma_{ij} - \delta_{ij} s_1 (\sigma_{11} + \sigma_{22} + \sigma_{33}) \quad (10)$$

$$\delta_{ij} = \begin{cases} 1 & \text{if } i = j \\ 0 & \text{if } i \neq j \end{cases}$$

formula (8) becomes

$$\varepsilon_{\varphi,\psi} = \frac{1}{2}s_2 (\sigma_{11} \cos^2 \varphi \sin^2 \psi + \sigma_{22} \sin^2 \varphi \sin^2 \psi + \sigma_{33} \cos^2 \psi) \\ + \frac{1}{2}s_2 (\sigma_{12} \sin 2\varphi \sin^2 \psi + \sigma_{13} \cos \varphi \sin 2\psi + \sigma_{23} \sin \varphi \sin 2\psi) \\ + s_1 (\sigma_{11} + \sigma_{22} + \sigma_{33}) \quad (11)$$

s_1 and $\frac{1}{2}s_2$ are the X-ray Elastic Constants (XEC) depending on the interference plane $\{hkl\}$ and the material phase. For cubic materials the following equation holds:

$$3s_1(hkl) + \frac{1}{2}s_2(hkl) = 3s_1 + \frac{1}{2}s_2 = \frac{1-2\nu}{E} = \frac{1}{3K} \quad (12)$$

The generalized transformation between specimen and measuring (laboratory) system is given by

$$\varepsilon'_{ij} = \omega_{ik} \omega_{jl} \varepsilon_{kl} \quad (13)$$

$$\omega = \begin{pmatrix} \cos \varphi \cos \psi & \sin \varphi \cos \psi & -\sin \varphi \\ -\sin \varphi & \cos \varphi & 0 \\ \cos \varphi \sin \psi & \sin \varphi \sin \psi & \cos \psi \end{pmatrix} \quad (14)$$

For the measuring direction within the specimen system it holds that

$$\varepsilon'_{33} = \omega_{3k} \omega_{3l} \varepsilon_{kl} = m_k m_l \varepsilon_{kl} \quad (15)$$

$$\begin{pmatrix} m_1 \\ m_2 \\ m_3 \end{pmatrix} = \begin{pmatrix} \cos \varphi \sin \psi \\ \sin \varphi \sin \psi \\ \cos \psi \end{pmatrix} \quad (16)$$

Referred to Equ. 11 and with the assumption or evidence that no shear components ε_{ik} or σ_{ik} ($i \neq k$) are present the formula reduces to

$$\varepsilon_{\varphi,\psi} = \frac{1}{2} s_2 \left[(\sigma_\varphi - \sigma_3) \sin^2 \psi + \sigma_3 \right] + s_1 (\sigma_1 + \sigma_2 + \sigma_3) \quad (17)$$

with

$$\sigma_\varphi = \sigma_1 \cos^2 \varphi + \sigma_2 \sin^2 \varphi \quad (18)$$

Neglecting σ_3 the formula simplifies for the azimuth $\varphi = 0^\circ$ and $\varphi = 90^\circ$ to

$$\varepsilon_{0,\psi} = \frac{1}{2} s_2 \sigma_1 \sin^2 \psi + s_1 (\sigma_1 + \sigma_2) \quad (19)$$

$$\varepsilon_{90,\psi} = \frac{1}{2} s_2 \sigma_2 \sin^2 \psi + s_1 (\sigma_1 + \sigma_2) \quad (20)$$

Measuring $D_{\varphi,\psi}$ in both directions $\psi > 0^\circ$, $\psi < 0^\circ$ versus different $\sin^2 \psi$ values, and regressing the averaged D vs. $\sin^2 \psi$ by a straight line the most-used formula holds:

$$\sigma_\varphi - \sigma_3 = \frac{1}{\frac{1}{2} s_2} \frac{\partial \varepsilon_{\varphi,\psi}}{\partial \sin^2 \psi} = \frac{1}{\frac{1}{2} s_2} \frac{1}{D_0} \frac{\partial D_{\varphi,\psi}}{\partial \sin^2 \psi} = - \frac{1}{\frac{1}{2} s_2} \frac{\cot \Theta_0}{2} \frac{\partial (2\Theta_{\varphi,\psi})}{\partial \sin^2 \psi} \quad (21)$$

For the determination of σ_3 the strain-stress-free lattice distance D_0 must be known exactly, see Fig. 5 and chapter 2.11.

The stress determined by diffraction methods from line shifts is, as already stated, an average $\bar{\sigma}^\alpha$, Equ. 1.

Two distinct directions within the D - or ε -vs.- $\sin^2 \psi$ diagram are of special interest. The strain-stress-free direction ψ^* to determine the lattice constant D_0 of the strain-stress-free state of the material according to the following equation:

$$\frac{1}{2} (\varepsilon_{+\psi} + \varepsilon_{-\psi}) = 0 \quad (22)$$

and the strain/stress-independent direction ψ' . The D -vs.- $\sin^2 \psi$ straight lines of different load stresses σ^L are crossing in the point D' , $\sin^2 \psi'$. The equation for this condition is

$$\frac{\partial \frac{1}{2} (\varepsilon_{+\psi} + \varepsilon_{-\psi})}{\partial \sigma^L} = 0 \quad (23)$$

2.071b Examples of XEC

The Table 3 summarizes the evaluation constants necessary for the stress determination from linear lattice-strain dependences. The XEC s_1 and $\frac{1}{2}s_2$ are calculated according to the model of Eshelby-Kröner by /25/. The data given are valid for the said elements and their base materials /26,27 supplemented/. The XEC of Ti are recalculated using newer monocrystal data. The Θ_0 -dependent data are derived from the XEC of this table and the Θ_0 -values of Tables 16-21 in paragraph 2.046. Significant deviations from the data of Table 3 may occur and noted by experimental tests. These deviations arise from textures, strong deformations, coarse grain sizes, alloying elements and second phases.

Table 3. XEC and related constants for the evaluation of lattice strain.

material	radiation	interference {hkl}	$-s_1$	$\frac{1}{2}s_2$	$-\frac{1}{2}\cot\Theta_0$	$\frac{1}{2}\cot\Theta_0$
			10^{-6} MPa^{-1}	10^{-6} MPa^{-1}	s_1	$\frac{1}{2}s_2$
					10^5 MPa	10^4 MPa
iron, ferritic-pearlitic iron-basis-materials	Ti- K_α	{200}	1.89	7.67	0.784	1.932
	Cr- K_α	{211}	1.25	5.76	0.848	1.840
	Fe- K_α	{220}	1.25	5.76	1.241	2.692
	Co- K_α	{211}	1.25	5.76	3.375	7.324
		{310}	1.66	6.98	0.495	1.178
	Cu- K_α	{222}	1.04	5.12	1.888	3.834
	Mo- K_α	{732}+{651}	1.34	6.05	0.866	1.917
retained austenite, austenitic iron-basis-materials	Ti- K_α	{111}	1.28	5.21	4.411	10.836
		{200}	2.40	8.56	1.751	4.908
	Cr- K_β	{311}	1.87	6.98	0.748	2.004
	Cr- K_α	{220}	1.56	6.05	1.534	3.956
	Mn- K_α	{311}	1.87	6.98	0.660	1.769
	Fe- K_α	{311}	1.87	6.98	1.338	3.585
		{222}	1.28	5.21	1.494	3.670
	Cu- K_α	{420}	1.86	6.95	0.789	2.111
		{331}	1.48	5.81	1.279	3.258
	Mo- K_α	{844}	1.56	6.05	0.833	2.147
aluminum, Al-basis-materials	Ti- K_α	{220}	4.97	19.07	0.294	0.765
	Cr- K_α	{222}	4.79	18.56	0.215	0.555
	Fe- K_α	{400}	5.51	20.60	0.278	0.743
	Co- K_α	{420}	5.17	19.62	0.152	0.400
		{331}	4.92	18.93	0.285	0.740
	Cu- K_α	{111}	4.79	18.56	2.991	7.719
		{511}+{333}	5.22	19.77	0.147	0.388
		{422}	4.97	19.07	0.392	0.102
	Mo- K_α	{880}	4.97	19.07	0.137	0.357
		{1111}+{775}	5.15	19.57	0.238	0.625
		{1042}	5.20	19.75	0.283	0.744

Table 3 continued. XEC and related constants for the evaluation of lattice strain.

material	radiation	interference $\{hkl\}$	$-s_1$	$\frac{1}{2}s_2$	$-\frac{1}{2}\cot\Theta_0$	$\frac{1}{2}\cot\Theta_0$
			10^{-6} MPa^{-1}	10^{-6} MPa^{-1}	s_1	$\frac{1}{2}s_2$
copper, Cu-basis-materials	Ti-K $_{\alpha}$	{111}	2.02	8.52	2.829	6.708
		{200}	3.75	13.71	1.139	3.114
	Fe-K $_{\alpha}$	{222}	2.02	8.52	0.995	2.360
	Co-K $_{\alpha}$	{400}	3.75	13.71	0.190	0.521
	Cu-K $_{\alpha}$	{420}	2.92	11.22	0.544	1.415
		{331}	2.33	9.44	0.855	2.110
	Mo-K $_{\alpha}$	{844}	2.45	9.82	0.584	1.457
nickel, Ni-basis-materials	Ti-K $_{\alpha}$	{200}	1.99	8.14	2.016	4.929
	Cr-K $_{\alpha}$	{220}	1.24	5.88	1.731	3.651
	Fe-K $_{\alpha}$	{222}	1.01	5.20	1.599	3.106
	Cu-K $_{\alpha}$	{420}	1.48	6.61	0.728	1.631
		{331}	1.17	5.68	1.362	2.805
	Mo-K $_{\alpha}$	{844}	1.24	5.88	0.681	1.435
titanium, Ti-basis-materials	Ti-K $_{\alpha}$	{110}	2.94	11.98	0.664	1.630
	Cr-K $_{\alpha}$	{201}	2.93	11.94	0.681	1.671
	Cr-K $_{\alpha}$	{004}	2.25	9.96	0.476	1.076
	Fe-K $_{\alpha}$	{203}	2.79	11.54	0.373	0.901
	Co-K $_{\alpha}$	{114}	2.64	11.11	0.429	1.019
		{213}	2.85	11.73	0.649	1.578
	Cu-K $_{\alpha}$	{006}	2.25	9.96	0.365	0.824
		{302}	2.91	11.90	0.486	1.188

2.071c Textured material

The XEC are substituted by the stressfactors F_{ij} . They are in addition dependent on the direction (ϕ, ψ). Experimentally they are evaluated by a uniaxial tension or bending test. Also the stress factors can be calculated using the monocrystal data and a model of crystallite coupling (Voigt, Reuss, Eshelby-Kröner) and the ODF as the weight function, chapter 2.13. For non-textured materials the F_{ij} correspond with combinations of XEC.

In the case of materials with strongly oriented crystallites there is no common relationship of the strain-, stress-free direction with the appropriate parameters. As Fig. 1 shows, there exist more than one point of the strain-stress-independent direction in the D-vs.- $\sin^2\psi$ diagram /28/. Kind and intensity of texture are the influences. More on that subject in chapter 2.16.

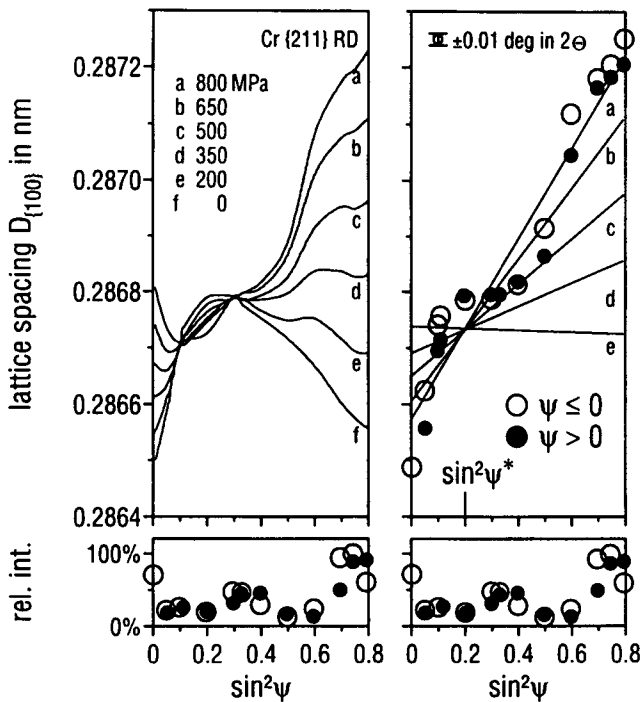


Figure 1. $D_{\{100\}}$ and relative intensity versus $\sin^2\psi$ of a textured steel strip, left: decreasing loads after regression analysis, right: points at maximum load and straight lines after linear-regression analysis $/2\theta/$.

2.072 Stress evaluation of mechanically isotropic materials

2.072a The principal D-vs.- $\sin^2\psi$ distributions

The numerous applications of RSA with X- and neutron-rays on mechanically isotropic and textured multiphase materials determining the LS- and RS-profiles led to different methods of stress evaluation. Also different measuring devices require different methods to determine the stress state. In the following, presently used methods of evaluation stresses from strain measurements are dealt with taking into account quasiisotropic and textured materials. The basic formulae $\epsilon_{\varphi,\psi}$ as function of ϵ_{ij} and ϵ_{ik} will be used. It is obvious that the ψ - or the φ -evaluations, the other coordinate constant or a (φ,ψ) -evaluation must tend toward the same result. The number of lattice-distance measurements have been increased with the years. XSA started with one film exposure and perpendicular ray incidence, followed by the two exposure technique. Since 1952 Hauk et al. made a lot of exposures and counter registrations to get the linear dependence D-vs.- $\sin^2\psi$ as accurately as possible. It serves also to identify oscillations by micro-RS. The procedure to evaluate stresses from linear ϵ -vs.- $\sin^2\psi$ dependences was given 1961 by Macherauch and Müller. Since 1964 Hauk et al. made measurements in both

directions $\psi \leq 0$ to ensure the correctness of alignment. Later (1975) it was found that ψ -splitting requires this procedure. The method to evaluate the strain/stress tensor was published by Dölle and Hauk. Details and references will follow. In the following, the evaluation of measured lattice-strain distributions to obtain stress components and/or the stress tensor will be dealt with. In the main cases the geometrical interpretation of the appropriate equations will be discussed showing the data that can be evaluated from the measured $D_{\phi,\psi}$ -values. Although the formulae are usually written in terms of ϵ_{ik} and σ_{ik} we prefer to use $D_{\phi,\psi}$ as the parameter in question, calculated from the measured $2\Theta_{\phi,\psi}$. The reason for that is the contemplation, especially if shown up as $D_{\phi,\psi}\{100\}$, the lattice constant of cubic materials. Another reason is the possibility to plot results gained from measurements with different wavelengths on different peaks. The discussion of results in ϵ_{ik} are only possible if the lattice-plane distance D_0 of the strain-stress-free state is accurately known.

There exist four basic D-vs.- $\sin^2\psi$ distributions /29/: linear dependence, ψ -splitting, curved lines and oscillations, Fig. 2. Combinations of these basic distributions are possible and are being observed. Fig. 3 shows more details in respect to mechanically isotropic and textured materials, eight D-vs.- $\sin^2\psi$ dependences /30/. As indicated in the figure there are two distributions with ψ -splitting, which means that measurements in the $-\psi$ and in the $+\psi$ range yield different curves. In all other cases the results in $\pm\psi$ directions fall within one straight or one curved line.

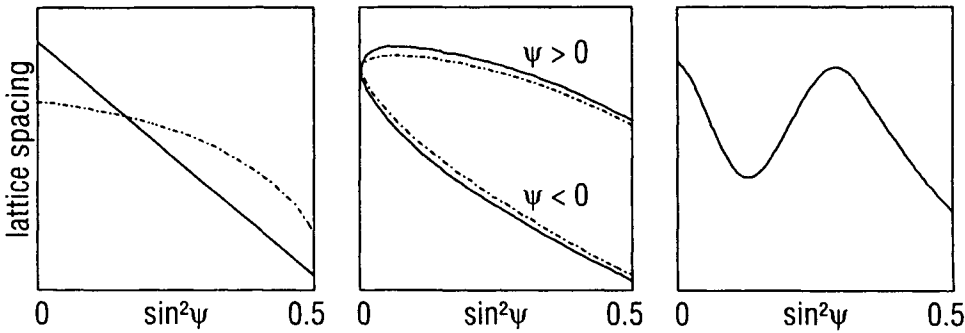


Figure 2. Major types of lattice-strain distributions /29/.

Modern diffractometer installations deliver plots with the following parameters, each versus $\sin^2\psi$: penetration depth, lattice spacing $D_{\{100\}}$, FWHM, intensity as well as the error bar of the calibration accuracy ± 0.01 degree in 2Θ . A linear regression analysis show up deviations of the measured lattice-strain distributions. Fig. 4 left demonstrates a linear; Fig. 4 right shows a D-vs.- $\sin^2\psi$ distribution with oscillations. The captions give the details.

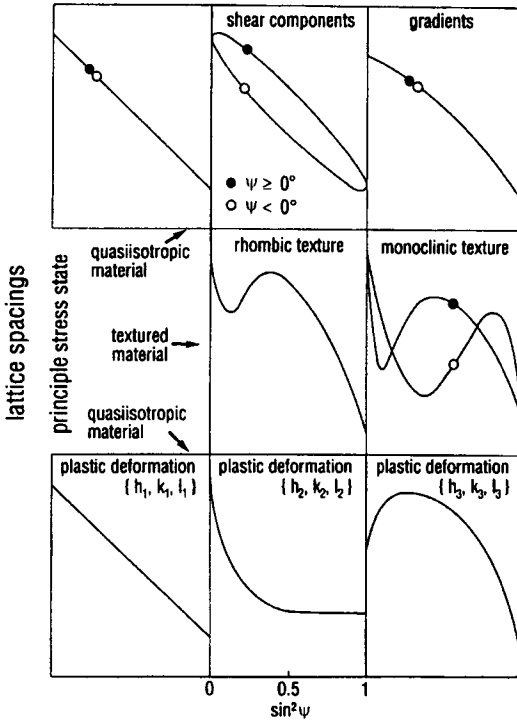


Figure 3. Different lattice-strain distributions as originating from certain phenomena /30/.

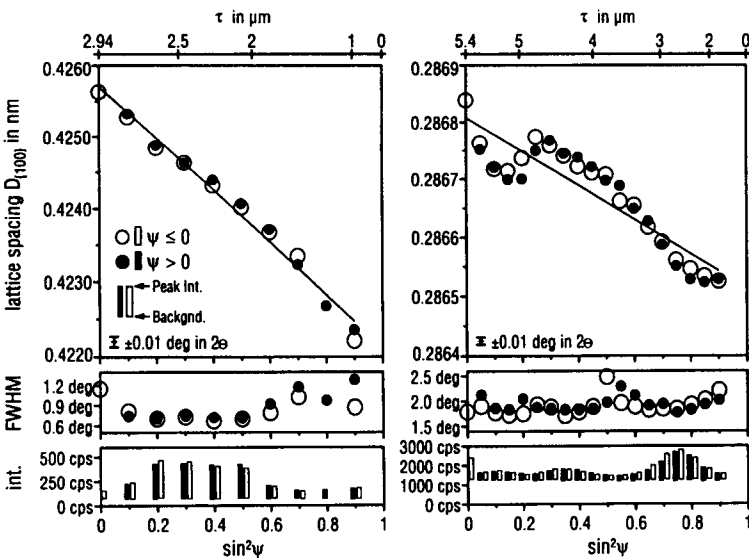


Figure 4. left: Linear D -vs.- $\sin^2\psi$ dependence, TiN-layer-steel composite, Cu- K_{α} radiation, $\{220\}$, fiber texture $\langle 111 \rangle$; right: D -vs.- $\sin^2\psi$ distribution with oscillations, rolled unalloyed steel, Cr- K_{α} radiation, $\{211\}$, rolling texture.

2.072b The linear D-vs.-sin²ψ dependence

Fig. 5 and 6 exhibit the graphical interpretation of the above explained formula Equ. 8, which here has been simplified for principal stresses /26,27 supplemented/:

$$\begin{aligned}\varepsilon_{\varphi,\psi} &= \frac{D_{\varphi,\psi} - D_0}{D_0} = \varepsilon_1 \cos^2 \varphi \sin^2 \psi + \varepsilon_2 \sin^2 \varphi \sin^2 \psi + \varepsilon_3 \cos^2 \psi \\ &= \frac{1}{2} s_2 (\sigma_1 \cos^2 \varphi \sin^2 \psi + \sigma_2 \sin^2 \varphi \sin^2 \psi + \sigma_3 \cos^2 \psi) + s_1 (\sigma_1 + \sigma_2 + \sigma_3)\end{aligned}\quad (8')$$

$\varphi = 0^\circ$ and 90°

The indicated distances and the zero intercepts serve to evaluate the strains and stresses, the lattice constant of the strain-stress-free state and the control of the experimental results. The D-vs.-sin²ψ linear dependences of different azimuths must meet in one point $D_{\varphi,\psi=0}$. In case D_0 is known the regression analysis allows one to determine the stress component σ_3 in thickness direction.

The most conspicuous advantage of the sin²ψ method is its lucidity. Deviations from the linear D-vs.-sin²ψ dependence can easily be detected. Furthermore, only one of the XEC, namely $\frac{1}{2} s_2$, is needed. The influence of an unknown σ_3 -component can be neglected in many practical cases, in particular for measurements to study the effect of a special parameter:

$$\Delta \sigma_\varphi = \frac{1}{\frac{1}{2} s_2} \frac{1}{D_0} \Delta \frac{\partial D_{\varphi,\psi}}{\partial \sin^2 \psi} \quad (21')$$

In this case an approximate value of D_0 is sufficient, also in all checked circumstances where σ_3 is zero or can be neglected. In many cases, especially in the neutron-ray technique the regression analysis D vs. sin²ψ is replaced by two strain measurements at $D_{\varphi=0,\psi=0}$ and $D_{\varphi,\psi=90}$ with the supposition these two points represent the linear dependence. This procedure may be erroneous since deviations of the linear dependence often occur at low and high ψ-values.

The film technique was the only method available from the beginning of the strain-stress determination up to the introduction of the different counters. But also today it remains useful to show up spotty images of single crystallites and their alterations by different influences as dynamic loads, recrystallisation, plastic deformations and others. A new variation is the image plate, mentioned in chapter 2.05.

Different cameras can be used, their shape may be cylindrical, conical or in most cases plane. The usefulness of the film registration is limited by the intensity ratio of the peak to the background which is often low when the peaks are broad. Nevertheless the evaluation method will be described in the following. Fig. 7 shows the principle of the measuring arrangement and the symbols used. An appropriate fine-grained calibration material is necessary to determine the distance from the irradiated point on the specimen to the film. The measurements on the film will be done by appropriate magnification of both the material (index ψ_+) and the calibration (c) $K_{\alpha 1}$ -rings.

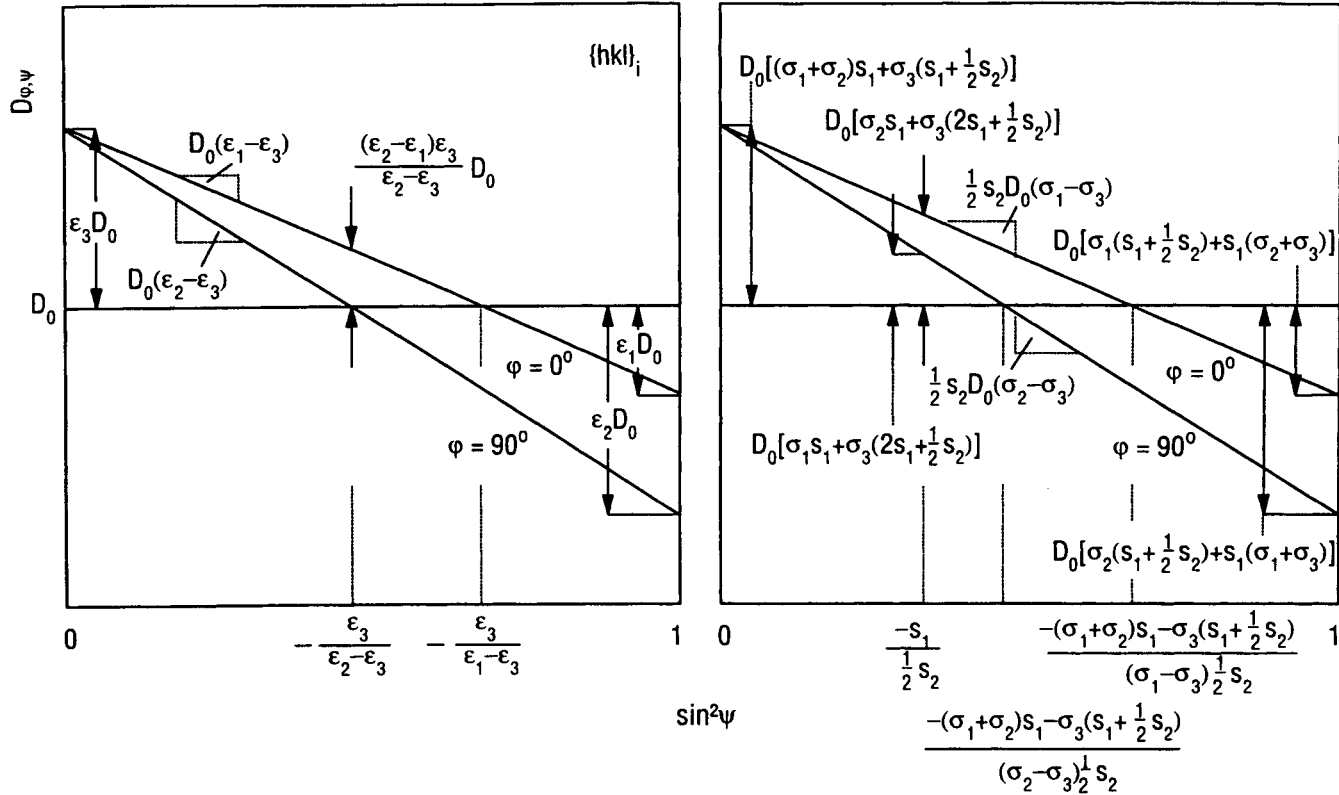


Figure 5 (left). $D_{\phi, \psi}$ -vs.- $\sin^2 \psi$ dependences for $\phi = 0^\circ$ and 90° of a triaxial strain state with the principal strains ϵ_1 and ϵ_2 parallel to the surface of the specimen. For arbitrary azimuth ϕ : $\epsilon_1 \rightarrow \epsilon_\phi$, $\epsilon_2 \rightarrow \epsilon_{\phi+90^\circ}$.

Figure 6 (right). $D_{\phi, \psi}$ -vs.- $\sin^2 \psi$ dependences for $\phi = 0^\circ$ and 90° of a triaxial stress state with the principal stresses σ_1 and σ_2 parallel to the surface of the specimen. For arbitrary azimuth ϕ : $\sigma_1 \rightarrow \sigma_\phi$, $\sigma_2 \rightarrow \sigma_{\phi+90^\circ}$.

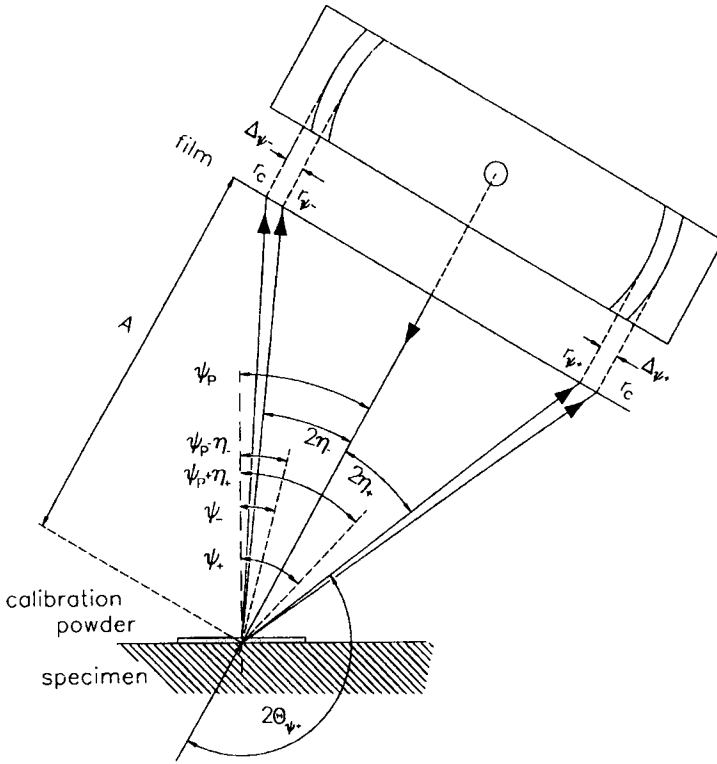


Figure 7. Principle of film-exposure method, diffraction patterns of material and calibration powder; $\eta = 90 - \Theta$, $\psi_{\pm} = \psi_p \pm \eta_{\pm}$.

$$r_c = A \cdot \tan 2\eta_c = -A \cdot \tan 2\Theta_c \quad (24)$$

$$r_{\psi} = A \cdot \tan 2\eta_{\psi} = -A \cdot \tan 2\Theta_{\psi}$$

$$\frac{\partial r_{\psi}}{\partial \Theta_{\psi}} = -\frac{2A}{\cos^2 2\Theta_{\psi}} \quad (25)$$

$$\begin{aligned} \varepsilon &= \varepsilon(\Theta_0) + \left. \frac{\partial \varepsilon}{\partial \Theta} \right|_{\Theta=\Theta_0} \Delta\Theta = 0 - \cot \Theta_0 \Delta\Theta = \cot \Theta_0 \frac{\cos^2 2\Theta_0}{2A} \Delta r \\ &= -\frac{\tan 2\Theta_c}{2r_c} \cot \Theta_0 \cos^2 2\Theta_0 \Delta r = \frac{\tan 2\Theta_c}{2r_c} \cot \Theta_0 \cos^2 2\Theta_0 (\Delta\psi - \Delta_0) \end{aligned} \quad (26)$$

The measured $\Delta\psi$ -values have to be normalized to a fixed $r_c = \text{const.}$ value, for example $r_c = 25 \text{ mm}$. To evaluate the stresses the foregoing formulae between strain and stresses have to be used. Measuring direction ($\varphi, \psi = 0^\circ$):

$$(\sigma_{11} + \sigma_{22})s_1 + \sigma_{33}\left(s_1 + \frac{1}{2}s_2\right) = \frac{\tan 2\Theta_c}{2r_c} \cot \Theta_0 \cos^2 2\Theta_0 (\Delta_{\varphi, \psi=0} - \Delta_0) \quad (27)$$

This formula was usually used for $\sigma_3 = 0$. Measuring direction ($\varphi = 0^\circ$ or 90° , ψ):

$$(\sigma_{11,22} - \sigma_{33}) = \frac{1}{\frac{1}{2}s_2} \frac{\tan 2\Theta_c}{2r_c} \cot \Theta_0 \cos^2 2\Theta_0 \cdot \frac{\partial \Delta_{\varphi=0,90,\psi}}{\partial \sin^2 \psi} \quad (28)$$

An example will illustrate the evaluation method:

Nonalloyed steel, RS-state $\sigma_{11} \neq 0$, Cr-K α , {211}, Cr powder {211},

$$D_{0,Fe} = \frac{0.28665}{\sqrt{6}} \text{ nm} ; D_{0,Cr} = \frac{0.28844}{\sqrt{6}} \text{ nm} ; s_1 = -1.25 \cdot 10^{-6} \text{ MPa}^{-1} ; \frac{1}{2}s_2 = 5.76 \cdot 10^{-6} \text{ MPa}^{-1}$$

The prime incidence angles should be $\psi_p = 12^\circ, 33^\circ, 39^\circ, 45^\circ$. The measuring results $D_{\varphi=0^\circ,\psi}$ are plotted in Fig. 8 and the regression straight line is drawn.

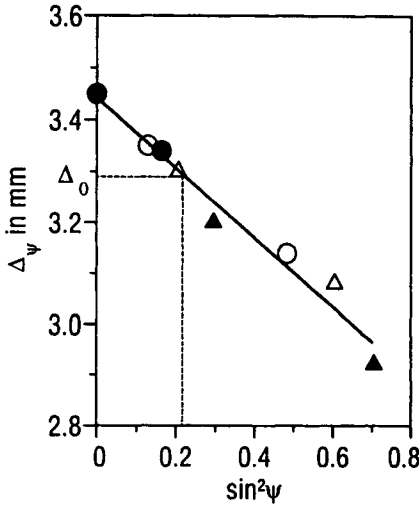


Figure 8. Plot for stress evaluation from several film measurements.

The result ($\varphi, \psi = 0^\circ$) for $\sigma_3 = 0$ is:

$$\begin{aligned} (\sigma_{11} + \sigma_{22}) &= \frac{1}{s_1} \frac{\tan 2\Theta_c}{2r_c} \cot \Theta_0 \cos^2 2\Theta_0 (\Delta_{\varphi,\psi=0} - \Delta_0) \\ &= 1448 \cdot (3.44 - 3.29) = 217 \text{ MPa} \end{aligned}$$

The result ($\varphi = 0^\circ, \psi$) for $\sigma_3 = 0$ is:

$$\sigma_{11} = -314 \cdot \frac{\partial \Delta_{\varphi=0,\psi}}{\partial \sin^2 \psi} = 314 \cdot (0.64 \pm 0.03) = 214 \pm 9 \text{ MPa}$$

The results are the same indicating that $\sigma_{22} = 0$. If the room or outdoor temperature is very different from normal temperature, corrections must be introduced, especially if the expansion coefficients of the material and the calibration powder are very different from each other. A simple formula using the difference of expansion coefficients does not hold, see /31/.

Two examples of older results demonstrate the numerous measurements and their scatter, Fig. 9 and 10 /32,33/.

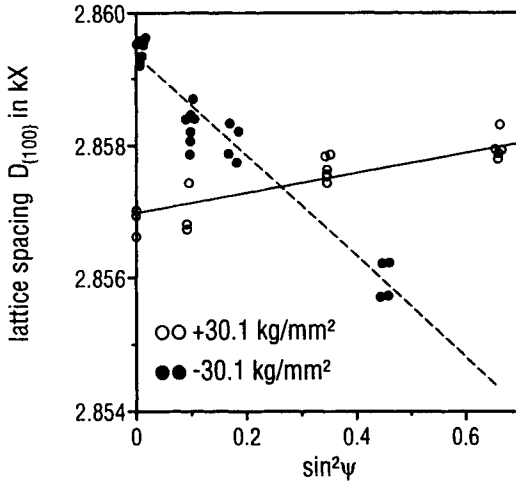


Figure 9. Bending stress on cast iron evaluated by the film back-reflection method, Co-K α , {310} /32/.

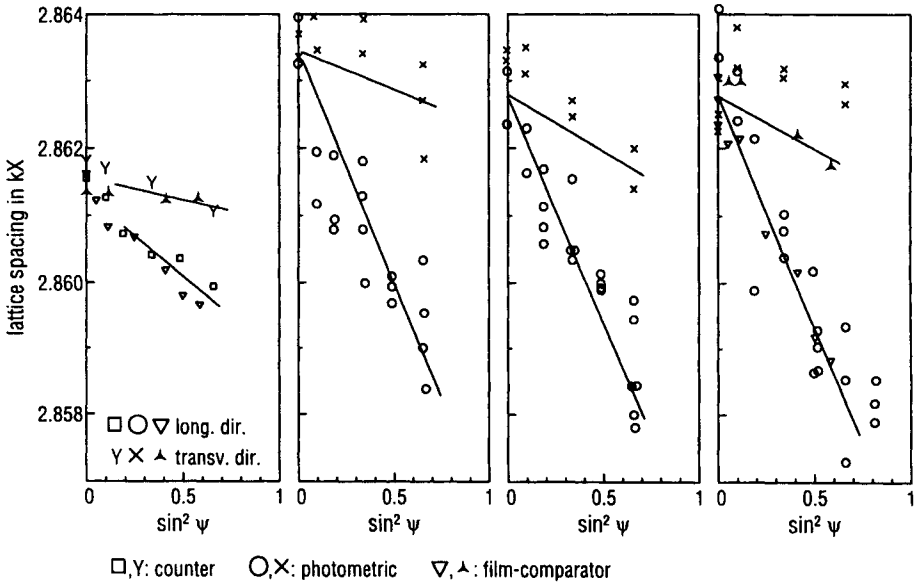


Figure 10. D-vs.- $\sin^2\psi$ plot of four plastically elongated steel samples, Co-K α radiation, {310} peak /33/.

To get an impression of the measuring volume and the scatter of the results using a diffractometer counter method the lattice strains of a TiAl6V4 sheet are plotted for the azimuths $\varphi = 0^\circ, 45^\circ, 90^\circ$, Fig. 11.

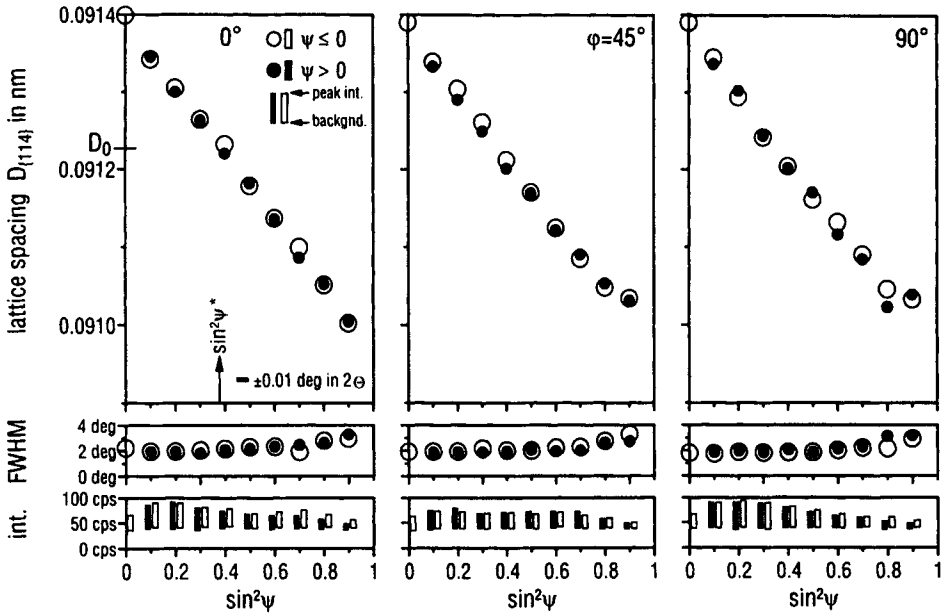


Figure 11. $D_{\{114\}}$ -vs.- $\sin^2\psi$ diagrams of a shot-peened TiAl6V4 sample, azimuth 0° , 45° , 90°

The stress evaluation was done using a linear-regression analysis up to $\sin^2\psi \leq 0.8$ using the equations in Fig. 5. The result is $\sigma_{11} = -570 \pm 12$ MPa, $\sigma_{22} = -580 \pm 14$ MPa, $\sigma_{33} = -150 \pm 11$ MPa using the strain-stress-free lattice distance $D_0\{114\} = 0.091227$ nm.

To get an impression of the effect of a σ_{33} -component, Fig. 12 illustrates the following example: steel, $D_0 = 0.28665$ nm, Cr- $K_{\alpha 1}$ radiation, $\{211\}$, $\sigma_{11} = -300$ MPa, $\sigma_{22} = 0$, $\sigma_{33} = 0 \pm 100$ MPa. The error of $D_{\phi,\psi}$ -measurement is $\pm 1 \cdot 10^{-5}$ nm. The influence of the σ_{33} can be determined in the low- $\sin^2\psi$ range. The σ_{33} -independent direction

$$\sin^2\psi'' = \frac{\left(s_1 + \frac{1}{2}s_2\right)}{\frac{1}{2}s_2} = 0.783 \overset{mech.}{\approx} \frac{1}{1+\nu}$$

is clearly to be noticed.

If the stress component in the thickness direction equals zero, this holds of course on the outermost surface, the following equation can be derived using Hooke's law:

$$\sigma_{33} = 0 = \frac{1}{\frac{1}{2}s_2} \left[\epsilon_{33} - \frac{s_1}{\frac{1}{2}s_2 + 3s_1} (\epsilon_{11} + \epsilon_{22} + \epsilon_{33}) \right]$$

$$\frac{\epsilon_{33}}{\epsilon_{11} + \epsilon_{22} + \epsilon_{33}} = \frac{s_1}{\frac{1}{2}s_2 + 3s_1} = \frac{-\nu\{hkl\}}{1 - 2\nu\{hkl\}}$$

$$\nu\{hkl\} = \frac{\epsilon_{33}}{\epsilon_{33} - \epsilon_{11} - \epsilon_{22}} \quad (29)$$

This needs very accurate lattice distance measurements, otherwise the error is very big. To use this value of ν together with an experimentally determined Young's modulus E at least may lead to big errors in the determined stress state.

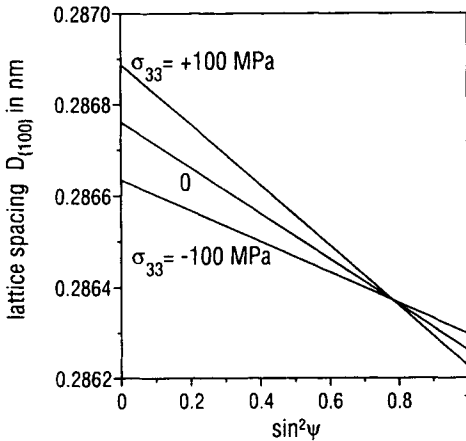


Figure 12. Influence of σ_{33} on the D -vs.- $\sin^2\psi$ dependences.

Early in the history of XSA the method to evaluate the biaxial stress state at the surface region ($\sigma_3 = 0$) from a single film exposure was introduced by /34/. The Debye ring was measured at four points: $(\varphi_p \pm \eta_{\pm}, \psi_p)$, $(\varphi_p, \psi_p \pm \eta_{\pm})$ with index p for primary incident. The method was not often used. But in recent times with the introduction of the imaging plate the method was further developed /35/. The Debye ring will now be measured at many points. The result is the stress component σ_{φ} . The method of evaluating the stress state from a single exposure cannot be very accurate since only the strains in the limit of $(\varphi_p \pm \eta, \psi_p \pm \eta)$, i.e. only the small range $\Delta \sin^2\psi \approx 0.4$ is used. Furthermore, texture and gradient effects will have a large influence on this determination of the stress state. Therefore linearity of D vs. $\sin^2\psi$ is an essential assumption. The calibration of the measuring data on the film will be performed with the help of an exact circle, for example produced by the interference of a metallic powder.

Fig. 13 shows the incident X-ray beam, the reflected Debye-Scherrer ring, the definition of α and the other parameters /35/. The direction (φ_p, ψ_p) of the incident beam is the L'_3 axis and the L'_2 axis lies in the surface plane of the specimen system. The transformation matrix from the specimen to the system L' , Equ. 14, is in this case

$$\begin{pmatrix} \cos\varphi_p \cos\psi_p & \sin\varphi_p \cos\psi_p & -\sin\psi_p \\ -\sin\varphi_p & \cos\varphi_p & 0 \\ \cos\varphi_p \sin\psi_p & \sin\varphi_p \sin\psi_p & \cos\psi_p \end{pmatrix} \quad (14')$$

$$\varepsilon_{\varphi,\psi} = \frac{D_{\varphi,\psi} - D_0}{D_0} = \frac{\cos \eta_0 - \cos \eta}{\cos \eta}$$

The index zero indicates again the strain/stress-free parameter. Now we introduce

$$\eta = \eta_0 + \delta(\alpha)$$

$$\sin \eta = \sin \eta_0 + \cos \eta_0 \cdot \delta \quad (32)$$

$$\cos \eta = \cos \eta_0 - \sin \eta_0 \cdot \delta$$

Inserting Equ. 32 in the above formulae we get:

$$\begin{aligned} \varepsilon_{\varphi,\psi} = \varepsilon(\alpha) &= \frac{\sin \eta_0 \cdot \delta}{\cos \eta_0 - \sin \eta_0 \cdot \delta} \quad , \quad \sin \eta_0 \cdot \delta \ll \cos \eta_0 \\ &= \tan \eta_0 \cdot \delta(\alpha) \\ &= \varepsilon_x (\cos \psi_p \sin \eta_0 \cos \alpha - \sin \psi_p \cos \eta_0)^2 + \varepsilon_y (\sin \eta_0 \sin \alpha)^2 \\ &\quad + \varepsilon_z (\sin \psi_p \sin \eta_0 \cos \alpha + \cos \psi_p \cos \eta_0)^2 \\ &\quad + (\varepsilon_1 - \varepsilon_2) \sin 2\varphi_p \sin \eta_0 \sin \alpha (\cos \psi_p \sin \eta_0 \cos \alpha - \sin \psi_p \cos \eta_0) \end{aligned} \quad (33)$$

As in the case of /34/ the $\delta(\alpha)$ will be measured at $\alpha = 0, \pi/2, \pi, 3\pi/2$, and therefore $\varepsilon_1, \varepsilon_2, \varepsilon_3$ and the $D_0 - \sigma_{33}$ relation may be determined with corresponding errors. Following the paper /35/ $\delta(\pm\alpha)$ and $\delta(\pm\alpha + \pi)$ will be measured and the following function used for further evaluation of σ_φ in the plane stress state:

$$\begin{aligned} \varepsilon_\alpha &= \frac{1}{2} [\varepsilon(\alpha) - \varepsilon(\alpha + \pi) + \varepsilon(-\alpha) - \varepsilon(-\alpha + \pi)] \\ &= -\frac{1}{2} s_2 \sigma_\varphi [\sin^2(\psi_p + \eta_0) - \sin^2(\psi_p - \eta_0)] \cos \alpha \end{aligned} \quad (34)$$

With l being the distance from the center of the ring, R the distance between film and specimen, and with the abbreviation

$$\Delta\alpha = \frac{1}{2} [l(\alpha) - l(\alpha + \pi) + l(-\alpha) - l(-\alpha + \pi)] \quad (35)$$

we get the final relationship /35/

$$\sigma_\varphi = \frac{1}{2} \frac{\cot \Theta}{s_2} \frac{\cos^2 2\Theta}{2R [\sin^2(\psi_p + \eta) - \sin^2(\psi_p - \eta)] \cos \alpha} \frac{\Delta\alpha}{\partial \cos \alpha} = \text{const.} \frac{\partial(\Delta\alpha)}{\partial \cos \alpha} \quad (36)$$

An example of measurements on a high-strength steel (HT80) under bending load with Cr-K $_{\alpha}$ radiation on the $\{211\}$ peak is shown in Fig. 14 /35/.

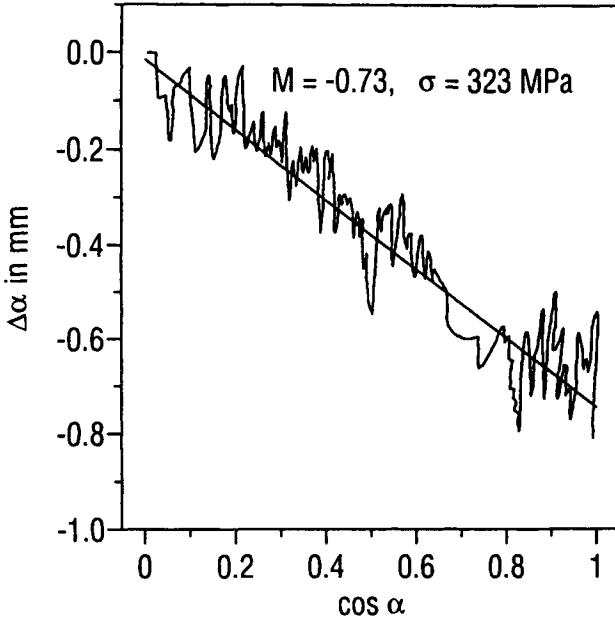


Figure 14. $\Delta\alpha - \cos\alpha$ diagram of the measurement on a high-strength steel (HT80) under bending load, M slope /35/.

2.072c The tensor evaluation, ψ -splitting, Dölle-Hauk method

It was shown by /24/ that the linear D -, $\varepsilon\text{-}\sin^2\psi$ relationship is a special case of the basic relation

$$\varepsilon_{\varphi,\psi} = \varepsilon_{ij} [(\varphi, \psi), \{hkl\}] m_i m_j.$$

This relation or Equ. 8 was written in the form

$$\varepsilon_{j,y} = \varepsilon'_{33} = \frac{D_{\varphi,\psi} - D_0}{D_0} = A + B \sin^2 \psi + C \sin \psi \cos \psi \quad (37)$$

$$A = \varepsilon_{33}$$

$$B = \varepsilon_{11} \cos^2 \varphi + \varepsilon_{22} \sin^2 \varphi - \varepsilon_{33} + (\varepsilon_{12} + \varepsilon_{21}) \sin \varphi \cos \varphi \quad (38)$$

$$C = (\varepsilon_{13} + \varepsilon_{31}) \cos \varphi + (\varepsilon_{23} + \varepsilon_{32}) \sin \varphi$$

The geometrical interpretation is shown in Fig. 15. In case that there are no shear components the usual $\sin^2\psi$ -law is obtained. The ψ -splitting and the determination of the complete strain tensor were thereby introduced in the XSA.

The practical evaluation of the stress tensor was brought forward and developed by /36/.

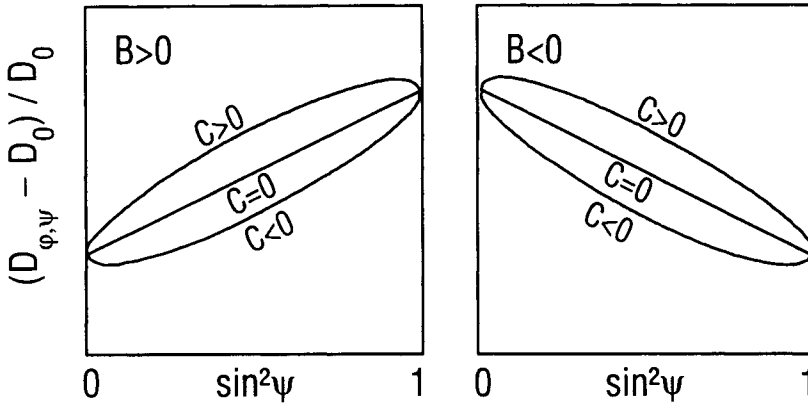


Figure 15. Possible lattice-strains versus $\sin^2\psi$ distributions with ψ -splitting /24/.

The $D_{\phi,\psi}$ -vs.- $\sin^2\psi$ dependences have to be measured in planes through the normal to the specimen and the three azimuths $\phi = 0^\circ, 45^\circ, 90^\circ$, sometimes in addition $\phi = -45^\circ$. The combinations of ϵ_{ij} and ϵ_{ik} can be determined from the D -vs.- $\sin^2\psi$ measurement, which can be seen in the diagrams of Fig. 16. The stresses σ_{ik} can be evaluated using Hooke's law, Equ. 9, 10 or Equ. 91, 92, 93 and 96. The accurate value of D_0 is necessary to evaluate σ_{33} . In this way the complete stress tensor is determined.

The advantage of this evaluation method is the fact that only linear regression analyses of the determined dependences D -vs.- $\sin^2\psi$ and ΔD -vs.- $\sin^2\psi$ are used. Furthermore, the evaluation of the averaged D -values for $\psi < 0^\circ$ and $\psi > 0^\circ$ come up to the usually used method of the normal-stress determination. The errors can be determined easily. Also here the experimental results can be easily checked, especially by exploiting the fact that all $D_{\phi,\psi}$ -vs.- $\sin^2\psi$ dependences have to meet at one point $D_{\phi,\psi=0}$.

The tilt $\Delta\psi$ of the principal stress components may be calculated. If ϵ_{33} and ϵ_{13} are non-zero the following equation holds:

$$\Delta\psi = \frac{1}{2} \arctan\left(-\frac{2\epsilon_{13}}{\epsilon_{11} - \epsilon_{33}}\right) = \frac{1}{2} \arctan\left(-\frac{2\sigma_{13}}{\sigma_{11} - \sigma_{33}}\right) \quad (39)$$

There exists a strain circle similar to the Mohr's stress circle with

$$\begin{aligned} \epsilon_1 &= \frac{1}{2}(\epsilon_{11} + \epsilon_{33}) - r \\ \epsilon_2 &= \epsilon_{22} \\ \epsilon_3 &= \frac{1}{2}(\epsilon_{11} + \epsilon_{33}) + r \\ r^2 &= \frac{1}{4}(\epsilon_{33} - \epsilon_{11})^2 + \epsilon_{13}^2 \end{aligned} \quad (40)$$

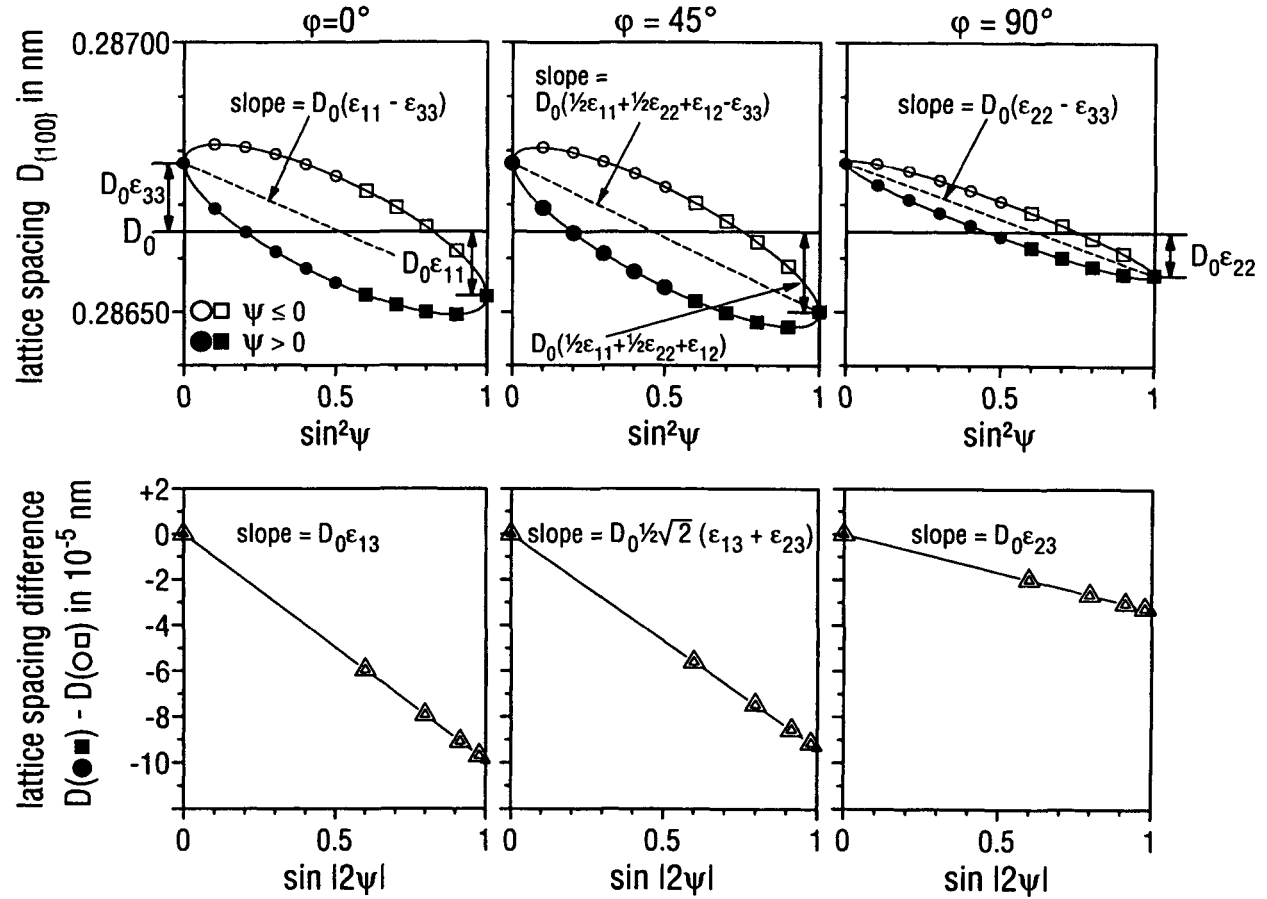


Figure 16. Principle of strain-tensor evaluation, Dölle-Hauk method, example ferritic steel /36/.

Since this method /36/ is generally used throughout the world, numerous experimental results of evaluating stress tensors are published. Here is one example, Fig. 17.

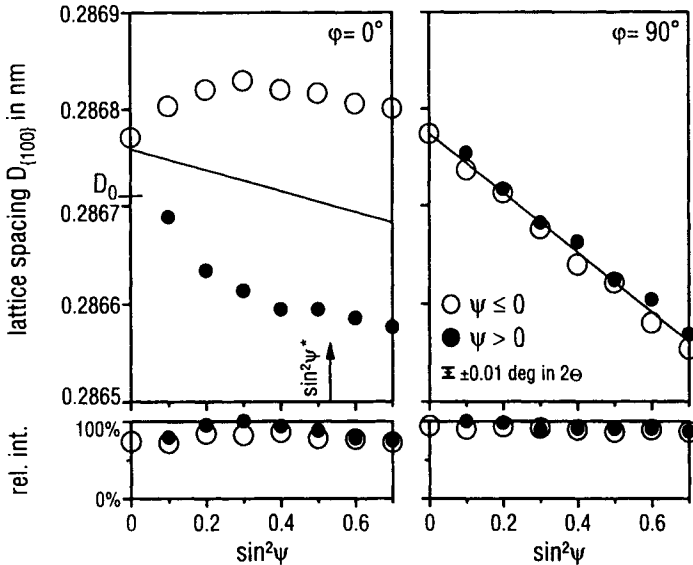


Figure 17. D-vs.- $\sin^2\psi$ distributions of a ground steel sample, Cr-K α , {211}.

The non-linear, elliptic D-vs.- $\sin^2\psi$ distribution for azimuth $\varphi = 0^\circ$ (Cr-K α , {211}), is given in Fig. 17. The C45 steel specimen was ground in azimuth $\varphi = 0^\circ$. The transverse direction shows linearity. The RS-evaluation with $D_0 = 0.28671 \text{ nm}$ and $s_1 = -1.25 \cdot 10^{-6} \text{ MPa}^{-1}$, $\frac{1}{2}s_2 = 5.76 \cdot 10^{-6} \text{ MPa}^{-1}$ results in

$$\begin{pmatrix} -123 & -71 \\ -242 & 4 \\ -71 & 4 & -58 \end{pmatrix} \pm \begin{pmatrix} 10 & 4 \\ 7 & 3 \\ 4 & 3 & 7 \end{pmatrix} \text{ MPa}^{-1}$$

The strain-free direction for $\varphi = 0^\circ$ will be $\sin^2\psi^* = 0.53$ as indicated in Fig. 17.

Another evaluation method was proposed by /36a/. The first application of the Dölle-Hauk method were published by /36b,36c/.

2.072d Other evaluations of the fundamental strain-stress equation

There are some constructions of diffractometer known where the main rotation is done by varying the azimuth φ but holding the pole angle ψ constant /37/. The main advantage of the ψ -methods is the fact that oscillations can be detected obviously. This is not the case here. The often mentioned feature of the measuring in the same depth is not reliable.

When the lattice-strain measurement will be done through variation of φ, ψ_1 the fundamental Equ. 11 applies:

$$\begin{aligned}\varepsilon_{\varphi, \psi_1} = & \frac{1}{2} s_2 \left[(\sigma_{11} \cos^2 \varphi + \sigma_{22} \sin^2 \varphi - \sigma_{33}) \sin^2 \psi_1 + \sigma_{33} \right] \\ & + \frac{1}{2} s_2 (\sigma_{12} \sin 2\varphi \sin^2 \psi_1 + \sigma_{13} \cos \varphi \sin 2\psi_1 + \sigma_{23} \sin \varphi \sin 2\psi_1) \\ & + s_1 (\sigma_{11} + \sigma_{22} + \sigma_{33})\end{aligned}$$

For two values, measured at azimuth 0° and 90° for example, it follows that

$$\varepsilon_{0, \psi_1} - \varepsilon_{90, \psi_1} = \frac{1}{2} s_2 (\sigma_{11} - \sigma_{22}) \sin^2 \psi_1 + \frac{1}{2} s_2 (\sigma_{13} - \sigma_{23}) \sin 2\psi_1 \quad (41)$$

Only the differences of stress components appearing in Equ. 41 can be determined. But only with measurements at two ψ_1 and ψ_2 the usual $(\sigma_{11} - \sigma_{33})$ term can be evaluated. The supposed feature of measuring in one penetration depth is gone. This is shown in the following

$$\varepsilon_{0, \psi_1} - \varepsilon_{0, \psi_2} = \frac{1}{2} s_2 (\sigma_{11} - \sigma_{33}) (\sin^2 \psi_1 - \sin^2 \psi_2) + \frac{1}{2} s_2 \sigma_{13} (\sin 2\psi_1 - \sin 2\psi_2) \quad (42)$$

Another evaluation method was developed and used by /38/ for thin polymeric plates in transmission technique where $D_{\varphi, \psi=90}\{100\}$ can directly be measured.

For $\sigma_{ij} = 0 \quad i \neq j$, the fundamental equation is

$$\varepsilon_{\varphi, \psi} = \frac{1}{2} s_2 \left[(\sigma_1 \cos^2 \varphi + \sigma_2 \sin^2 \varphi - \sigma_3) \sin^2 \psi + \sigma_3 \right] + s_1 (\sigma_1 + \sigma_2 + \sigma_3) \quad (43)$$

$$\frac{\partial \varepsilon_{\varphi, \psi}}{\partial \sin^2 \varphi} = \frac{1}{2} s_2 (\sigma_2 - \sigma_1) \sin^2 \psi$$

For $\psi = \text{const.}$ one gets the difference of the main stresses in the surface plane. Another way is to determine the XEC in the case of $\sigma_1 = \sigma_L, \sigma_2 = \sigma_3 = 0$ and $\psi = 90^\circ$

$$\frac{\partial \varepsilon_{\varphi, \psi=90}}{\partial \sigma_L} = \frac{1}{2} s_2 \{100\} \cos^2 \varphi + s_1 \{100\}$$

$$s_1 \{100\} = \frac{\partial \varepsilon_{\varphi=90, \psi=90}}{\partial \sigma_L} \quad ; \quad \frac{1}{2} s_2 \{100\} = \frac{\partial^2 \varepsilon_{\varphi, \psi=90}}{\partial \cos^2 \varphi \partial \sigma_L}$$

2.072e A method using at least six different $D_{\varphi, \psi}$ -measurements

W. Reimers

Basically, the strain and stress tensor can be calculated using a data set of at least six non-coplanar $D_{\varphi, \psi}$ -spacings according to the basic formula. The following suppositions must be fulfilled: linear D-vs.- $\sin^2 \psi$ dependences, that means no oscillations, no orientation-dependent micro-RS and no gradients with the depth from the surface. The method was introduced by /39/. The authors claim to get results of less scatter by choosing optimal measuring directions (φ, ψ) .

The basic Equ. 11 may be written as follows:

$$\begin{aligned}
 D_{\varphi, \psi} = & \sigma_{11} \left(\frac{1}{2} s_2 \sin^2 \psi \cos^2 \varphi + s_1 \right) D_0 + \sigma_{22} \left(\frac{1}{2} s_2 \sin^2 \psi \sin^2 \varphi + s_1 \right) D_0 \\
 & + \sigma_{33} \left(s_1 + \frac{1}{2} s_2 - \frac{1}{2} s_2 \sin^2 \psi \right) D_0 + \sigma_{12} \left(\frac{1}{2} s_2 \sin^2 \psi \sin 2\varphi \right) D_0 \\
 & + \sigma_{13} \left(\frac{1}{2} s_2 \sin^2 \psi \cos \varphi \right) D_0 + \sigma_{23} \left(\frac{1}{2} s_2 \sin 2\psi \sin \varphi \right) D_0 + D_0
 \end{aligned} \quad (11')$$

Equ. 11' can be used to calculate the stress tensor components σ_{ij} using a least-squares procedure as follows. The interplanar lattice spacing D is written as $D = D(\sigma_{11}, \sigma_{22}, \sigma_{33}, D_0, \varphi, \psi)$, where D_0 is supposed to be kept fixed in the calculation algorithm. Furthermore, a set of n measured data points $D_{\varphi, \psi}$ with experimental errors Δ_i is given. The stress tensor can be evaluated by minimizing the following function:

$$\chi^2 \equiv \sum_{i=1}^n \frac{(D_{\varphi, \psi_i} - D_i)^2}{\Delta_i^2} = \min. \quad (44)$$

For finding the minimum of χ^2 the partial derivations have to vanish. Thus, the corresponding condition is:

$$\frac{\partial \chi^2}{\partial \sigma_{ij}} = 0 \quad i, j = 1, 2, 3, \quad i \leq j \quad (45)$$

To make the following calculations more transparent, Equ. 11' can be written as:

$$D_i = a_i \sigma_{11} + b_i \sigma_{22} + c_i \sigma_{33} + d_i \sigma_{12} + e_i \sigma_{13} + f_i \sigma_{23} + D_0 \quad (46)$$

$a_i, \dots, f_i$ describe the individual orientations $(\varphi, \psi)_i$ of the specimen during the measurement of D_{φ, ψ_i} . Equ. 45 can be explicitly written:

$$\begin{aligned}
 \frac{\partial \chi^2}{\partial \sigma_{11}} & \equiv \sum_{i=1}^n \frac{(D_{\varphi, \psi_i} - D_i)}{\Delta_i^2} \cdot a_i = 0 \\
 & \vdots \\
 \frac{\partial \chi^2}{\partial \sigma_{23}} & \equiv \sum_{i=1}^n \frac{(D_{\varphi, \psi_i} - D_i)}{\Delta_i^2} \cdot f_i = 0
 \end{aligned} \quad (47)$$

Expanding Equ. 47 leads to a set of simultaneous equations, the "normal equations", which can be written as

$$\underline{K} \cdot \vec{p} = \vec{l} \quad (48)$$

$\bar{p}$ is the unknown vector $(\sigma_{11}, \sigma_{22}, \sigma_{33}, \sigma_{12}, \sigma_{13}, \sigma_{23})^T$ and $\bar{l}$ the vector

$$\left(\sum_{i=1}^n \frac{a_i D_{\varphi, \psi_i}}{\Delta_i^2}, \dots, \sum_{i=1}^n \frac{f_i D_{\varphi, \psi_i}}{\Delta_i^2} \right)^T.$$

The matrix $\underline{K}$ of known coefficients contains all permutations of

$$\sum_{i=1}^n \frac{x_i \cdot y_i}{\Delta_i^2}; \quad x_i, y_i = a_i \dots f_i$$

in alphabetical order. The solution of Equ. 48 yields the full stress tensor σ_{ij} , $i, j = 1, 2, 3$. Generally, the present formalism allows the calculation of three-dimensional stress states. Absolute values for particular components of the stress tensor can be given, if the stress-free lattice parameter D_0 is known precisely. In the case of X-ray diffraction, one can often assume plane-stress conditions because of the negligible penetration depth. Here, the procedure at hand opens the possibility for the calculation of the stress-free lattice parameter D_0 (near the surface). Equ. 11' shows the dependence of $D_{\varphi, \psi}$ on σ_{ij} , φ , ψ and D_0 . On the other hand, σ_{33} can be understood as a function of D_0 : $\sigma_{33} = F(D_0)$. This function contains *the whole* least-squares algorithm, and the direction of σ_{33} is perpendicular to the surface of the specimen. At the surface $\sigma_{33} = 0$ must be fulfilled. If D_0 is not known precisely, the procedure $\sigma_{33} = F(D_0)$ yields a value of σ_{33} not equal to zero. Using a simple numerical method like the "regula falsi" for the determination of the roots of F , the exact value for D_0 , which leads to a vanishing σ_{33} , can be found. The regula falsi method needs two initial or starting values $D_0^{(i-2)}$ and $D_0^{(i-1)}$ with $F(D_0^{(i-2)}) \cdot F(D_0^{(i-1)}) < 0$. Now, the new value of $D_0^{(i)}$ can be calculated by applying the following iteration rule:

$$D_0^{(i)} = D_0^{(i-2)} - F\left(D_0^{(i-2)}\right) \cdot \frac{D_0^{(i-1)} - D_0^{(i-2)}}{F\left(D_0^{(i-1)}\right) - F\left(D_0^{(i-2)}\right)} \quad (49)$$

After a few iterations, the condition $\sigma_{33} = 0$ is fulfilled, and the corresponding value for D_0 is found. Then, the resulting stress tensor takes the following form:

$$\sigma_{ij} = \begin{pmatrix} \sigma_{11} & \sigma_{12} & \approx 0 \\ \sigma_{12} & \sigma_{22} & \approx 0 \\ \approx 0 & \approx 0 & 0 \end{pmatrix} \quad (50)$$

If the off-diagonal elements σ_{i3} are not equal to zero within their error bars, the sample possibly exhibits stress gradients or gradients of D_0 in the near-surface region. In this case the procedure does not work properly.

2.072f Accuracy of stress evaluation, the errors

The following considerations and formulae have been summarized and supplemented by B. Krüger /40/ using books of mathematics and statistics /41,42,43/, of physics and chemistry /44,45/ and those reflections of errors that are related to XSA /26,46,47,48,49/. Emphasis is laid on practical use. Therefore general considerations and the appropriate equations for the evaluations of XSA are explained by practical examples.

Kinds of errors

Repetition of measurements under identical conditions results in an average value with a variance, also called an error. Results of repeated measurements will be in the confidence range with a defined probability. Several parameters influence the value of the error, therefore the chosen parameters and the method of calculation should be named. Besides the statistical errors there exist the systematic errors. Fig. 18 demonstrates the possible cases of a series of identical measurements namely with small and large variance, with and without systematic error.

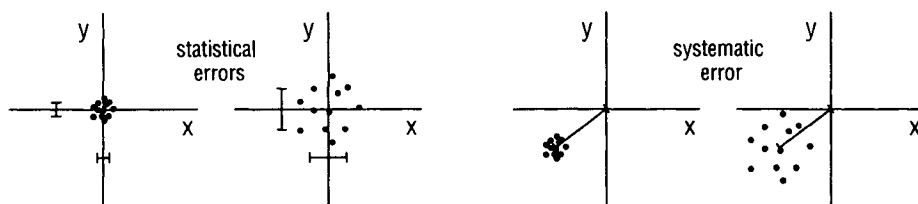


Figure 18. Principal results of a series of measurements with small and large statistical errors without (left) and with (right) systematic error /41/.

The following explanation differentiates between the effect and the determination of the statistical and of the systematic error.

The **statistical** error depends as well on the counting statistics as on the procedure to evaluate the position of an interference line, for example using the parabola method /47,48/. A repetition of a measurement will give in general a variation of the previous result. Supposing that systematic errors can be neglected, the statistical errors are demonstrated in Fig. 19 (left). It is obvious that considering those statistical errors, also the slope of the D -vs.- $\sin^2\psi$ line is connected with a statistical error. An increase of the counting rate (a higher precision of the position determination of each interference line) will cause smaller errors.

A further kind of statistical error, but in a local manner, is given because of the heterogeneity of a multicrystalline material within the irradiated volume. The reason for this may be the grain microstructure, local variations of chemical composition or different grades of mechanical treatment /50/. Only a series of identical measurements with local variations can remove these influences.

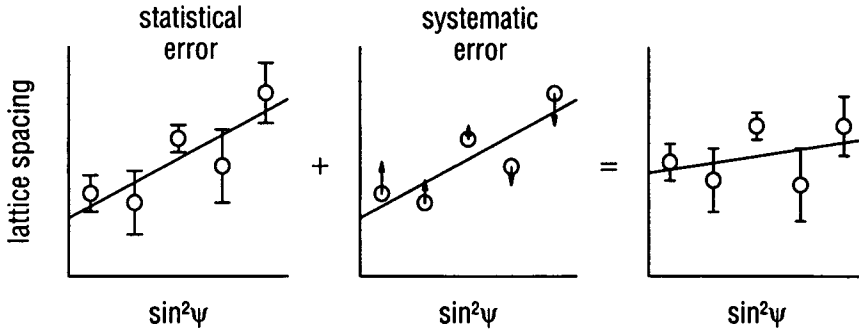


Figure 19. Effects on the regression line caused by the two kinds of errors and their superposition.

The **systematic** errors, which superimpose linearly, belong to different parameters of the measuring and evaluation procedure. The accuracy of the alignment of the measuring device and the measuring of the lattice parameter D_0 influences the D -vs.- $\sin^2\psi$ line, Fig. 19, also done by local alterations of the material properties, which cause a systematic error in a single measurement. Additional errors are caused by the evaluation data and the evaluation procedure when calculating the stress components and finally by measuring on different samples of the same material. Neglecting the statistical error, a repetition of a measurement will give always the same result.

The systematic errors of a single measurement cannot be determined. So it is necessary to cross-check results, especially strange ones, before making conclusions about possible reasons and effects. Repeated precise alignment of the measuring device is appropriate to avoid experimental errors.

In the following, some well-known formulae will be introduced that are necessary for further explanations.

$$\sum_{i=1}^N y_i = \sum_{i=1}^N y_i \quad (N = \text{number of measuring values } y_i)$$

The variance var , the square of the standard deviation, is a measure of the variations of the single values /41,42,43,44,45/.

$$\text{var}(y) = \frac{\sum (y_i - \bar{y})^2}{N-1} = \frac{\sum y_i^2 - \frac{1}{N}(\sum y_i)^2}{N-1} \quad (51)$$

$$\bar{y} = \frac{1}{N} \sum y_i \quad \text{arithmetic average of all measuring values } y_i$$

The statistical error err also known as standard deviation is defined :

$$\text{err}(y) = \sqrt{\text{var}(y)} \quad (52)$$

The average squared error of the average value called standard error is defined /44,45/:

$$\text{var}(\bar{y}) = \frac{\text{var}(y)}{N} \quad (53)$$

The parameter covariance cov (sum of the differential products) will be used for simplification and calculation of the regression lines and their errors /41,42,43,44,45/.

$$\text{cov}(x, y) = \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{N - 1} = \frac{\sum x_i y_i - \frac{1}{N} \sum x_i \sum y_i}{N - 1} \quad (54)$$

Equ. 51 and Equ. 53 hold for weighted measuring data as well /44,45/:

$$\text{var}(y) = \frac{\sum w_i (y_i - \bar{y})^2}{\left(1 - \frac{1}{N}\right) \sum w_i} \quad \bar{y} = \frac{\sum w_i y_i}{\sum w_i} \quad w_i = \text{weighting factors} \quad (55)$$

The naming of errors makes sense only if the number of measured values exceeds the number of parameters.

Linear regression

The consideration of statistical errors is twofold: One method describes the direct relation between the measuring data and the regression result, Fig. 20 (left); the other takes the error propagation into account, Fig. 20 (center).

The regression on the data points will be done by the minimization of the sum of squares of the errors, which are the differences between the y -values of the measured data and the linear regression resulting from the same x -values.

$$Y(X) = a_0 + a_1 X \quad \text{or} \quad Y(X) = \bar{y} + a_1 (X - \bar{x}) \quad (56)$$

The linear regression coefficients a_0 and a_1 result in the values /41,42,43,44,45/

$$a_1 = \frac{\text{cov}(x, y)}{\text{var}(x)} = \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{\sum (x_i - \bar{x})^2} = \frac{N \sum x_i y_i - \sum x_i \sum y_i}{N \sum x_i^2 - \left(\sum x_i\right)^2} \quad (57)$$

$$a_0 = \frac{\sum y_i \sum x_i^2 - \sum x_i \sum x_i y_i}{N \sum x_i^2 - \left(\sum x_i\right)^2} \quad \text{and} \quad \bar{y} = \bar{y}(\bar{x}) \quad (58)$$

The variance of all measured values y_i relating to the linear regression /41,42/

$$\text{var}(y, x) = \frac{\sum (y_i - Y)^2}{N - 2} = \frac{\sum y_i^2 - a_0 \sum y_i - a_1 \sum x_i y_i}{N - 2} \quad (59)$$

says, that in case of $N = \infty$ measurements approximately 68% of the measuring values are within a range of width $\pm \sqrt{\text{var}(y, x)}$ around the regression linear dependence.

The variance of the regression values $Y(X)$ describes a nonlinear confidence range with a minimal width at the center of gravity of the measured data /44/.

$$\text{var}(Y) = \text{var}(a_1) \cdot \left(\frac{N - 1}{N} \text{var}(x) + (X - \bar{x})^2 \right) \quad (60)$$

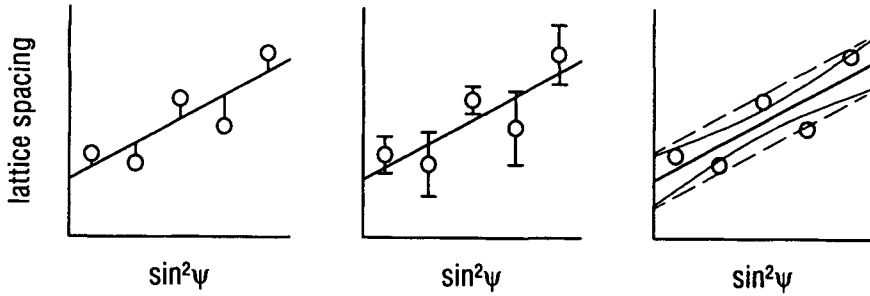


Figure 20. Calculation of the statistical errors of the regression line and confidence ranges. left: direct relation between measuring data and regression result; center: error propagation taking into account the known variances of the measuring data; right: confidence ranges $\text{var}(y,x)$, dashed lines, and $\text{var}(Y)$, continuous lines.

The resulting variances of a_1 and a_0 are /44,45/:

$$\text{var}(a_1) = \frac{N}{N-2} \frac{\sum (y_i - Y)^2}{N \sum x_i^2 - (\sum x_i)^2} = \frac{1}{N-2} \left(\frac{N \sum y_i^2 - (\sum y_i)^2}{N \sum x_i^2 - (\sum x_i)^2} - a_1^2 \right) \quad (61)$$

$$\text{and } \text{var}(a_0) = \text{var}(a_1) \cdot \frac{\sum x_i^2}{N} \quad (62)$$

The correlation coefficient r is often used to express the quality of a regression analysis (0 means no correlation, 1 means perfect correlation) /42,44/.

$$r^2(x,y) = \frac{\text{cov}^2(x,y)}{\text{var}(x) \cdot \text{var}(y)} \cong 1 - \frac{\text{var}(y,x)}{\text{var}(y)} \quad (63)$$

If the result depends on several parameters $y(a,b,c,\dots)$ the total variance can be calculated from the single variances by error propagation /41,44,45,47/.

$$\begin{aligned} \text{var}(y) = & \left(\frac{\partial y}{\partial a} \right)^2 \text{var}(a) + \left(\frac{\partial y}{\partial b} \right)^2 \text{var}(b) + \left(\frac{\partial y}{\partial c} \right)^2 \text{var}(c) + \dots \\ & + 2 \left(\frac{\partial^2 y}{\partial a \partial b} \text{cov}(a,b) + \frac{\partial^2 y}{\partial a \partial c} \text{cov}(a,c) + \frac{\partial^2 y}{\partial b \partial c} \text{cov}(b,c) \right) + \text{further differential terms} \end{aligned} \quad (64)$$

The differential terms yield zero, if the parameters are independent from each other. For comparative reflections the relative variance is advantageous:

$$\text{var}(y)_{\text{rel}} = \frac{\text{var}(y)}{\bar{y}^2} \quad (65)$$

The variances of the slope a_1 , of the intercept a_0 and any y-value of the regression straight line Y using the error propagation theory are

$$\begin{aligned} \text{var}(a_1) = & \frac{\sum (x_i - \bar{x})^2 \text{var}(y_i)}{\left(\sum (x_i - \bar{x})^2\right)^2} + \frac{\sum (y_i - \bar{y})^2 \text{var}(x_i)}{\left(\sum (x_i - \bar{x})^2\right)^2} + 4 a_1 \frac{\sum (x_i - \bar{x})^2 \text{var}(x_i)}{\left(\sum (x_i - \bar{x})^2\right)^2} \\ & - 4 a_1 \sum (x_i - \bar{x})^2 \frac{\sum (x_i - \bar{x})(y_i - \bar{y}) \text{var}(x_i)}{\left(\sum (x_i - \bar{x})^2\right)^3} \end{aligned} \quad (66)$$

$$\begin{aligned} \text{var}(a_0) = & \frac{\sum \text{var}(y_i) + a_1^2 \sum \text{var}(x_i)}{N^2} + \bar{x}^2 \text{var}(a_1) - 2\bar{x} \frac{\sum (x_i - \bar{x}) \text{var}(y_i)}{N \sum (x_i - \bar{x})^2} \\ & - 4\bar{x} a_1 \sum (x_i - \bar{x})(y_i - \bar{y}) \frac{\sum (x_i - \bar{x}) \text{var}(x_i)}{N \left(\sum (x_i - \bar{x})^2\right)^2} \\ & + 2\bar{x} a_1 \sum (x_i - \bar{x})^2 \frac{\sum (y_i - \bar{y}) \text{var}(x_i)}{N \left(\sum (x_i - \bar{x})^2\right)^2} \end{aligned} \quad (67)$$

$$\text{var}(Y) = \text{var}(a_0) + \text{var}(a_1) \cdot X^2 \quad (68)$$

If the errors of all data of a measuring series are the same, then the two last terms of Equ. 66 and the very last one of Equ. 67 are zero.

Determination of the position of the interference with the center-of-gravity method

This method will be discussed in detail because the simple calculation algorithm is fit for automatic measurement data evaluation. The intensity distribution should be registered in N equidistant steps $\Delta 2\Theta$

$$I_i(2\Theta_i) = I_i(2\Theta_1 + (i-1)\Delta 2\Theta). \quad (69)$$

The center of gravity $2\Theta_{\text{gravity}}$ of this intensity distribution is /46/

$$2\Theta_{\text{gravity}} = 2\Theta_1 + \frac{\Delta 2\Theta \sum (i-1)I_i}{\sum I_i} \quad (70)$$

By the use of the error propagation theory and neglecting the errors in $\Delta 2\Theta$ one gets

$$\text{var}(2\Theta_{\text{gravity}}) = \frac{\sum (2\Theta_i - 2\Theta_{\text{gravity}})^2 \text{var}(I_i)}{(\sum I_i)^2} \quad (71)$$

Supposing the Gaussian distribution it follows /41,42,43,44/

$$\text{var}(I_i) = I(2\Theta_i) \quad (72)$$

All procedures done before the determination of the center of gravity must be taken into account. There are to be mentioned the background and the PLA-corrections as well as smoothing procedures.

$$\text{var}(I_i) = \text{PLA}(2\Theta_i) \cdot I(2\Theta_i) + I_{\text{background}}(2\Theta_i) \quad (73)$$

Furthermore the following variances hold if the symmetrization or K_α -separation of the lines take into account the doublet distances $\Delta_{K\alpha}$ respectively $\Delta_{i,K\alpha}$:

$$\begin{aligned} \text{var}(I_{\text{sym}}(2\Theta_i)) &= \text{var}(I_i(2\Theta_i)) + 0.25 \text{var}(I_i(2\Theta_i + \Delta_{K\alpha})) \\ \text{with } \Delta_{K\alpha} &= 2\Theta_{K\alpha 2} - 2\Theta_{K\alpha 1} \text{ and } 2\Theta_{K\alpha} = 2 \arcsin\left(\frac{\lambda_{K\alpha}}{2D}\right) \end{aligned} \quad (74)$$

$$\begin{aligned} \text{var}(I_{\text{sep}}(2\Theta_i)) &= \text{var}(I_i(2\Theta_i)) + 0.25 \text{var}(I_i(2\Theta_i - \Delta_{i,K\alpha})) \\ \text{with } \Delta_{i,K\alpha} &= 2\Theta_i - 2 \arcsin\left(\frac{\lambda_{K\alpha 1}}{\lambda_{K\alpha 2}} \sin \Theta_i\right) \end{aligned}$$

An example demonstrates the method and shows the different influences, Fig. 21. After background- and PLA-correction the K_α -doublet was symmetrized numerically as well as separated. The centers of gravity were determined by varying the threshold. The symmetrized interference line was evaluated in 5% steps in the range of thresholds between 55% to 80% of the maximum intensity, the K_α -separated one in 10% steps in the range of 30% to 80% of the maximum intensity. Both averaged results are $2\Theta_{\text{sym}} = 114.817^\circ \pm 0.010^\circ$ and $2\Theta_{\text{sep}} = 114.806^\circ \pm 0.007^\circ$, a very good agreement. The errors implied by the counting statistics of the single centers of gravity are of the symmetrisation 0.0007° (80%) up to 0.0009° (55%) and of the K_α -separation 0.0009° (80%) up to 0.0018° (30%). They are one order of magnitude smaller than the standard deviation of the 2Θ -average values, calculated from all intensity values. Using a larger scatter of the intensities, that means more than 68% of all intensity values are within the scatter range, the error of the peak position will increase according to Equ. 71.

To demonstrate the influence of the intensity, of the peak-to-background ratio and of the 2Θ step width, the intensities of the measured interference line have been altered by calculation (Fig. 21) and the errors of the centers of gravity have been calculated (K_α -separation), Table 4.

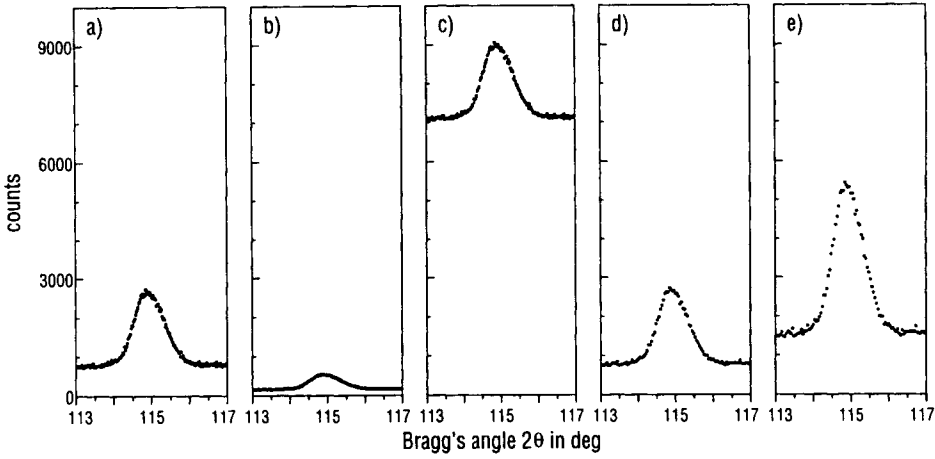


Figure 21. a) {114}-interference, TiAl6V4, Cu- K_{α} radiation, $\psi = 0^{\circ}$; b – e) numerically altered to show up the following influences: b) intensity, c) peak-to-background ratio, d) 2θ step width, e) intensity and 2θ step width.

Table 4. Error in 2θ of the center-of-gravity evaluation, K_{α} separation, thresholds 0.3 to 0.8 times the maximum intensity, 10% step, different measurement conditions.

peak	$I_{\max}$ [cts]	$I_{\text{background}}$ [cts]	$\frac{I_{\max.}}{I_{\text{background}}}$	stepwidth 2θ [deg]	error of 2θ [deg]
a	2725	704	3.87	0.02	0.004
b	545	140.8	3.87	0.02	0.009
c	9061	7040	1.29	0.02	0.009
d	2725	704	3.87	0.04	0.006
e	5450	1408	3.87	0.04	0.004

D-versus- $\sin^2\psi$ method

The D-vs.- $\sin^2\psi$ dependence is a straight line if the normal stress component in the irradiated volume of a nontextured material is constant. The formulae are repeated here /26/:

$$\sigma_{\varphi} = \frac{1}{D_0 \frac{1}{2} s_2} \frac{\partial D_{\varphi, \psi}}{\partial (\sin^2 \psi)} = \frac{m_{\varphi}}{D_0 \frac{1}{2} s_2} \quad (75)$$

$$D_{\varphi, \psi=0} = D_0 \left[1 + s_1 (\sigma_{11} + \sigma_{22} + \sigma_{33}) + \frac{1}{2} s_2 \sigma_{33} \right] \quad (76)$$

According to the Equ. 57 to 63 and supposing that N measurements were made at different ψ -angles the two coefficients of the regression straight line and their variances are given by

$$D_{\varphi,\psi} = D_{\varphi,\psi=0} + m_{\varphi} \sin^2 \psi \quad (77)$$

$$\begin{aligned} m_{\varphi} &= \frac{\sum (\sin^2 \psi_i - \overline{\sin^2 \psi}) (D_{\varphi,\psi_i} - \overline{D_{\varphi,\psi}})}{\sum (\sin^2 \psi_i - \overline{\sin^2 \psi})^2} \\ &= \frac{N \sum (\sin^2 \psi_i D_{\varphi,\psi_i}) - \sum \sin^2 \psi_i \sum D_{\varphi,\psi_i}}{N \sum (\sin^2 \psi_i)^2 - (\sum \sin^2 \psi_i)^2} \end{aligned} \quad (78)$$

$$D_{\varphi,\psi=0} = \frac{\sum D_{\varphi,\psi_i} \sum (\sin^2 \psi_i)^2 - \sum \sin^2 \psi_i \sum (\sin^2 \psi_i D_{\varphi,\psi_i})}{N \sum (\sin^2 \psi_i)^2 - (\sum \sin^2 \psi_i)^2} \quad (79)$$

$$\text{var}(m_{\varphi}) = \frac{1}{N-2} \left(\frac{N \sum D_{\varphi,\psi_i}^2 - (\sum D_{\varphi,\psi_i})^2}{N \sum (\sin^2 \psi_i)^2 - (\sum \sin^2 \psi_i)^2} - (m_{\varphi})^2 \right) \quad (80)$$

$$\text{var}(D_{\varphi,\psi=0}) = \text{var}(m_{\varphi}) \cdot \frac{\sum (\sin^2 \psi_i)^2}{N} \quad (81)$$

Using the error propagation theory the variances of m_{φ} and $D_{\varphi,\psi=0}$ can be related to the 2Θ -variances. The error of $\sin^2 \psi$ will be neglected:

$$\text{var}(m_{\varphi}) = \frac{\sum (\sin^2 \psi_i - \overline{\sin^2 \psi})^2 \text{var}(D_{\varphi,\psi_i})}{\left(\sum (\sin^2 \psi_i - \overline{\sin^2 \psi})^2 \right)^2} \quad (82)$$

$$\text{var}(D_{\varphi,\psi=0}) = \frac{\sum \text{var}(D_{\varphi,\psi_i})}{N^2} + (\overline{\sin^2 \psi})^2 \text{var}(m_{\varphi}) - 2 \overline{\sin^2 \psi} \frac{\sum (\sin^2 \psi_i - \overline{\sin^2 \psi}) \text{var}(D_{\varphi,\psi_i})}{N \sum (\sin^2 \psi_i - \overline{\sin^2 \psi})^2} \quad (83)$$

$$\text{var}(D_{\varphi,\psi_i}) = \left(\frac{\lambda \cos \Theta}{2 \sin^2 \Theta} \right)^2 \left(\frac{\pi}{180} \right)^2 \frac{\text{var}(2\Theta)}{4} \quad (84)$$

For the variance of σ_{φ} follows:

$$\text{var}(\sigma_{\varphi}) = \frac{\text{var}(m_{\varphi})}{\left(D_0 \frac{1}{2} s_2 \right)^2} + \left(\frac{\sigma_{\varphi}}{D_0} \right)^2 \text{var}(D_0) + \left(\frac{\sigma_{\varphi}}{\frac{1}{2} s_2} \right)^2 \text{var}\left(\frac{1}{2} s_2\right) \quad (85)$$

Again a practical example. Fig. 22 shows errors according to the Equ. 82, 83, 84 of σ_φ and of $D_{\varphi,\psi=0}$ versus $\sin^2\psi$ range for different $\Delta\sin^2\psi$ steps. Details are given in the caption.

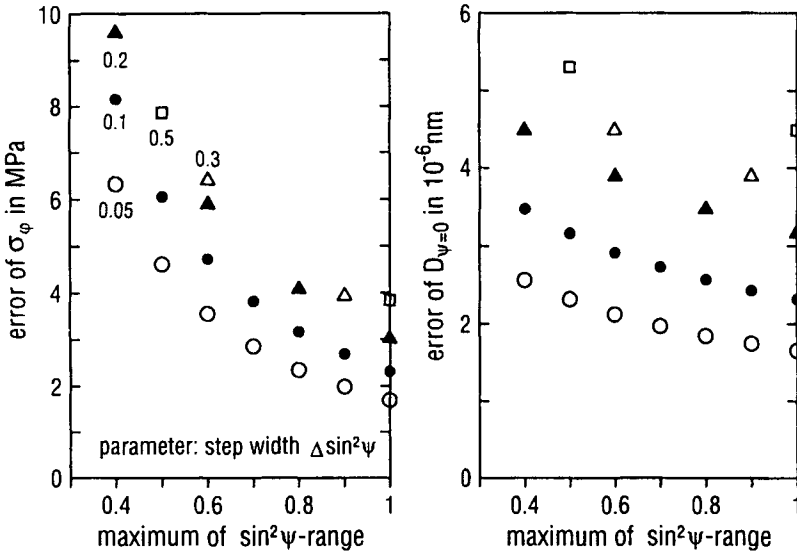


Figure 22. Errors in σ_φ (left) and in $D_{\varphi,\psi=0}$ (right) versus $\sin^2\psi$ range and $\Delta\sin^2\psi$ steps, error of determination of position of peak $\pm 0.01^\circ$ in 2Θ , Cr- K_α radiation, Fe{211}, $D_0 = 0.28665$ nm, $\frac{1}{2}s_2 = 5.76 \cdot 10^{-6}$ MPa $^{-1}$.

The tensor evaluation, the Dölle-Hauk method

The method is described in 2.072c. Lattice-strain measurements should be made at the azimuths $\varphi = 0^\circ, \pm 45^\circ, 90^\circ$. The following abbreviations are helpful.

$$D_{\varphi,\psi_i}^+, D_{\varphi,\psi_i}^- = \frac{1}{2} (D_{\varphi,+\psi} \pm D_{\varphi,-\psi}) \quad (86)$$

$$\text{var}(D_{\varphi,\psi_i}^+) = \text{var}(D_{\varphi,\psi_i}^-) = \frac{1}{4} [\text{var}(D_{\varphi,+\psi}) + \text{var}(D_{\varphi,-\psi})] \quad (87)$$

The advantage of this method is that only linear dependences are used. Therefore the explanations of the foregoing block hold. One should note that the number of the data points reduces after averaging the measured values, $M = \frac{1}{2}(N+1)$ or $M = \frac{N}{2}$, N being even or odd.

Using Equ. 57, 61 and 66 according to the regression line $D_{\varphi,\psi}^- = m_{\varphi}^- |\sin 2\psi|$ it follows that

$$\begin{aligned} m_{\varphi}^- &= \frac{\sum (|\sin 2\psi_i| - \overline{|\sin 2\psi|}) (D_{\varphi,\psi_i}^- - \overline{D_{\varphi,\psi}})}{\sum (|\sin 2\psi_i| - \overline{|\sin 2\psi|})^2} \\ &= \frac{M \sum (|\sin 2\psi_i| D_{\varphi,\psi_i}^-) - \sum |\sin 2\psi_i| \sum D_{\varphi,\psi_i}}{M \sum |\sin 2\psi_i|^2 - \left(\sum |\sin 2\psi_i| \right)^2} \end{aligned} \quad (88)$$

$$\text{var}(m_{\bar{\varphi}}) = \frac{1}{M-2} \left(\frac{M \sum D_{\varphi, \psi_i}^2 - \left(\sum D_{\varphi, \psi_i} \right)^2}{M \sum |\sin 2\psi_i|^2 - \left(\sum |\sin 2\psi_i| \right)^2} - (m_{\bar{\varphi}})^2 \right) \quad (89)$$

Using the error propagation theory and neglecting the error of $\sin 2\psi$ it follows that

$$\text{var}(m_{\bar{\varphi}}) = \frac{\sum \left(|\sin 2\psi_i| - \left| \overline{\sin 2\psi} \right| \right)^2 \text{var}(D_{\varphi, \psi})}{\left(\sum \left(|\sin 2\psi_i| - \left| \overline{\sin 2\psi} \right| \right)^2 \right)^2} \quad (90)$$

The following equations will be repeated from the previous explanations:

$$(\sigma_{\varphi} - \sigma_{33}) = \frac{1}{D_0 \frac{1}{2} s_2} \frac{\partial D_{\varphi, \psi}^+}{\partial (\sin^2 \psi)} = \frac{m_{\varphi}^+}{D_0 \frac{1}{2} s_2} \quad (91)$$

$$(\sigma_{13}, \sigma_{23}) = \frac{1}{D_0 \frac{1}{2} s_2} \frac{\partial D_{\varphi, \psi}^-}{\partial (|\sin 2\psi|)} = \frac{m_{\varphi}^-}{D_0 \frac{1}{2} s_2} \quad (92)$$

The appropriate variances follow from Equ. 85. The stress component in thickness direction σ_{33} can be calculated according to

$$\sigma_{33} = \frac{1}{D_0 \left(3s_1 + \frac{1}{2} s_2 \right)} \left(D_{\varphi, \psi=0}^+ - D_0 - \frac{s_1}{\frac{1}{2} s_2} (m_0^+ + m_{90}^+) \right) \quad (93)$$

The square errors of the normal components are as follows (N_{φ} : numbers of azimuths φ):

$$\begin{aligned} \text{var}(\sigma_{33}) = & \frac{s_1^2 \left(\text{var}(m_0^+) + \text{var}(m_{90}^+) \right)}{\left(D_0 \frac{1}{2} s_2 \left(3s_1 + \frac{1}{2} s_2 \right) \right)^2} + \frac{\sum \text{var}(D_{\varphi, \psi=0}^+)}{\left(N_{\varphi} D_0 \left(3s_1 + \frac{1}{2} s_2 \right) \right)^2} \\ & + \left(\overline{D_{\varphi, \psi=0}^+} - \frac{s_1}{\frac{1}{2} s_2} (m_0^+ + m_{90}^+) \right)^2 \frac{\text{var}(D_0)}{D_0^4 \left(3s_1 + \frac{1}{2} s_2 \right)^2} \\ & + \left(D_0 - \overline{D_{\varphi, \psi=0}^+} + (m_0^+ + m_{90}^+) \frac{s_1 \left(3s_1 + 2 \frac{1}{2} s_2 \right)}{\left(\frac{1}{2} s_2 \right)^2} \right)^2 \frac{\text{var}\left(\frac{1}{2} s_2\right)}{D_0^2 \left(3s_1 + \frac{1}{2} s_2 \right)^4} \\ & + \left(3 \left(D_0 - \overline{D_{\varphi, \psi=0}^+} \right) - (m_0^+ + m_{90}^+) \right)^2 \frac{\text{var}(s_1)}{D_0^2 \left(3s_1 + \frac{1}{2} s_2 \right)^4} \end{aligned} \quad (94)$$

$$\begin{aligned} \text{var}(\sigma_{11}) &= \text{var}(\sigma_{11} - \sigma_{33}) + \text{var}(\sigma_{33}) \\ &= \frac{\text{var}(m_0^+)}{(D_0 \frac{1}{2} s_2)} + \left(\frac{\sigma_{11} - \sigma_{33}}{D_0} \right)^2 \text{var}(D_0) + \left(\frac{\sigma_{11} - \sigma_{33}}{\frac{1}{2} s_2} \right)^2 \text{var}\left(\frac{1}{2} s_2\right) + \text{var}(\sigma_{33}) \end{aligned} \quad (95)$$

(analogous to σ_{22})

Measurements at $\varphi = 45^\circ$ or $\varphi = -45^\circ$ allow the determination of σ_{12} , Equ. 96 to 99.

$$\pm \sigma_{12} = \sigma_{\varphi=\pm 45} - \frac{1}{2}((\sigma_{11} - \sigma_{33}) + (\sigma_{22} - \sigma_{33})) = \frac{1}{D_0 \frac{1}{2} s_2} \left(m_{\pm 45}^+ - \frac{1}{2}(m_0^+ + m_{90}^+) \right) \quad (96)$$

$$\text{var}(\sigma_{12}) = \frac{\text{var}(m_{\pm 45}^+) + \frac{1}{4}(\text{var}(m_0^+) + \text{var}(m_{90}^+))}{(D_0 \frac{1}{2} s_2)^2} + (\sigma_{12})^2 \left(\frac{\text{var}(D_0)}{(D_0)^2} + \frac{\text{var}(\frac{1}{2} s_2)}{(\frac{1}{2} s_2)^2} \right) \quad (97)$$

$$\sigma_{12} = \frac{1}{2 D_0 \frac{1}{2} s_2} (m_{45}^+ - m_{-45}^+) \quad (98)$$

$$\text{var}(\sigma_{12}) = \frac{\text{var}(m_{\pm 45}^+) + \text{var}(m_{-45}^+)}{(D_0 \frac{1}{2} s_2)^2} + (\sigma_{12})^2 \left(\frac{\text{var}(D_0)}{(D_0)^2} + \frac{\text{var}(\frac{1}{2} s_2)}{(\frac{1}{2} s_2)^2} \right) \quad (99)$$

With the assumption of $\sigma_{33} = 0$ the lattice spacing of the stress-free state D_0 and the variance of it can be calculated.

$$D_0 = \overline{D_{\varphi, \psi=0}^+} - \frac{s_1}{\frac{1}{2} s_2} (m_0^+ - m_{90}^+) \quad (100)$$

$$\text{var}(D_0) = \frac{s_1^2 (\text{var}(m_0^+) + \text{var}(m_{90}^+))}{(\frac{1}{2} s_2)^2} + \frac{\sum \text{var}(D_{\varphi, \psi=0}^+)}{(N_\varphi)^2} \quad (101)$$

$$+ (D_0 (\sigma_{11} + \sigma_{22}))^2 \text{var}(s_1) + \left(\frac{D_0 s_1 (\sigma_{11} + \sigma_{22})}{\frac{1}{2} s_2} \right)^2 \text{var}\left(\frac{1}{2} s_2\right)$$

Using this assumption the variances of the normal stresses are as follows
 $\text{var}(\sigma_{11}) = \text{var}(\sigma_{11} - \sigma_{33})$ and $\text{var}(\sigma_{22}) = \text{var}(\sigma_{22} - \sigma_{33})$.

The following example will illustrate the last explanations. In Fig. 23 there are plotted the D-vs.- $\sin^2\psi$ distributions of a ground and roll-peened surface of a TiAl6V4 specimen, Cu-K α radiation, {114}. Table 5 represents the evaluation results, based on the equations of the last two blocks. The details are given in the caption.

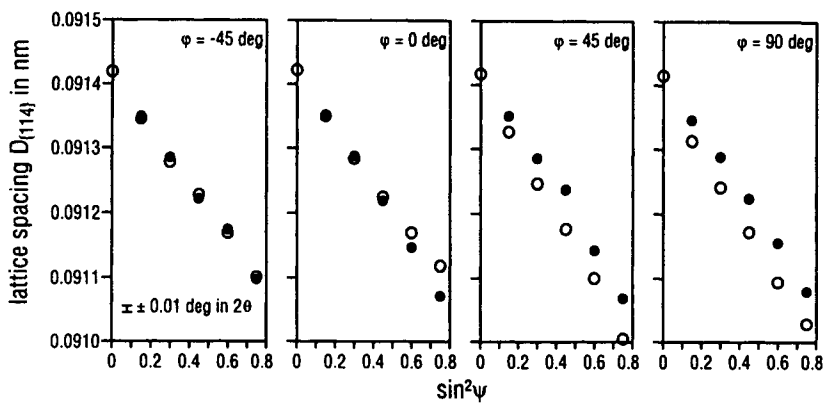


Figure 23. D-vs.-sin²ψ distribution, TiAl6V4, ground and roll-peened, Cu-K_α, {114} /51,52/.

Table 5. Evaluation of the lattice-strain measurements according to Fig. 23 and the equations of the last two blocks with the following assumptions: precision of the interference line position ±0.01° in 2Θ, D₀{114}= 0.091227 nm, s₁= -2.64 ·10⁻⁶ MPa⁻¹, $\frac{1}{2}s_2= 11.12 \cdot 10^{-6}$ MPa⁻¹. The errors considered are slope, intercept and ±4 ·10⁻⁶ nm in D₀{114}.

	σ [MPa]	regression error		error propagation ±0.01° in 2Θ	
		errors taken into account m_φ $D_{\varphi,\psi=0}$	m_φ $D_{\varphi,\psi=0}$ D_0	errors taken into account m_φ $D_{\varphi,\psi=0}$	m_φ $D_{\varphi,\psi=0}$ D_0
$\sigma_{11}-\sigma_{33}$	-428	4	4	7	7
$\sigma_{22}-\sigma_{33}$	-467	9	9		
σ_{33}	-96	11	18	9	17
σ_{11}	-524	12	18	12	18
σ_{22}	-563	14	20		
σ_{13}	-8	12	12	2	2
σ_{23}	28	3	3		
$\sigma(45^\circ)$	-491	13	13	7	7
σ_{12}	-44	14	14	8	8
$\sigma(-45^\circ)$	-410	10	10	7	7
σ_{12}	-38	11	11	8	8
$\sigma_{12}(\pm 45^\circ)$	-41	8	8	5	5
D ₀ {114} in nm					
$\sigma_{33}=0$	0.091199	3 ·10 ⁻⁶	3 ·10 ⁻⁶	3 ·10 ⁻⁶	3 ·10 ⁻⁶

XEC

Here again only straight lines are used to determine s_1 and $\frac{1}{2}s_2$. The necessary formulae are derived in chapter 2.13. The external load is signified by σ^L and N_σ gives the number of the applied loads. The following formulae need no special explanatory remarks:

$$s_1 = \frac{1}{D_0} \frac{\partial D_{\psi=0}^L}{\partial \sigma^L} \quad (102)$$

$$\frac{1}{2}s_2 = \frac{1}{D_0} \frac{\partial}{\partial \sigma^L} \frac{\partial D^L}{\partial \sin^2 \psi} = \frac{1}{D_0} \frac{\partial m^L}{\partial \sigma^L} \quad (103)$$

$$\frac{\partial D_{\psi=0}^L}{\partial \sigma^L} = \frac{\sum (\sigma_i^L - \overline{\sigma^L}) (D_{i,0}^L - \overline{D_0^L})}{\sum (\sigma_i^L - \overline{\sigma^L})^2} = \frac{N_\sigma \sum (\sigma_i^L D_{i,0}^L) - \sum \sigma_i^L \sum D_{i,0}^L}{N_\sigma \sum (\sigma_i^L)^2 - (\sum \sigma_i^L)^2} \quad (104)$$

$$\frac{\partial m^L}{\partial \sigma^L} = \frac{\sum (\sigma_i^L - \overline{\sigma^L}) (m_i^L - \overline{m^L})}{\sum (\sigma_i^L - \overline{\sigma^L})^2} = \frac{N_\sigma \sum (\sigma_i^L m_i^L) - \sum \sigma_i^L \sum m_i^L}{N_\sigma \sum (\sigma_i^L)^2 - (\sum \sigma_i^L)^2} \quad (105)$$

$$\text{var} \left(\frac{\partial D_{\psi=0}^L}{\partial \sigma^L} \right) = \frac{1}{N_\sigma - 2} \left(\frac{N_\sigma \sum (D_{i,0}^L)^2 - (\sum D_{i,0}^L)^2}{N_\sigma \sum (\sigma_i^L)^2 - (\sum \sigma_i^L)^2} - \left(\frac{\partial D_0^L}{\partial \sigma^L} \right)^2 \right) \quad (106)$$

$$\text{var} \left(\frac{\partial m^L}{\partial \sigma^L} \right) = \frac{1}{N_\sigma - 2} \left(\frac{N_\sigma \sum (m_i^L)^2 - (\sum m_i^L)^2}{N_\sigma \sum (\sigma_i^L)^2 - (\sum \sigma_i^L)^2} - \left(\frac{\partial m^L}{\partial \sigma^L} \right)^2 \right) \quad (107)$$

$$\text{var} \left(\frac{\partial D_{\psi=0}^L}{\partial \sigma^L} \right) = \frac{\sum (\sigma_i^L - \overline{\sigma^L})^2 \text{var}(D_{i,0}^L)}{\left(\sum (\sigma_i^L - \overline{\sigma^L})^2 \right)^2} + \frac{\sum (D_{i,0}^L - \overline{D^L})^2 \text{var}(\sigma_{i,0}^L)}{\left(\sum (\sigma_i^L - \overline{\sigma^L})^2 \right)^2} \quad (108)$$

$$\text{var} \left(\frac{\partial m^L}{\partial \sigma^L} \right) = \frac{\sum (\sigma_i^L - \overline{\sigma^L})^2 \text{var}(m_i^L)}{\left(\sum (\sigma_i^L - \overline{\sigma^L})^2 \right)^2} + \frac{\sum (m_i^L - \overline{m^L})^2 \text{var}(\sigma_i^L)}{\left(\sum (\sigma_i^L - \overline{\sigma^L})^2 \right)^2} \quad (109)$$

Using error propagation theory and Equ. 102 and 103 the square of standard deviation (variance) of s_1 and $\frac{1}{2}s_2$ are obtained as

$$\text{var}(s_1) = \frac{1}{D_0^2} \left(\text{var} \left(\frac{\partial D_{\psi=0}^L}{\partial \sigma^L} \right) + \left(\frac{s_1}{D_0} \right)^2 \text{var}(D_0) \right) \quad (110)$$

$$\text{var}\left(\frac{1}{2}s_2\right) = \frac{1}{D_0^2} \left(\text{var} \left(\frac{\partial m^L}{\partial \sigma^L} \right) + \left(\frac{\frac{1}{2}s_2}{D_0} \right)^2 \text{var}(D_0) \right) \quad (111)$$

The following example considers only errors associated with counting statistics (Fig.22). Fe{211}, Cr-K α , $D_0=0.28665$ nm, $0 \leq \sin^2\psi \leq 0.9$, $\Delta\sin^2\psi = 0.1$, 6 load steps σ^L from 300 down to 100 MPa in steps of 40 MPa, error of σ^L is ± 1 MPa, error of D_0 of $\pm 1 \cdot 10^{-5}$ nm can be neglected. Result: $s_1 = -(1.25 \pm 0.05) \cdot 10^{-6}$ MPa $^{-1}$, $\frac{1}{2}s_2 = (5.76 \pm 0.10) \cdot 10^{-6}$ MPa $^{-1}$. Taking into account the local variances of the material properties the error of the XEC will increase.

2.072g The D-, ϵ -, σ -, FWHM-polefigures

In analogy to intensity polefigures $2\Theta_{\varphi,\psi}$, $D_{\varphi,\psi}$, $\epsilon_{\varphi,\psi}$, FWHM-polefigures provide an overview of the lattice-strain state at certain points of a component. Numerous measurements of the specific {hkl} peak in the coordinates (φ,ψ) have to be performed to establish such a polefigure. The measured values of $2\Theta_{\varphi,\psi}$ or $D_{\varphi,\psi}$ or, if the D_0 -value is precisely known, of $\epsilon_{\varphi,\psi}$ will be plotted versus $\left(\varphi, \tan\left(\frac{\psi}{2}\right)\right)$ or (φ,ψ) or $(\varphi,\sin^2\psi)$.

Modern stress- and texture-diffractometers with adequate data processing and printing systems enable the production of such presentations. To show up the different distributions, appropriate polefigures are shown in Figures 24, 25 and 26. The first four polefigures are calculated ones, for stress tensors as indicated /19/. The scale in $\sin^2\psi$ enables one to read directly the strain dependences in the usual manner. The polefigures in Fig. 25 are experimentally determined ones /19/. Shown are the intensity- and the lattice-spacing polefigures of a rolled steel, also in a stereoscopic view /19/.

Fig. 26a shows a deformation state in which the axes of the stress tensor do not coincide with the axes of the specimen system /53,54/. The lattice-deformation polefigure observed on {211}-lattice planes of a ground-steel specimen (German grade Ck45) is presented. The patterns displayed belong to the near-surface state of stresses comprising the components σ_{11} , σ_{22} , σ_{13} determined with Cr-K α radiation. The lattice deformation polefigure is symmetrical with respect to the grinding direction GD. The tilt of the stress tensor around the transverse direction TD can clearly be seen. An example of a deformation polefigure of the 2nd kind is displayed in Fig. 26b. The patterns are valid for {211}-planes of a 78% cold-rolled Armco-iron sheet measured with Cr-K α radiation. The data were obtained from the total deformation polefigure of the {211}-planes and that of {721+633+552}-planes determined with Mo-K α radiation. The latter was assumed to be equal to the 1st-kind-deformation polefigure of the material investigated. 2nd-kind-deformation polefigures like that shown in Fig. 26b characterize the texture-related deviations from the quasiisotropic material behavior of the lattice planes {hkl} considered.

Fig. 26c and 26d demonstrate results obtained on a cold-rolled highly textured Ni-strip /55/. Fig. 26c shows the intensity and Fig. 26d the lattice-strain polefigure. The ideal orientations are indicated and the rolling direction is marked. The evaluation of these data revealed small macro-RS and big micro-RS with a steep gradient of the stress component in RD.

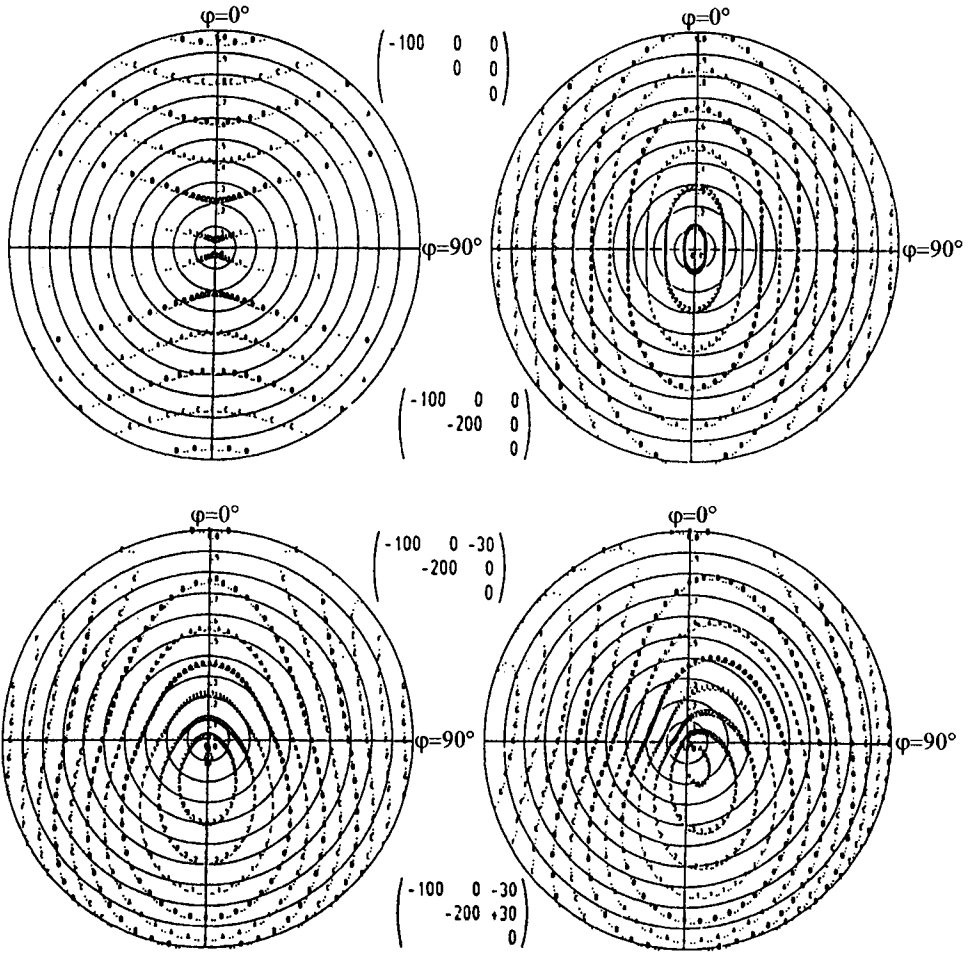


Figure 24. D-polefigures calculated for different stress tensors (with $\sin^2\psi$ as the distance from the center, the rim corresponds to $\sin^2\psi=1$). The symbol 0 represents $D_0 = 0.28665$ nm; 0 - G, $\Delta_D = -3 \cdot 10^{-5}$ nm; 0 - 4, $\Delta_D = 3 \cdot 10^{-5}$ nm /19/.

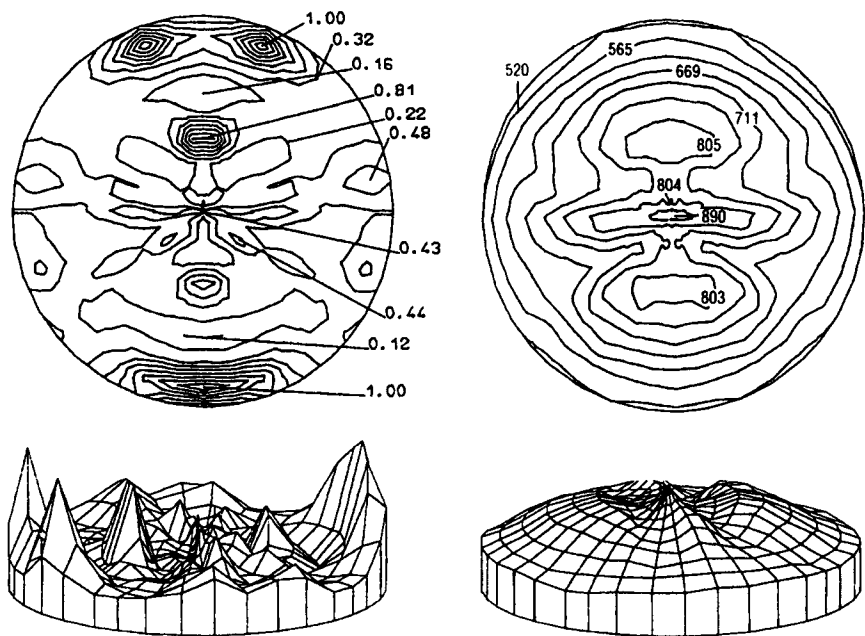


Figure 25. (above): {211} polefigures (with $\sin^2\psi$ as the distance from the center) of a rolled steel; left: Intensity polefigure; right: Lattice-spacing polefigure (D values should be read as $0.286 \text{ nm} + 10^{-6}$ times the numbers given) /19/. (below): The same polefigures in a stereoscopic view (75° left from RD and 15° inclined to the surface) /19/.

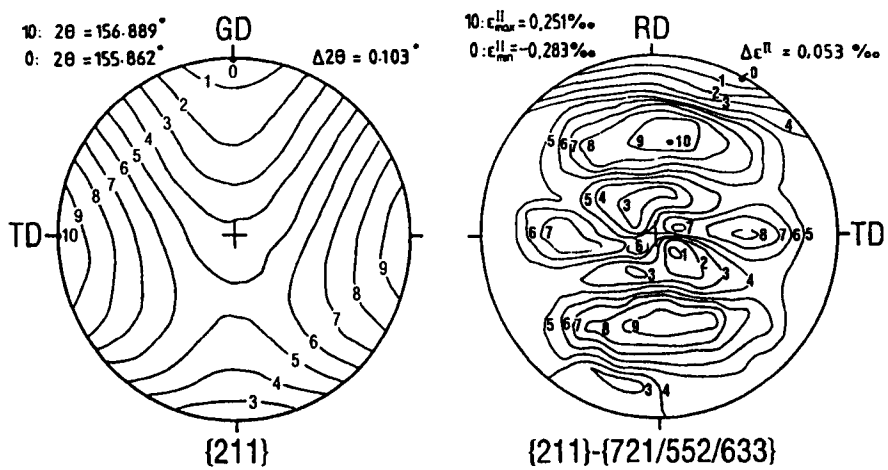


Figure 26a. 1st-kind-deformation polefigure of a surface residual stress state with $\sigma_{11} = 77 \text{ MPa}$, $\sigma_{22} = -216 \text{ MPa}$ and $\sigma_{13} = 50 \text{ MPa}$ determined with Cr- K_{α} -radiation (GD grinding direction) /54/.
Figure 26b. 2nd-kind-deformation polefigure of a 78% cold-rolled Armco-iron sheet /54/. Stereographic projections, the rim of both figures corresponds to $|\psi| = 70^\circ$

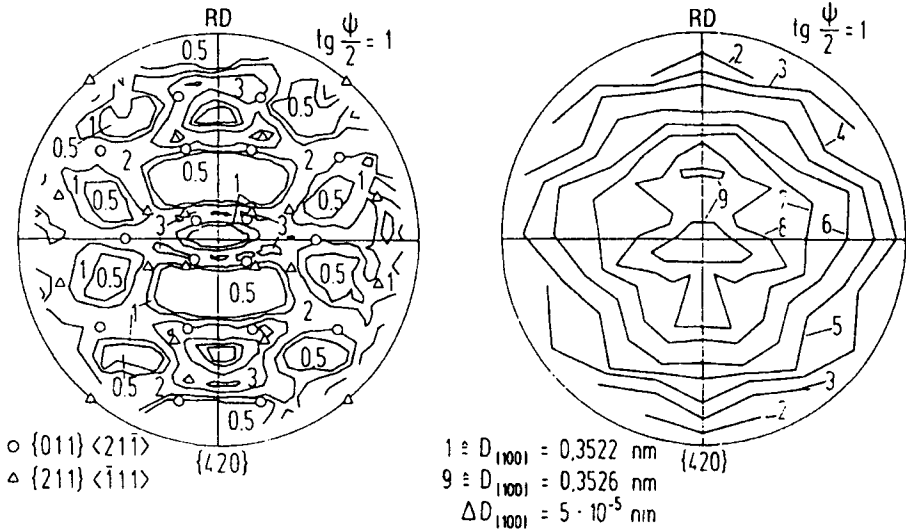


Figure 26c. {420} intensity polefigure, rolled Ni strip, Mo-K α -radiation /55/

Figure 26d. {420} lattice-distance polefigure, rolled Ni strip, Cu K α -radiation /55/.

2.072h The ϕ - and ψ -integral method

The conception, the basic formulae and a few examples of applications are published mostly by Lode and Peiter. This special literature can be found in the book /56/. In recent papers of these authors it is claimed that this method is the only one to get the total strain, stress tensor. But the evaluation method ignores the real material state and the details of the measuring techniques. A restriction of the method is the fact that mechanically isotropic materials and linear strain gradients are supposed. Furthermore detailed measuring instructions are not given. The method is not transparent. The use of the method is limited and some of the published results are somewhat doubtful. More in section 2.152h.

The following will follow the papers of /57,58/. The strain profile with depth from the surface z is developed into a Mac-Laurin-Taylor series

$$\langle \varepsilon_{ij} \rangle = \varepsilon_{ij}(z=0) + \sum_{k=1}^n \frac{d^k \varepsilon_{ij}(z=0)}{dz^k} \tau^k \quad (112)$$

For the ϕ -integral method the fundamental formula holds with

$$\varepsilon'_{ij} = \frac{d\varepsilon_{ij}(z=0)}{dz} \text{ and } \tau_{\psi} = \frac{\sin \Theta \sin \psi}{2\mu} \quad (113)$$

$$\varepsilon_{\phi, \psi} = \frac{A_0}{2} + A_1 \cos \phi + A_2 \cos 2\phi + B_1 \sin \phi + B_2 \sin 2\phi \quad (114)$$

$$\begin{aligned}
 A_0 &= (\varepsilon_{11}(z=0) + \tau\varepsilon'_{11} + \varepsilon_{22}(z=0) + \tau\varepsilon'_{22})\sin^2\psi + 2(\varepsilon_{33}(z=0) + \tau\varepsilon'_{33})\cos^2\psi \\
 A_1 &= (\varepsilon_{13}(z=0) + \tau\varepsilon'_{13})\sin 2\psi
 \end{aligned} \tag{115}$$

$$A_2 = \frac{1}{2}(\varepsilon_{11}(z=0) + \tau\varepsilon'_{11} - \varepsilon_{22}(z=0) - \tau\varepsilon'_{22})\sin^2\psi$$

$$B_1 = (\varepsilon_{23}(z=0) + \tau\varepsilon'_{23})\sin 2\psi$$

$$B_2 = (\varepsilon_{12}(z=0) + \tau\varepsilon'_{12})\sin^2\psi$$

$$A_n = \frac{1}{\pi} \int_0^{2\pi} \langle \varepsilon_{\varphi,\psi} \rangle \cos n\varphi d\varphi \tag{116}$$

$$B_n = \frac{1}{\pi} \int_0^{2\pi} \langle \varepsilon_{\varphi,\psi} \rangle \sin n\varphi d\varphi \tag{117}$$

The $2\Theta_{\varphi,\psi}$ respectively the $\langle D_{\varphi,\psi} \rangle$ are measured over the range $0^\circ \leq \varphi \leq 360^\circ$. To evaluate the twelve components $\varepsilon_{ij}(z=0)$ and ε'_{ij} at least three tilt angles ψ are necessary.

The often-praised possibility offered by the φ -integral method to measure in one depth from the surface is questionable. An error analysis by /59/ showed high unacceptable values when assuming typical measurement conditions. In general we do not recommend to use the φ -integral method in the present form to determine the strain, stress tensor and the linear gradients of the components. There is one application of a φ -method for samples with a fiber texture with the fiber axis perpendicular to the specimen surface. This will be reported in 2.073d.

The ψ -integral method is even more nontransparent. An example will be discussed in 2.15.

2.072i The deviatoric-hydrostatic-stress approach

A pure hydrostatic stress state is characterized by a horizontal $D_{\varphi,\psi}$ -vs.- $\sin^2\psi$ straight line parallel to that of the strain-stress-free state. The D_0 -constant has to be known very exactly to determine the hydrostatic stress state. For cubic materials there exists a special relation:

$\varphi = 0^\circ$:

$$\varepsilon_{0,\psi} = \frac{D_{0,\psi} - D_0}{D_0} = s_1(\sigma_1 + \sigma_2 + \sigma_3) + \frac{1}{2}s_2[(\sigma_1 - \sigma_3)\sin^2\psi + \sigma_3] \tag{117}$$

$$\sigma_1 = \sigma_2 = \sigma_3 = \sigma_H : \tag{118}$$

$$\varepsilon_{0,\psi} = \sigma_H \left(3s_1 + \frac{1}{2}s_2 \right) = \text{const.} \tag{119}$$

In /60/ the fact was discussed that each RS-state can be considered as a superposition of a hydrostatic stress state and a deviatoric stress state characterized by the deviator tensor. In /61/ the RS-state of the ceramic material $\text{Al}_2\text{O}_3 + \text{SiC}$ was evaluated in this way. The RS-state of $\alpha\text{-Fe}$ and Fe_3C was analyzed by /62/ by hydrostatic stress state and deviatoric stresses emphasizing that the exact D_0 -value is only necessary to determine the hydrostatic phase stresses.

In the following the basic formulae will be cited. It will be shown that the usual evaluation method /36/ and the deviatoric – hydrostatic method will deliver the same information. An example will illustrate the procedure and discuss the results.

Any stress tensor can be divided:

$$\begin{pmatrix} \sigma_1 & & \\ & \sigma_2 & \\ & & \sigma_3 \end{pmatrix} = \begin{pmatrix} \sigma_1^d & & \\ & \sigma_2^d & \\ & & \sigma_3^d \end{pmatrix} + \sigma_H \quad ; \quad \sum_i \sigma_i^d = 0 \quad (120)$$

deviator hydrostatic definition
stress

The same is valid for the phase stresses, index α . From $D_{\varphi,\psi}$ -vs.- $\sin^2\psi$ measurements one gets

$$\frac{\partial D_{\varphi=0,\psi}^\alpha}{\partial \sin^2 \psi} \Rightarrow \sigma_1^\alpha - \sigma_3^\alpha; \quad \frac{\partial D_{\varphi=90,\psi}^\alpha}{\partial \sin^2 \psi} \Rightarrow \sigma_2^\alpha - \sigma_3^\alpha$$

with the condition

$$\sum_i \sigma_i^{d,\alpha} = 0 \Rightarrow \sigma_1^{d,\alpha}, \sigma_2^{d,\alpha}, \sigma_3^{d,\alpha} \text{ and } \sigma_H^{d,\alpha} \text{ (the exact } D_0^\alpha \text{ must be known).} \quad (121)$$

With c_α the volume content the macrostress tensor is

$$\begin{pmatrix} \sigma_1 & & \\ & \sigma_2 & \\ & & \sigma_3 \end{pmatrix} = \begin{pmatrix} \sum_\alpha c_\alpha \sigma_1^{d,\alpha} & & \\ & \sum_\alpha c_\alpha \sigma_2^{d,\alpha} & \\ & & \sum_\alpha c_\alpha \sigma_3^{d,\alpha} \end{pmatrix} + \sum_\alpha c_\alpha \sigma_H^\alpha \quad (122)$$

$$\sigma_3 = \sigma_3^d + \sigma_H = \sum_\alpha c_\alpha \sigma_3^{d,\alpha} + \sigma_H = 0 \quad (123)$$

$$\sigma_H = - \sum_\alpha c_\alpha \sigma_3^{d,\alpha} \quad (124)$$

The usual method /36/ evaluates

$$\sigma_1^\alpha - \sigma_3^\alpha = \sigma_1^{d,\alpha} - \sigma_3^{d,\alpha} \quad \sigma_2^\alpha - \sigma_3^\alpha = \sigma_2^{d,\alpha} - \sigma_3^{d,\alpha} \quad (125)$$

The deviator is given by $\sum_i \sigma_i^{d,\alpha} = 0$; D_0^α must be known.

$$\sigma_1 - \sigma_3 = \sum_\alpha c_\alpha (\sigma_1^\alpha - \sigma_3^\alpha) \quad \sigma_2 - \sigma_3 = \sum_\alpha c_\alpha (\sigma_2^\alpha - \sigma_3^\alpha) \quad (126)$$

The result of the evaluation with $\sigma_3 = 0$ yields the same information:

$$\begin{pmatrix} \sigma_1 & & \\ & \sigma_2 & \\ & & 0 \end{pmatrix} = \frac{1}{3} \cdot \begin{pmatrix} 2\sigma_1 - \sigma_2 & & \\ & 2\sigma_2 - \sigma_1 & \\ & & -(\sigma_1 + \sigma_2) \end{pmatrix} + \frac{1}{3}(\sigma_1 + \sigma_2) \quad (127)$$

An example of evaluating RS of an $\text{Al}_2\text{O}_3 + \text{SiC} + \text{ZrO}_2$ specimen:

The RS-state of a sintered $\text{Al}_2\text{O}_3 + \text{SiC}$ two-phase material has been studied with X-rays /61,63/ and with neutron-rays /64/. Part of the following evaluation has been published /65/; here are the detailed results of a thorough evaluation of the RS-state.

The X-ray study was done on a three-phase material from which the powder mixture and the sintered specimen, a slice 5 mm thick, diameter 60 mm, were available. The lattice strains were measured by X-rays in the center of the slice. The composition is 73 wt% Al_2O_3 , 17 wt% SiC and 10wt% ZrO_2 .

The peaks of ZrO_2 could not be measured. There are three possibilities to deal with a three-phase material in which only two phases are measurable, (chapter 2.12).

- Neglect the third phase because of its small amount. This was done in the following.
- Determine the macro-RS by another method; not possible here.
- Determine the phase stresses of only two phases, but in this case only differences of macro-stresses are available.

The X-ray measurements of the lattice strains in both phases of the powder and of the specimen for the azimuths $\varphi = 0^\circ$ and 90° are shown in Fig. 27. All data used for the evaluation and the results of the stress evaluation are listed in Table 6.

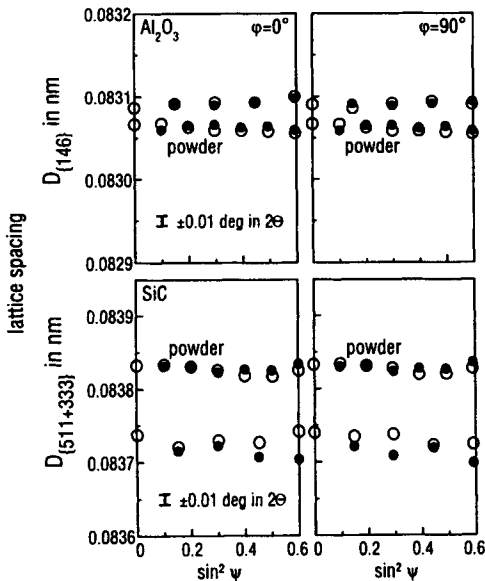


Figure 27. Lattice spacings of the powder and the sintered sample, 73 wt% $\text{Al}_2\text{O}_3 + 17$ wt% SiC + 10 wt% ZrO_2 , above $\text{Al}_2\text{O}_3\{146\}$ Cu- K_α , below $\text{SiC}\{511+333\}$ Cu- K_α .

From the slopes of $D_{\phi,\psi}$ vs. $\sin^2\psi$ the differences $(\sigma_1 - \sigma_3)^{I+II}$ and $(\sigma_2 - \sigma_3)^{I+II}$ are determined. Index (I + II) means macro- + micro-RS. σ_3^{I+II} is evaluated using the relation between σ_3 and D_0 , as given in chapter 2.11. For separating macro- and micro-RS the formulae Equ. 2, 4, chapter 2.06 will be used.

The results are given as the following tensors.

$$\begin{pmatrix} 81 & & \\ & 19 & \\ & & 41 \end{pmatrix} \text{MPa} \quad \begin{pmatrix} 259 & & \\ & 277 & \\ & & 236 \end{pmatrix} \text{MPa} \quad \begin{pmatrix} -933 & & \\ & -998 & \\ & & -853 \end{pmatrix} \text{MPa}$$

Macro-RS Micro-RS Al_2O_3 Micro-RS SiC

Table 6. Data and results of an evaluation of a sintered three-phase material, with the third phase neglected.

phase		Al_2O_3	SiC
vol. %		0.783	0.217
{hkl}		{146}	{511+333}
powder- $D_0\{\text{hkl}\}$	D_0 in nm	0.083062	0.083827
XEC	$-s_1$	0.54	0.49
in 10^{-6}MPa^{-1}	$\frac{1}{2}s_2$	2.92	3.00
macro- and micro-RS in MPa	$(\sigma_1 - \sigma_3)^{I+II}$	62 ± 10	-41 ± 91
	$(\sigma_2 - \sigma_3)^{I+II}$	18 ± 10	-167 ± 77
	σ_3^{I+II}	278 ± 10	-812 ± 34
	σ_1^{I+II}	340	-852
	σ_2^{I+II}	296	-979
macro-RS in MPa	σ_1^I	81	
	σ_2^I	19	
	σ_3^I	41	
micro-RS in MPa	σ_1^{II}	259	-933
	σ_2^{II}	277	-998
	σ_3^{II}	236	-853

As a check test the vol.% weighted sum of the micro-RS should be zero:

$$c_{\alpha}\sigma_i^{\alpha} + c_{\beta}\sigma_i^{\beta} = 0$$

As an example, we obtain for σ_1 with the not-rounded values

$$0.783 \cdot 258.7 - 0.217 \cdot 933.3 = 202.6 - 202.5 \text{ MPa}$$

These results of the example are valid only if it is assumed that both lattice constants of the phases $D_0^{\alpha,\beta}$ and the concentrations $c^{\alpha,\beta}$ are not altered during the manufacturing of the sample. It is recommended to determine the $D_0^{\alpha,\beta}$ values of the sample by means that are pointed out by /65/. But care should be taken of the possibilities to make experimental mistakes that are listed in chapter 2.10.

The method to neglect σ_3 to determine an unknown D_0 leads to the following RS.

macro- and micro-stresses

$$\begin{pmatrix} 62 & & \\ & 18 & \\ & & 0 \end{pmatrix} \quad \begin{pmatrix} -41 & & \\ & -167 & \\ & & 0 \end{pmatrix}$$

Al_2O_3 SiC

and after separation into macro-micro-RS

$$\begin{pmatrix} 40 & & \\ & -22 & \\ & & 0 \end{pmatrix} \quad \begin{pmatrix} 22 & & \\ & 40 & \\ & & 0 \end{pmatrix} \quad \begin{pmatrix} -80 & & \\ & -145 & \\ & & 0 \end{pmatrix}$$

macro-RS micro-RS Al_2O_3 micro-RS SiC

The values of $D_0^{\alpha,\beta}$ for $\sigma_3 = 0$ follow from the measurements according to the ψ^* -method

$$\sin^2 \psi^* = \left(\frac{-s_1}{\frac{1}{2}s_2} \right) \left(1 + \frac{\sigma_2}{\sigma_1} \right)$$

to 0.0831nm and 0.0837nm.

$$\text{Al}_2\text{O}_3 \quad \sin^2 \psi^* = \left(\frac{0.54}{0.92} \right) \left(1 + \frac{18}{62} \right) = 0.24$$

$$\text{SiC} \quad \sin^2 \psi^* = \left(\frac{0.49}{3.00} \right) \left(1 + \frac{167}{41} \right) = 0.83$$

Going back to the above-mentioned results for the two-phase Al_2O_3 +SiC material with known $D_0^{\alpha,\beta}$ powder values. The tensors of macro- and micro-RS can be transformed to hydrostatic stresses and deviator tensors as follows:

Phase stresses in MPa

$$\text{Al}_2\text{O}_3: \quad 305 + \begin{pmatrix} 35 & & \\ & -9 & \\ & & -27 \end{pmatrix} ; \quad 305 = \frac{340 + 296 + 278}{3}$$

$$\text{SiC:} \quad -881 + \begin{pmatrix} 29 & & \\ & -98 & \\ & & 69 \end{pmatrix} ; \quad -881 = \frac{-852 - 979 - 812}{3}$$

Macro-RS

$$47 + \begin{pmatrix} 34 & & \\ & -28 & \\ & & -6 \end{pmatrix} ; \quad 47 = \frac{81 + 19 + 41}{3}$$

Micro-RS

$$\text{Al}_2\text{O}_3: \quad 257 + \begin{pmatrix} 1 & & \\ & 19 & \\ & & -21 \end{pmatrix} ; \quad 257 = \frac{259 + 277 + 236}{3}$$

$$\text{SiC:} \quad -928 + \begin{pmatrix} -5 & & \\ & -70 & \\ & & +75 \end{pmatrix} ; \quad -928 = \frac{-933 - 998 - 853}{3}$$

The evaluating manner of /62/ following the results of the strain measurements is as follows:
First the deviator tensors of the phase stresses will be determined ($\alpha \triangleq \text{Al}_2\text{O}_3$):

$$(\sigma_1^{d,\alpha} - \sigma_3^{d,\alpha}) = (\sigma_1^\alpha - \sigma_3^\alpha)^{I+II} = 62 \text{ MPa}$$

$$(\sigma_2^{d,\alpha} - \sigma_3^{d,\alpha}) = (\sigma_2^\alpha - \sigma_3^\alpha)^{I+II} = 18 \text{ MPa}$$

$$\sigma_3^{d,\alpha} = -\sigma_1^{d,\alpha} - \sigma_2^{d,\alpha} = -62 - \sigma_3^{d,\alpha} - 18 - \sigma_3^{d,\alpha} \Rightarrow \sigma_3^{d,\alpha} = -\frac{1}{3} \cdot 80 = -27 \text{ MPa}$$

With the value of D_0 the σ_3^α -component can be determined to 278 MPa. Therefore the result for the macro- and micro-phase-stresses is:

$$\begin{pmatrix} \sigma_1^\alpha & & \\ & \sigma_2^\alpha & \\ & & \sigma_3^\alpha \end{pmatrix} = 305 + \begin{pmatrix} 35 & & \\ & -9 & \\ & & -27 \end{pmatrix}$$

the same as previously determined. All the other results can be verified in the same way.

2.072j The ψ -method with ω' -tilt

To study entirely the near-surface region especially of surface deformed materials the specimen in a Ψ -diffractometer will be tilted by the angle ω' as indicated in Fig. 28. The method ω' - ψ was experimentally and mathematically developed by /66/. The formula of the penetration depth considers first the ψ -independent tilt ω' of the Ω -mode and then the ψ -tilt of the Ψ -diffractometer, here called ψ_1 , Table 12, paragraph 2.046.

$$\tau_{\psi_1, \omega'} = \tau_{0, \omega'} \cos \psi_1 = \frac{\sin^2 \Theta - \sin^2 \omega'}{2\mu \sin \Theta \cos \omega'} \cos \psi_1 \quad (128)$$

The material-independent expression $2\mu\tau$ of the penetration depth depends only on functions of the angles Θ , ψ_1 , ω' . The discussion of the formula can be done for $\psi_1 = 0^\circ$ taking $\omega' = \text{const.}$ or $\Theta - \omega'_{\max} = \text{const.}$, Fig. 28. If the angle of incidence α of the X-ray beam is small, the penetration depth is nearly constant versus 2Θ . This can be checked by the following: for $\psi_1 = 0^\circ$ and $\omega' = \Theta - \alpha$ Equ. 128 looks like

$$2\mu\tau_{0\omega'} = \frac{\sin^2 \Theta - \sin^2 (\Theta - \alpha)}{\sin \Theta \cos (\Theta - \alpha)} = \frac{[\sin \Theta + \sin (\Theta - \alpha)] \cdot [\sin \Theta - \sin (\Theta - \alpha)]}{\sin \Theta \cos (\Theta - \alpha)} \quad (129)$$

$$= \frac{1}{\sin \Theta \cos (\Theta - \alpha)} \cdot \left[2 \sin \left(\frac{2\Theta - \alpha}{2} \right) \cos \frac{\alpha}{2} \cdot 2 \cos \left(\frac{2\Theta - \alpha}{2} \right) \sin \frac{\alpha}{2} \right]$$

$$= \frac{\sin \alpha \sin (2\Theta - \alpha)}{\sin \Theta \cos (\Theta - \alpha)} = \frac{\sin \alpha}{\sin \Theta \cos (\Theta - \alpha)} \cdot [\sin \Theta \cos (\Theta - \alpha) + \cos \Theta \sin (\Theta - \alpha)] \quad (130)$$

$$= \sin \alpha [1 + \cot \Theta \tan (\Theta - \alpha)]$$

$$\text{for } \alpha \ll \Theta: \quad 2\mu\tau_{0, \omega'} \approx 2 \sin \alpha$$

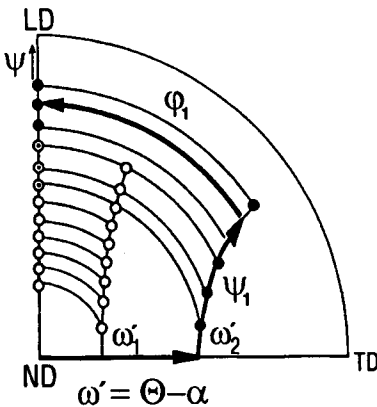


Figure 28. Representation of the simultaneous ω' - and ψ_1 -tilting and ϕ_1 -rotation by the help of stereographic projection /67/.

To establish the strain formula for an ω' -tilt a tensor transformation has to be made. The usual transformation between specimen- and laboratory-system (ω' -tilt = 0°) has to be modified when $\omega' \neq 0$.

$$\begin{aligned}\cos\varphi \sin\psi &= \sin\omega' \sin\varphi_1 + \cos\omega' \sin\psi_1 \cos\varphi_1 \\ \sin\varphi \sin\psi &= -\sin\omega' \cos\varphi_1 + \cos\omega' \sin\psi_1 \sin\varphi_1 \\ \cos\psi &= \cos\omega' \cos\psi_1\end{aligned}\quad (131)$$

The strain equation $\varepsilon_{\varphi,\psi,\omega'}$ as a function of the ε_{ik} is the following /66/:

$$\begin{aligned}\varepsilon_{\varphi_1,\psi_1,\omega'} &= \varepsilon'_{33} \\ &= \left[\cos^2\omega' \sin^2\psi_1 \cos^2\varphi_1 + \frac{1}{2}\sin(2\omega')\sin(2\varphi_1)\sin\psi_1 + \sin^2\omega' \sin^2\varphi_1 \right] \cdot \varepsilon_{11} \\ &\quad + \left[\sin(2\varphi_1)(\cos^2\omega' \sin^2\psi_1 - \sin^2\omega') + \sin(2\omega') \sin\psi_1 (2\sin^2\varphi_1 - 1) \right] \cdot \varepsilon_{12} \\ &\quad + \left[\sin(2\psi_1) \cos^2\omega' \cos\varphi_1 + \sin(2\omega') \sin\varphi_1 \cos\psi_1 \right] \cdot \varepsilon_{13} \\ &\quad + \left[\cos^2\omega' \sin^2\psi_1 \sin^2\varphi_1 - \frac{1}{2}\sin(2\omega')\sin(2\varphi_1)\sin\psi_1 + \sin^2\omega' \cos^2\varphi_1 \right] \cdot \varepsilon_{22} \\ &\quad + \left[\sin(2\psi_1) \cos^2\omega' \sin\varphi_1 - \sin(2\omega') \cos\varphi_1 \cos\psi_1 \right] \cdot \varepsilon_{23} \\ &\quad + \left[\cos^2\omega' \cos^2\psi_1 \right] \cdot \varepsilon_{33}\end{aligned}\quad (132)$$

with the limiting conditions

$$\omega' = \Theta - \alpha, \quad \omega' \leq \psi \leq 90^\circ \quad (133)$$

$$\sin^2\psi_1 = 1 - \frac{1 - \sin^2\psi}{\cos^2\omega'} \quad (134)$$

Fig. 29 demonstrates the relationships.

An example will illustrate the above mentioned thoughts. The following stress and strain tensors will be illustrated in D-vs.- $\sin^2\psi$ diagrams for ω' -tilts of 0° , 30° , 60° , 75° and azimuths φ of 0° , 45° and 90° .

$$\underline{\underline{\sigma}} = \begin{pmatrix} -300 & 0 & -100 \\ 0 & -400 & 0 \\ -100 & 0 & 20 \end{pmatrix} \text{ MPa} \quad \underline{\underline{\varepsilon}} = \begin{pmatrix} -9.22 & 0 & -6.10 \\ 0 & -15.31 & 0 \\ -6.10 & 0 & 10.29 \end{pmatrix} \cdot 10^{-4}$$

with the mechanical values of $E = 210$ GPa and $\nu = 0.28$. The variety of $D_{\varphi,\psi}$ vs. $\sin^2\psi$ decreases rapidly with increasing ω' , Fig. 30. The limiting positions for Cr-K α {211} peak of iron specimen are reached with $\alpha = 0^\circ$, $\omega'_{\max} = 78^\circ$. To determine the total stress tensor the

ω' -tilt should be restricted to approximately 30° . At least six measurements $(\varphi_1, \psi_1, \omega')$ should be made to evaluate the strain tensor, see section 2.072e. With Hooke's law the stress tensor can be calculated.

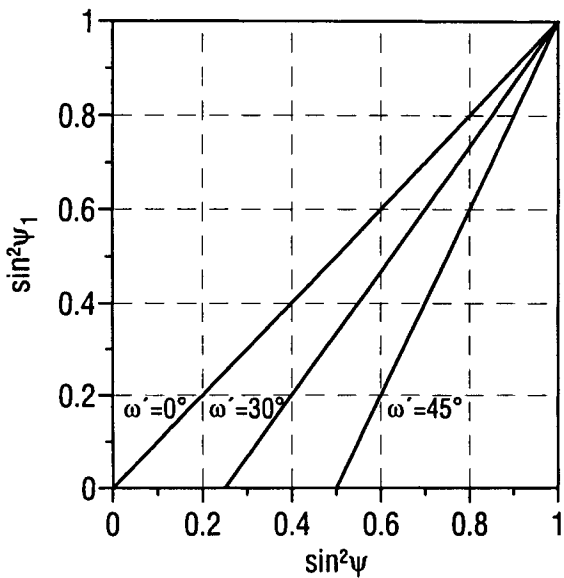


Figure 29. Relation between the ψ_1 tilt of the diffractometer system and the ψ -tilt of the specimen system

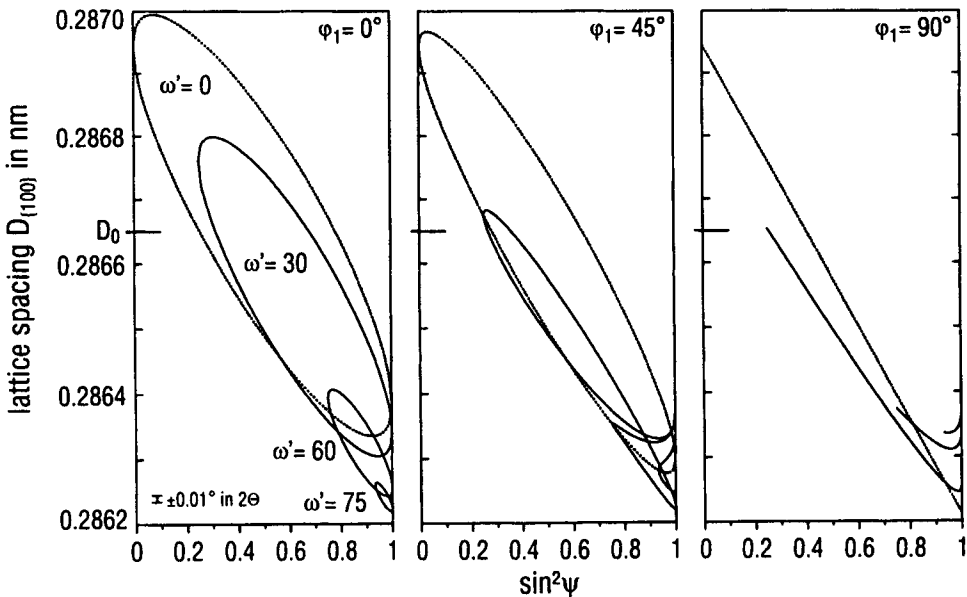


Figure 30. Example of the influence of the tilt ω' ; details in the text.

2.072k A method for the analysis of surface layers

A method was published by /68/ that allows one to determine the RS-state of a removed surface layer. This procedure can be used for thin layer-substrate composites or on bulk material with very steep stress gradients. The principle is to subtract the intensity versus 2Θ -peak of the remainder material from the respective dependence measured on the original surface. These difference peaks versus $\sin^2\psi$ enables one to evaluate the RS-state of the surface layer. An example will illustrate the foregoing /69/.

Fig. 31 shows a theoretical RS-state of -500 MPa compression constant over a thickness of $3\ \mu\text{m}$ and the case where a surface layer of $2\ \mu\text{m}$ thickness is removed. The following data are taken to calculate the D-vs.- $\sin^2\psi$ dependences of the original surface a, the surface after removing a $2\ \mu\text{m}$ thick layer b and of the layer c itself:

$\text{Fe}\{211\}$, $\text{Cr-K}\alpha$, $D_0\{100\} = 0.286648\ \text{nm}$, $\mu = 905\ \text{cm}^{-1}$, Ψ -diffractometer. The material will be mechanically isotropic. The formulae are

$$I_{z=0-c}(2\Theta) = \frac{I_{z=0}(2\Theta) - I_{z=c}(2\Theta) \exp\left\{-\frac{c}{\tau}\right\}}{1 - \exp\left\{-\frac{c}{\tau}\right\}} \quad ; \quad \frac{c}{\tau} = \frac{4\mu}{\sin\Theta \cos\psi} \quad (135)$$

The D-vs.- $\sin^2\psi$ distributions having a curvature depending on the RS-gradient are illustrated in Fig. 32 for the cases a and b but is linear for the $2\ \mu\text{m}$ thick layer as shown in Fig. 32,c. A correct value of RS = -500 MPa is obtained.

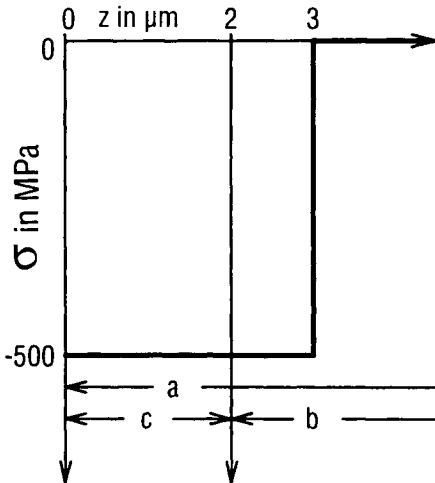


Figure 31. Compressive RS-state of -500 MPa in a surface layer of $3\ \mu\text{m}$ thickness before and after removal of a $2\ \mu\text{m}$ thick layer.

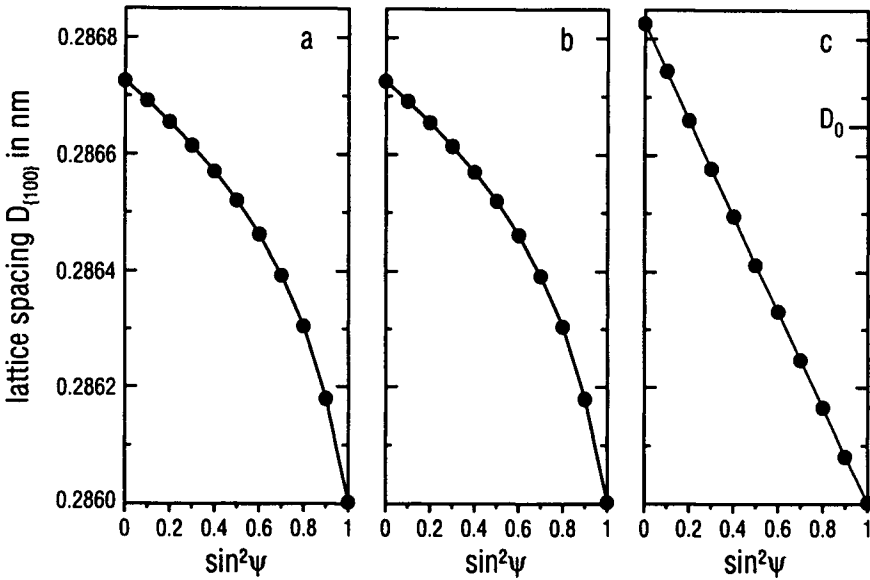


Figure 32. D-vs.- $\sin^2 \psi$ distributions (a) before and (b) after removal achieved at the respective surface and the D-vs.- $\sin^2 \psi$ dependence of the removed layer.

2.0721 The low-angle-incidence method

The low-angle-incidence XSA was introduced to study the RS in thin films /70/. Recently published papers give new results /71, 72/. A special case is the grazing incidence (angle γ_0) where total reflection occurs:

$$\gamma_0 = \sqrt{2(1-n)} = \frac{e \cdot \lambda}{c} \sqrt{\frac{N}{\pi \cdot m}}$$

This formula was already cited in paragraph 2.041. The refraction coefficient is n , e and m are the charge and the mass of an electron, N is the number of electrons per volume, c the velocity of light and λ the wavelength.

The arrangement is shown in Fig. 6, paragraph 2.041, the diffractometer used is an Ω -diffractometer, the focusing condition is not the Bragg-Brentano method but the Seemann-Bohlin one will be used /73/. The very small incidence angle $\alpha > \gamma_0$ is kept constant during the measurement. Measurements at different ψ -angles have to be realized by monitoring a few interference lines at different Bragg's angles 2Θ .

The formula for the penetration depth (Ω -mode) is /71, 72/

$$\tau_\alpha = \frac{1}{\mu} \cdot \frac{\sin \alpha \sin(2\Theta - \alpha)}{\sin \alpha + \sin(2\Theta - \alpha)} \quad \text{for } \alpha = \Theta - \omega' \quad (136)$$

This is the formula for $\psi = 0$:

$$\begin{aligned}
 \mu \cdot \tau_\alpha &= \frac{\sin(\Theta - \omega') \sin(\Theta + \omega')}{\sin(\Theta - \omega') + \sin(\Theta + \omega')} = \frac{-\frac{1}{2}(\cos 2\Theta - \cos 2\omega')}{2 \sin \Theta \cos \omega'} \\
 &= \frac{-\frac{1}{2}(\cos^2 \Theta - \sin^2 \Theta - \cos^2 \omega' + \sin^2 \omega')}{2 \sin \Theta \cos \omega'} \\
 &= \frac{\sin^2 \Theta - \sin^2 \omega'}{2 \sin \Theta \cos \omega'}
 \end{aligned} \tag{137}$$

For Bragg-Brentano-focusing principle the penetration depth is obtained according to Equ. 136 and /70/

$$\alpha = \Theta: \tau_\alpha = \frac{\sin \alpha}{2\mu}, \quad \psi = 0 \tag{138}$$

and for a low-incidence arrangement

$$\text{small } \alpha = \Theta - \psi: \tau_\alpha \approx \frac{\sin \alpha}{\mu}, \quad \psi \approx \Theta \tag{139}$$

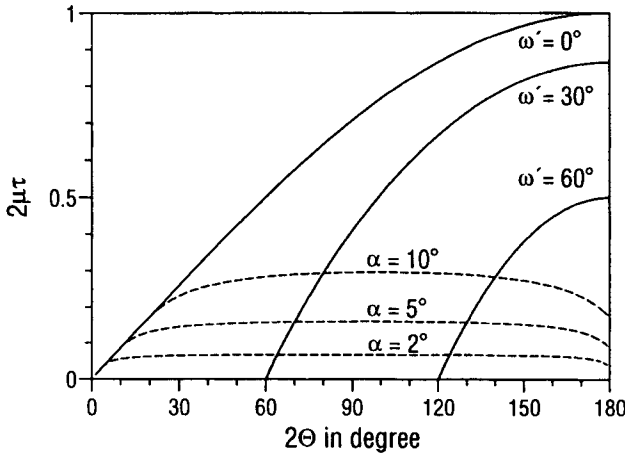


Figure 33. Penetration depth (attenuation-factor normalized) versus 2Θ with the ω' -tilt as well as with α the low incidence angle as parameters.

Fig. 33 shows the dependence of the penetration depth of the two methods on 2Θ . For a fixed incidence angle α the penetration depth is nearly constant. In contrast to the usual technique, τ does not depend on the measurement direction $\psi = \Theta - \alpha$, but it can be varied with α . This allows one to study the stresses in very small but distinct depths. Surface gradients can be tested by successive measurements varying the angle of incidence.

From Equ. 8 it follows for a biaxial RS-state: $\sigma_1 = \sigma_2 = \sigma$, $\sigma_3 = 0$

$$\varepsilon_\psi = \frac{1}{2} s_2 \cdot \sigma \sin^2 \psi + 2s_1 \cdot \sigma$$

The rotationally symmetric RS σ will be determined from the slope of the linear dependence

$$D_\psi \text{ vs. } \frac{1}{2}s_2 \sin^2 \psi + 2s_1.$$

The strain-free and stress-independent direction is given by

$$\sin^2 \psi^* = \frac{-2s_1}{\frac{1}{2}s_2}, \text{ that is at } \frac{1}{2}s_2 \sin^2 \psi^* + 2s_1 = 0.$$

This procedure may be explained by an example: Ni-layer, $\sigma = -300$ MPa, $D_0 = 0.35238$ nm, Cu-K α -radiation, Fig. 34. The positions of the peaks are plotted for the angle α between the X-ray beam and the surface of 0° , 5° and 10° respectively.

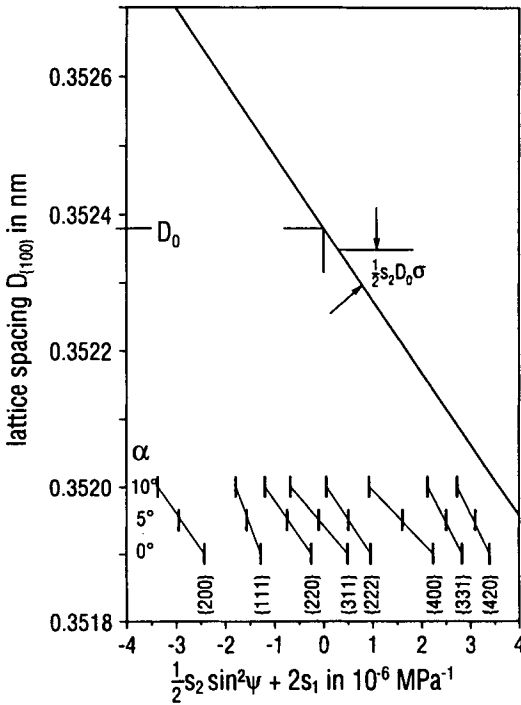


Figure 34. Calculated result of a low-incidence measurement (Seemann-Bohlin-focusing principle), details of rotationally symmetric biaxial stress state in the text.

From Equ. 8 it follows for a triaxial RS-state: $\sigma_1 = \sigma_2 = \sigma$, $\sigma_3 \neq 0$

$$\varepsilon_\psi = \frac{1}{2}s_2 [(\sigma - \sigma_3) \sin^2 \psi + \sigma_3] + s_1 (2\sigma + \sigma_3)$$

From this one gets the strain-free direction:

$$\sin^2 \psi^* = \frac{-\frac{1}{2}s_2 \sigma_3 - s_1 (2\sigma + \sigma_3)}{\frac{1}{2}s_2 (\sigma - \sigma_3)}$$

and the σ_1 -independent direction:

$$\sin^2 \psi' = \frac{-2s_1}{\frac{1}{2}s_2}$$

In this case the σ_3 -independent direction should be considered, too:

$$\sin^2 \psi'' = \frac{\frac{1}{2}s_2 + s_1}{\frac{1}{2}s_2}$$

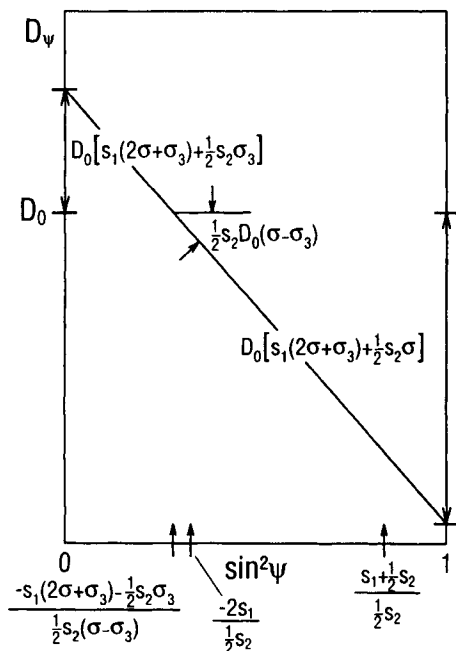


Figure 35. Triaxial ($\sigma_1 = \sigma_2 = \sigma$, σ_3) stress state: the D -vs.- $\sin^2 \psi$ dependence, the strain-free- and the stress-independent-direction as well as the σ_3 -independent direction.

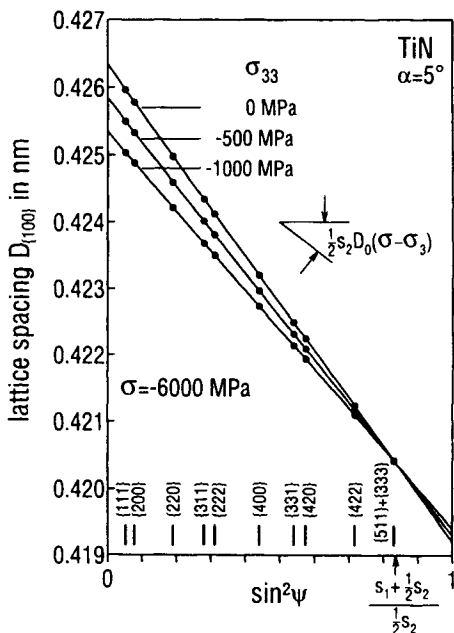


Figure 36. $D_{\{100\}}$ -vs.- $\sin^2 \psi$ straight lines with σ_3 as parameter; details in the text.

The graphical representation of the above explained formulae is plotted in Fig. 35. To calculate the stresses σ and σ_3 , the slope and the indicated distance on the ordinate are to be evaluated. To find the exact D_0 -value the values of the stresses are necessary.

An example will illustrate the above mentioned considerations: TiN layer, $D_0 = 0.42400$ nm, $\sigma = -6000$ MPa, $\sigma_3 = 0, -500$ MPa, -1000 MPa, $\alpha = 5^\circ$, Fig. 36. The mechanical XEC are used, otherwise there would not be straight-line dependences. This is not a severe restriction because the elastic anisotropy of ceramics and hard materials is small.

A very low-angle-incidence method with decreasing α down to 0.6° (total reflection angle $\gamma_0 = 0.5^\circ$) was used to determine the micro-strain profile from the FWHM observed at the surface of mechanical treated ceramics /74/. Fig. 37 shows the relation of micro-strain and the residual stress determined by the usual method of two surface-treated ceramics.

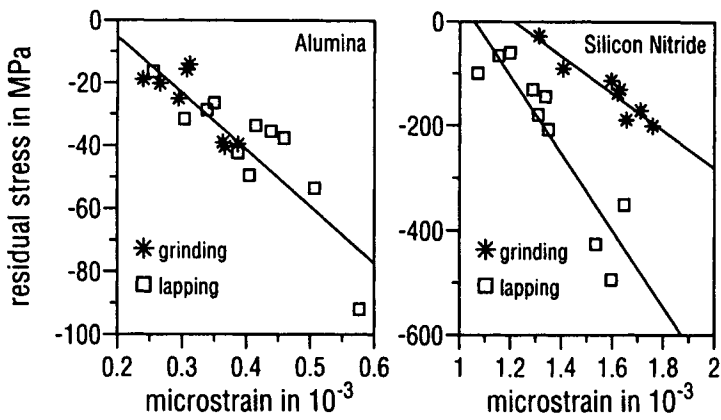


Figure 37. Relationship between micro-strains and residual stresses of surface layers of machined Al_2O_3 and Si_3N_4 /74/.

The characteristics of the ψ - and the grazing-incidence X-ray diffraction (GIXD) are listed in the following.

characteristics	ψ -method	GIXD
mode	Ω, Ψ	Ω
$\Theta/2\Theta$ -rotation of specimen	yes	no
focusing	Bragg-Brentano	Seemann-Bohlin, Bragg-Brentano defocusing
scan range 2Θ of detector	small	large, $\alpha = \text{const.}$
penetration depth	relatively large	small
gauge area	increasing with ψ	constant
interference line	one	several
ψ -values	$\pm\psi$ arbitrary in Ψ -mode	$\psi = \alpha - \Theta(\{hkl\}, \lambda)$
elastic anisotropy effects from stress evaluation from D vs. $\sin^2\psi$ depends on	one $\{hkl\}$ respective XEC	several $\{hkl\}$ averaged XEC
D vs. $\sin^2\psi$ for quasiisotropic material	linear	deviations (elastic anisotropy)
possible influence of texture on D vs. $\sin^2\psi$	oscillations	"scatter"

2.072m An ultra-low-angle-incidence method

For extremely thin layers the incidence angle α has to be reduced to nearly zero degrees. Therefore the divergence of the X-ray beam must be also made sufficiently small by using a special Soller slit and a mirror, Fig. 38 /75/. Fig. 39 shows a diffraction pattern from a Au film only 1.3 nm thick. The evaluation result is a high stress state of $1.6 \cdot 10^3$ MPa and a relatively small lattice constant of 0.405 nm.

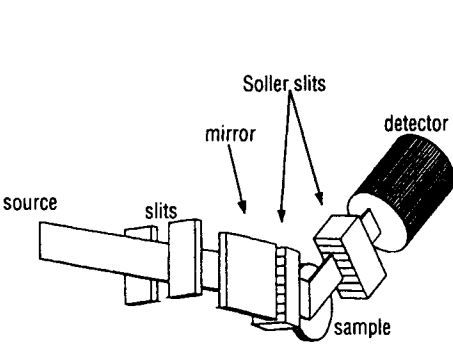


Figure 38. Schematic diagram of major components of a thin-film diffractometer used for GIXD (Grazing Incidence X-ray Diffraction) /75/.

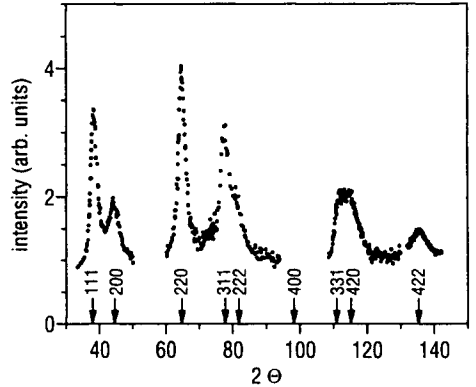


Figure 39. GIXD scan for a Au film of 13 Å average thickness. Each reflection is indexed as indicated /75/.

2.073 Textured materials, strongly deformed materials, lattice-strain distributions with oscillations

Many studies have been made on strongly nonlinear lattice-strain distributions dealing with the types of oscillations, their origins and the methods to evaluate stresses, especially for the RS-state of textured and/or severely deformed materials. Here the methods to evaluate the stresses will be considered. More about the physical background of the oscillations will be dealt with in the respective chapters.

The Figures 40, 41 demonstrate the variety of D-vs.- $\sin^2\psi$ distributions measured on different peaks of the ferritic and the austenitic phase of plastically elongated steels. The X-ray measurements were made at the center of the specimen on a thin plate; the neutron results are the average of RS II over the cross section when the specimen was totally bathed in the neutron beam /76/. The different D-vs.- $\sin^2\psi$ distributions demonstrate very clearly

- good agreement between X- and neutron-ray measurements
- very different shapes of the curves for different $\{hkl\}$
- when the measurements – especially with X-rays – do not cover a very large part of the range $0 \leq \sin^2\psi \leq 1$, unreliable and erroneous stresses will be evaluated.

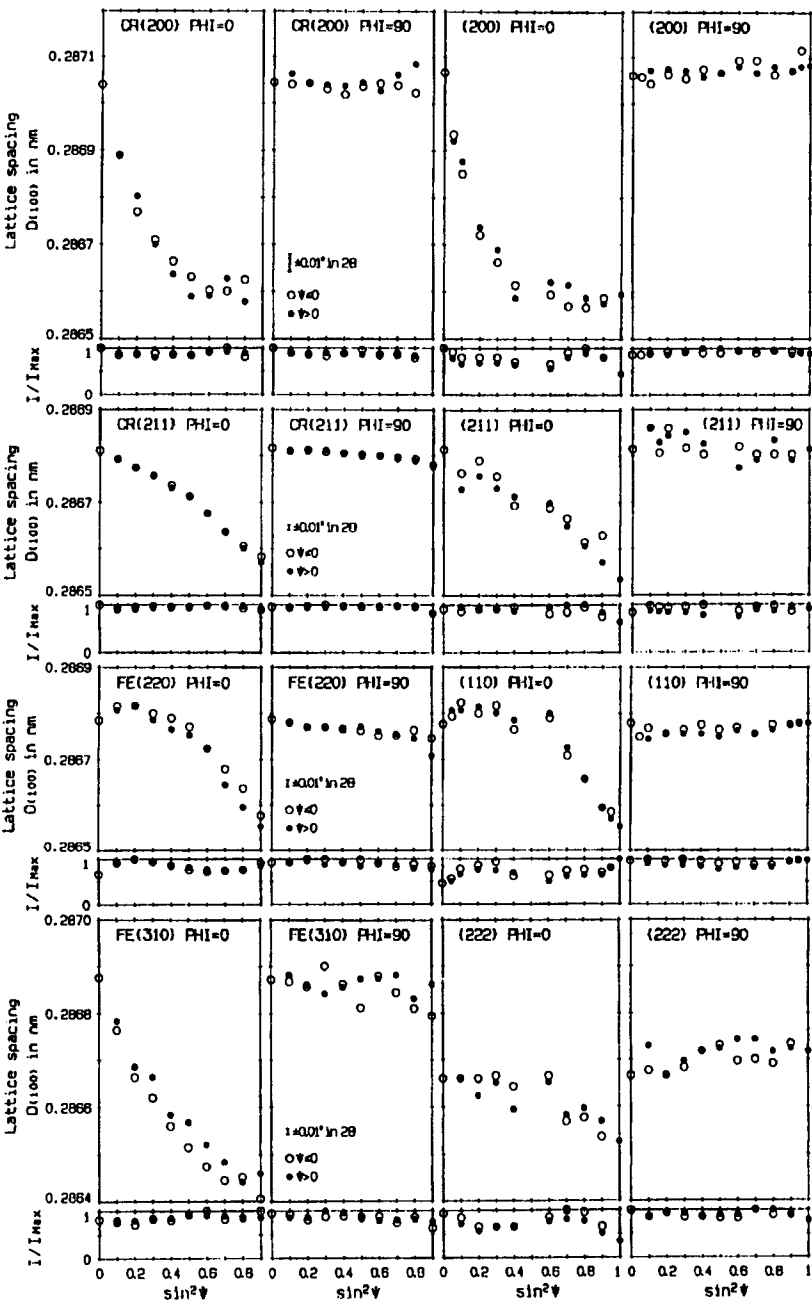


Figure 40. Lattice parameter and relative intensity versus $\sin^2\psi$ as measured on a 25CrMo4 steel after 8% plastic strain using different {hkl} peaks. Left-hand side: X-ray results for a specimen after removing 770 μm by electropolishing. Right-hand side: neutron results, which represent averages over the whole cross section /76/.

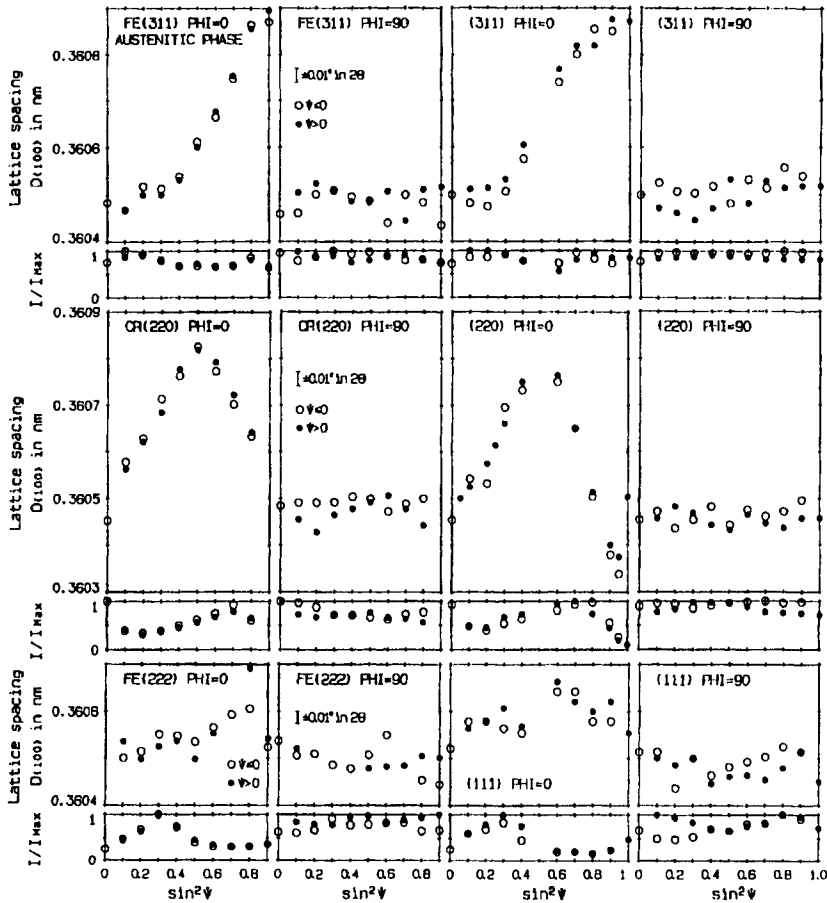


Figure 41. Lattice parameter and relative intensity versus $\sin^2\psi$ as measured on the austenitic phase of a X2CrNiMoN22-5 steel after 12% plastic strain using different $\{hkl\}$ peaks. Left-hand side: X-ray results obtained at the center of the specimen. Right-hand side: neutron results, which represent averages over the whole cross section /76/.

2.073a Linearization of D-vs.- $\sin^2\psi$ distributions with oscillations

The most experimentally investigated peak is the $\{211\}$ one measured with Cr- K_α radiation on the ferritic phase of steels. The next frequently used interference may be the double peak $\{732+651\}$ also on the ferritic phase of steels but registered with Mo- K_α radiation. In general they will have a different shape, characteristic oscillations at the $\{211\}$ peak and an approximately linear dependence in case of the $\{732+651\}$ interference. Fig. 42, 43 show examples of those D-vs.- $\sin^2\psi$ distributions /76/. The linear regression analysis of the $\{211\}$ peak shifts in connection with the XEC of the quasiisotropic iron $\frac{1}{2}s_2 = 5.76 \cdot 10^{-6} \text{ MPa}^{-1}$ results in values discussed in /4/, Table 7. The linearity of the Mo- K_α peak $\{732+651\}$ is explicable by

the presence of many texture poles in the neighborhood of the rolling or the strain direction, i.e. azimuth 0°, Fig. 44, the high multiplicity of this twofold peak and the larger penetration depth. The RS of the explained example using the averaged XEC are also listed in Table 7.

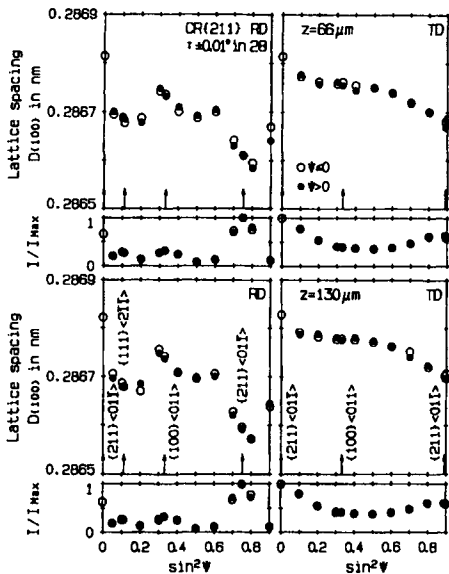


Figure 42. Distributions of D vs. $\sin^2\psi$ and intensity vs. $\sin^2\psi$ for the {211} peak in RD and TD for two different depths z, two specimens of rolled steel 4/.

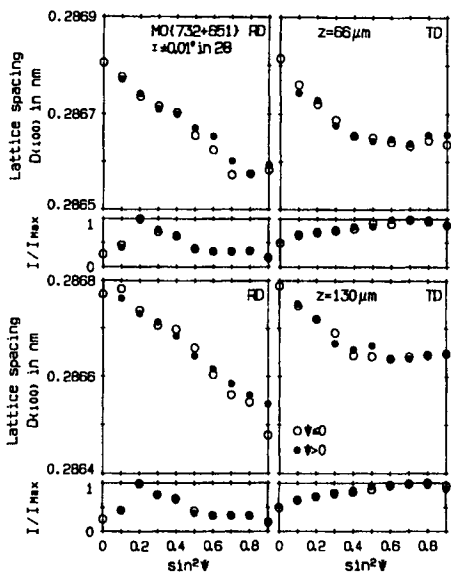


Figure 43. Distributions of D vs. $\sin^2\psi$ and intensity vs. $\sin^2\psi$ for the {732+651} peak in RD and TD for two different depths z, two specimens of rolled steel 4/.

Table 7. RS (in MPa) evaluated by the crystallite-group method and by linear regression analysis of the D-vs.- $\sin^2\psi$ distributions, rolled steel 4/.

	crystallite-group method			linear regression analysis			
	$\{100\}\langle 011 \rangle$	$\{111\}\langle 2\bar{1}\bar{1} \rangle$	$\{211\}\langle 01\bar{1} \rangle$	Cr $\{211\}$	Fe $\{220\}$	Cu $\{220\}$	Mo $\{732+651\}$
$\sigma_1\text{-}\sigma_3$	-183	-185	-218	-182 ± 21	-213 ± 26	-162 ± 13	-216 ± 10
$\sigma_2\text{-}\sigma_3$	-162	-221	-211	-176 ± 80	-166 ± 5	-161 ± 10	-161 ± 7
σ_3	41	24	36				

When lattice strain distributions with oscillation are linearized and the stress state calculated using the XEC of mechanically isotropic material the result has to be checked carefully. The evaluated macrostress should be checked either by other measurements on different peaks (see next section) or by a mechanical method. Experience has shown that linearization often works quite well but no physical proof is known. Table 8 shows the extent of nonlinearities observed on different peaks grouped according to their origin /78/. D-vs.- $\sin^2\psi$ distributions of peaks with strongly preferred orientations should not be evaluated by linearization.

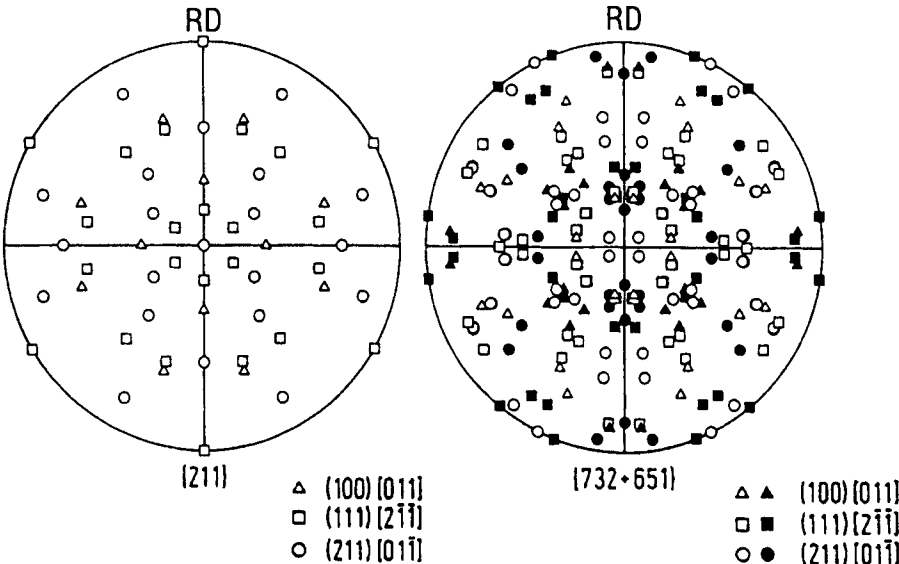


Figure 44. Calculated polefigures showing ideal orientations of rolled steel, left {211}, right {732+651} peak /77/.

Table 8. Qualitative classification of the nonlinearities observed in D-vs.-sin²ψ distributions of cubic materials caused by texture, uniaxial plastic straining or different kinds of rolling /78/.

hkl	Texture (+ elastical tension)	Deformation	
		uniaxially tensile deformed	rolled
200	linear	strongly convex	oscillating
311	oscillating	slightly convex	slightly oscillating
211	strongly oscillating	≈ linear	slightly oscillating
220	≈ linear	strongly concave	oscillating
222	linear	≈ linear	oscillating

Linear D-vs.-sin²ψ dependences can be achieved if those of different peaks are averaged. The multiplicity factors of the lattice planes and the relative intensities as well as the XEC for the stress evaluation should be used. Two examples will illustrate this procedure. Fig. 45, 46 show the weighted averaged D- and intensity-versus-sin²ψ linear dependences, Fig. 45 of a quenched-and-tempered steel, and Fig. 46 of the ferritic and the austenitic phases of a duplex steel, both after plastic elongation /76/. The details of the figures are selfexplaining.

The multi-peak summation is a useful method to evaluate the macro- plus microstress from several individual D-vs.-sin²ψ distributions. The method is time consuming but helpful to solve some severe problems of lattice-strain distributions with strong oscillations. Details on the nature of micro-RS states cannot be found out in this way. To do that other ways must be followed; see the chapter on texture and plastic deformation 2.16.

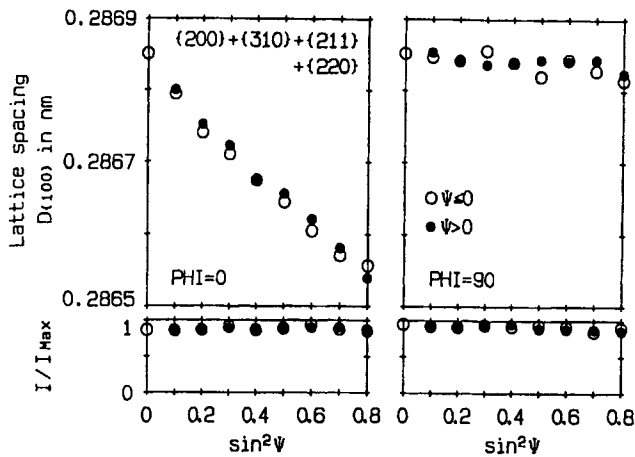


Figure 45. D-vs.- $\sin^2\psi$ distribution averaged over four lattice planes of an 8% uniaxial plastically deformed but still nearly untextured 25CrMo4 steel, the individual distributions of which are partly extremely nonlinear, Figure 40 /76/.

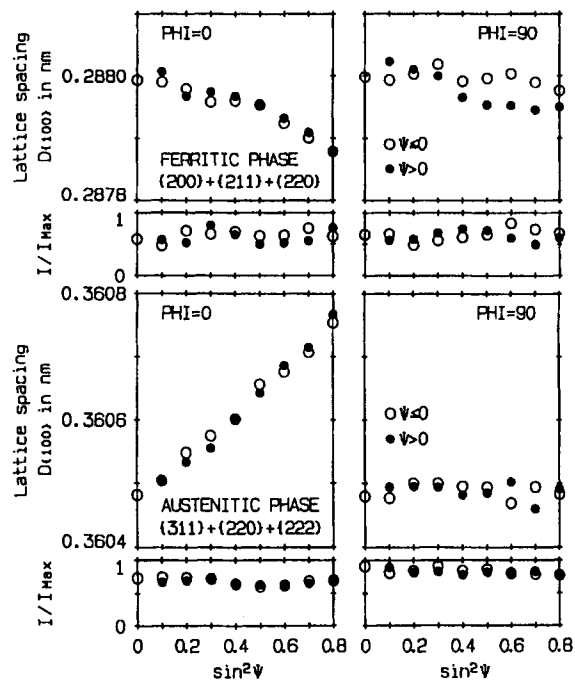


Figure 46. Lattice parameter and relative-intensity-versus- $\sin^2\psi$ measured by X-rays on the central part of a X2CrNiMoN22-5 steel specimen after 12% uniaxial plastic deformation in the elongation direction. The plotted values are weighted averages of those obtained for the {200}, {211} and {220} peaks of the ferritic phase respectively the {311}, {220} and {222} peaks of the austenitic phase /76/.

2.073b The crystallite-group method.

This method was introduced by /79,80/ evaluating RS-states of drawn wires. The procedure was further developed for rolled materials by /81,82,83/ and for fiber texture by /84/. Theoretical thoughts were published by /85/.

Stress evaluation by the crystallite-group method is confined to materials with strong textures. The texture has to be described by ideal orientations. The crystallite groups, i.e. all crystallites with the same orientation, are treated as being one crystal. That means, the method presumes the stresses within all crystals of one crystallite group being the same, whereas other crystallite groups may have different stresses.

After determining the strains at the intensity poles of a crystallite group, its stress state can be evaluated using the monocrystal elastic data and the respective formulae developed in the following.

The strain measured in a direction $\underline{m}$ is always the average of that value of all crystallites having the lattice plane $\{hkl\}$ under study oriented perpendicular to $\underline{m}$. The strains are weighted by the frequency of the orientation (ODF, orientation-distribution function).

The main presupposition of the crystallite-group method is that in direction corresponding to the intensity poles of the ideal orientations only the considered group contributes to the respective interference line, and therefore one determines the exact strain value of the group. All influences of other crystallites reflecting in this direction $\underline{m}$ too, are neglected. This assumption is justified only if the texture is very strong. This was discussed in /86/. The strains in the different intensity poles have to be determined very accurately. Different radiations and measurements on different peaks in different 2Θ -ranges may be necessary.

The formulae for evaluating the strains of a crystallite group are developed beginning with the strain of a crystal in a certain measuring direction $\underline{m}$ and with Hooke's law (chapter 2.03):

$$\epsilon_{\underline{m}} = \epsilon_{ij} m_i m_j = s_{ijmn}^c \sigma_{mn}^c m_i^c m_j^c \quad (140)$$

The index "C" refers to the crystal system C. However stresses should be determined with respect to the specimen system S, here denoted by the index "S". The relationship between directions in C and S is given by the transformation matrix π . The notation of the components of π will be used as introduced by /81/:

$$(\pi_{ij}) = \begin{pmatrix} \pi_{11} & \pi_{12} & \pi_{13} \\ \pi_{21} & \pi_{22} & \pi_{23} \\ \pi_{31} & \pi_{32} & \pi_{33} \end{pmatrix} = \begin{pmatrix} \alpha_1 & \beta_1 & \gamma_1 \\ \alpha_2 & \beta_2 & \gamma_2 \\ \alpha_3 & \beta_3 & \gamma_3 \end{pmatrix} \quad ; \quad \underline{m}^c = \begin{pmatrix} \alpha \\ \beta \\ \gamma \end{pmatrix} \quad (141)$$

The stresses σ^C and the measuring direction $\underline{m}$ can now be expressed with respect to the specimen system, $\pi_{ij}^T = \pi_{ji}$.

$$\sigma_{mn}^C = \pi_{mk}^T \pi_{nl}^T \sigma_{kl}^S = \pi_{km} \pi_{ln} \sigma_{kl}^S \quad (142)$$

$$m_i^C = \pi_{ir}^T m_r^S = \pi_{ri} m_r^S \quad (143)$$

With Equ. 142 and Equ. 143 one gets from Equ. 140:

$$\begin{aligned}\varepsilon_{\underline{m}} &= s_{ijmn}^C (\pi_{km} \pi_{ln} \sigma_{kl}^S) m_i^C m_j^C = s_{ijmn}^C (\pi_{km} \pi_{ln} \sigma_{kl}^S) (m_r^S \pi_{ri} m_s^S \pi_{sj}) \\ &= s_{ijmn}^C \sigma_{kl}^S m_r^S m_s^S \pi_{km} \pi_{ln} \pi_{ri} \pi_{sj}\end{aligned}\quad (144)$$

Equ. 144 is the general relation between the crystallite-group strains and the stresses in the specimen system. It is valid for all crystal symmetries, all stress states and all crystallite groups /87/. For a similar formula see /88/. The measuring direction is given by the angles φ, ψ :

$$\underline{m}^S = \begin{pmatrix} \cos \varphi \sin \psi \\ \sin \varphi \sin \psi \\ \cos \psi \end{pmatrix} \quad (145)$$

Introducing Equ. 145 into Equ. 144 one gets for $\varphi = 0^\circ$ an equation with terms in $\sin^2 \psi$ and $\sin \psi \cos \psi = \frac{1}{2} \sin 2\psi$. This means a linear dependence or an elliptical dependence on $\sin^2 \psi$ as it is the case in the usual stress evaluation procedure. The coefficients of the terms depend on the stresses σ_{ij} , the monocrystal compliances and the components of the transformation matrix π . The strains of the crystallite group which are determined at special azimuths φ can be drawn as usual versus $\sin^2 \psi$ and $\sin 2\psi$ respectively. From the slopes and the intercepts the stresses can be derived.

In case of cubic materials only the compliances s_{11} , s_{12} and s_{44} are independent. Furthermore a principal stress state is assumed. From Equ. 144 with the notation of Equ. 141 the following relation holds /89,90,91/:

$$\varepsilon_{\underline{m}} = \left[s_{12} + s_0 (\alpha^2 \alpha_i^2 + \beta^2 \beta_i^2 + \gamma^2 \gamma_i^2) + \frac{1}{2} s_{44} (\alpha \alpha_i + \beta \beta_i + \gamma \gamma_i)^2 \right] \cdot \sigma_i \quad (146)$$

with $s_0 = s_{11} - s_{12} - \frac{1}{2} s_{44}$.

$\underline{m}^C = \begin{pmatrix} \alpha \\ \beta \\ \gamma \end{pmatrix}$ is given by the normal to the studied lattice plane $\{hkl\}$. Transformed to the specimen system it is given by Equ. 145. The row vectors of π are the specimen axes expressed in the crystal system. Therefore the following relations hold /81/:

	$\varphi = 0^\circ$	$\varphi = 90^\circ$
$\alpha \alpha_1 + \beta \beta_1 + \gamma \gamma_1 =$	$\sin \psi$	0
$\alpha \alpha_2 + \beta \beta_2 + \gamma \gamma_2 =$	0	$\sin \psi$
$\alpha \alpha_3 + \beta \beta_3 + \gamma \gamma_3 =$	$\cos \psi$	$\cos \psi$

(147)

And Equ. 143 reads in more detail:

	$\varphi = 0^\circ$	$\varphi = 90^\circ$	
$\alpha =$	$\alpha_1 \sin \psi + \alpha_3 \cos \psi$	$\alpha_2 \sin \psi + \alpha_3 \cos \psi$	(148)
$\beta =$	$\beta_1 \sin \psi + \beta_3 \cos \psi$	$\beta_2 \sin \psi + \beta_3 \cos \psi$	
$\gamma =$	$\gamma_1 \sin \psi + \gamma_3 \cos \psi$	$\gamma_2 \sin \psi + \gamma_3 \cos \psi$	

Inserting Equ. 147 and 148 into Equ. 146 we again get the dependence of the crystallite-group strain versus $\sin^2 \psi$ and $\sin 2\psi$, but now for the special case of cubic symmetry.

The components of the transformation matrix depend on the crystallite group itself. The ideal orientations are described by $\{mnr\}\langle uvw \rangle$, $\{mnr\}$ are the Miller's indices of the lattice planes lying parallel to the specimen surface and $\langle uvw \rangle$ the indices of the specimen's 1-axis in the crystal system. For cubic materials, the direction $\langle mnr \rangle$ is aligned with the normal to the lattice plane $\{mnr\}$. Therefore the specimen's third axis is $\langle mnr \rangle$.

The transverse direction follows from

$$\begin{pmatrix} x \\ y \\ z \end{pmatrix} = \begin{pmatrix} m \\ n \\ r \end{pmatrix} \times \begin{pmatrix} u \\ v \\ w \end{pmatrix} \quad (149)$$

The transformation matrix π between the crystal and the specimen system is built up by the unit vectors in the directions $\langle uvw \rangle$, $\langle xyz \rangle$ and $\langle mnr \rangle$.

$$\pi = \begin{pmatrix} \frac{u}{U} & \frac{v}{U} & \frac{w}{U} \\ \frac{x}{X} & \frac{y}{X} & \frac{z}{X} \\ \frac{m}{M} & \frac{n}{M} & \frac{r}{M} \end{pmatrix} \quad (150)$$

with

$$U = \sqrt{u^2 + v^2 + w^2}, \quad X = \sqrt{x^2 + y^2 + z^2}, \quad M = \sqrt{m^2 + n^2 + r^2} \quad (151)$$

Three common crystallite groups in rolled steels are $\{100\}\langle 011 \rangle$, $\{111\}\langle 2\bar{1}\bar{1} \rangle$ and $\{211\}\langle 01\bar{1} \rangle$. The respective transformation matrices are disposed in the following table with the notation of the π_{ij} according to Equ. 141. The relationship between specimen and crystallite systems of the ideal orientations of iron are as follows /81, supplemented/:

Table 9. Components of transformation matrices between crystal and specimen system /81, supplemented/.

specimen system		crystal system		
		{100}	{010}	{001}
{100}<011>				
RD	$\frac{1}{\sqrt{2}}[011]$	$\alpha_1 = 0$	$\beta_1 = \frac{1}{\sqrt{2}}$	$\gamma_1 = \frac{1}{\sqrt{2}}$
TD	$\frac{1}{\sqrt{2}}[0\bar{1}1]$	$\alpha_2 = 0$	$\beta_2 = \frac{-1}{\sqrt{2}}$	$\gamma_2 = \frac{1}{\sqrt{2}}$
ND	[100]	$\alpha_3 = 1$	$\beta_3 = 0$	$\gamma_3 = 0$
{111}<2\bar{1}\bar{1}>				
RD	$\frac{1}{\sqrt{6}}[2\bar{1}\bar{1}]$	$\alpha_1 = \frac{2}{\sqrt{6}}$	$\beta_1 = \frac{-1}{\sqrt{6}}$	$\gamma_1 = \frac{-1}{\sqrt{6}}$
TD	$\frac{1}{\sqrt{2}}[01\bar{1}]$	$\alpha_2 = 0$	$\beta_2 = \frac{1}{\sqrt{2}}$	$\gamma_2 = \frac{-1}{\sqrt{2}}$
ND	$\frac{1}{\sqrt{3}}[111]$	$\alpha_3 = \frac{1}{\sqrt{3}}$	$\beta_3 = \frac{1}{\sqrt{3}}$	$\gamma_3 = \frac{1}{\sqrt{3}}$
{211}<01\bar{1}>				
RD	$\frac{1}{\sqrt{2}}[01\bar{1}]$	$\alpha_1 = 0$	$\beta_1 = \frac{1}{\sqrt{2}}$	$\gamma_1 = \frac{-1}{\sqrt{2}}$
TD	$\frac{1}{\sqrt{3}}[\bar{1}11]$	$\alpha_2 = \frac{-1}{\sqrt{3}}$	$\beta_2 = \frac{1}{\sqrt{3}}$	$\gamma_2 = \frac{1}{\sqrt{3}}$
ND	$\frac{1}{\sqrt{6}}[211]$	$\alpha_3 = \frac{2}{\sqrt{6}}$	$\beta_3 = \frac{1}{\sqrt{6}}$	$\gamma_3 = \frac{1}{\sqrt{6}}$

With Table 9, Equ. 146, 147 and 148 the $\epsilon(\sin^2\psi, \sin 2\psi)$ relations of the crystallite groups are fixed, and they are collected in Table 10.

Knowing the ϵ -vs.- $\sin^2\psi$ relation of the crystallite group the (φ, ψ) angles of the poles, to measure the strains, are to be calculated. From Equ. 143 follows

$$\underline{m}_i^S = \pi_{ik} m_k^C$$

$$\text{with } \underline{m}^C = \frac{1}{\sqrt{h^2 + k^2 + l^2}} \begin{pmatrix} h \\ k \\ l \end{pmatrix} \quad \underline{m}^S = \begin{pmatrix} \cos\varphi \sin\psi \\ \sin\varphi \sin\psi \\ \cos\psi \end{pmatrix} \quad (152)$$

and π_{ik} according to Equ. 150.

There are two independent equations for the unknown φ and ψ , the third being dependent on the other two because of the unit length of the vector $\underline{m}$.

Table 10. Formulae for strain stress relationship of five ideal orientations, RD ($\varphi = 0^\circ$) and TD ($\varphi = 90^\circ$) /82,83/.

Ideal orientation	$\mathcal{E}_{\varphi=0,\psi}$	Zone axis	Phase, solid solution	{hkl}
$\{211\}\langle 01\bar{1}\rangle$	$\left(s_{12} + \frac{1}{6}s_0\right)\sigma_1 + \left(s_{12} + \frac{1}{3}s_0\right)\sigma_2 + \left(s_{12} + \frac{1}{2}s_{44} + \frac{1}{2}s_0\right)\sigma_3$ $+ \frac{1}{2}\left[\left(s_{44} + \frac{2}{3}s_0\right)\sigma_1 - \left(\frac{2}{3}s_0 + s_{44}\right)\sigma_3\right]\sin^2\psi$	$[\bar{1}11]$	Fe	$\{110\}\{211\}\{220\}$ $\{541\}\{642\}$
			Cu Zn	$\{211\}$
$\{011\}\langle 21\bar{1}\rangle$		$[\bar{1}1\bar{1}]$	Cu	$\{220\}\{844\}$
			Ni	
$\{111\}\langle 2\bar{1}\bar{1}\rangle$	$\left(s_{12} + \frac{1}{3}s_0\right)\sigma_1 + \left(s_{12} + \frac{1}{3}s_0\right)\sigma_2 + \left(s_{12} + \frac{1}{2}s_{44} + \frac{1}{3}s_0\right)\sigma_3$ $+ \frac{1}{6}\left[(3s_{44} + s_0)\sigma_1 - s_0\sigma_2 - 3s_{44}\sigma_3\right]\sin^2\psi + \frac{1}{6}\sqrt{2}s_0(\sigma_1 - \sigma_2)\sin 2\psi$	$[01\bar{1}]$	Fe	$\{110\}\{200\}\{211\}$ $\{220\}\{222\}$
$\{100\}\langle 011\rangle$	$s_{12}\sigma_1 + s_{12}\sigma_2 + s_{11}\sigma_3 + \frac{1}{2}\left[(s_{44} + s_0)\sigma_1 + s_0\sigma_2 - (2s_0 + s_{44})\sigma_3\right]\sin^2\psi$	$[0\bar{1}1]$	Fe	$\{110\}\{200\}\{211\}$ $\{222\}$
$\{211\}\langle \bar{1}11\rangle$	$\left(s_{12} + \frac{1}{3}s_0\right)\sigma_1 + \left(s_{12} + \frac{1}{6}s_0\right)\sigma_2 + \left(s_{12} + \frac{1}{2}s_{44} + \frac{1}{2}s_0\right)\sigma_3$ $+ \frac{1}{2}\left[s_{44}\sigma_1 + \frac{1}{3}s_0\sigma_2 - \left(\frac{1}{3}s_0 + s_{44}\right)\sigma_3\right]\sin^2\psi + \frac{1}{6}\sqrt{2}s_0(\sigma_2 - \sigma_3)\sin 2\psi$	$[0\bar{1}1]$	Cu	$\{220\}\{311\}\{222\}$
			Ni	$\{311\}$

Table 10 continued.

Ideal orientation	$\mathcal{E}\varphi=90,\psi$	Zone axis	Phase, solid solution	{hkl}
$\{211\}\langle 01\bar{1}\rangle$	$\left(s_{12} + \frac{1}{6}s_0\right)\sigma_1 + \left(s_{12} + \frac{1}{3}s_0\right)\sigma_2 + \left(s_{12} + \frac{1}{2}s_{44} + \frac{1}{2}s_0\right)\sigma_3$ $+ \frac{1}{2}\left[\frac{1}{3}s_0\sigma_1 + s_{44}\sigma_2 - \left(\frac{1}{3}s_0 + s_{44}\right)\sigma_3\right]\sin^2\psi + \frac{1}{6}\sqrt{2}s_0(\sigma_1 - \sigma_3)\sin 2\psi$	$[01\bar{1}]$	Fe	$\{110\}\{200\}\{211\}$ $\{220\}\{222\}$
			Cu Zn	$\{211\}$
$\{011\}\langle 21\bar{1}\rangle$	$\left(s_{12} + \frac{1}{6}s_0\right)\sigma_1 + \left(s_{12} + \frac{1}{3}s_0\right)\sigma_2 + \left(s_{12} + \frac{1}{2}s_{44} + \frac{1}{2}s_0\right)\sigma_3$ $+ \frac{1}{2}\left[\frac{1}{3}s_0\sigma_1 + s_{44}\sigma_2 - \left(\frac{1}{3}s_0 + s_{44}\right)\sigma_3\right]\sin^2\psi$	$[21\bar{1}]$	Cu	$\{220\}\{311\}\{420\}$
			Ni	
$\{111\}\langle 2\bar{1}\bar{1}\rangle$	$\left(s_{12} + \frac{1}{3}s_0\right)\sigma_1 + \left(s_{12} + \frac{1}{3}s_0\right)\sigma_2 + \left(s_{12} + \frac{1}{2}s_{44} + \frac{1}{3}s_0\right)\sigma_3$ $+ \frac{1}{6}\left[-s_0\sigma_1 + (3s_{44} + s_0)\sigma_2 - 3s_{44}\sigma_3\right]\sin^2\psi$	$[2\bar{1}\bar{1}]$	Fe	$\{110\}\{222\}$
$\{100\}\langle 011\rangle$	$s_{12}\sigma_1 + s_{12}\sigma_2 + s_{11}\sigma_3 + \frac{1}{2}\left[s_0\sigma_1 + (s_{44} + s_0)\sigma_2 - (2s_0 + s_{44})\sigma_3\right]\sin^2\psi$	$[011]$	Fe	$\{110\}\{200\}\{211\}$ $\{222\}$
$\{211\}\langle \bar{1}11\rangle$	$\left(s_{12} + \frac{1}{3}s_0\right)\sigma_1 + \left(s_{12} + \frac{1}{6}s_0\right)\sigma_2 + \left(s_{12} + \frac{1}{2}s_{44} + \frac{1}{2}s_0\right)\sigma_3$ $+ \frac{1}{2}\left[\left(s_{44} + \frac{2}{3}s_0\right)\sigma_2 - \left(\frac{2}{3}s_0 + s_{44}\right)\sigma_3\right]\sin^2\psi$	$[\bar{1}11]$	Cu	$\{220\}\{844\}$
			Ni	

The transverse direction $\langle xyz \rangle$ is parallel to all those lattice planes $\{hkl\}$ showing poles in $(\varphi = 0^\circ, \psi)$ directions, that means for cubic materials $\langle xyz \rangle$ is perpendicular to the directions $\langle hkl \rangle$. $\langle xyz \rangle$ is the zone axis for those lattice planes. For measurements in $(\varphi = 90^\circ, \psi)$ the 1-axis $\langle uvw \rangle$ is the zone axis for the respective poles.

From Equ. 152 these relations follow:

$$\pi \cdot \begin{pmatrix} h \\ k \\ l \end{pmatrix} \cdot \frac{1}{\sqrt{h^2 + k^2 + l^2}} = \begin{cases} \begin{pmatrix} \sin \psi \\ 0 \\ \cos \psi \end{pmatrix} & \varphi = 0^\circ \\ \begin{pmatrix} 0 \\ \sin \psi \\ \cos \psi \end{pmatrix} & \varphi = 90^\circ \end{cases} \quad (153)$$

The first two rows of the Equations 153 run in detail:

$$(\alpha_1 h + \beta_1 k + \gamma_1 l) \cdot \frac{1}{\sqrt{h^2 + k^2 + l^2}} = \begin{cases} \sin \psi & \varphi = 0^\circ \\ 0 & \varphi = 90^\circ \end{cases} \quad (154)$$

$$(\alpha_2 h + \beta_2 k + \gamma_2 l) \cdot \frac{1}{\sqrt{h^2 + k^2 + l^2}} = \begin{cases} 0 & \varphi = 0^\circ \\ \sin \psi & \varphi = 90^\circ \end{cases}$$

As an example one gets for the $\{211\}\langle 01\bar{1} \rangle$ group

$$\frac{(k-l)}{\sqrt{2}} \cdot \frac{1}{\sqrt{h^2 + k^2 + l^2}} = \begin{cases} \sin \psi & \varphi = 0^\circ \\ 0 \ (\Rightarrow k = l) & \varphi = 90^\circ \end{cases}$$

$$\frac{(-h+k+l)}{\sqrt{3}} \cdot \frac{1}{\sqrt{h^2 + k^2 + l^2}} = \begin{cases} 0 \ (\Rightarrow h = k+l) & \varphi = 0^\circ \\ \sin \psi & \varphi = 90^\circ \end{cases}$$

$$\sin^2 \psi = \begin{cases} \frac{(k-l)^2}{2(h^2 + k^2 + l^2)} & \text{with } (h = k+l) & \varphi = 0^\circ \\ \frac{(-h+k+l)^2}{3(h^2 + k^2 + l^2)} & \text{with } (k = l) & \varphi = 90^\circ \end{cases}$$

The Table 11 contains the data for iron which help to select the radiation, peak, and direction $\sin^2\psi$ to evaluate strain distributions with oscillations /92 supplemented/. Fig. 47 and Fig. 50 show D-vs.- $\sin^2\psi$ results of a rolled unalloyed steel /4/. The ideal orientations are indicated. The specific D, $\sin^2\psi$ values are plotted in Fig. 48, also the D_0 -values are noted for RD ($\varphi = 0^\circ$) and TD ($\varphi = 90^\circ$) and all three major crystallite groups. The evaluation of the RS will be done in the usual manner using the compliances of the monocrystal. In Table 7 the RS evaluated by the crystallite-group method and by linearization of the D-vs.- $\sin^2\psi$ distributions are listed /4/.

The method to get the strain-free direction $\sin^2\psi^*$ is principally the same as previously indicated:

$$\frac{1}{2}(\epsilon_{0,+ \psi} + \epsilon_{0,- \psi}) = 0$$

The formulae can be found in section 2.112b.

Table 11. Data of the poles of the three ideal orientations in RD and TD, iron-base material, $D_0 = 0.28665 \text{ nm}$.

{hkl}	$K_{\alpha 1}$	2θ in degree	ψ in degree		$\sin^2\psi$	
			RD	TD	RD	TD
{100}{011}						
110	Ti Cr	85.37 68.78	90	90	1	1
200	Ti Cr Fe Co Cu	146.99 106.02 84.97 77.23 65.02	0	0	0	0
211	Cr Fe Co Cu	156.07 111.62 99.69 82.32	35.26	35.26	0.33	0.33
220	Fe Co Cu	145.54 123.91 98.93	90	90	1	1
222	Cu	137.13	54.74	54.74	0.67	0.67

Table 11 continued.

{hkl}	$K\alpha_1$	2θ in degree	ψ in degree		$\sin^2\psi$	
			RD	TD	RD	TD
{111}{2 $\bar{1}\bar{1}$ }						
110	Ti Cr	85.37 68.78	35.26	90	0.33	1
200	Ti Cr Fe Co Cu	146.99 106.02 84.97 77.23 65.02	54.74	—	0.67	—
211	Cr Fe Co Cu	156.07 111.62 99.69 82.32	19.47, 90	—	0.11, 1	—
220	Fe Co Cu	145.54 123.91 98.93	35.26	90	0.33	1
222	Cu	137.13	0, 70.53	—	0, 0.89	—
{211}{01 $\bar{1}$ }						
110	Ti Cr	85.37 68.78	30, 90	54.74	0.25, 1	0.67
200	Ti Cr Fe Co Cu	146.99 106.02 84.97 77.23 65.02	—	35.26	—	0.33
211	Cr Fe Co Cu	156.07 111.62 99.69 82.32	0, 60	70.53	0, 0.75	0.89
220	Fe Co Cu	145.54 123.91 98.93	30, 90	54.74	0.25, 1	0.67
222	Cu	137.13	—	19.47, 90	—	0.11, 1

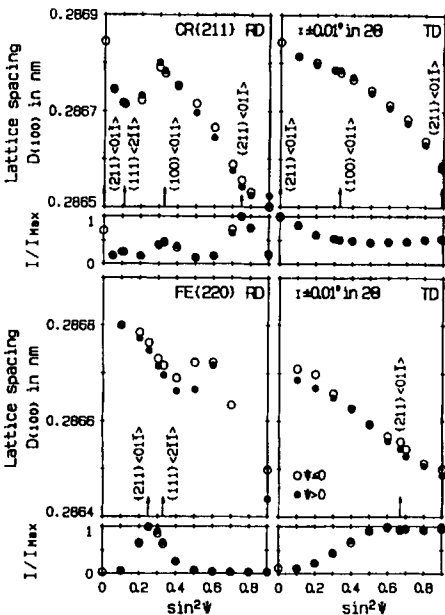


Figure 47. Distributions of D vs. $\sin^2\psi$ and intensity vs. $\sin^2\psi$ for the peaks {211} and {220} in RD and TD, 88%-cold-rolled unalloyed steel. The positions of the poles of three crystallite groups are indicated /4/.

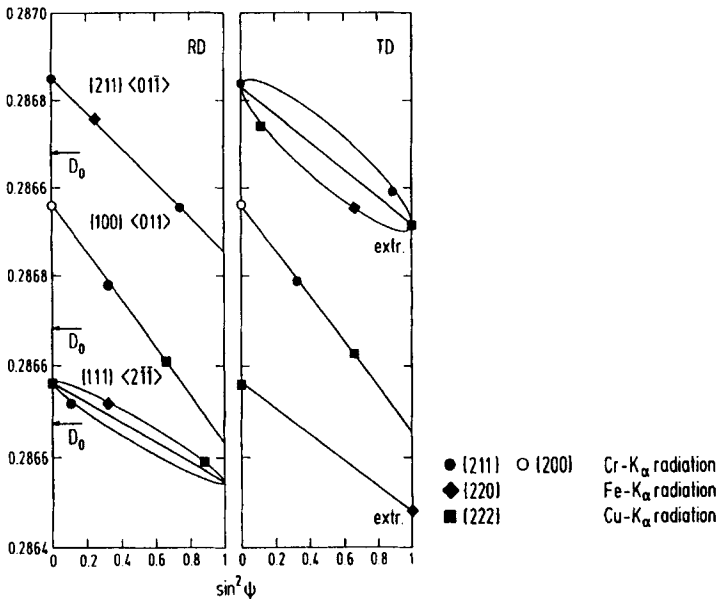


Figure 48. Lattice distances of the three crystallite groups vs. $\sin^2\psi$ taken from the respective poles of different D-vs.- $\sin^2\psi$ distributions in RD and TD, 88%-cold-rolled unalloyed steel; extr. (extrapolated). /4/

2.073c The crystallite-group method, fiber texture

The stress evaluation in fiber-textured materials showing nonlinear D-vs.- $\sin^2\psi$ distributions can also be done by the crystallite-group method. The procedure is described in /84/. Here are the main points. The ideal orientation has the form $\{mnr\}\langle uvw \rangle$, with $\{mnr\}$ being the fiber axis and $\langle uvw \rangle$ the $\varphi = 0^\circ$ direction. Table 12 shows the formulae for the main fiber crystallite groups for the strain and the strain-stress-free and -independent direction. The suppositions are $\sigma_1 = \sigma_2$, $\sigma_3 = 0$; the fiber axis is perpendicular to the surface of the film or the specimen:

$$K = \alpha_3^2(\alpha_3^2 - \alpha_1^2) + \beta_3^2(\beta_3^2 - \beta_1^2) + \gamma_3^2(\gamma_3^2 - \gamma_1^2) \quad (155)$$

$$\begin{pmatrix} \alpha_1 \\ \beta_1 \\ \gamma_1 \end{pmatrix} = \frac{1}{\sqrt{u^2 + v^2 + w^2}} \begin{pmatrix} u \\ v \\ w \end{pmatrix} \quad (156)$$

$$\begin{pmatrix} \alpha_3 \\ \beta_3 \\ \gamma_3 \end{pmatrix} = \frac{1}{\sqrt{m^2 + n^2 + r^2}} \begin{pmatrix} m \\ n \\ r \end{pmatrix} \quad (157)$$

α_i , β_i , γ_i are the cosines of the angles between crystal- and specimen-systems.

Table 12. Formulae to evaluate fiber-textured cubic materials, details in the text /84/.

crystallite group	$\varepsilon_\psi =$	$\sin^2 \psi^* =$
$\langle 100 \rangle$	$\left[2s_{12} + \left(\frac{1}{2}s_{44} + s_0 \right) \sin^2 \psi \right] \cdot \sigma_1$	$\frac{-2s_{12}}{\frac{1}{2}s_{44} + s_0}$
$\langle 110 \rangle$	$\left[2s_{12} + \frac{1}{2}s_0 + \left(\frac{1}{2}s_{44} + K \cdot s_0 \right) \sin^2 \psi \right] \cdot \sigma_1$	$\frac{-2s_{12} - \frac{1}{2}s_0}{\frac{1}{2}s_{44} + K \cdot s_0}$
$\langle 111 \rangle$	$\left[2s_{12} + \frac{2}{3}s_0 + \frac{1}{2}s_{44} \sin^2 \psi \right] \cdot \sigma_1$	$\frac{-2s_{12} - \frac{2}{3}s_0}{\frac{1}{2}s_{44}}$

The representations of the poles of different peaks are shown in Fig. 49 for the three main fiber textures for a specific material. Examples of single and double fiber textures are discussed by /84/. In /93/ the formulae were given for hexagonal-crystal films with the fiber axis $\langle 001 \rangle$ perpendicular to the surface. The formula is

$$\varepsilon'_{33} = \left[(s_{11} + s_{12} + 2s_{13}) \sin^2 \psi + 2s_{13} \right] \cdot \sigma_1 \quad (158)$$

Measurement at different distinct poles given by $\sin^2\psi$ result in a linear D-vs.- $\sin^2\psi$ distribution. A recent paper /88/ extended the formulae to a triaxial principal stress state.

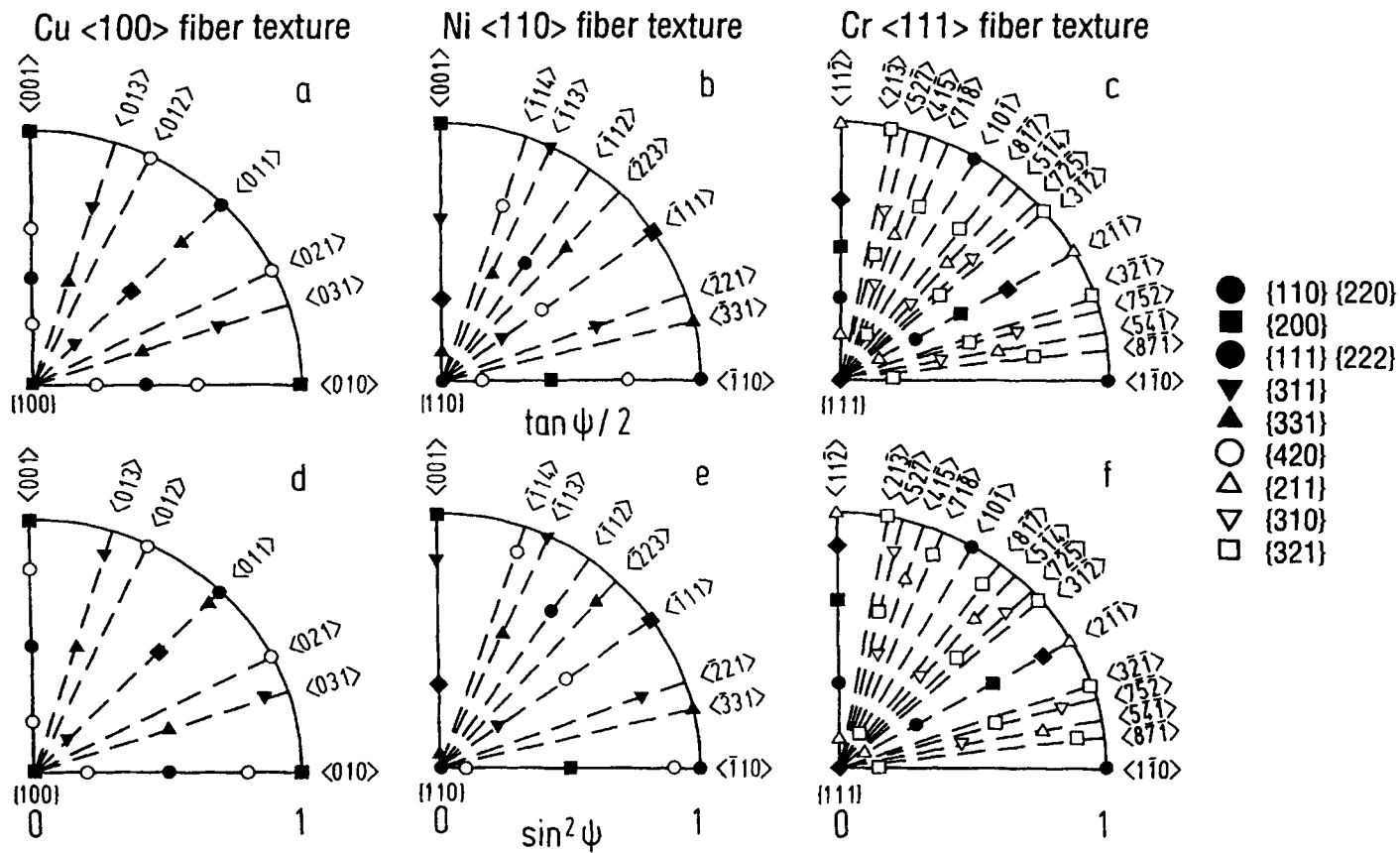


Figure 49. Stereographic projections of the poles of particular crystallite groups and fiber textures with the fiber axis perpendicular to the surface /84/.

Also /88/ extended the formulae for a fiber texture of a cubic material with the $\langle 111 \rangle$ axis perpendicular to the surface for a general stress state as follows. The evaluation of σ_{11} , σ_{22} , σ_{33} , σ_{12} will be done according to the Dölle-Hauk method /36/. In case of a rotational biaxial stress state $\sigma_1 = \sigma_2$, $\sigma_3 = 0$ the formulae simplify to those previous given by /84/.

2.073d The φ -integral method for a fiber texture

RS in specimen with a fiber texture may also be determined by using the φ -integral method, which will be outlined in the following /94/. The basic formulae can be found in section 2.072h. The measurements will be done at least at two ψ -values for $\varphi = 0^\circ$ to 360° . With these measurement results the coefficients A_n and B_n can be evaluated and therewith the equation system for the ε_{ik} ; least-square error method should be used. From the ε_{ik} tensor, the σ_{ik} tensor can be evaluated using the general Hooke's law and appropriate XEC and neglecting the texture or in case of low anisotropic materials (as for example most of the ceramics) the Young's modulus E and the Poisson's ratio ν .

The fiber texture should be not too weak to get strong circles in the polefigure with the fiber axis perpendicular to the surface of the specimen.

The method is applicable only when linear D-vs.- $\sin^2\psi$ dependences exist. It is a lucky circumstance that despite of strong fiber textures the D-vs.- $\sin^2\psi$ dependences of ceramic materials and ceramic layers are often practically linear because of the weak elastic anisotropy.

2.073e Evaluation of D-vs.- $\sin^2\psi$ distributions with texture conditioned oscillations, the σ -modeling

The evaluation of LS and RS from D-vs.- $\sin^2\psi$ distributions with oscillations will be described in chapter 2.16. Here the case will be handled where the values of the stresses will be given and the strain distributions modeled. The oscillations have their origin in the strongly preferred material state (texture) and/or, not considered here, plastic deformation.

The suppositions are: experimentally evaluated ODF, the assumption of a model of the crystallite coupling, the elastic coefficients of the monocrystal, the Young's modulus and the compression modulus of the polycrystalline materials. The formula

$$\varepsilon_{\varphi,\psi} = F_{ij}\sigma_{ij} \quad (159)$$

holds.

The stress factors F_{ij} will be calculated using the ODF and the model of coupling. Usually the well-known models according to Voigt (homogeneous strain), Reuss (homogeneous stress) and Eshelby-Kröner (anisotropic ellipsoid in homogeneous matrix) will be taken into account. With the assumption of the RS or LS state the D-vs.- $\sin^2\psi$ distribution will be calculated. By comparison with the experimental D-vs.- $\sin^2\psi$ the real stress state can be evaluated by multilinear regression (least-squares method) /28,95,96,97,98,99,100/.

In order to check the validity of the operation the $\{h00\}$ and $\{hhh\}$ peaks should be linear. The following example is described in detail in /28/. The specimen is a cold-rolled highly textured unalloyed steel strip. Figures 47, 50, 51 demonstrate the comparison between measured and calculated D-vs.- $\sin^2\psi$ distributions of different $\{hkl\}$ in the RD and TD.

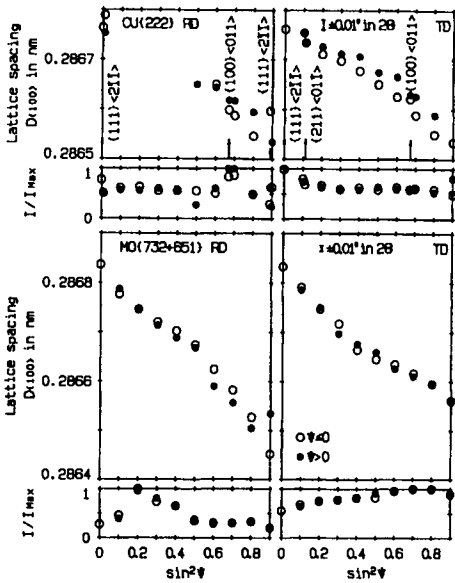


Figure 50. Measured D-vs.-sin²ψ and intensity-vs.-sin²ψ distributions for the peaks {211} see Fig. 47, {222}, {732+651} observed at the surface of the steel strip /4/.

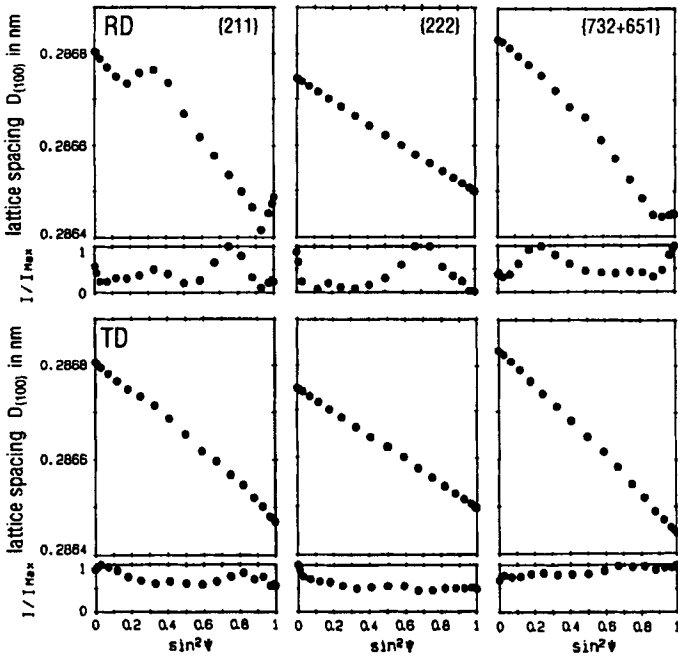


Figure 51. Calculated D-vs.-sin²ψ and intensity-vs.-sin²ψ distributions for the peaks {211}, {222}, {732+651} and a RS-state with $\sigma_{11} = \sigma_{22} = -200$ MPa, $\sigma_{12} = \sigma_{13} = \sigma_{23} = \sigma_{33} = 0$ /4/.

2.073f The σ - and the D_0 -modeling

An interesting fact is that in many cases D-vs.- $\sin^2\psi$ distributions calculated on the basis of the Reuss model fit the experimental ones better than that using the Eshelby-Kröner model. The latter is in good agreement with measurement results for quasiisotropic materials, Fig. 52 and Fig. 53 show that even the Reuss model does not demonstrate all the oscillations. The reason for this could be that the calculation takes into account only the texture induced oscillations but plastic deformation will have an effect too /78/.

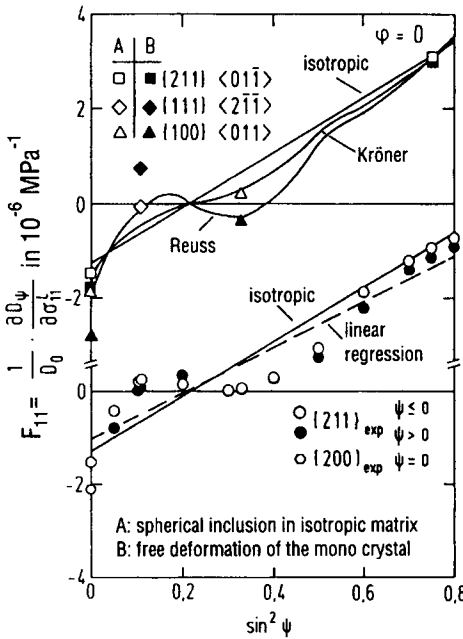


Figure 52. Anisotropic stress factors F_{11} -vs.- $\sin^2\psi$. Above: Calculations of an isotropic material and for two assumptions of the coupling and two descriptions of texture. Below: Experimental results, regression line for the $\{211\}$ peak shifts and for the isotropic material /4/.

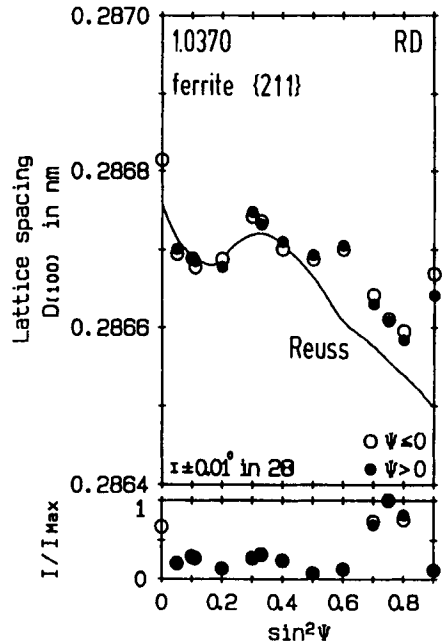


Figure 53. Comparison between experimentally determined and calculated D-vs.- $\sin^2\psi$ distribution using F_{ij} from the Reuss model as well as the stresses $\sigma_{11} = -150$ MPa and $\sigma_{22} = -120$ MPa /78/.

In most practical cases of LS and RS problems well-separated interference lines are observed, there are no gradients of the strain, the stress, the composition, the microstructure as well as the texture within the effective penetration depth of the radiation. But there are a lot of possibilities to make the situation less ideal. Peaks may be overlap, there may be pronounced changes of the strains, the stresses, the microstructure, the composition of the materials, D_0 -profiles, and the texture as a function of depth and moreover, these quantities may depend on the depth in a nonlinear way. In case that there is a simple relationship between the different parameters, it may be possible to find an appropriate data evaluation procedure. However, there is no universal way to attack this problem.

In the event of overlapping peaks, one may have recourse to commonly used deconvolution procedures. In case that D_0 and/or the RS show a rapid variation with depth, model calculations should be performed to establish the relationship between the depth dependence of these quantities and the D-vs.- $\sin^2\psi$ distributions observed for different $\{hkl\}$ peaks.

The chosen example belongs to an Fe-ion implanted copper-foil /98/. Fig. 54 shows the lattice strains measured with Co- K_α radiation on the $\{400\}$ peak and with Ti- K_α radiation on the $\{111\}$ peak for azimuth $\varphi = 0^\circ$ and 45° . Fig. 55 illustrates the D_0 - and σ -profiles. The arrows indicate the parameters which will be varied /99/.

The following formulae (paragraph 2.036, 2.152) will be used:

$$D_\psi = \frac{\int_0^t D_0(z) \exp\left(-\frac{z}{\tau}\right) dz}{\tau \cdot \left[1 - \exp\left(-\frac{t}{\tau}\right)\right]} \tag{160}$$

$$\langle \sigma_{ij} \rangle = \frac{\int_0^t \sigma_{ij}(z) \exp\left(-\frac{z}{\tau}\right) dz}{\tau \cdot \left[1 - \exp\left(-\frac{t}{\tau}\right)\right]} \tag{161}$$

The lattice-strain profiles versus $\sin^2\psi$ were calculated using the shown models and formulae. It is a tedious and obviously an inexact method to evaluate the D-vs.- $\sin^2\psi$ profiles for the two peaks of the two radiations. Fig. 56 shows a typical result of the modeling. The data used are indicated in the caption /98/. The final solution of such problems is of course a consideration of the confidence range of all parameters D , σ , z and $\frac{\partial \sigma}{\partial z}$.

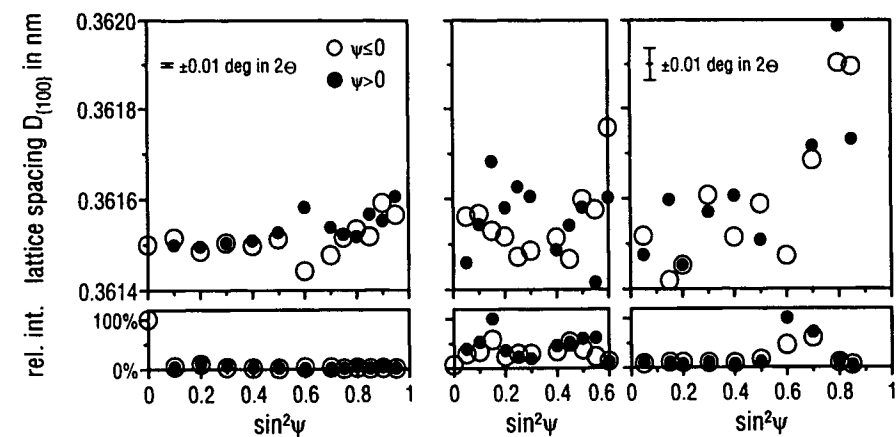


Figure 54. D-vs.- $\sin^2\psi$ distributions for the ion-implanted area. Left: four measurements averaged, Co- K_α radiation, $\{400\}$ peak, $2\Theta = 163.5^\circ$, $\varphi = 0^\circ$; center and right: three measurements averaged, Ti- K_α radiation, $\{111\}$ peak, $2\Theta = 82.4^\circ$, $\varphi = 0^\circ$ and 45° /98/.

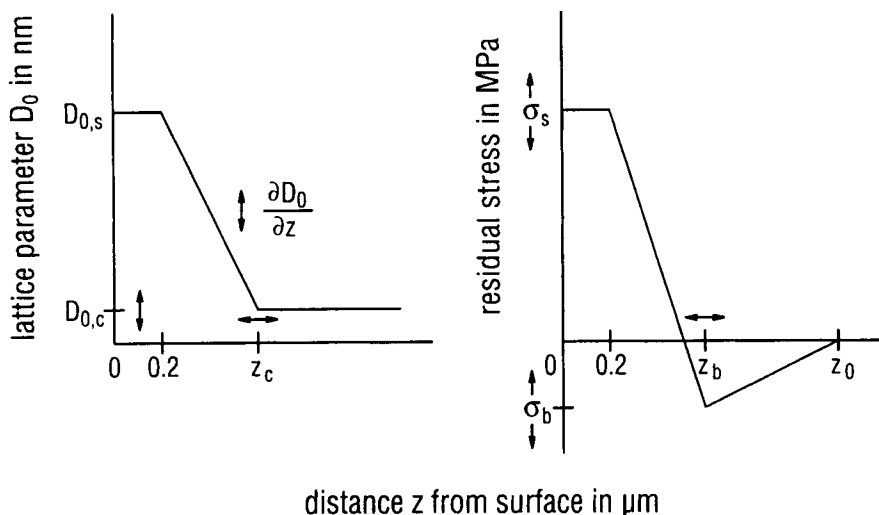


Figure 55. Models for lattice-spacing and residual-stress profiles with indication of the varied parameters. $D_{0,s}$, $D_{0,c}$: lattice parameter at the surface and for the core up to the depth z_c ; σ_s residual stress at the surface balanced by σ_b (equilibrium of forces) at the distance z_b from surface /98/.

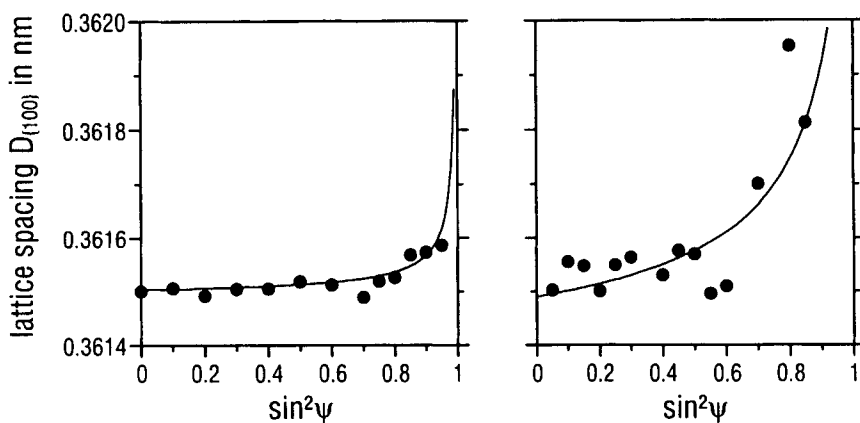


Figure 56. D-vs.- $\sin^2\psi$ distributions for the $\{400\}$ peak observed with Co- K_α radiation (left) and for the $\{111\}$ peak with Ti- K_α radiation (right).

Points: measured and averaged values; curve: calculated by combination of both models (D_0 and residual-stress profile) with the following parameters, $D_{0,c} = 0.361498$ nm, $D_{0,s} = 0.361898$ nm, $z_c = 0.22$ μm , $\sigma_s = 550$ MPa, $\sigma_b = -400$ MPa, $z_b = 0.7$ μm ,

$$z_0 = 1.44$$
 μm , $\frac{\partial \sigma}{\partial z} = -1900 \frac{\text{MPa}}{\text{mm}}$ /98/.

2.074 References 2.06, 2.07

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2.08 Peak width and its relation to different parameters

The full width at half maximum of a peak (FWHM) is determined by the distribution of the randomly oriented lattice plane distances. There are many causes for the spread of the interplanar spacings: distributions of the lattice parameters by alloying, interstitial site occupation, precipitations, undirected micro distortion created by micro stresses. The non directed micro stresses can originate from thermal or/and plastic deformation (relaxation, slip, twinning, stacking faults, orientation differences). Small crystallite sizes are also contributing to the broadening of the interference lines.

The experimental determination of the FWHM is demonstrated in Figure 1. The background may be constant or increasing with 2θ and should be taken into account within a range of five times the half width. The integral width is also of physical significance.

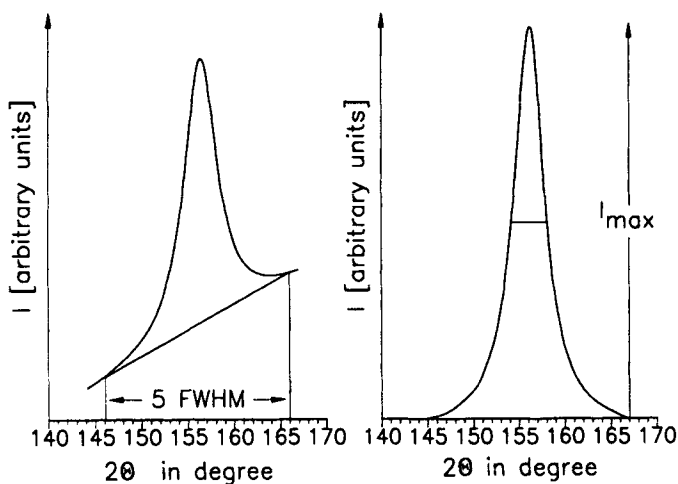


Figure 1. Determination of the full width at half maximum of peak interference profile.

Different procedures for a thorough analysis of the line broadening are discussed in chapter 2.17. Some information, however, can already be obtained by merely considering the values of the FWHM. Therefore, the measurements on the sample material should be corrected for the instrumental peak broadening by subtracting the FWHM-values of a calibration powder of the same base material measured under the same experimental conditions and at the same $\sin^2\psi$ orientations. If the FWHM of different $\{hkl\}$ are necessary in appropriate studies, the values of FWHM times $\cot \Theta$ should be compared. In many papers, the corrected FWHM values are used to relate them to one systematically varied parameter, whereas the remaining parameters are kept constant. So Figure 2 shows the relationship between hardness and half width for steels [1].

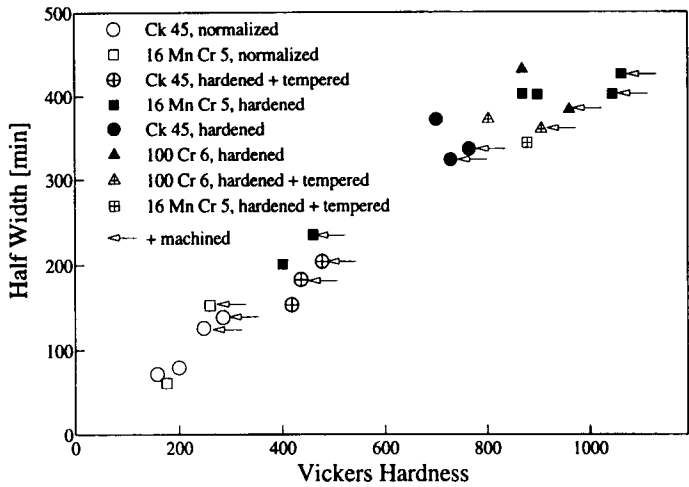


Figure 2. Full width at half maximum of X-ray diffraction lines of steels in different states [1].

It is often observed that the RS and the FWHM are correlated in the case of plastically deformed metals and also in mechanically surface treated metals and ceramics. The sign of the correlation depends on the hardness of the material. Whereas in the case of ground Si_3N_4 the FWHM increases with increasing RS (Figure 3), the opposite behavior is observed for shot peened steel samples (Figure 4) and also for machined steel samples (Figure 5).

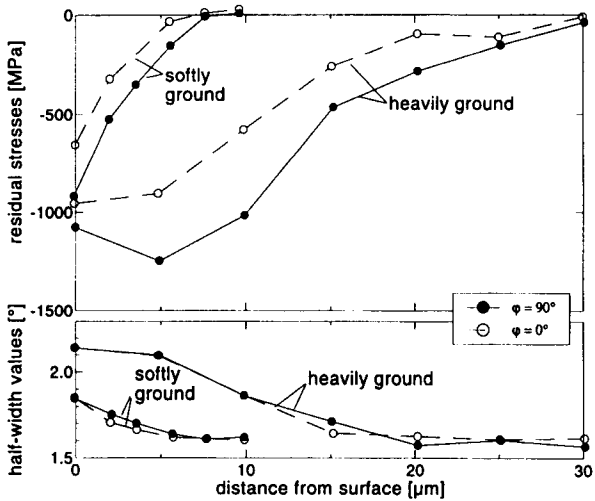


Figure 3. Depth distributions of residual stresses and half-width values of differently ground Si_3N_4 obtained with V K_α -radiation [2].

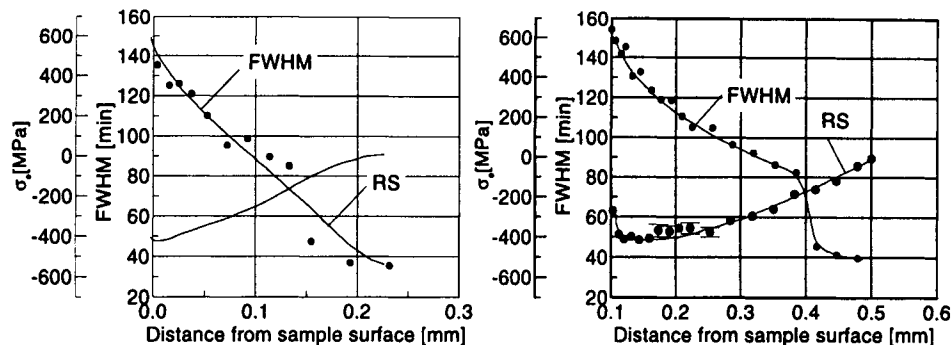


Figure 4. Residual stress and full width at half maximum FWHM at various sample depths in normalized and shot peened samples (Steel, German grade: 16 Mn Cr 5) [3].

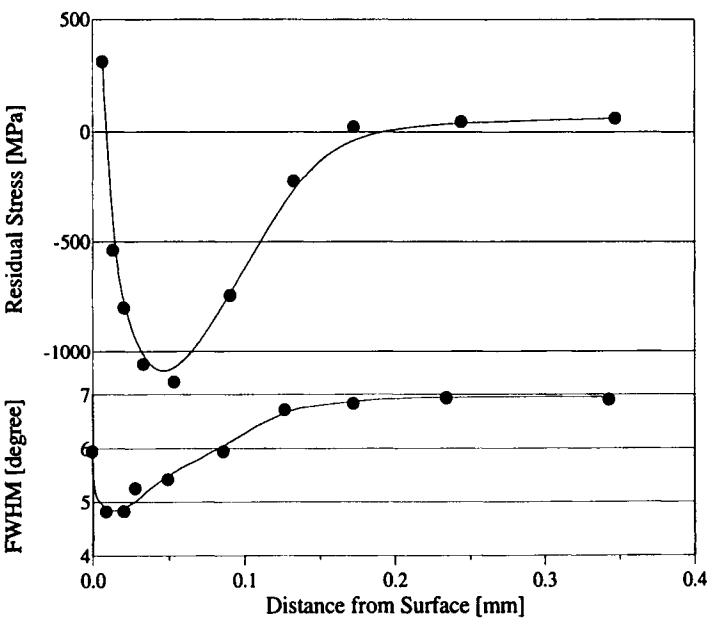


Figure 5. Turning of hardened steel, German grade: 100 Cr Mn 6 [4].

Further examples for relationships between different parameters and the FWHM-values are given in the literature [5-8].

An interesting aspect is the influence of a texture formation on the FWHM-value. The corresponding results on rolled, textured materials prove that in the direction of the ideal orientation the undirected micro-stresses are smaller than in the other directions. Figure 6 shows experimental results for a ferritic unalloyed steel [9].

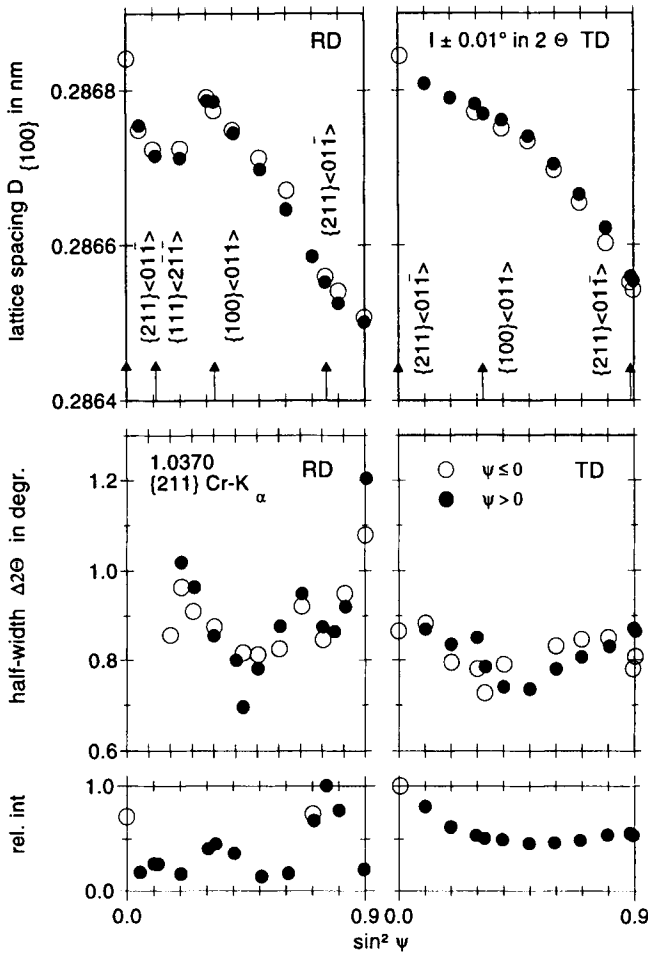


Figure 6. Distribution of D-values, half-widths, and intensity versus $\sin^2\psi$; ferritic steel 1.0370 [9].

In Figure 6 the lattice distance versus $\sin^2\psi$ is displayed and the ideal orientations are marked. Furthermore, the distributions of FWHM as well as the relative intensities are plotted versus $\sin^2\psi$. In the RD, the minima of the FWHM are clearly related to the maxima of the intensities. Also for rolled Ni sheets, the minima of the FWHM are found in the directions of the ideal orientations, Figure 7 [10].

In [11], the $\{211\}$ intensity- and the interference line broadening pole figures of a steel are presented. Three different deformation modes were considered: 70% cold worked, 5% uniaxially elongated and 0.6% biaxially expanded. The authors conclude „that the main texture orientations have stored less energy due to plastic deformation than the others“. The influence of the deformation modes on the results is not discussed in detail.

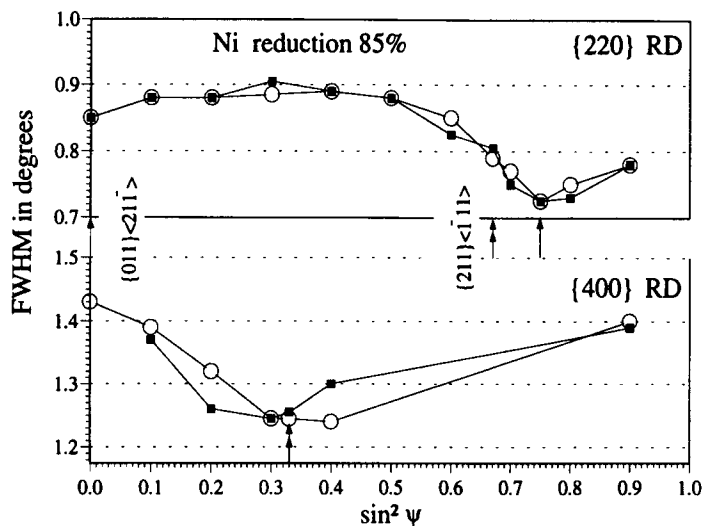


Figure 7. Minima of the FWHM in the directions of ideal orientations in rolled Ni-sheets [10].

Different results were reported for uniaxially elongated specimens. Older studies with film registration and no texture information showed the dependencies of FWHM versus $\beta = 90 - \psi$ displayed in Figure 8 and Figure 9.

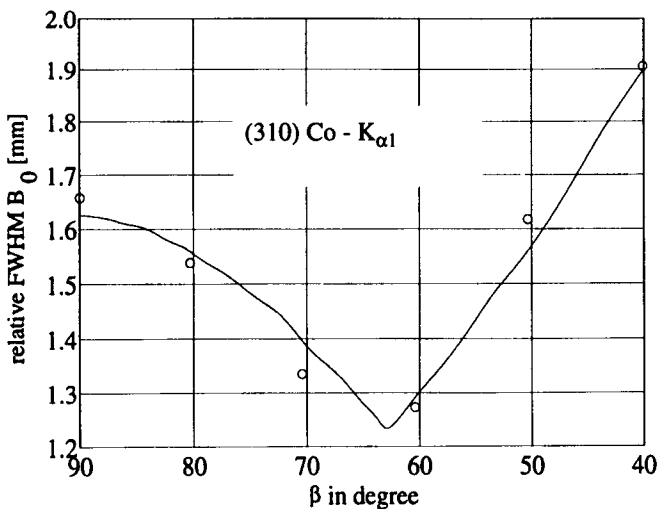


Figure 8. Full width at half maximum versus β ($\beta = 90 - \psi$), iron [12].

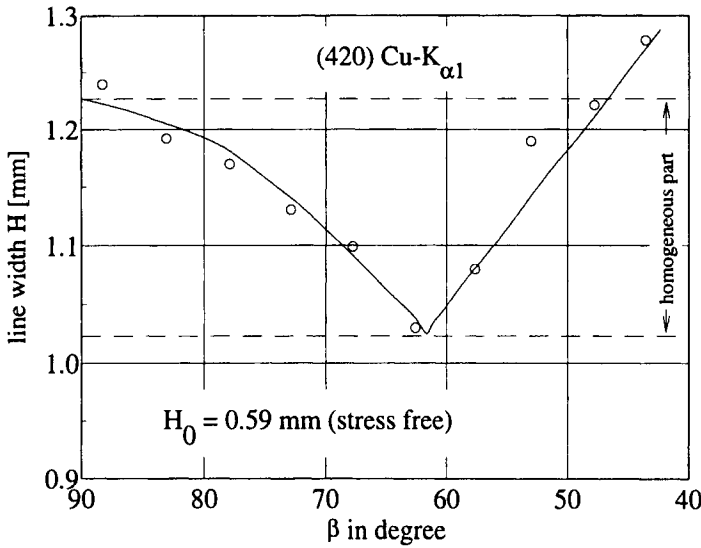


Figure 9. Line width of Cu (420) versus β ($\beta = 90 - \psi$), nickel [13].

The minima of FWHM are found in the strain free direction $\sin^2\psi^* = -s_1/\frac{1}{2}s_2$.

The authors conclude that the minimum FWHM relates to the broadening of the peak by random distortion and particle size, and that the directed micro RS result in an additional broadening. Studies of the crystal orientation and the micro structure in the surface region are not reported. From today's point of view, it seems probable that strong texture and very sharp stress gradient are the reasons for a minimum of the FWHM. A RS gradient might be the consequence of a work hardened surface layer.

It can be stated that the values of the FWHM are generally valuable characteristics of the material. It is useful to exploit them systematically in view of their relations to different material parameters. The FWHM should always be measured and registered, not only at $\psi = 0$ but also versus $\sin^2\psi$. For the interpretation of the values of the FWHM, however, besides the lattice distortions, the domain size has to be considered, too. There are programs available to analyse the interference lines in regard to lattice distortion and domain size. They should be used more often than it is done today.

2.09 Stacking faults

W. Reimers

For cold worked metals and alloys in addition to the broadening of X-ray diffraction lines due to a reduction in size of the coherently diffracting domains and from the inhomogeneous

directions due to deformation stacking faults. In fact, a peak shift may originate from a high twin stacking fault density, too, but it is negligibly small [16]. Equation 1 is only valid for one point (hkl) in reciprocal space. In a powder or polycrystal, all interferences $\{hkl\}$ are observed simultaneously in one line. Therefore, one has to use an average value for $\Delta(2\Theta^\circ)$ to calculate the stacking fault density α :

$$\langle \Delta(2\Theta^\circ) \rangle_{hkl} = \pm \frac{270}{\pi^2 l_0} \alpha \langle \cos \phi \rangle_{hkl} j \tan \Theta \quad (2)$$

where j is the fraction of lattice planes affected by stacking faults and $\langle \cos \phi \rangle_{hkl}$ the mean cosine of ϕ of the shifted lines only. In Table 1, the peak shifts for the lines with $h^2 + k^2 + l^2 \leq 27$ are listed as a function of the deformation stacking fault density α . According to Bragg's law the peak shift is related to a fictitious strain $da/a = -\cot \Theta \, d\Theta$ which is also listed in Table 1 [18]:

$$\frac{da}{a} = \mp \frac{3}{4\pi l_0} \alpha \langle \cos \phi \rangle_{hkl} j \quad (3)$$

Table 1. $\langle \cos \phi \rangle_{hkl}$, peak shifts and fictitious strains for various X-ray lines due to deformation stacking faults [18].

hkl^x	$\langle \cos \phi \rangle_{hkl}$	$\Delta(2\Theta^\circ) \cdot \alpha^{-1}$	$da/a \cdot \alpha^{-1}$
111	1/3	$+3.95 \cdot \tan \Theta$	$-3.45 \cdot 10^{-2}$
200	$1/\sqrt{3}$	$-7.90 \cdot \tan \Theta$	$+6.90 \cdot 10^{-2}$
220	$2/\sqrt{6}$	$+3.95 \cdot \tan \Theta$	$-3.45 \cdot 10^{-2}$
311	$\sqrt{3}/\sqrt{11}$	$-1.43 \cdot \tan \Theta$	$+1.26 \cdot 10^{-2}$
222	1/3	$-1.97 \cdot \tan \Theta$	$+1.27 \cdot 10^{-2}$
400	$1/\sqrt{3}$	$+3.95 \cdot \tan \Theta$	$-3.45 \cdot 10^{-2}$
331	$7/\sqrt{57}$	$+0.83 \cdot \tan \Theta$	$-0.73 \cdot 10^{-2}$
420	$1/\sqrt{15}$	$-0.79 \cdot \tan \Theta$	$+0.69 \cdot 10^{-2}$
422 ^{xx}	0	0	0
511	17/18	$-0.33 \cdot \tan \Theta$	$+0.29 \cdot 10^{-2}$

x Note that positive as well as negative hkl have to be considered

xx Positive and negative shifts add up to zero

An example for the influence of deformation stacking faults on lattice strain measurements is given in Figure 11. The plot shows lattice strains calculated from the peak shifts of different reflections for tensile deformed α -brass. The differences between the

shifts of different reflections for tensile deformed α -brass. The differences between the different orders of the same reflections should not occur for real lattice strains. According to Table 1 they can be attributed to deformation stacking faults. The stacking fault density α calculated from these differences is shown in Figure 12. Compared to filings, the stacking fault density of tensile deformed α -brass of 10^{-2} is about two orders of magnitude lower.

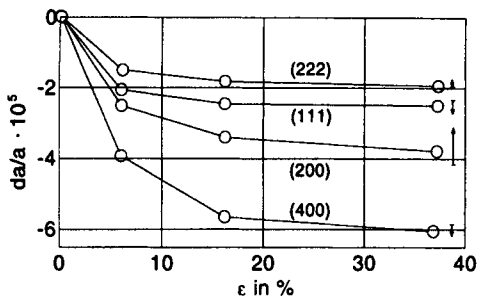


Figure 11. Fictitious strains in tensile deformed α -brass 70 as function of deformation [18].

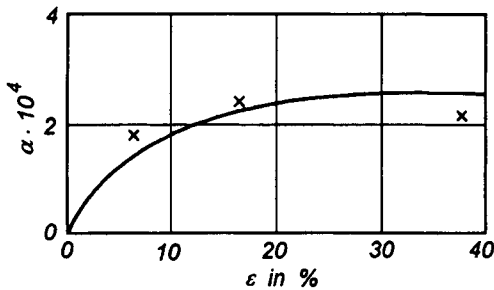


Figure 12. Deformation stacking fault density as a function of the deformation of tensile deformed α -brass 70 [18].

Furthermore, peak broadening is symmetrical in the case of deformation stacking faults and asymmetrical if twin stacking faults are present. It can be interpreted in terms of a stacking fault induced reduction of the average coherent domain size (chapter 2.17) [16]:

$$\frac{1}{L_{\text{eff}}} = \frac{1}{L} + \frac{1.5\alpha + \beta}{D(111)} \langle \cos\phi \rangle_{hkl} j \tag{4}$$

where $D(111)$ is the lattice spacing of the (111)-planes and β the twin stacking fault density. Typical for the influence of stacking faults on the coherent domain size is its anisotropy due to the $\langle \cos\phi \rangle_{hkl}$ -term (Table 1). It allows one to estimate the influence of stacking faults as opposed to a dislocation induced reduction of the coherent domain size [17, 18].

The influence of stacking faults is most important for alloys with a low stacking fault energy (e.g. austenitic steels). If the deformation stacking faults density is to be determined from peak shifts and Equation 2, additional peak shifts, due to the influence of macroscopical residual stresses, have to be considered, too. Therefore, a lot of investigations were performed on filings. In the case of compact samples (e.g. after tensile deformation or cold working) the influence of macroscopic residual stresses can be eliminated by considering different orders of the same diffraction line (e.g. (200)-(400), (111)-(222)) [17, 18]. In contrast to residual stresses, which cause a shift of each line of these pairs into the same direction, the peak shifts due to deformation stacking faults show opposite signs (Tab. 1). From another point of view, residual stress determination might become erroneous due to fictitious strains from the stacking fault peak shifts [18, 19]. In those cases, the comparison of different orders of the same line is helpful, this time for eliminating the stacking faults effects.

According to Equation 3, the value $1.5\alpha + \beta$ may also be evaluated by a detailed line profile analysis (chapter 2.17). A separate evaluation of β by a careful analysis of the peak asymmetry is in principle possible but in general difficult to perform [16, 18].

As pointed out above, the Equations 2 and 4 for peak shift and coherent domain size are obtained under the assumption of infinite large stacking faults distributed randomly on one (111)-plane. If more than one {111}-plane contains stacking faults, the total stacking fault density can be calculated as the sum of the densities of single planes. If the stacking faults are not distributed randomly but clustered or ordered, a further asymmetry in peak broadening occurs [18, 20]. A finite width of the stacking faults must be considered if the width is lower than the coherent domain size [18, 21].

2.10 Recommendations for strain measurement and stress evaluation

In principle, XSA aims at the determination of the triaxial stress state, considering, if necessary, gradients of stress, texture and D_0 values. Such a comprehensive question, however, leads to extensive and complex procedures - during the actual measurements as well as when evaluating the data. Thus, if the technical problems are to be solved in decent time, simplifications are normally called for. Of course, those simplifications, like e.g. the assumption of a biaxial stress state, or of an isotropic orientation distribution of the crystallites under study, can only be made after assuring their applicability for the special case. Especially when using the software that is offered for the determination of RS by service companies and equipment manufacturers, it is necessary to check whether the implicit requirements of the program are indeed fulfilled for the specimen in question, and whether the evaluation procedures represent the state of the art.

Continuing the statements made in [22, 23], recommendations for strain measurements will be listed in the following order: material characteristics, choice of measuring conditions, alignment and calibration, number of measurements to be performed, X-ray elastic constants (XEC).

It is recommended

1. to consider the characteristic parameters of the material state of the specimen or component in question. Significant factors are e.g.: chemical composition, phase

composition, microstructure, texture. Important information concerning, for example, the presence of gradients, can be derived from the history of the specimen - heat treatment, plastic deformation, mechanical surface treatment, etc. An additional factor that needs to be considered for the experimental setup as well as in the interpretation of the results is the roughness of the surface [24-27]. In order to avoid misinterpretations, the static and dynamic strength properties like E , ν , $R_{p0.2}$, R_m , A and strain stress diagrams should also be taken into account if they are available.

2. to define the type of stresses to be investigated. If possible, micro and macro residual stresses should be separated. In multiphase materials, the XSA should be performed in all phases detectable with X-ray diffraction. D_0 should be evaluated on that spot (or equivalent place) where XSA is performed, otherwise erroneous results due to decomposition by heat may occur.

3. to select the appropriate X-ray wavelength. The wavelength substantially influences the penetration depth, thus determining the depth of information for the registered stresses. The availability of reflections $\{hkl\}$ with their different intensities (atomic form factor, structure factor, multiplicity), and the line positions in 2Θ also depend on the wavelength. Furthermore, choosing the wavelength entails the choice of filtermaterials and of the suitable calibration powder for an optimum diffractometer adjustment. The angular distances between the reflections of the sample and the calibration powder should be as small as possible. A summary of the necessary X-ray data for measurements on ferritic and austenite steels, aluminum, copper, nickel and titanium is presented in chapter 2.04.

4. to align and calibrate the diffractometer such that the peak positions of the $\{hkl\}$ -lines of the calibration powder do not differ more than $\pm 0.01^\circ$ in 2Θ over the whole $\pm \psi$ -range chosen for the intended measurements.

5. to check the necessity to install a monochromator, additional soller slits or a multilayer mirror. The peak/background ratio and the FWHM-value should be considered for correct assessment.

6. to register and to control the temperature during the complete time of the measurements for XSA. If the temperature is not kept constant throughout the measurements, corrections with respect to a reference temperature have to be applied.

7. to measure $2\Theta_{\phi\psi}$, $D_{\phi,\psi}$ versus $\sin^2 \psi$ in steps of $\Delta \sin^2 \psi = 0.1$ for the positive and negative ψ -branch up to the highest possible value, because nonlinearities in the $D_{\phi,\psi}$ versus $\sin^2 \psi$ diagram often show up for high $\sin^2 \psi$ -values only.

8. to check the linearity of the $D_{\phi,\psi}$ versus $\sin^2 \psi$ -distribution. Only if the linearity is proven, the number of measuring directions can be reduced to a minimum of at least four different positions in ψ in the same azimuth ϕ . If nonlinear $D_{\phi,\psi}$ versus $\sin^2 \psi$ -distributions are observed, the intervals of $\Delta \sin^2 \psi$ should be reduced and additional measurements with different wavelengths on several types of lattice planes are very useful.

9. to analyze the interference line intensity versus $\sin^2 \psi$. For Ψ -mode measurements the observed intensity should be constant over the whole ψ -range. For Ω -mode measurements a continuous decrease of the intensity is expected with increasing inclination angle ψ . If further intensity variations are observed, the sample should be checked for coarse grains or texture. Coarsely grained specimen should be moved relative to the X-ray beam during the

measurement in order to sample a larger number of grains. If texture is present, the strain measurement should be performed in the texture poles and their vicinity.

10. to study the interference line profile. The analysis of the full width at half maximum value may give important hints about the material state under study. For comparing the FWHM-values determined from different interference lines, a correction in the form of a multiplication with $\cot \Theta$ has to be made. After having performed the correction for the $K_{\alpha 1}$, $K_{\alpha 2}$ doublet splitting, a symmetric profile should be obtained. Asymmetries may indicate a stress- or a D_0 - gradient within the information depth of the measurements. An asymmetric interference profile may also be due to overlapping reflections. However, if possible, the interference line chosen for the strain measurement should be free from overlapping with other peaks. Truncated interference profiles as a consequence of a limited measuring range (for example $\leq 165^\circ$ in 2Θ) may result in important errors. Peaks at lower Bragg's angles should be chosen.

11. to apply the intensity correction factors and, subsequently, to use K_α separation methods when determining the peak position. For the peak position determination methods taking into account the full reflection profile are preferable to methods where only a part of the available information is used. If the statistical scatter of the obtained $D_{\varphi, \psi}$ -values is too important, the measuring conditions and the alignment of the diffractometers should be checked. When the measurement has to be repeated, the irradiated area should be changed instead of merely increasing the counting statistics.

12. to state the standard deviations of multiple measurements (parameters: ψ -values included, azimuths φ selected, counting time, $\Delta(2\Theta) = \pm 0.01^\circ$). When different (hkl)-planes with different radiations are measured, the obtained $D_{\varphi, \psi(hkl)}$ - values should be converted into $D_{(100)}$ to facilitate the detection of errors of alignment and measurement.

13. to take into consideration the consequences of elastic anisotropy. The XEC should be calculated from the mono crystal compliances according to the model of Eshelby-Kröner or to the models of Voigt and Reuss (average value). When only a small anisotropy is present, the mechanical XEC can be used. If the mono crystal data are not available, the XEC should be determined experimentally.

14. to complement, if possible, the results of X-ray stress analysis with additional physical or mechanical data for practical assessment.

2.101 References 2.08 to 2.10

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2.11 Determination of the lattice distance of the strain-stress-free state D_0 and the relation with the stress component in the thickness direction σ_{33}

2.111 Historical review

The idea to determine the lattice distance of the stress-free state D_0 (in older papers d_0) in a stressed material was first put forward by /1/ and by /2/ for a rotationally symmetric stress state, but Glocker /3/ stated the formulae for some uni- and biaxial stress states. Binder and Macherauch /4/ treated this problem basically for the general plane stress state.

The importance of knowing the correct D_0 for material physics and for the determination of the exact value of the strain is obvious. The necessity to use the precise value of D_0 was notorious when the triaxial strain and stress tensors was introduced in XSA /5,6/. Here, the dilemma of the diffraction method was evident: the connection between D_0 and σ_{33} . Additional considerations or experimental efforts are necessary to separate the influences of D_0 and σ_{33} . Different methods, both theoretical and experimental, to determine D_0 and the possible errors of the tests will be dealt with in the following /7/. It is important to consider the presence of micro-RS.

2.112 Theoretical aspects

2.112a Non-zero σ_{33} -components

The following thoughts and equations are valid for isotropic and anisotropic, mechanically isotropic and textured materials. Since σ_{i3} ($i = 1, 2, 3$) are zero at the very surface of the material, non-zero values are related to gradients. The following formulae of elasticity theory are the basis of discussion of several examples

$$\operatorname{div} \sigma = f \quad f: \text{load density} \quad (1)$$

For free outside surfaces but not for inner ones like in tubes or in a pipe it holds:

$$\operatorname{div} \sigma_{ij} = 0 \quad (2)$$

$$\sigma_{ij} n_j = 0 \quad (3)$$

There is a further condition for each cross section:

$$\int_A \sigma_{ij} n_A dA = 0 \quad (4)$$

Equ. 2 means in detail:

$$\begin{aligned}
 \frac{\partial \sigma_{11}}{\partial x} + \frac{\partial \sigma_{12}}{\partial y} + \frac{\partial \sigma_{13}}{\partial z} &= 0 \\
 \frac{\partial \sigma_{12}}{\partial x} + \frac{\partial \sigma_{22}}{\partial y} + \frac{\partial \sigma_{23}}{\partial z} &= 0 \\
 \frac{\partial \sigma_{13}}{\partial x} + \frac{\partial \sigma_{23}}{\partial y} + \frac{\partial \sigma_{33}}{\partial z} &= 0
 \end{aligned} \tag{5}$$

If there are no gradients of the stress components in the surface plane $z = 0$, then

$$\frac{\partial \sigma_{13}}{\partial z} = \frac{\partial \sigma_{23}}{\partial z} = \frac{\partial \sigma_{33}}{\partial z} = \frac{\partial \sigma_{i3}}{\partial z} = 0 \tag{6}$$

Therefore, there are no macrostress components σ_{i3} at the surface and inside the specimen. An example, which is often misunderstood, is the bending specimen with a prismatic cross section. There are no gradients at the plane surface, but $\frac{\partial \sigma'_{11}}{\partial z} \neq 0$ and nevertheless $\frac{\partial \sigma'_{33}}{\partial z} = 0$ and of course $\sigma'_{33} = 0$. But if in contrast, there is a gradient in the surface plane, for example $\frac{\partial \sigma_{11}}{\partial x} \neq 0$ or $\frac{\partial \sigma_{22}}{\partial y} \neq 0$ there may be gradients in the z -direction, for example $\frac{\partial \sigma_{13}}{\partial z} \neq 0$ or $\frac{\partial \sigma_{33}}{\partial z} \neq 0$.

When micro-RS are present, the σ_{i3} -components are still zero at the surface, but the microstresses vary in x - and y -direction. Therefore gradients of σ''_{i3} -components may occur. Additionally the following condition must be fulfilled in each depth: $c_\alpha < \sigma'' >^\alpha + c_\beta < \sigma'' >^\beta + \dots = 0$

In the following, some examples for applications will be discussed.

Examples for LS are tubes loaded from inside, where $\sigma_{33}^L = -p_i$ at the inside surface and $\sigma_{11}^L = \sigma_{22}^L = \sigma_{33}^L = 0$ at the outside surface. A bent flat specimen with a gradient $\frac{\partial \sigma_{11}^L}{\partial z} \neq 0$ has $\frac{\partial \sigma_{22}^L}{\partial z} = \frac{\partial \sigma_{33}^L}{\partial z} = 0$.

A specimen with RS I only and no gradients of $\sigma_{11}^L, \sigma_{22}^L, \sigma_{12}^L$ in any depth z shows $\sigma_{33}^L = 0$ and $\frac{\partial \sigma_{33}^L}{\partial z} = 0$ at the surface, and therefore $\sigma_{33}^L(z) = 0$ at any depth z . If no RS II are present in a single-phase material then σ_{33} is 0. But in a two-phase and multiphase material, there are always RS II and RS III present and therefore $\sigma_{i3}''(z=0)$ but $\frac{\partial \sigma_{i3}''(z=0)}{\partial z} \neq 0$ and $\frac{\partial \sigma_{i3}''}{\partial z} \neq 0$ though there are no gradients of averaged stresses $\langle \sigma'' \rangle^\alpha$ at the surface.

But if there are gradients of σ_{ij}^I at the surface in the x- and the y-direction, then $\frac{\partial \sigma_{i3}^I}{\partial z} \neq 0$ may not be zero:

$$\frac{\partial \sigma_{11}^I}{\partial x} \neq 0 \quad \frac{\partial \sigma_{12}^I}{\partial y} = 0 \Rightarrow \frac{\partial \sigma_{i3}^I}{\partial z} \neq 0 \quad \frac{\partial^2 \sigma_{11}}{\partial x^2} \neq 0 \Rightarrow \frac{\partial^2 \sigma_{33}^I}{\partial z^2} \neq 0 \quad (7)$$

In general, the diffraction methods determine the sum of macro- and average micro-RS. The following holds for the σ_{33} -components:

$$\langle \sigma_{33} \rangle^\alpha = \sigma_{33}^I + \langle \sigma_{33}^{II} \rangle^\alpha = \sigma_H^\alpha + \sigma_{33}^{d,\alpha} \quad (8)$$

since $\sigma_{33}^I = 0$

$$c_\alpha < \sigma_{33}^{II} >^\alpha + c_\beta < \sigma_{33}^{II} >^\beta = 0$$

Experimental results for cylindrical samples of an $\text{Al}_2\text{O}_3 + \text{SiC}$ composite show that σ_{ij} is zero near the surface /8/ and not zero in the center of the cross section /9/. This can be explained since $\frac{\partial \sigma_{22}^I}{\partial y} \neq 0$, therefore $\frac{\partial \sigma_{23}^I}{\partial z} \neq 0$ and $\frac{\partial \sigma_{33}^I}{\partial z} \neq 0$.

A bending beam with rectangular cross section will show that σ_{i3} is zero and $\frac{\partial \sigma_{33}^I}{\partial z} = 0$, although $\frac{\partial \sigma_{11}^I}{\partial z} \neq 0$ and $\frac{\partial \sigma_{22}^I}{\partial z} = 0$. The same stress state holds for RS I after plastic bending with a small curvature. An RS II state may superpose onto the bending stress state. Other examples are notches in specimens of a single-phase material with LS or RS I (macro-RS) state: $\frac{\partial \sigma_{11}^I}{\partial x} \neq 0$ and a triaxial stress state below the notch ground.

In the following, some examples of micro-RS in two- and multiphase materials will be discussed. When macrostresses σ^L and/or σ^I are present, there are micro-RS gradients at the surface and can be calculated as the average over the penetration depth. In a multiphase material without macrostresses, $\langle \sigma^{II} \rangle^\alpha$ may be non-zero from previous plastic deformation. If such a material with macrostresses is loaded, $\langle \sigma_{33}^{II} \rangle^\alpha$ may be non-zero too.

Table 1 summarizes the stress states for one- and two- (multi-) phase materials and the σ_{33} -gradients to be evaluated.

2.112b Material state, the cases handled

The experimental methods use powder or small parts of the sample. Errors may arise from an improper choice or cutting or grinding of the sample material, so that additional micro-RS may feign unrealistic values. It has to be distinguished, whether the following parameters are zero, are constant versus the depth from the surface or have a gradient. The parameters to be checked are: D_0 , σ_{33} , σ_{11} , σ_{22} , micro-RS, σ_{13} , content of phases, μ , texture. In some cases a few parameters can be determined by calculation or by modeling.

Table 2 gives a survey of the different possibilities of determining D_0 . The relation between D_0 and σ_{33} should be taken with care.

Table 1. Appearance of load-, macro- and microstresses in one- and in two-phase materials after different material treatments. Those kinds of microstresses II and III are considered, that cause line shifts as do macrostresses. Stresses that are negligible are written in parentheses.

material	geometry	treatment	LS			RS I			microstress II			microstress III		
			$\sigma_{11/12}^L$	σ_{33}^L	σ_{13}^L	σ_{11}^I	σ_{33}^I	σ_{13}^I	σ_{11}^{II}	σ_{33}^{II}	σ_{13}^{II}	σ_{11}^{III}	σ_{33}^{III}	σ_{13}^{III}
one phase	plane (initially)	elastic bending	×	(×)										
		plastic bending				×	×					×	×	
		plastic elongation										×	×	
		grinding				×								×
		shot-peening				×								
		roll-peening				×						×	×	×
two phases		elastic bending	×	(×)					×	×				
		plastic bending				×	×		×	×		×	×	
		plastic elongation							×	×		×	×	
		grinding				×			×	×	×	×	×	×
		shot-peening				×			×	×		×	×	
		roll-peening				×			×	×	×	×	×	×
one phase	cylindrical	heat treatment				×	×							
two phases	cylindrical	heat treatment				×	×		×	×				

Table 2. Determination of D_0 , possible sources of errors.

procedure	characterization
$\left. \begin{array}{l} \text{strain - stress free direction} \\ \text{relationship } \sigma_{33} - D_0 \end{array} \right\} \text{ with } \sigma_{33} = 0$ $\sigma_{33} \neq 0$	mechanically isotropic and textured materials D_0 from other material test
$\Delta\sigma_{ii}$	exact D_0 not necessary
σ_H, σ^d	exact D_0 only for σ_H necessary
stress-free heat treatment	decomposition at the surface
powder	micro-RS $\leftrightarrow$ hydrostatic stress state
thinned plate	micro-RS, hydrostatic stress state
cube (n-diffraction)	micro-RS, hydrostatic stress state
stress-free zone	hydrostatic stress state
stress state on surface $z = 0$	extrapolation necessary

Note: pure hydrostatic stress state: $\sigma_1 = \sigma_2 = \sigma_3$ only in two-phase and multiphase material

2.112c The strain-stress-free direction ψ^*

The fundamental equation and the definition of the strain-stress free direction

$$\frac{1}{2} (\varepsilon_{\varphi, \psi > 0} + \varepsilon_{\varphi, \psi < 0}) = 0 \quad \text{for } \psi = \psi^* \quad (9)$$

yields for a mechanically isotropic material with no steep gradient:

$$\begin{aligned} \frac{1}{2} (\varepsilon_{\varphi, +\psi} + \varepsilon_{\varphi, -\psi}) = \frac{1}{2} s_2 [\bar{\sigma}_{11} \cos^2 \varphi \sin^2 \psi + \bar{\sigma}_{22} \sin^2 \varphi \sin^2 \psi \\ + \bar{\sigma}_{12} \sin 2\varphi \sin^2 \psi + \bar{\sigma}_{33} \cos^2 \psi] + s_1 \cdot [\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33}] = 0 \end{aligned} \quad (10)$$

$$\varphi = 0: \quad \frac{1}{2} s_2 [(\bar{\sigma}_{11} - \bar{\sigma}_{33}) \cdot \sin^2 \psi + \bar{\sigma}_{33}] + s_1 \cdot [\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33}] = 0 \quad (11)$$

$$\sin^2 \psi^* = \frac{-s_1 (\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33}) - \frac{1}{2} s_2 \bar{\sigma}_{33}}{\frac{1}{2} s_2 (\bar{\sigma}_{11} - \bar{\sigma}_{33})} \quad (12)$$

$$\sin^2 \psi^* = \frac{-s_1}{\frac{1}{2}s_2} \left[1 + \frac{\bar{\sigma}_{22} - \bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}} + \frac{\left(3 + \frac{\frac{1}{2}s_2}{s_1}\right) \cdot \bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}} \right] \quad (13)$$

This equation reduces to

$$\sin^2 \psi^* = \frac{-s_1}{\frac{1}{2}s_2} \left[1 + \frac{\bar{\sigma}_{22} - \bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}} \right] = \frac{-s_1}{\frac{1}{2}s_2} \left[1 + \frac{\frac{\partial D_{\varphi=90^\circ, \psi}}{\partial \sin^2 \psi}}{\frac{\partial D_{\varphi=0^\circ, \psi}}{\partial \sin^2 \psi}} \right] \quad (14)$$

when $|\bar{\sigma}_{11} + \bar{\sigma}_{22}| \gg |\bar{\sigma}_{33}|$.

If $\bar{\sigma}_{33} = 0$, the formula reduces to

$$\sin^2 \psi^* = \frac{-s_1}{\frac{1}{2}s_2} \left(1 + \frac{\bar{\sigma}_{22}}{\bar{\sigma}_{11}} \right) \xrightarrow{\text{mech.}} \frac{\nu}{1+\nu} \left(1 + \frac{\bar{\sigma}_{22}}{\bar{\sigma}_{11}} \right) \quad (15)$$

$$\varphi = 45^\circ: \quad \frac{1}{2}s_2 \left[\frac{1}{2}(\bar{\sigma}_{11} + \bar{\sigma}_{22} + 2\bar{\sigma}_{12} - 2\bar{\sigma}_{33}) \cdot \sin^2 \psi + \bar{\sigma}_{33} \right] + s_1(\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33}) = 0 \quad (16)$$

$$\sin^2 \psi^* = \frac{-2s_1(\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33}) - \frac{1}{2}s_2\bar{\sigma}_{33}}{\frac{1}{2}s_2(\bar{\sigma}_{11} + \bar{\sigma}_{22} + 2\bar{\sigma}_{12} - 2\bar{\sigma}_{33})} \quad (17)$$

The ψ^* -direction is independent of the stresses if $\sigma_{33} = \sigma_{12} = 0$. In this case, the formula reduces to

$$\sin^2 \psi^* = \frac{-2s_1}{\frac{1}{2}s_2} \xrightarrow{\text{mech.}} \frac{2\nu}{1+\nu} \quad (18)$$

$$\varphi = 90^\circ \quad \frac{1}{2}s_2 \left[(\bar{\sigma}_{22} - \bar{\sigma}_{33}) \cdot \sin^2 \psi \right] + s_1 \left[\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33} \right] = 0 \quad (19)$$

$$\sin^2 \psi^* = \frac{-s_1(\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33}) - \frac{1}{2}s_2\bar{\sigma}_{33}}{\frac{1}{2}s_2(\bar{\sigma}_{22} - \bar{\sigma}_{33})} \quad (20)$$

$$\text{if } \bar{\sigma}_{33} = 0 \quad \sin^2 \psi^* = \frac{-s_1}{\frac{1}{2}s_2} \left(1 + \frac{\bar{\sigma}_{11}}{\bar{\sigma}_{22}} \right) \xrightarrow{\text{mech.}} \frac{\nu}{1+\nu} \left(1 + \frac{\bar{\sigma}_{11}}{\bar{\sigma}_{22}} \right) \quad (21)$$

Table 3 contains the formulae for the triaxial stress state and the formulae for the two-dimensional general, rotationally symmetrical and the torsional stress state, and the formulae for the uniaxial stress state.

Table 3. Formulae of $\sin^2 \psi^*$ for different stress states. $\bar{\sigma}_\varphi = \bar{\sigma}_{11} \cos^2 \varphi + \bar{\sigma}_{22} \sin^2 \varphi$

stress state	φ azimuth		$\varphi = 0^\circ$	
	$\bar{\sigma}_{33} \neq 0$	$\bar{\sigma}_{33} = 0$	$\bar{\sigma}_{33} \neq 0$	$\bar{\sigma}_{33} = 0$
$\bar{\sigma}_{11} \neq \bar{\sigma}_{22} \neq \bar{\sigma}_{33}$	$-\frac{\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33}}{\bar{\sigma}_\varphi - \bar{\sigma}_{33}} \frac{s_1}{\frac{1}{2}s_2} - \frac{\bar{\sigma}_{33}}{\bar{\sigma}_\varphi - \bar{\sigma}_{33}}$	$-\frac{\bar{\sigma}_{11} + \bar{\sigma}_{22}}{\bar{\sigma}_\varphi} \frac{s_1}{\frac{1}{2}s_2}$	$-\frac{\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}} \frac{s_1}{\frac{1}{2}s_2} - \frac{\bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}}$	$-\frac{\bar{\sigma}_{11} + \bar{\sigma}_{22}}{\bar{\sigma}_{11}} \frac{s_1}{\frac{1}{2}s_2}$
$\bar{\sigma}_{11} = \bar{\sigma}_{22} \neq \bar{\sigma}_{33}$	$-\frac{2\bar{\sigma}_{11} + \bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}} \frac{s_1}{\frac{1}{2}s_2} - \frac{\bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}}$	$-2 \frac{s_1}{\frac{1}{2}s_2}$	$-\frac{2\bar{\sigma}_{11} + \bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}} \frac{s_1}{\frac{1}{2}s_2} - \frac{\bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}}$	$-2 \frac{s_1}{\frac{1}{2}s_2}$
$\bar{\sigma}_{11} = -\bar{\sigma}_{22} \neq \bar{\sigma}_{33}$	$-\frac{\bar{\sigma}_{33}}{\bar{\sigma}_\varphi - \bar{\sigma}_{33}} \left(\frac{s_1}{\frac{1}{2}s_2} + 1 \right)$	0	$-\frac{\bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}} \left(\frac{s_1}{\frac{1}{2}s_2} + 1 \right)$	0
$\bar{\sigma}_{11} \neq \bar{\sigma}_{22} = \bar{\sigma}_{33} = 0$		$-\frac{1}{\cos^2 \varphi} \frac{s_1}{\frac{1}{2}s_2}$		$-\frac{s_1}{\frac{1}{2}s_2}$

The value of $D_0(\psi^*)$ found by an approximate formula ($\bar{\sigma}_{33} = 0$) can per definition not be inserted for a triaxial stress evaluation. The errors would be:

$$D_0(\psi^*) - D_0 = D_0 \left(2s_1 + \frac{\bar{\sigma}_{22}}{\bar{\sigma}_{11}} s_1 + \frac{1}{2} s_2 \right) \bar{\sigma}_{33} \frac{\text{mech.}}{D_0} \frac{1 - \nu - \nu \frac{\bar{\sigma}_{22}}{\bar{\sigma}_{11}}}{E} \bar{\sigma}_{33} \quad (22)$$

$$D_0(\psi^*) - D_0 = D_0 \left(3s_1 + \frac{1}{2} s_2 \right) \bar{\sigma}_{33} \frac{\text{mech.}}{D_0} \frac{1 - 2\nu}{E} \bar{\sigma}_{33} \quad \text{with } \bar{\sigma}_{11} = \bar{\sigma}_{22} \quad (23)$$

The real purpose of this procedure which has been applied for many years ($\bar{\sigma}_{33} = 0$) is to obtain an indication about the presence of $\bar{\sigma}_{33}$ or an alteration of D_0 .

Equ. 10 with $\bar{\sigma}_{33} = 0$ was discussed by /4/. Fig. 1 shows the stress-free directions in a polar diagram $\sin \psi^*$ versus azimuth φ for $\nu = 0.28$ (steels) and $m = \frac{\bar{\sigma}_{22}}{\bar{\sigma}_{11}}$ /4/.

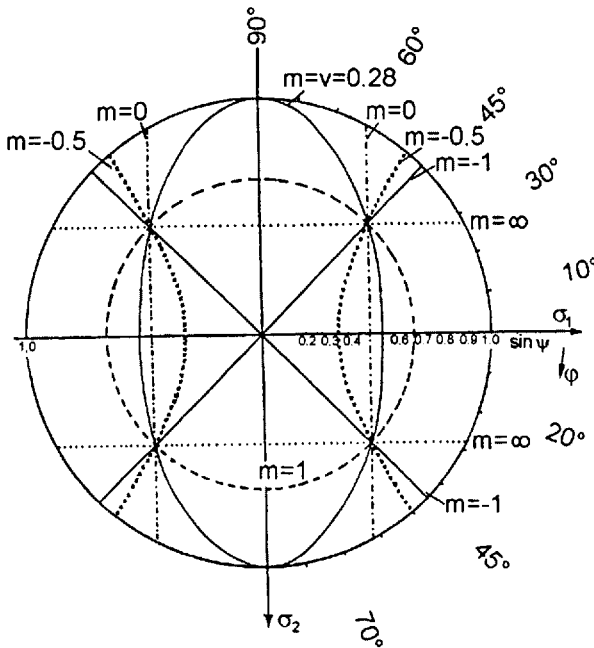


Figure 1. Stress-free directions for steels ($\nu = 0.28$) and $\bar{\sigma}_{33} = 0$ with the parameter $m = \frac{\bar{\sigma}_{22}}{\bar{\sigma}_{11}}$ /4/.

2.112d The $D_0 - \sigma_{33}$ relation

Corresponding to the fundamental equation, the exact value of D_0 is necessary to determine σ_{33} . The methods to measure or to evaluate D_0 and possible errors will be dealt with in the following. First of all, a survey of the measuring methods is given in Table 4. The material parameters that can vary are listed in Table 4. Distinction has been made between stress states

with and without a gradient as a function of the distance from the surface. The methods that can be used to get the exact D_0 -value are indicated. In some cases, the relation between σ_{33} and D_0 has to be used in discussing the presence of a D_0 -, a σ_{33} -, or a combined profile. The possible error due to a hydrostatic pressure state should be considered.

To use this table, further assumptions must be made. A gradient is confirmed if the alteration of the respective parameter is clearly measurable within the penetration depth τ_0 for $\psi = 0$. For textured materials or plastically deformed materials that show D-oscillations versus $\sin^2\psi$, the same procedures should be applicable if the influence of texture (oscillations) can be separated. In other words: the D-vs.- $\sin^2\psi$ distributions should be linearized. Methods for this purpose can be found in chapter 2.16. If the previously described methods fail, measurements on powdered material are recommended.

Table 4. Different material states, methods to evaluate D_0 . + recommended, ± manyfold determination after removing layers may be necessary, – not applicable.

material	σ_{11}, σ_{22}	σ_{33}	D_0	method					
				strain-free direction	powder, thin plate	stress-free region	surface extrapolation	second phase etching	relation $D_0 - \sigma_{33}$
one-phase and multiphase,	constant	0	constant	+	+	+	+	+	+
		≠ 0		–	±	+	+	+	+
not textured, no oscillations	or	0	gradient	±	±	+	+	–	–
	gradient	≠ 0		–	–	–	+	–	±

For mechanically isotropic materials and linear D-vs.- $\sin^2\psi$ dependences, the intercept for $\psi = 0$, $D_{\psi=0} - D_0$ can be used to get the relation between D_0 and σ_{33} :

$$D_0 = \frac{D_{\psi=0^\circ}}{1 + s_1 (\bar{\sigma}_{11} + \bar{\sigma}_{22}) + (s_1 + \frac{1}{2} s_2) \bar{\sigma}_{33}} \overset{mech.}{\frac{D_{\psi=0^\circ}}{E - \nu (\bar{\sigma}_{11} + \bar{\sigma}_{22}) + \bar{\sigma}_{33}}} E \tag{24}$$

$$\sigma_{33} = \frac{(D_{\psi=0^\circ} - D_0) - D_0 s_1 (\bar{\sigma}_{11} + \bar{\sigma}_{22})}{D_0 (s_1 + \frac{1}{2} s_2)} \overset{mech.}{\frac{D_{\psi=0^\circ} - D_0}{D_0}} E + \nu (\bar{\sigma}_{11} + \bar{\sigma}_{22}) \tag{25}$$

If one uses mechanical constants, knowing of Young's modulus E and the Poisson's constant ν is necessary. A relation between $\bar{\sigma}_{33}$ and D_0 can generally be found by the

following consideration. According to the basic formula of XSA, the following equation, with $\sigma_{ik} = 0$ ($i \neq k$), is obtained:

$$\begin{aligned} \varepsilon_{\varphi=0^\circ, \psi} &= s_1 (\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33}) + \frac{1}{2} s_2 \bar{\sigma}_{33} + \frac{1}{2} s_2 (\bar{\sigma}_{11} - \bar{\sigma}_{33}) \sin^2 \psi \\ &= s_1 (\bar{\sigma}_{11} - \bar{\sigma}_{33} + \bar{\sigma}_{22} - \bar{\sigma}_{33}) + \left(3s_1 + \frac{1}{2}s_2\right) \bar{\sigma}_{33} + \frac{1}{2} s_2 (\bar{\sigma}_{11} - \bar{\sigma}_{33}) \sin^2 \psi \end{aligned} \quad (26)$$

$$\varepsilon_{\varphi=90^\circ, \psi} = s_1 (\bar{\sigma}_{11} - \bar{\sigma}_{33} + \bar{\sigma}_{22} - \bar{\sigma}_{33}) + \left(3s_1 + \frac{1}{2}s_2\right) \bar{\sigma}_{33} + \frac{1}{2} s_2 (\bar{\sigma}_{22} - \bar{\sigma}_{33}) \sin^2 \psi \quad (27)$$

$$\begin{aligned} \frac{1}{2} (\varepsilon_{\varphi=0^\circ, \psi} + \varepsilon_{\varphi=90^\circ, \psi}) &= \frac{\frac{1}{2} (D_{\varphi=0^\circ, \psi} + D_{\varphi=90^\circ, \psi}) - D_0}{D_0} \\ &= \left(3s_1 + \frac{1}{2}s_2\right) \bar{\sigma}_{33} + \left(2s_1 + \frac{1}{2}s_2 \sin^2 \psi\right) (\bar{\sigma}_{11} - \bar{\sigma}_{33} + \bar{\sigma}_{22} - \bar{\sigma}_{33}) \end{aligned} \quad (28)$$

With $\sin^2 \psi^* = -2 \frac{s_1}{\frac{1}{2}s_2}$, the value for the biaxial stress state, one gets

$$\bar{\sigma}_{33} = \frac{1}{3s_1 + \frac{1}{2}s_2} \frac{\frac{1}{2} (D_{\varphi=0^\circ, \psi^*} + D_{\varphi=90^\circ, \psi^*}) - D_0}{D_0} \quad (29)$$

the relation between D_0 and $\bar{\sigma}_{33}$ in the stress-free state of $\bar{\sigma}_{11} + \bar{\sigma}_{22}$ state /10/, $D_{\varphi=0^\circ, \psi^*}$ and $D_{\varphi=90^\circ, \psi^*}$ stands for the average of $\frac{1}{2} (D_{0^\circ, \psi^*} + D_{180^\circ, \psi^*})$ respectively of $\frac{1}{2} (D_{90^\circ, \psi^*} + D_{270^\circ, \psi^*})$.

A relation between $\bar{\sigma}_{33}$ and D_0 can also be taken into account in the following way. The lattice strain measurement is made at azimuth $\varphi = \pm 45^\circ$. Thus,

$$\bar{\varepsilon}_{\varphi=\pm 45^\circ, \psi} = s_1 (\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33}) + \frac{1}{2} s_2 \left(\frac{1}{2} \bar{\sigma}_{11} \sin^2 \psi + \frac{1}{2} \bar{\sigma}_{22} \sin^2 \psi + \bar{\sigma}_{33} \cos^2 \psi \right) \quad (30)$$

$$= \left(s_1 + \frac{1}{2}s_2 \cos^2 \psi\right) \bar{\sigma}_{33} + \left(s_1 + \frac{\frac{1}{2}s_2 \sin^2 \psi}{2}\right) (\bar{\sigma}_{11} + \bar{\sigma}_{22}) \quad (31)$$

With $\sin^2 \psi^* = -2 \frac{s_1}{\frac{1}{2}s_2}$ it holds that

$$\bar{\sigma}_{33} = \frac{1}{3s_1 + \frac{1}{2}s_2} \frac{D_{\varphi=\pm 45^\circ, \psi^*} - D_0}{D_0} \quad (32)$$

In Fig. 2 different stress states with linear $D_{\varphi, \psi}$ -vs.- $\sin^2 \psi$ dependences are drawn, and the D_0 -determination is outlined. The data used are given in the caption.

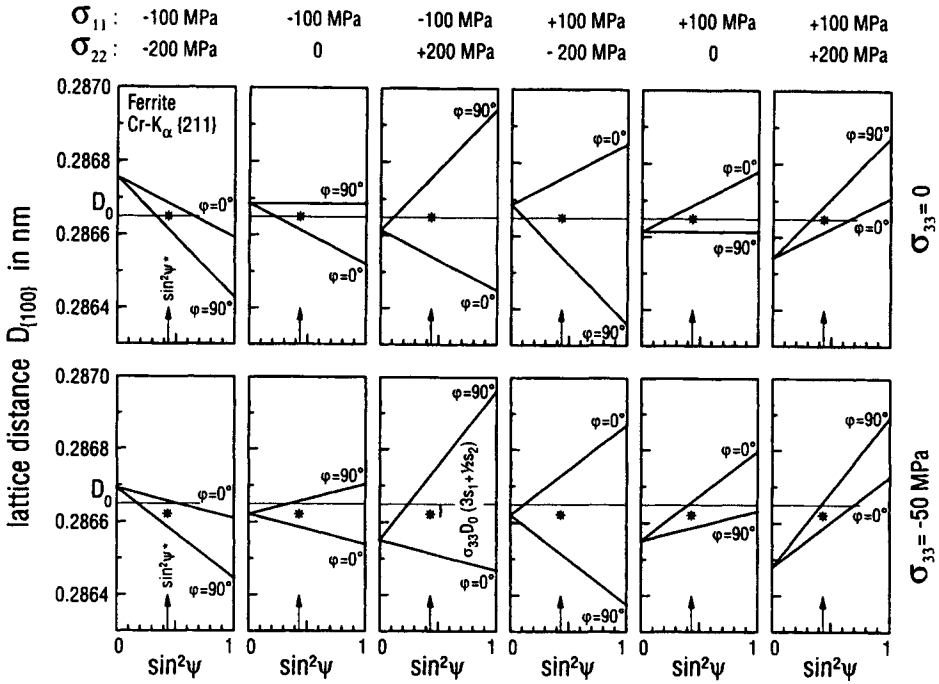


Figure 2. Determination of D_0 for linear $D_{\phi,\psi}$ -vs.- $\sin^2\psi$ dependences

When this evaluation method is applied, the accuracy of $\bar{\sigma}_{33}$ depends of course on the accuracy of the D_0 -determination, on the choice of the XEC and on the precision of the $D_{\phi,\psi}$ -measurements.

In case of thin film-substrate composites, there exists an elegant procedure to determine D_0 without knowing the XEC of the layer material [11].

The obvious assumptions are $\sigma_1 = \sigma_2 = \sigma$, $\sigma_3 = 0$, $\sigma_{ik} = 0$, $D_0(z) = \text{const.}$

For different specimens at any azimuth ϕ , measurements are made of $D_{\phi,\psi}$ vs. $\sin^2\psi$. The

intercept $D_{\phi,\psi=0^\circ}$ and the slope $\frac{\partial D_{\phi,\psi}}{\partial \sin^2\psi}$ are determined by linear regression.

For $\phi = 0^\circ$ and the above assumptions, the basic equation can be expressed

$$\phi = 0^\circ: \quad \frac{D_\psi - D_0}{D_0} = 2s_1 \sigma + \frac{1}{2}s_2 \sigma \sin^2 \psi \quad (33)$$

$$\phi = 0^\circ, \psi^*: \quad \frac{D_{\psi^*} - D_0}{D_0} = 0; \quad 2s_1 \sigma = -\frac{1}{2}s_2 \sigma \sin^2 \psi^* \quad (34)$$

$$D_{\psi=0^\circ} = D_0 - \frac{1}{2}s_2 D_0 \sigma \sin^2 \psi^* = D_0 - \frac{\partial D}{\partial \sin^2 \psi} \sin^2 \psi^* \quad (35)$$

$$\frac{\partial D}{\partial \sin^2 \psi} = 0; \quad D_{\psi=0^\circ} = D_0$$

Fig. 3 /11/ and Fig. 4 /12/ show two examples of a D_0 -determination for a CVD-TiC coating on steel and a CVD-TiN coating on Mo.

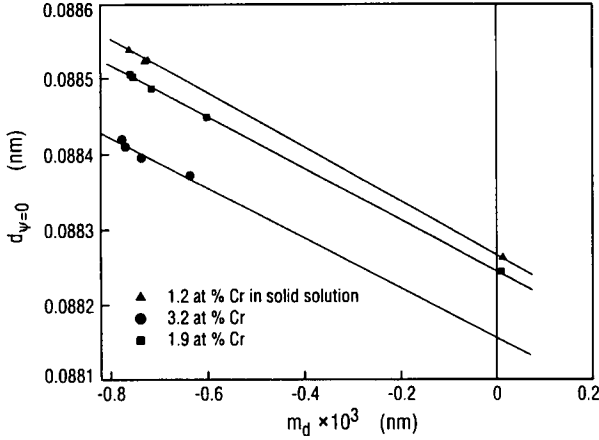


Figure 3. The spacing $d_{\psi=0^\circ}$ versus the slope m_d for CVD-TiC coatings (TiC {422}-Cu K_α reflection) /11/.

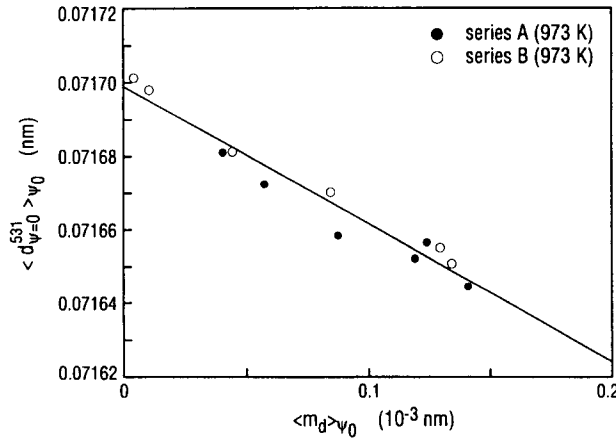


Figure 4. Plot of $\langle d_{\psi=0}^{531} \rangle_{\psi_0}$ versus $\langle m_d \rangle_{\psi_0}$ /12/.

2.112e Nonlinear D-vs.- $\sin^2\psi$ distributions

For stress states with shear components, the ψ -splitting should be ignored and the averaged $\frac{1}{2}(D_{\varphi, \psi > 0} + D_{\varphi, \psi < 0})$ values taken to perform the linear regression. Further procedure is described in 2.112c and d. There is no general method to determine D_0 if there are nonlinear D-vs.- $\sin^2\psi$ distributions with oscillations. As shown in paragraph 2.073, it is possible to

average the $D_{\phi\psi}$ -vs.- $\sin^2\psi$ measurements of different lattice planes. The $\sin^2\psi^*$ -value should be calculated with averaged XEC in this case. Some effort is necessary to get a sufficiently exact value.

The crystallite-group method to evaluate stresses in textured materials is mentioned in chapter 2.07. Here, linear and elliptic dependences D -vs.- $\sin^2\psi$ exist. Therefore, the method in 2.112c and d can be used. The formulae are listed in Table 5.

In the cases of stress gradients in the z -direction, the lattice-strain measurements should be made on thin plates cut out parallel to the surface of the specimen. The measurements should be performed at the surface, in the initial state and then the plate should be thinned by etching on the backside. The D_0 -value can then be evaluated by using the appropriate method mentioned above.

In case of a D_0 -profile at the surface of a specimen, the strain measurement should be made by using different radiations and different peaks to probe different surface regions. The results of the D_0 -profile must be corrected by deconvoluting the specific integrated thickness.

Another way is using a soft radiation with a small penetration depth and an appropriate peak to measure at the outermost surface. Thin layers of different thickness must be etched to get the D_0 -profile.

2.112f Correcting the approximately assumed value of D_0

The stress-tensor evaluation requires the correct D_0 -value. If D_0 is known only approximately, an improved value can be evaluated by the following iteration. If the depth profiles of for example σ_{13} and σ_{33} seem to be unrealistic, the σ_{33} -values in a special depth should be corrected to zero. Hence the D_0 -value will be altered and becomes the correct value. Here we give a procedure based on the general Hooke's law:

$$\sigma_{33} = \frac{1}{\frac{1}{2}s_2} \left[\epsilon_{33} - \frac{s_1(\epsilon_{11} + \epsilon_{22} + \epsilon_{33})}{3s_1 + \frac{1}{2}s_2} \right] ; \quad \epsilon_{ii} = \frac{D_{ii} - D_0}{D_0} \quad (36)$$

$$D_0 \sigma_{33} = \frac{D_{33} - D_0}{3s_1 + \frac{1}{2}s_2} - (D_{11} + D_{22} - 2D_{33}) \frac{s_1}{\frac{1}{2}s_2 (3s_1 + \frac{1}{2}s_2)} \quad (37)$$

If σ_{33} is set to zero, the strain-stress-free lattice constant will become D'_0

$$0 = \frac{D_{33} - D'_0}{3s_1 + \frac{1}{2}s_2} - (D_{11} + D_{22} - 2D_{33}) \frac{s_1}{\frac{1}{2}s_2 (3s_1 + \frac{1}{2}s_2)} \quad (38)$$

Combining both equations,

$$D_0 \sigma_{33} (3s_1 + \frac{1}{2}s_2) = D'_0 - D_0 \quad (39)$$

$$D'_0 = D_0 + D_0 (3s_1 + \frac{1}{2}s_2) \sigma_{33} \frac{mech.}{D_0 + D_0 \frac{1-2\nu}{E} \sigma_{33}} \quad (40)$$

$$\text{With } \frac{\Delta D}{D} = -\frac{\Delta 2\Theta}{2 \tan \Theta} \quad \text{it follows} \quad 2\Theta'_0 = 2\Theta_0 - 2\sigma_{33} \frac{1-2\nu}{E} \tan \Theta_0$$

That is the formula that was previously derived by /13/.

Table 5. Relation between the lattice constant of the stress-free state D_0 and the stress component in the thickness-direction $\bar{\sigma}_{33}$ for cubic materials and for a crystallite group of the ideal rolling texture of iron. XEC $\{211\}$ in 10^{-6} MPa $^{-1}$: $s_1 = -1.25$ and $\frac{1}{2} s_2 = 5.76$; coefficients of the single crystal in 10^{-6} MPa $^{-1}$: $s_{11} = 7.67$, $s_{12} = -2.83$, $s_{44} = 8.57$ and $s_0 = 6.22/7$.

	quasiisotropic $\{211\}$ - lattice plane	textured $\{211\}\langle 01\bar{1} \rangle$ crystallite group
$D_0 =$	$\frac{D_{\psi=0^\circ}}{1 + s_1(\bar{\sigma}_{11} + \bar{\sigma}_{22}) + (s_1 + \frac{1}{2} s_2) \bar{\sigma}_{33}}$ $\frac{D_{\psi=0^\circ}}{1 - 1.25(\bar{\sigma}_{11} + \bar{\sigma}_{22}) + 4.51 \bar{\sigma}_{33}}$	$\frac{D_{\psi=0^\circ}}{1 + (s_{12} + \frac{1}{6} s_0) \bar{\sigma}_{11} + (s_{12} + \frac{1}{3} s_0) \bar{\sigma}_{22} + (s_{12} + \frac{1}{2} s_{44} + \frac{1}{2} s_0) \bar{\sigma}_{33}}$ $\frac{D_{\psi=0^\circ}}{1 - 1.79 \sigma_{11} - 0.76 \sigma_{22} + 4.56 \bar{\sigma}_{33}}$
$\bar{\sigma}_{33} =$	$\frac{\epsilon_{33}}{3 s_1 + \frac{1}{2} s_2} - \frac{s_1 (\epsilon_{11} + \epsilon_{22} - 2 \epsilon_{33})}{\frac{1}{2} s_2 (3 s_1 + \frac{1}{2} s_2)}$ $\frac{\epsilon_{11}}{2.01} + \frac{\epsilon_{11} + \epsilon_{22} - 2 \epsilon_{33}}{9.26}$	$\frac{\epsilon_{33}}{s_{11} + 2 s_{12}} - \frac{2(\epsilon_{11} - \epsilon_{33}) \left[s_{44} (s_{12} + \frac{1}{6} s_0) - \frac{1}{3} s_0 (s_{12} + \frac{1}{3} s_0) \right]}{s_{44} (s_{11} + s_{12}) (s_{44} + \frac{2}{3} s_0)} - \frac{2(\epsilon_{22} - \epsilon_{33}) (s_{12} + \frac{1}{3} s_0)}{s_{44} (s_{11} + 2 s_{12})}$ $\frac{\epsilon_{33}}{2.01} + \frac{\epsilon_{11} - \epsilon_{33}}{7.95} + \frac{\epsilon_{22} - \epsilon_{33}}{11.36}$
$\sin^2 \psi^* =$ $\varphi = 0^\circ$	$\frac{-s_1}{\frac{1}{2} s_2} \left[\frac{\bar{\sigma}_{11} + \bar{\sigma}_{22} + \left(1 + \frac{\frac{1}{2} s_2}{s_1} \right) \bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}} \right]$ $\frac{0.22 (\bar{\sigma}_{11} + \bar{\sigma}_{22}) - 0.78 \bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}}$	$\frac{-(s_{12} + \frac{1}{6} s_0) \sigma_{11} - (s_{12} + \frac{1}{3} s_0) \bar{\sigma}_{22} - (s_{12} + \frac{1}{2} s_{44} + \frac{1}{2} s_0) \bar{\sigma}_{33}}{\frac{1}{2} (s_{44} + \frac{2}{3} s_0) (\bar{\sigma}_{11} - \bar{\sigma}_{33})}$ $\frac{0.28 \bar{\sigma}_{11} + 0.12 \bar{\sigma}_{22} - 0.72 \bar{\sigma}_{33}}{\bar{\sigma}_{11} - \bar{\sigma}_{33}}$

Table 5 continued.

	textured $\{111\}\langle 2\bar{1}\bar{1}\rangle$ crystallite group	textured $\{100\}\langle 011\rangle$ crystallite group
$D_0 =$	$\frac{D_{\psi=0^\circ}}{1 + (s_{12} + \frac{1}{3}s_0)(\bar{\sigma}_{11} + \bar{\sigma}_{22}) + (s_{12} + \frac{1}{2}s_{44} + \frac{1}{3}s_0)\bar{\sigma}_{33}}$ $\frac{D_{\psi=0^\circ}}{1 - 0.76(\bar{\sigma}_{11} + \bar{\sigma}_{22}) + 3.53\bar{\sigma}_{33}}$	$\frac{D_{\psi=0^\circ}}{1 + s_{12}(\bar{\sigma}_{11} + \bar{\sigma}_{22}) + s_{11}\bar{\sigma}_{33}}$ $\frac{D_{\psi=0^\circ}}{1 - 2.83(\bar{\sigma}_{11} + \bar{\sigma}_{22}) + 7.67\bar{\sigma}_{33}}$
$\bar{\sigma}_{33} =$	$\frac{\epsilon_{33}}{s_{11} + 2s_{12}} - \frac{s_{12} + \frac{1}{3}s_0}{\frac{1}{2}s_{44}(s_{11} + 2s_{12})}(\epsilon_{11} + \epsilon_{22} - 2\epsilon_{33})$ $\frac{\epsilon_{33}}{2.01} + \frac{\epsilon_{11} + \epsilon_{22} - 2\epsilon_{33}}{11.36}$	$\frac{\epsilon_{33}}{s_{11} - 2s_{12}} - \frac{s_{12}}{(s_{11} - s_{12})(s_{11} + 2s_{12})}(\epsilon_{11} + \epsilon_{22} - 2\epsilon_{33})$ $\frac{\epsilon_{33}}{2.01} + \frac{\epsilon_{11} + \epsilon_{22} - 2\epsilon_{33}}{7.46}$
$\sin^2\psi^* =$	$\frac{-(s_{12} + \frac{1}{3}s_0)(\bar{\sigma}_{11} + \bar{\sigma}_{22}) - (s_{12} + \frac{1}{2}s_{44} + \frac{1}{3}s_0)\bar{\sigma}_{33}}{(\frac{1}{2}s_{44} + \frac{1}{6}s_0)\bar{\sigma}_{11} - \frac{1}{6}s_0\bar{\sigma}_{22} - \frac{1}{2}s_{44}\bar{\sigma}_{33}}$	$\frac{-s_{12}(\bar{\sigma}_{11} + \bar{\sigma}_{22}) - s_{11}\bar{\sigma}_{33}}{\frac{1}{2}[(s_{44} + s_0)\bar{\sigma}_{11} + s_0\bar{\sigma}_{22} - (2s_0 + s_{44})\bar{\sigma}_{33}]}$
$\varphi = 0^\circ$	$\frac{0.14(\bar{\sigma}_{11} + \bar{\sigma}_{22}) - 0.66\bar{\sigma}_{33}}{\bar{\sigma}_{11} - 0.19\bar{\sigma}_{22} - 0.81\bar{\sigma}_{33}}$	$\frac{0.38(\bar{\sigma}_{11} + \bar{\sigma}_{22}) - 1.04\bar{\sigma}_{33}}{\bar{\sigma}_{11} + 0.42\bar{\sigma}_{22} - 1.42\bar{\sigma}_{33}}$

2.113 Experimental results

2.113a Examples of D_0 determination and of correlation between D_0 and σ_{33}

Fig. 5 demonstrates examples of D -vs.- $\sin^2\psi$ plots for different stress states of mechanically isotropic materials and the relation of the stress-independent point to the D_0 -value. The details are given in the figure and in the caption.

For a textured material, an example for strain-stress-independent points and for the determination of the D_0 -value according to the crystallite-group method are given in Fig. 6.

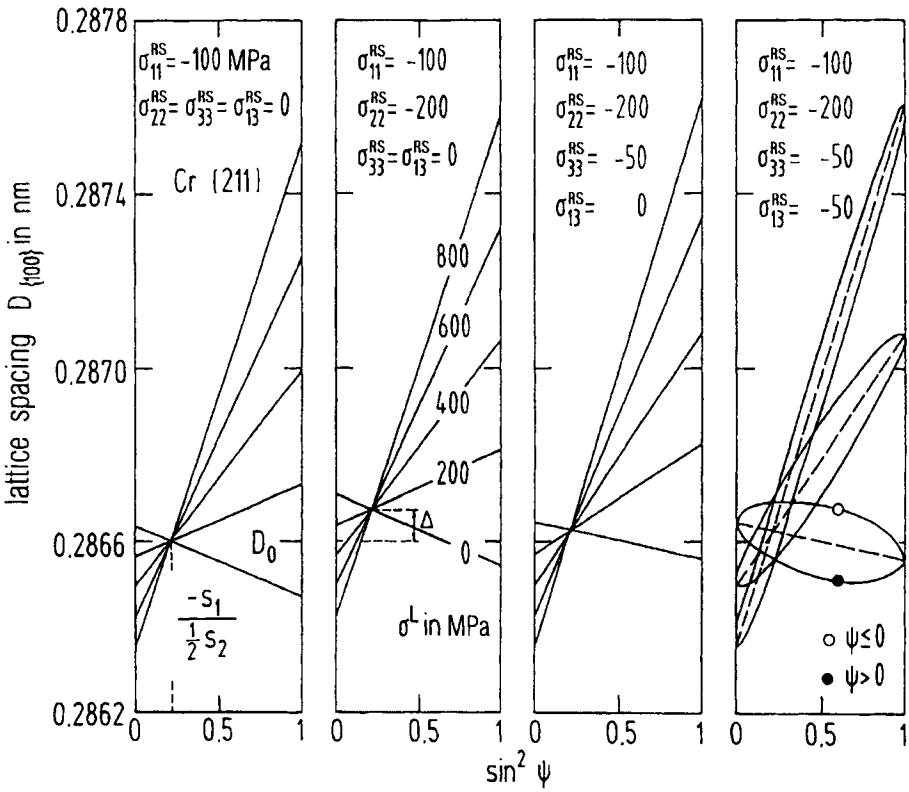


Figure 5. Lattice-strain distribution, stress-independent direction and load stress as a parameter superimposed onto different residual-stress states, calculated for quasiisotropic iron, Cr- $K\alpha$ radiation and $\{211\}$ lattice plane, $\Delta = D' - D_0/7$

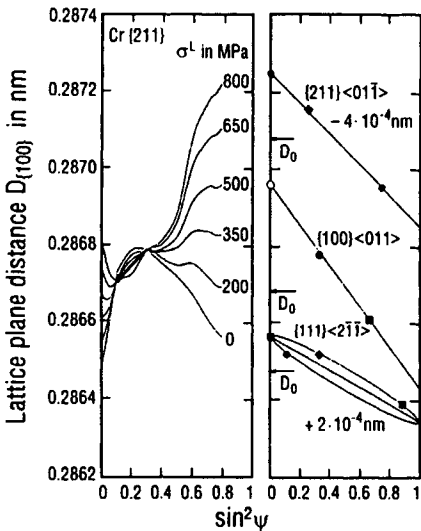


Figure 6. Measured lattice-strain distribution of a textured steel strip, load stress as a parameter; left: stress-independent directions, Cr- K_{α} radiation and $\{211\}$ -lattice plane; right: crystallite-group elaboration and strain-free directions, different radiations and lattice planes /14,15/.

2.113b Heat treatment of materials

Heat treatment is often used for stress relief. Alteration of the microstructure due to decomposition of the alloy may be counteracted by removing (etching) the surface zone of the material. An example demonstrates the care that should be taken in determining the correct value of D_0 . Test specimens were made, and after that two cycles of stress relief were performed. To remove the decomposed regions, 200 μm of the surface layer was etched. As Fig. 7 demonstrates, the D_0 -values determined with different wavelengths are not constant and show gradients.

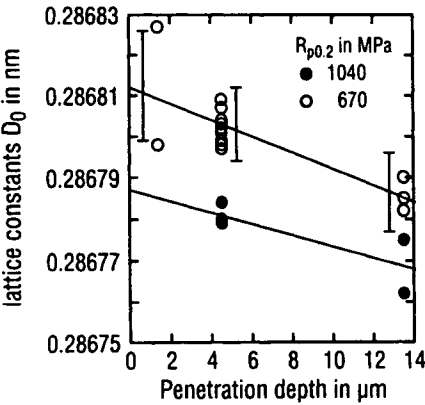


Figure 7. Lattice constants determined by Cu-, Cr- and Mo- K_{α} radiations on a 54NiCrMoV6 steel in two states after stress-relief treatments /16/.

In the case of multiphase materials, the difference between the expansion coefficients of the phases is the origin of micro-RS. The direct measurement of the exact value of D_0 is not possible. Fig. 8 demonstrates an example of a duplex steel containing, approximately 50 vol.% of ferrite and austenite each /17/. After every thermal treatment, a surface layer of 200 μm thickness was etched.

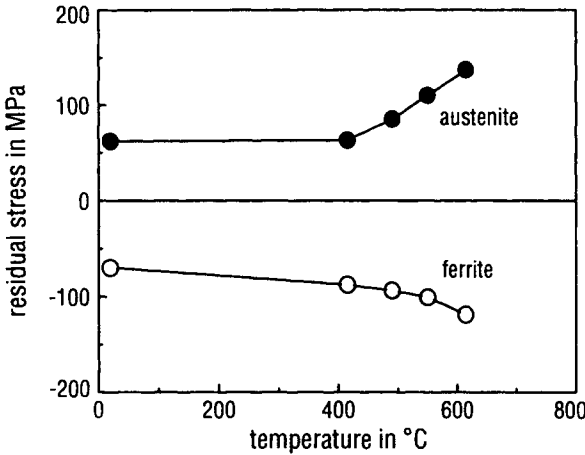


Figure 8. Phase-RS of a duplex steel after annealing for 2 hours; Cr- K_{α} radiation, ferrite $\{211\}$, austenite $\{220\}$ /17/.

2.113c Filings, thin plates

An often-applied method to obtain the strain-stress-free lattice distance is based on measurements on thin filings. However, the following problems may arise: The filing manufacturing may introduce micro-RS and alter the lattice parameter. Second, thermal stress relief may alter the composition and thus the lattice parameter. The D_0 -evaluation on filings is influenced by a hydrostatic stress state. In multiphase materials, the micro-RS in the phases compensate each other. The relationship between the hydrostatic stress state σ_H and the deviation from the strain-stress-free lattice distance $D_H - D_0$ is given by the following equation with $\epsilon_1 - \epsilon_3 = \epsilon_2 - \epsilon_3 = 0$:

$$\sigma_H = \frac{\epsilon_{33}}{\frac{1}{2}s_2} \left(1 - \frac{3s_1}{3s_1 + \frac{1}{2}s_2} \right) = \frac{\epsilon_{33}}{3s_1 + \frac{1}{2}s_2} \quad (41)$$

$$\sigma_H = \frac{D_H - D_0}{D_0 \left(3s_1 + \frac{1}{2}s_2 \right)} \frac{mech.}{D_0} \frac{D_H - D_0}{D_0} \frac{E}{1 - 2\nu} \quad (42)$$

This equation is valid for single-phase and multiphase, mechanically isotropic and textured materials. Examples are given in Table 6.

Thin plates are often used, because no macrostresses are present. But triaxial micro-RS may be present, partly of them as hydrostatic stress. Both have to be considered when evaluating the exact value of D_0 .

Table 6. Lattice constants of the stress-free state of steel and the apparent hydrostatic-RS state of filings /7/.

steel	condition	D in nm		apparent hydrostatic stresses in MPa
		compact specimen	filings	
1.0370	cold rolled	0.286680	0.286658	- 40
25CrMo4	hot rolled, quenched and tempered	0.286750	0.286702	- 80
X2CrNiMoV22-5	hot rolled, 1050°C/H ₂ O	ferrite 0.287967 austenite 0.360578	0.287888 0.360611	- 140 + 70

2.113d Stress-free zone in material

The determination of D_0 in mechanically surface treated materials is only possible if the stressed material region is removed. The D_0 -value in the unstressed zone may be influenced by a hydrostatic stress. This method assumes that no alteration of the D_0 -value takes place in the surface region during the manufacturing of the component or the test specimen. The result of the measurement must be assessed with care, because diffraction methods always evaluate a stress difference, for example $\sigma_{11} - \sigma_{33}$ in special front cuts, or $\sigma_{22} - \sigma_{33} = 0$ in the center of bars.

2.113e D_0 -value by extrapolating the stress-free state to $z = 0$ (surface)

Efforts to determine the RS-tensor of the surface region of surface treated or plastically deformed materials, and here especially the knowledge of the stress-free D_0 -value and the biaxial RS-state at the surface ($z = 0$) itself were enriched by the papers /10,18,19/, see also section 2.153e. The idea is to determine the triaxial RS-state in dependence on the penetration depth τ and to extrapolate to $\tau = 0$ where $\sigma_{33} = 0$. The procedure will be explained on the basis of an example /20/. Further tests will show whether the accuracy of measurements in the front region of 2θ with X-rays is sufficient to determine reliable results.

The assumptions are $\sigma_{33}(z) \neq 0$, $D_0(z) = \text{const.}$, and no D-oscillations versus $\sin^2\psi$. D-vs.- $\sin^2\psi$ distributions are needed for different $\{hkl\}$, for different radiations by means of Ω - and Ψ -diffractometers, particularly using synchrotron radiation.

The goal is to select appropriate $\{hkl\}$ to get a wide range of D-vs.- $\sin^2\psi$ dependences at different and especially at small penetration depths. Fig. 9 illustrates some of the possibilities for the iron phase ferrite. The penetration depth-vs.- $\sin^2\psi$ curves for Ω - and Ψ -diffractometers and for the Cr-peaks $\{110\}$ and $\{211\}$ are plotted. As indicated, the D-values will be taken for the same penetration depth on the $\{110\}$ peak measured in the Ω - and the Ψ -mode /19/. According to /20/, the measurements are done on different peaks in Ω -mode. The goal is to measure as many D-vs.- $\sin^2\psi$ data as possible within the margins of Ω - and Ψ -curves.

The approach is outlined in the following:

$$\frac{D_{\varphi=0^\circ, \psi} - D_0}{D_0} = \varepsilon_{0^\circ, \psi} = \frac{1}{2}s_2[(\sigma_1 - \sigma_3)\sin^2\psi + \sigma_3] + s_1(\sigma_1 + \sigma_2 + \sigma_3) \quad (43)$$

$$\frac{D_{\varphi=90^\circ, \psi} - D_0}{D_0} = \varepsilon_{90^\circ, \psi} = \frac{1}{2}s_2[(\sigma_2 - \sigma_3)\sin^2\psi + \sigma_3] + s_1(\sigma_1 + \sigma_2 + \sigma_3) \quad (44)$$

$$(\sigma_1 - \sigma_3)(\tau) = \frac{1}{\frac{1}{2}s_2} \frac{\partial \varepsilon_{0^\circ, \psi}(\tau)}{\partial \sin^2\psi} = \frac{1}{D_0 \frac{1}{2}s_2} \frac{\partial D_{0^\circ, \psi}(\tau)}{\partial \sin^2\psi} \quad (45)$$

$$(\sigma_2 - \sigma_3)(\tau) = \frac{1}{\frac{1}{2}s_2} \frac{\partial \varepsilon_{90^\circ, \psi}(\tau)}{\partial \sin^2\psi} = \frac{1}{D_0 \frac{1}{2}s_2} \frac{\partial D_{90^\circ, \psi}(\tau)}{\partial \sin^2\psi} \quad (46)$$

$$\frac{D_{\varphi=0^\circ}(\tau) - D_0}{D_0} = \frac{s_1}{D_0 \frac{1}{2}s_2} \left(\frac{\partial D_{0^\circ, \psi}(\tau)}{\partial \sin^2\psi} + \frac{\partial D_{90^\circ, \psi}(\tau)}{\partial \sin^2\psi} \right) + \left(3s_1 + \frac{1}{2}s_2 \right) \sigma_3 \quad (47)$$

$$\sigma_3 = \frac{1}{3s_1 + \frac{1}{2}s_2} \left[\frac{D_{\varphi=0^\circ}(\tau) - D_0}{D_0} - \frac{s_1}{D_0 \frac{1}{2}s_2} \left(\frac{\partial D_{0^\circ, \psi}(\tau)}{\partial \sin^2\psi} + \frac{\partial D_{90^\circ, \psi}(\tau)}{\partial \sin^2\psi} \right) \right] \quad (48)$$

The results are $\sigma_1(\tau)$, $\sigma_2(\tau)$ and $\sigma_3(\tau)$. By extrapolating to $\tau = 0$, $\sigma_1(\tau = 0)$, $\sigma_2(\tau = 0)$ and D_0 can be calculated. The formula (48) for σ_3 can be transformed to the appropriate formula given by /19/.

The example of the evaluation is based on measurements of an electroerosive cut of a Ck45 steel using the $\{110\}$ and $\{211\}$ peaks with Cr- K_α radiation and an Ω -diffractometer. The points at which D-measurements were made are indicated on the penetration curves, left $\{211\}$ and right $\{211\}$, Fig. 10 left below. The measured lattice spacings belonging to these above penetration depths are plotted on the left-hand side of Fig. 10. For specific penetration depths, here 1, 2 and 3 μm , the lattice spacings are plotted, right above. The slopes of these straight lines are used to calculate the RS. They are plotted in the last diagram right below, to get the extrapolation $\tau = 0$. The results for the surface are: $\sigma_{11} = \sigma_{22} = 270 \text{ MPa}$, $\sigma_{33} = 0$, $D_0 = 0.28689 \text{ nm}$.

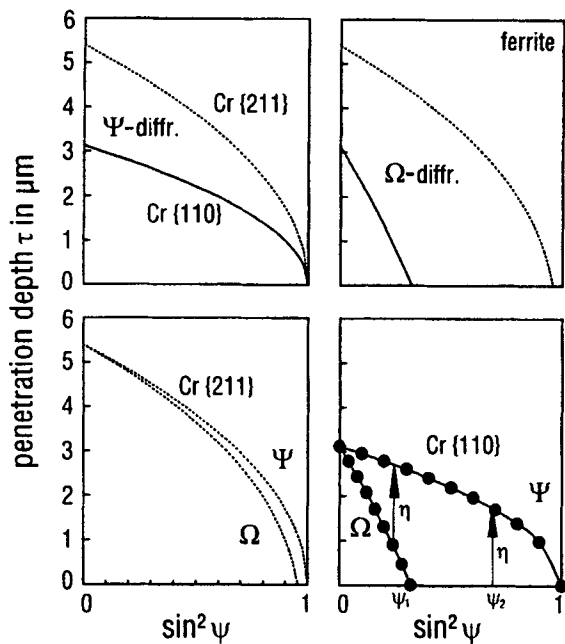


Figure 9. Penetration depths versus $\sin^2\psi$ for Ω - and Ψ -geometry measurements /20/.

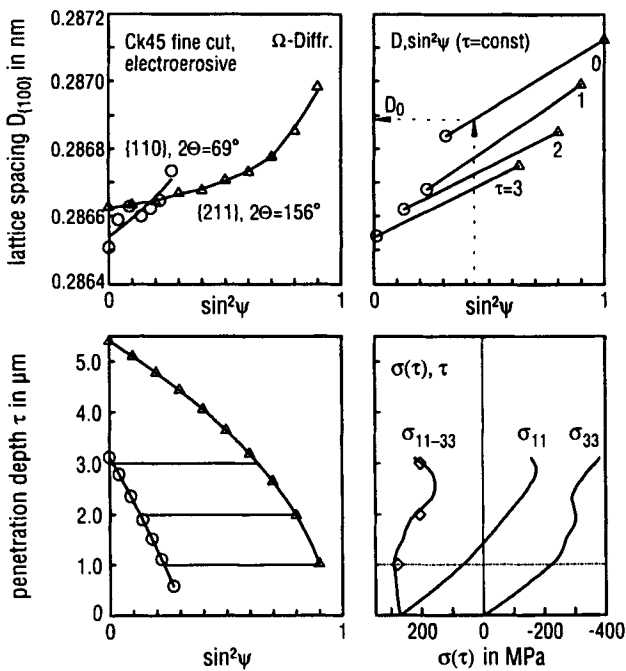


Figure 10. Measurements on Ck 45 /20/.

2.113f RS in the thickness direction

Mechanical surface treatment of materials is usually connected with the presence of RS in the thickness direction. The measured D_0 -profile can be converted into a σ_{33} -profile if there are no gradients of microstructure or composition of the alloy. Examples for RS-gradients in the thickness directions are known for the mechanical treatment of the surface by grinding, shot peening and roll peening /7/. Fig. 11 shows experimental results, including some details. This example demonstrates the case of σ_{33} -profiles, other effects are excluded, /21/.

Interesting examples of triaxial studies on polymeric materials are discussed in section 2.123b.

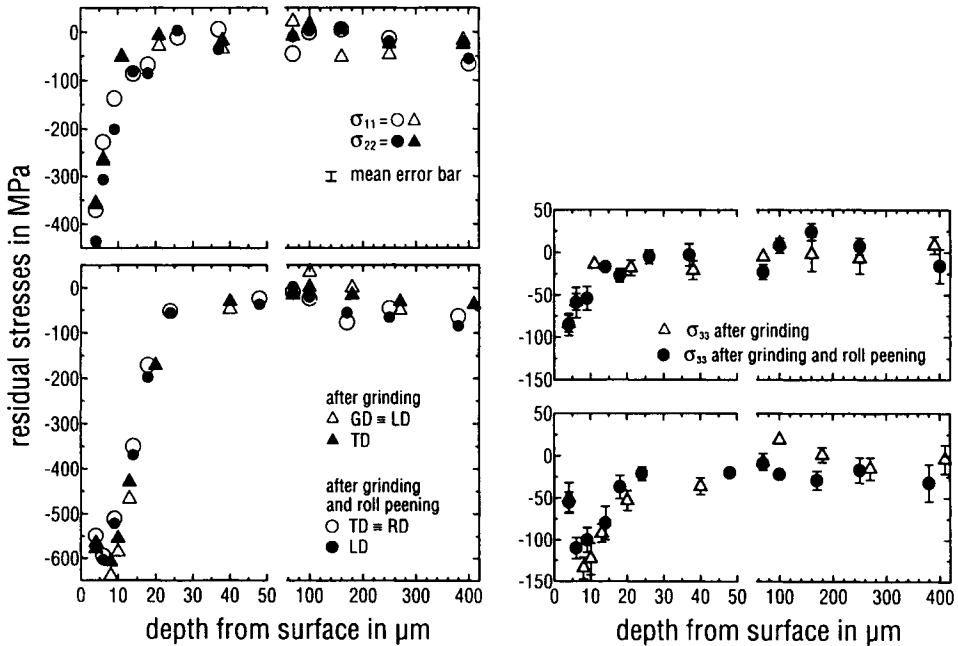


Figure 11. RS-profiles of two differently ground and roll peened (10 kN rolling force, 40 passes) TiAl6V4 specimens; depth from surface equals etching depth plus penetration depth $\tau_{0,3}$ ($\approx 4 \mu\text{m}$), $\{114\}$ -plane, Cu- $K\alpha$ radiation /21/.

2.114 Recommendations

- The relation between D_0 and σ_{33} according to the fundamental equation of XSA may be the cause of misinterpreting the experimental results if influencing parameters are ignored, or if the experiments are not performed carefully. Additional tests should be performed to decide whether there is a D_0 -profile or a σ_{33} -profile or both, influencing the measured lattice strains.
- If there is no microstructure gradient within the cross section of the specimen, the following recommendations should be helpful for experimental studies.

- Assume as correct that value of D_0 , that can be measured or evaluated at the stress-free or stress-poor zones of the specimen.
- Integrating methods over the cross section ($\Sigma\sigma^I = 0$) yield the correct value only if no microstresses are present.
- When using a calibrating specimen to measure D_0 , it should be checked carefully whether there may be differences in heat treatment, chemical composition, mechanical treatment and others.
- In using powders or filings of the same material, it should be checked carefully whether there are micro-RS from producing the powder, or differences in the chemical compositions or/and the microstructures after stress relaxation caused by heat treatment.
- In case of lateral or depth gradients in the composition and the microstructure of the material, it is obvious that the D_0 -value must be taken from the same volume element for which the stress state will be measured.
- Special care is necessary in dealing with problems of steep gradients of strain-stress-chemical composition and microstructure.
- If experimental studies reveal σ_{33} -components of some hundred MPa or more, the procedure should be checked carefully, also the problem and experimental performance should be rechecked.
- In many cases, only a biaxial stress state can be assumed or no information about the stress component in thickness direction is needed. In these cases, the exact and correct value of D_0 is not necessary to determine the stresses. Otherwise, the knowledge of the complete triaxial strain-stress tensors is of principal and practical importance. Stress-states and D_0 -value are characteristic data of materials and of components.

Table 7 summarizes the methods of determining D_0 and possible errors.

Table 7. Methods of D_0 -determination, assumptions, and parameters of influence.

method	assumption		influencing parameters			
	$\sigma_{33} = 0$	$D_0 = \text{const.}$	micro-RS	stress gradient	D_0 -gradient	hydrostatic stress state
strain-free direction, λ_z	×	×		×	×	×
powder, thin plate			×			×
stress-free region			×		×	×
surface extrapolation		×			×	
second phase etching						×

2.115 References

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2.12 Strains and stresses in the phases of dual- / multiphase and of heterogeneous materials

2.121 Historical review

Experimental and theoretical studies on the phases of multiphase and of heterogeneous materials go back to the sixties. There are principally three methods to calculate strains and stresses in the phases (which will be understood in the wide sense) of a material. The first one is based on a theory of Oldroyd, the second one assumes homogeneous strain (Voigt) or homogeneous stress (Reuss) or deals with a weighted average of parameters, calculated according to both assumptions, and the third one is based on the model of Eshelby. It is obvious that studies on the strains and stresses in the phases are the basis of XEC-calculations.

The theory of Oldroyd /1/ was established to calculate the influence of small viscous inclusions on the mechanical parameters of materials. Stroppe /2/ used, developed and adjusted the results on problems of spheroidal wrought iron. Hoffmann and Blumenauer /3/ published experimental results on the elastic behavior of sintered two-phase materials. Hoffmann et al. /4,5/ reported recently on the influence of glass spheres and glass fibers in the polymeric material PBT (polybutyleneterephthalat) on the triaxial stress state. Hauk and Kockelmann /6/ reported on the calculation of the stress distribution of different two-phase materials as layer composite, fiber-reinforced composite, melted and sintered materials.

The model of Eshelby, based on an ellipsoidal, anisotropic inclusion, surrounded by a matrix of a quasiisotropic multiphase material /7/, was used to calculate LS and RS after cooling fiber- (whisker-) reinforced metals and ceramics, taking into account different elastic moduli, thermal expansion coefficients, and yield stresses /8/. The model of Eshelby was also used to calculate macro-RS and different kinds of micro-RS of multiphase and heterogeneous materials /9/. Diffraction methods are the choice of the procedures in evaluating the stress distribution between the phases of a material.

2.122 Calculation of phase stresses

2.122a Method of Oldroyd-Stroppe

The equations developed by Oldroyd-Stroppe were reported by Hauk and Kockelmann /6/ for a two-phase material in a triaxial stress state. The presumptions are:

- Homogeneous stress (Formulae for homogeneous strain /10/)
- Inclusion and matrix are quasiisotropic phases
- The stress component in the radial direction on the border between the matrix and the inclusion is steady/homogeneous
- The deformations at any place are steady

It will be distinguished between the volume, hydrostatic, and the deformation, deviatoric parts of the stresses. The derivation of the formulae is too lengthy for this publication; therefore reference is made to the paper /6/.

2.122b Coupling of phases using the Voigt and the Reuss model

As pointed out previously three models of crystallite coupling are mainly used to describe the elastic behavior of materials. Experimental results of XEC-determination of quasi-single-phase materials show agreement with the calculation using the Eshelby-Kröner model and lie between the calculated values according to the Voigt and the Reuss model. When the following formula is postulated the coupling can be evaluated

$$\frac{1}{2}s_2 = q^V \frac{1}{2}s_2^V + q^R \frac{1}{2}s_2^R \quad (1)$$

$$q^V + q^R = 1 \quad (2)$$

q^V , q^R being the Voigt and the Reuss coupling ratios.

Using Equ. 11 and the calculation results of /12/ one gets $q^V \approx 0.60 \pm 0.05$ for different metals. Besides the coupling of the crystallites it is of interest to study the coupling of the phases in a two-phase material too. According to the Voigt model of homogeneous strain, the compound (index C) Lamé constants λ^C and μ^C are the weighted constants of the phases α and β with phase contents c^α , c^β .

$$\lambda^{C,V} = c^\alpha \lambda^\alpha + c^\beta \lambda^\beta \quad ; \quad \mu^{C,V} = c^\alpha \mu^\alpha + c^\beta \mu^\beta \quad ; \quad c^\alpha + c^\beta = 1 \quad (3)$$

$$E^{C,V} = \frac{(3\lambda^{C,V} + 2\mu^{C,V})\mu^{C,V}}{\lambda^{C,V} + \mu^{C,V}} \quad (4)$$

$$\nu^{C,V} = \frac{\lambda^{C,V}}{2(\lambda^{C,V} + \mu^{C,V})} \quad (5)$$

The analogous formulae for the Reuss model of homogeneous stress use the combinations

$\frac{1}{E}$ and $\frac{-\nu}{E}$ for the averaging of Young's moduli and Poisson's ratios. That yields:

$$E^{C,R} = \frac{E^\alpha E^\beta}{E^\alpha + E^\beta} \quad (6)$$

$$\nu^{C,R} = E^{C,R} \left(c^\alpha \frac{\nu^\alpha}{E^\alpha} + c^\beta \frac{\nu^\beta}{E^\beta} \right) \quad (7)$$

According to Hill /11/, the real coupling of the phases is between those calculated according to the models of Voigt and Reuss, therefore

$$E^C = q^V E^V + q^R E^R \approx \frac{1}{2} (E^V + E^R) \quad (8)$$

$$\nu^C = q^V \nu^V + q^R \nu^R \approx \frac{1}{2} (\nu^V + \nu^R) \quad (9)$$

For the respective averages of the XEC Evenschor and Hauk /10/ derived (in the following shown for phase α , phase β analogous):

$$s_1^{C,V,\alpha} = \left(s_1^\alpha \frac{1-2\nu^C}{1-2\nu^\alpha} + \frac{1}{2} s_2^\alpha \frac{\nu^\alpha - \nu^C}{(1+\nu^\alpha)(1-2\nu^\alpha)} \right) \frac{E^\alpha}{E^C} \quad (10)$$

$$\frac{1}{2} s_2^{C,V,\alpha} = \frac{1}{2} s_2^\alpha \frac{E^\alpha}{E^C} \frac{1+\nu^C}{1+\nu^\alpha} = \frac{1}{2} s_2^\alpha \frac{\frac{1}{2} s_2^C(mech.)}{\frac{1}{2} s_2^\alpha(mech.)} \quad (11)$$

$$s_1^{C,R,\alpha} = s_1^\alpha \quad ; \quad \frac{1}{2} s_2^{C,R,\alpha} = \frac{1}{2} s_2^\alpha \quad (12)$$

/13,14/ have studied the phase coupling by measurements of the applied-stress sharing:

$$m^\alpha = \frac{\partial(\sigma_{11} - \sigma_{33})^\alpha}{\partial \sigma^L} = 2\mu^\alpha \frac{\partial(\varepsilon_{11} - \varepsilon_{33})^\alpha}{\partial \sigma^L} \quad (13)$$

$$m = c^\alpha m^\alpha + c^\beta m^\beta = 1 \quad (14)$$

However, the obtained formula is not invariant in respect to the interchange of the phase notation. The following shows another way of evaluation /15/.

$$\text{With } m^{R,\alpha} = m^{R,\beta} = 1 \quad (15)$$

$$m^{V,\alpha} = \frac{\mu^\alpha}{c^\alpha \mu^\alpha + c^\beta \mu^\beta} \quad (16)$$

and the assumption

$$m^\alpha = q^V m^{V,\alpha} + q^R m^{R,\alpha} \quad (17)$$

one gets after some transformations:

$$q^V = (m^\alpha - 1) \frac{\mu^{C,V}}{\mu^\alpha - \mu^{C,V}} = (m^\beta - 1) \frac{\mu^{C,V}}{\mu^\beta - \mu^{C,V}} \quad (18)$$

Regarding experimental errors of the m^α , m^β -determinations the average of the calculated $q^{V,\alpha}$ and $q^{V,\beta}$ should be taken for further considerations. The differences in the numerator and the denominator of the final formula and the selection of the values of the material parameters imply a large scatter.

The ceramics Al_2O_3 with different contents of ZrO_2 were some of the most tested materials. They had been studied concerning to triaxial states of macro- and micro-RS after sintering /16,17,13,14,18/ and the XEC of different peaks /16,17,13,14/. In the last mentioned papers, the constance of the plastically and thermally induced micro-RS, RS II is supposed. Studies on the mechanical surface treatment of these ceramics will not be dealt with. With the results of measurement and evaluation of m^α , m^β by /16/ and /13,14/ we get the following figure according to the above mentioned formula.

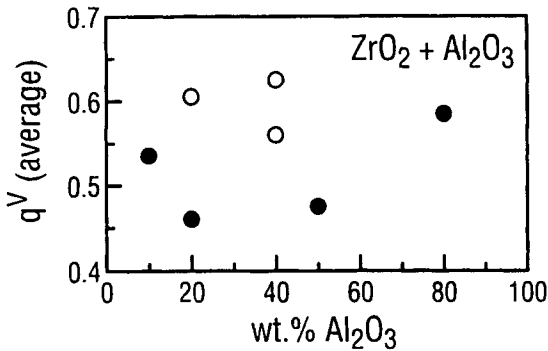


Figure 1. Coupling factor q^V of two-phase materials $\text{ZrO}_2 + \text{Al}_2\text{O}_3$,
O according to /16/, ● to /13,14/.

It seems that there is no principal difference in the coupling between crystallites of different orientations in a single-phase material and the average coupling between phases. The coupling will be altered if cracks or pores are present which create additional inner surfaces.

2.122c Definitions of stresses and transfer factor for multiphase materials

Behnken, Chauhan and Hauk /19/ have developed the methods to determine the different macro- and micro-stresses in multiphase materials. The set of data needed to determine RS and LS in multiphase materials was published by Behnken and Hauk /9/. Diffraction methods detect the average stress $\bar{\sigma}^\alpha$ in each phase:

$$\bar{\sigma}^\alpha = \sigma^L + \sigma^I + \langle \sigma^{II} \rangle^\alpha + \langle \sigma^{III} \rangle^\alpha \quad (19)$$

If we sum up the $\bar{\sigma}^\alpha$ values for the different phases, each weighted with the volume ratio c^α , the macrostress will be obtained

$$\sum_{\alpha=1}^n c^{\alpha} \bar{\sigma}^{\alpha} = \sigma^L + \sigma^I \quad (20)$$

because the average weighted micro-RS are zero

$$\sum_{\alpha=1}^n c^{\alpha} \left(\langle \sigma^{II} \rangle^{\alpha} + \langle \sigma^{III} \rangle^{\alpha} \right) = 0 \quad (21)$$

The phases will have different stresses in relation to the macrostress. The micro-RS have many origins. The micro-RS are proportional to the macrostress as a result of the stresses between the phases and may be altered by relaxation. The other part of the micro-RS is independent of the macrostress. It originates from the different thermal expansion coefficients and different plastic deformations of the phases and of subgrains.

$$\bar{\sigma}^{\alpha} = \bar{\sigma}_0^{\alpha} + f^{\alpha} (\sigma^L + \sigma^I) = \sigma^L + \sigma^I + \langle \sigma^{II} \rangle^{\alpha} + \langle \sigma^{III} \rangle^{\alpha} \quad (22)$$

$$\langle \sigma^{II} \rangle^{\alpha} + \langle \sigma^{III} \rangle^{\alpha} = \bar{\sigma}_0^{\alpha} + (f^{\alpha} - 1) (\sigma^L + \sigma^I) \quad (23)$$

With the tensor f^{α} (transfer tensor) and 1 (unit tensor)

$$(f^{\alpha})_{ijkl} = \frac{\partial (\bar{\sigma})_{ij}^{\alpha}}{\partial (\sigma^L + \sigma^I)_{kl}} \quad (24)$$

The f tensor is isotropic if the material is not textured and if there is no influence of the surface.

$$f_{11} = f_{22} = f_{33} \quad ; \quad f_{12} = f_{13} = f_{23} \quad ; \quad f_{44} = f_{55} = f_{66} \quad (25)$$

$$f_{ij} = f_{ji} \quad i, j = 1, 2, \dots, 6 \quad (26)$$

The relation of the XEC^{α} of the phase α in a single-phase material to the $\text{XEC}^{\text{C},\alpha}$ of the compound-XEC in a multiphase material is as follows:

$$s_1^{C,\alpha} = \frac{\partial \varepsilon_{\varphi, \psi=0}}{\partial \sigma_{11}^L} = \frac{\partial}{\partial \sigma_{11}^L} \left[s_1 (\sigma_{11} + \sigma_{22} + \sigma_{33} + \frac{1}{2} s_2 \sigma_{33}) \right] \quad (27)$$

$$= s_1 \left(\frac{\partial \sigma_{11}}{\partial \sigma_{11}^L} + \frac{\partial \sigma_{22}}{\partial \sigma_{11}^L} \right) + \left(s_1 + \frac{1}{2} s_2 \right) \frac{\partial \sigma_{33}}{\partial \sigma_{11}^L}$$

$$s_1^{C,\alpha} = s_1^{\alpha} (f_{11}^{\alpha} + f_{21}^{\alpha}) + \left(s_1^{\alpha} + \frac{1}{2} s_2^{\alpha} \right) f_{31}^{\alpha} \quad (28)$$

and after similar derivation

$$\frac{1}{2} s_2^{C,\alpha} = \frac{1}{2} s_2^{\alpha} (f_{11}^{\alpha} - f_{31}^{\alpha}) \quad (29)$$

2.122d Separation of macro- and micro-RS in multiphase materials

With the following formulae, which have been discussed in the previous chapters, the determination of RS- and LS-macro- and microstress in two-phase and multi(up to four)-phase materials is possible /9,20,21/. It is distinguished between phases measurable and not measurable by diffraction, between an absolute and a relative determination of the stresses, between load-dependent and load-independent microstresses:

$$\bar{\sigma}^{\alpha} = \sigma^L + \sigma^I + \langle \sigma^{II} \rangle^{\alpha} \quad (30)$$

$$\text{with } \langle \sigma^{III} \rangle^{\alpha} = 0 \quad (31)$$

$$\sum_{\alpha=1}^n c^{\alpha} \langle \sigma^{II} \rangle^{\alpha} = 0 \quad (32)$$

$$\sum_{\alpha=1}^n c^{\alpha} \bar{\sigma}^{\alpha} = \sigma^L + \sigma^I \quad (33)$$

$$\langle \sigma^{II} \rangle_{11}^{\alpha} = (f^{\alpha} - 1)(\sigma^L + \sigma^I)_{11} + \bar{\sigma}_{0,11}^{\alpha} \quad (34)$$

$$\sum_{\alpha=1}^n c^{\alpha} (f^{\alpha} - 1) = 0 \quad (35)$$

$$\bar{\sigma}_{0,11}^{\alpha} = \frac{1}{\frac{1}{2}s_2^{\alpha}} \frac{1}{D_0^{\alpha}} \frac{\partial D_{\phi,\psi}^{\alpha}}{\partial \sin^2 \psi} - \frac{\frac{1}{2}s_2^{C,\alpha}}{\frac{1}{2}s_2^{\alpha}} (\sigma_{11}^L + \sigma_{11}^I) \quad (36)$$

$$\text{if } \bar{\sigma}_0^{\alpha} = 0 \quad \sigma_{11}^I = \frac{1}{\frac{1}{2}s_2^{\alpha}} \frac{1}{D_0^{\alpha}} \frac{\partial D_{\phi,\psi}^{\alpha}}{\partial \sin^2 \psi} \quad (37)$$

It is possible to distinguish between micro-RS caused by thermal and plastical deformation. Table 1 in chapter 2.06 gives a summary of the different micro-RS, their notation, origin, appearance in D-vs.- $\sin^2\psi$ diagrams and their compensation /20/. The above listed formulae are combined in the following three Tables 1, 2 and 3 /20/.

For a successful use of the tables in dealing with polymeric materials, the following should be observed. If there is more than one amorphous phase, all amorphous phases are dealt with as *one* phase. The measurable phase may be a crystalline powder added to an amorphous polymeric material or the crystalline part within a semicrystalline polymer. Examples will be discussed to explain the tables /19/.

Polyethylene (PE) consists of the crystalline α -phase (70 vol.%) and the amorphous phase. The phase-XEC is known from calculations /22/. The details to evaluate LS and RS in PE + glass filler are dealt with in line three of Table 1. The amorphous PE-phase and the glass filler will be handled as the not measurable β -phase.

A different case is the semicrystalline polypropylene (PP) polymer. It consists of two crystalline α - and β -phases (45 vol.% and up to 10 vol.%) within the amorphous phase. The compound-XEC has been experimentally determined several times /23,24/. PP + Al-powder is referred to in Table 3, line three.

Table 1. Determination of load stress as well as macro- and micro-RS on one and two-phases materials from linear lattice strain dependences. Stress data are understood as $\sigma_{11}-\sigma_{33}$. $\bar{\sigma}^n$: average value over the volume of the appropriate phase probed by the X-rays; σ^I, σ^{II} : macro-, micro-RS. Stresses of kind III within the phases according to the deformation are not considered. α, β : crystalline phases; upper index n: general phase; n.m.: not measurable; n.d.: not determinable. **) determined on a separate specimen; ***) not determinable and negligible.

material	phase	macro-RS σ^I	total stress determined $\bar{\sigma}^n$	load stress σ^L	micro-RS σ^{II} ; $\sigma^L = 0$		
					total	of macro-RS σ^I dependent independent ($\bar{\sigma}_0^n$)	
one phase material α	α	$\bar{\sigma}^\alpha$	$\sigma^L + \bar{\sigma}^\alpha$	$\bar{\sigma}^\alpha - \sigma^I$	0	0	0
quasi-single- phase material	α	mech. **)	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\alpha$	$\bar{\sigma}^\alpha - \sigma^L - \langle \sigma^{II} \rangle_\alpha$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - \sigma^I$	$(f^\alpha - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - f^\alpha \cdot \sigma^I$
one phase α measurable β small	β n.m.		n.d. ***)		n.d. ***)	n.d. ***)	n.d. ***)
two phases material	α	mech. **)	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\alpha$	$\bar{\sigma}^\alpha - \sigma^L - \langle \sigma^{II} \rangle_\alpha$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - \sigma^I$	$(f^\alpha - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - f^\alpha \cdot \sigma^I$
only one phase measurable	β n.m.		n.d.		$\sum_{n=1}^2 c^n \langle \sigma^{II} \rangle_n = 0$	$\sum_{n=1}^2 c^n (f^n - 1) = 0$	$\sum_{n=1}^2 c^n \bar{\sigma}_0^n = 0$
both phases measurable	α	$\sum_{n=1}^2 c^n \langle \sigma^I + \sigma^{II} \rangle_n$	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\alpha$	$\bar{\sigma}^\alpha - \sigma^L - \langle \sigma^{II} \rangle_\alpha$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - \sigma^I$	$(f^\alpha - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - f^\alpha \cdot \sigma^I$
	β		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\beta$	$\bar{\sigma}^\beta - \sigma^L - \langle \sigma^{II} \rangle_\beta$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - \sigma^I$	$(f^\beta - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - f^\beta \cdot \sigma^I$

Table 2. Determination of load stress as well as macro- and micro-RS on three-phases materials from linear lattice strain dependences. Stress data are understood as $\sigma_{11}-\sigma_{33}$. $\bar{\sigma}^n$: average value over the volume of the appropriate phase probed by the X-rays; σ^I , σ^{II} : macro-, micro-RS. Stresses of kind III within the phases according to the deformation are not considered. α , β , γ : crystalline phases; upper index n: general phase; n.m.: not measurable; n.d.: not determinable. *) semicrystalline polymer with α crystalline, β metal powder and δ amorphous phase, no load-independent micro-RS between α and δ phases; compound-XEC of $(\alpha+\delta)$ phase known; **) determined on a separate specimen; ***) not determinable and negligible.

material	phase	macro-RS σ^I	total stress determined $\bar{\sigma}^n$	load stress σ^L	micro-RS σ^{II} ; $\sigma^L=0$		
					total	of macro-RS σ^I dependent independent ($\bar{\sigma}_0^n$)	
only one phase α measurable γ small	α	mech. **)	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\alpha$	$\bar{\sigma}^\alpha - \sigma^L - \langle \sigma^{II} \rangle_\alpha$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - \sigma^I$	$(f^\alpha - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - f^\alpha \cdot \sigma^I$
	β n.m.		n.d.		$\sum_{n=1}^2 c^n \langle \sigma^{II} \rangle_n = 0$	$\sum_{n=1}^2 c^n (f^n - 1) = 0$	$\sum_{n=1}^2 c^n \bar{\sigma}_0^n = 0$
	γ n.m.		n.d. ***)		n.d. ***)	n.d. ***)	n.d. ***)
two phases measurable	α	mech. **)	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\alpha$	$\bar{\sigma}^\alpha - \sigma^L - \langle \sigma^{II} \rangle_\alpha$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - \sigma^I$	$(f^\alpha - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - f^\alpha \cdot \sigma^I$
	β		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\beta$		$\langle \sigma^I + \sigma^{II} \rangle_\beta - \sigma^I$	$(f^\beta - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - f^\beta \cdot \sigma^I$
	γ n.m.		n.d.		$\sum_{n=1}^3 c^n \langle \sigma^{II} \rangle_n = 0$	$\sum_{n=1}^3 c^n (f^n - 1) = 0$	$\sum_{n=1}^3 c^n \bar{\sigma}_0^n = 0$
two phases α' and β measurable	$\alpha' = \alpha + \delta$ *)	$\sum_{n=1}^2 c^n \langle \sigma^I + \sigma^{II} \rangle_n$	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_{\alpha'}$	$\bar{\sigma}^{\alpha'} - \sigma^L - \langle \sigma^{II} \rangle_{\alpha'}$	$\langle \sigma^I + \sigma^{II} \rangle_{\alpha'} - \sigma^I$	$(f^{\alpha'} - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_{\alpha'} - f^{\alpha'} \cdot \sigma^I$
	β		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\beta$		$\langle \sigma^I + \sigma^{II} \rangle_\beta - \sigma^I$	$(f^\beta - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - f^\beta \cdot \sigma^I$
all three phases measurable	α	$\sum_{n=1}^3 c^n \langle \sigma^I + \sigma^{II} \rangle_n$	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\alpha$	$\bar{\sigma}^\alpha - \sigma^L - \langle \sigma^{II} \rangle_\alpha$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - \sigma^I$	$(f^\alpha - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - f^\alpha \cdot \sigma^I$
	β		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\beta$		$\langle \sigma^I + \sigma^{II} \rangle_\beta - \sigma^I$	$(f^\beta - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - f^\beta \cdot \sigma^I$
	γ		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\gamma$		$\langle \sigma^I + \sigma^{II} \rangle_\gamma - \sigma^I$	$(f^\gamma - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\gamma - f^\gamma \cdot \sigma^I$

Table 3. Determination of load stress as well as macro- and micro-RS on four-phases materials from linear lattice strain dependences. Stress data are understood as $\sigma_{11}-\sigma_{33}$. $\bar{\sigma}^n$: average value over the volume of the appropriate phase probed by the X-rays; σ^I , σ^{II} : macro-, micro-RS. Stresses of kind III within the phases according to the deformation are not considered. α , β , γ , η : crystalline phases; upper index n: general phase; n.m.: not measurable; n.d.: not determinable. *) semicrystalline polymer with α and γ crystalline, β metal powder and δ amorphous phase, no load-independent micro-RS between α , γ and δ phases; compound-XEC of $(\alpha+\gamma+\delta)$ phase known; **) determined on a separate specimen; ***) not determinable and negligible.

material	phase	macro-RS σ^I	total stress determined $\bar{\sigma}^n$	load stress σ^L	micro-RS σ^{II} ; $\sigma^L=0$		
					total	of macro-RS σ^I	
						dependent	independent ($\bar{\sigma}_0^n$)
two phases α and β measurable η small	α	mech. **)	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\alpha$	$\bar{\sigma}^\alpha - \sigma^L - \langle \sigma^{II} \rangle_\alpha$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - \sigma^I$	$(f^\alpha - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - f^\alpha \cdot \sigma^I$
	β		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\beta$	$\bar{\sigma}^\beta - \sigma^L - \langle \sigma^{II} \rangle_\beta$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - \sigma^I$	$(f^\beta - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - f^\beta \cdot \sigma^I$
	γ n.m.		n.d.		$\sum_{n=1}^3 c^n \langle \sigma^{II} \rangle^n = 0$	$\sum_{n=1}^3 c^n (f^n - 1) = 0$	$\sum_{n=1}^3 c^n \bar{\sigma}_0^n = 0$
	η n.m.		n.d. ***)		n.d. ***)	n.d. ***)	n.d. ***)
three phases measurable	α	mech. **)	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\alpha$	$\bar{\sigma}^\alpha - \sigma^L - \langle \sigma^{II} \rangle_\alpha$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - \sigma^I$	$(f^\alpha - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - f^\alpha \cdot \sigma^I$
	β		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\beta$	$\bar{\sigma}^\beta - \sigma^L - \langle \sigma^{II} \rangle_\beta$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - \sigma^I$	$(f^\beta - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - f^\beta \cdot \sigma^I$
	γ		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\gamma$	$\bar{\sigma}^\gamma - \sigma^L - \langle \sigma^{II} \rangle_\gamma$	$\langle \sigma^I + \sigma^{II} \rangle_\gamma - \sigma^I$	$(f^\gamma - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\gamma - f^\gamma \cdot \sigma^I$
	η n.m.		n.d.		$\sum_{n=1}^4 c^n \langle \sigma^{II} \rangle^n = 0$	$\sum_{n=1}^4 c^n (f^n - 1) = 0$	$\sum_{n=1}^4 c^n \bar{\sigma}_0^n = 0$
two phases α' and β measurable	$\alpha' = \alpha + \gamma + \delta$ *)	$\sum_{n=1}^2 c^n \langle \sigma^I + \sigma^{II} \rangle^n$	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_{\alpha'}$	$\bar{\sigma}^{\alpha'} - \sigma^L - \langle \sigma^{II} \rangle_{\alpha'}$	$\langle \sigma^I + \sigma^{II} \rangle_{\alpha'} - \sigma^I$	$(f^{\alpha'} - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_{\alpha'} - f^{\alpha'} \cdot \sigma^I$
	β		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\beta$	$\bar{\sigma}^\beta - \sigma^L - \langle \sigma^{II} \rangle_\beta$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - \sigma^I$	$(f^\beta - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - f^\beta \cdot \sigma^I$
	γ n.m.						
all four phases measurable	α	$\sum_{n=1}^4 c^n \langle \sigma^I + \sigma^{II} \rangle^n$	$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\alpha$	$\bar{\sigma}^\alpha - \sigma^L - \langle \sigma^{II} \rangle_\alpha$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - \sigma^I$	$(f^\alpha - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\alpha - f^\alpha \cdot \sigma^I$
	β		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\beta$	$\bar{\sigma}^\beta - \sigma^L - \langle \sigma^{II} \rangle_\beta$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - \sigma^I$	$(f^\beta - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\beta - f^\beta \cdot \sigma^I$
	γ		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\gamma$	$\bar{\sigma}^\gamma - \sigma^L - \langle \sigma^{II} \rangle_\gamma$	$\langle \sigma^I + \sigma^{II} \rangle_\gamma - \sigma^I$	$(f^\gamma - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\gamma - f^\gamma \cdot \sigma^I$
	η		$\langle \sigma^L + \sigma^I + \sigma^{II} \rangle_\eta$	$\bar{\sigma}^\eta - \sigma^L - \langle \sigma^{II} \rangle_\eta$	$\langle \sigma^I + \sigma^{II} \rangle_\eta - \sigma^I$	$(f^\eta - 1) \cdot \sigma^I$	$\langle \sigma^I + \sigma^{II} \rangle_\eta - f^\eta \cdot \sigma^I$

2.122e The Eshelby's inclusion model

The inclusion theory of Eshelby /7/ is one of the most often applied theoretical methods to study two-phase and multiphase material problems. The principles are outlined in the paper of Withers /25/ and in /26,27/. The following blocks are taken from Withers paper /25/.

When a misfitting homogeneous ellipsoidal inclusion is concentrated to lie inside an ellipsoidal hole in a matrix of the same material the stress and strain within it is always uniform. Eshelby realised that because of the simple nature of the constrained inclusion shape and stress state, the solution to this one problem could be translated across to a wide variety of different situations. This is because whatever the stresses generated by the misfit, one can always generate the same stress state for another similarly constrained inclusion of a different material, given that one has complete freedom over the choice of the original shape of the second inclusion. Conversely, for a reinforcing inclusion of particular elastic constants, no matter what the matrix/inclusion misfit is, it is always possible to imagine an equivalent inclusion made of the matrix material which will generate the same inclusion stress when constrained to the same shape. Because the two inclusions have the same constrained shape and stress state they can be interchanged without disturbing the matrix, and thus solutions for the stress state of the elastically homogeneous problem can be used to solve the reinforcing problem. The trick, given that the elastically homogeneous problem is easily solved, is to calculate the elastically homogeneous inclusion/matrix shape misfit equivalent to the elastically inhomogeneous problem of interest.

$$\text{Misfit } \epsilon^T = (\alpha_M - \alpha) \Delta T$$

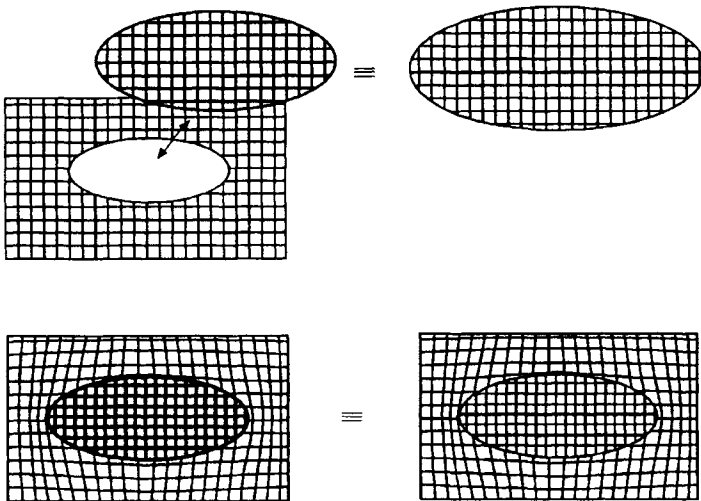


Figure 2: Eshelby's approach is most easily understood by considering a thermal residual stress state. Here the misfit between the matrix and the reinforcing particle is simply the difference in expansion coefficients multiplied by the temperature drop. Naturally, in this case the equivalent inclusion, having the same elastic constants as the matrix, is larger than the reinforcing inclusion. /25/

For a dilute composite the approach is mathematically rigorous. In order to satisfy the boundary conditions at the external surfaces of a non-dilute composite, Eshelby introduces the idea of an image stress (used in a similar way to the image techniques in electrostatics). In this form the model assumes that the inclusions are randomly distributed within the matrix. Because the stress in the inclusion is uniform, the mean inclusion stress is easily calculated (see below). Using the condition of microstress balance expressed in Equ. 38 this leads directly to the mean matrix stress, without the need to explicitly average over the locally fluctuation matrix stress field.

$$(1 - f) \langle \sigma_M \rangle + f \langle \sigma_I \rangle = 0$$

f fraction of inclusion I in a matrix M

(38)

The model has the following attributes:

- it has a rigorous analytical basis for low inclusion concentrates
- it is valid for all aspect ratios
- it can be computed on a programmable calculator or personal computer
- it applies to thermal and to load-induced stress fields
- it is rigorous only for ellipsoidal particles
- it is conceptually difficult to grasp
- additional assumptions are required to model plastic flow

For further study and use of the Eshelby's method some references are listed in Table 4 of applications and results of this method.

Table 4. Examples of applications of the Eshelby's theory

application	reference
macroscopic elastic constants	/28,29/
X-ray elastic constants	/12,30,31/
thermal stress	/25,26,32/
thermal RS	/26,33,34/
RS in phases after plastic strain	/35,36/
orientation dependent RS after plastic strain	/37/

The practical use and applications of this theory are described in the following. Two results for Al or Fe inclusions in different matrices (nontextured, quasiisotropic) are given in /39/ and one for Al in a PP-matrix is published in /19/.

Fig. 3 shows the transfer functions f_{11} and f_{21} of Al versus different matrices characterized by the logarithm of the ratio of Young's modulus of the matrix over that of the inclusion. The following data are used: $E^{Al} = 704 \text{ GPa}$, $E^{Fe} = 212 \text{ GPa}$; $\nu^{Al} = 0.35$, $\nu^{Fe} = 0.29$.

The Poisson ratio of the matrix was set equal to that of the inclusion. Wörtler /38/ arrived at practically the same result using another way of calculation and keeping ν of the matrix constant while varying ν of the inclusion. The relationship between the transfer factor and $\log [E(\text{matrix})/E(\text{inclusion})]$ seems to be nearly identical for different material combinations.

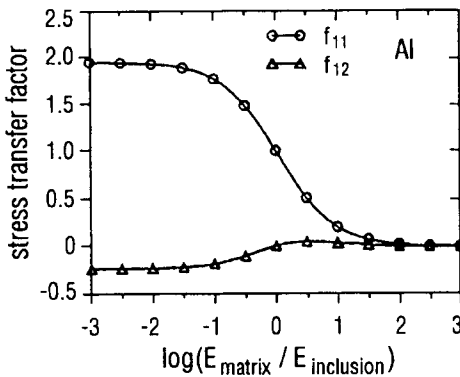


Figure 3. Stress transfer factors f_{11} and f_{12} of a spherical grain, which is surrounded by a homogeneous, elastic isotropic matrix, versus logarithm of the ratio Young's moduli (matrix / inclusion). Eshelby's model, Al-grains /39/.

From the dependences in Fig. 3 it follows that in the case of a very strong matrix, the inclusive material is nearly stress free in spite of the outside load. If the elastic behavior of the inclusion and the matrix are the same, both phases are under the same macrostress. If the inclusion is much harder than the matrix, the stress of the included material is approximately two times the macrostress in the longitudinal direction and -0.3 times the macrostress in the transverse direction. This was experimentally observed for Al-powder in PP /40/.

2.122f Stresses in layer-substrate composites

A schematic specimen with substrate and layer materials, as well as the stress state and the XEC will be treated in section 2.134e /6/.

2.122g Stresses in fiber-reinforced composite materials

In the special case of fibers in the form of thin wires, each of the same thickness and of high strength in the longitudinal direction within a ductile matrix, the following conditions must be fulfilled to apply the theory outlined in /6/:

- No texture in the fibers and the matrix material
- The shape of the fibers are thin, continuous, uniaxially oriented wires
- The load is applied parallel to the fiber axis
- The radial stress components are continuous on the border between fiber and matrix
- The distortions should be steady
- The strains of both phases should be homogeneous

With these assumptions and using linear elasticity theory, the stresses are given in cylindrical coordinates. The derivation and the formulae themselves are too voluminous to be reproduced here. They can be found in paper /6/.

2.123 Examples of stresses in different phases of multiphase materials

2.123a Two-phase materials

The load distribution between the phases in the elastic and plastic deformation ranges are of importance in the field of metallurgy and metal physics. In the following, some examples of phase stresses versus the applied stress are presented. The two-phase materials studied are WC-Co (three compositions) /3/ Fig. 4, Fe-Fe₃C (C130 steel) /3/ Fig. 5, and ferrite-austenite (each 50 vol.%) /41/ Fig. 6.

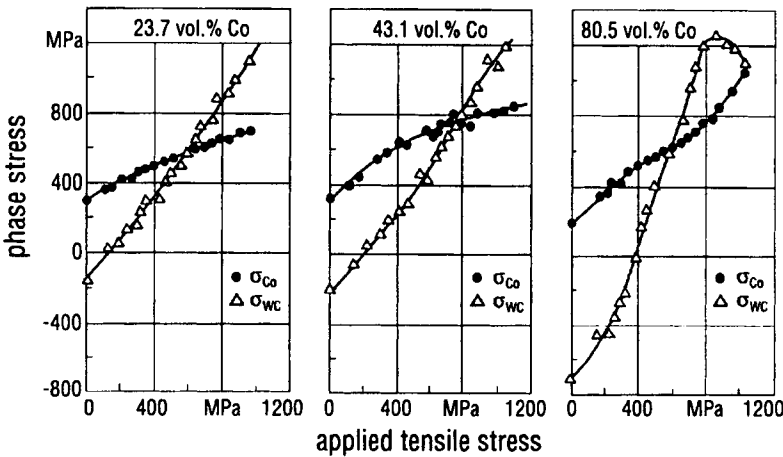


Figure 4. Phase stresses versus applied stresses in WC-Co compositions /3/.

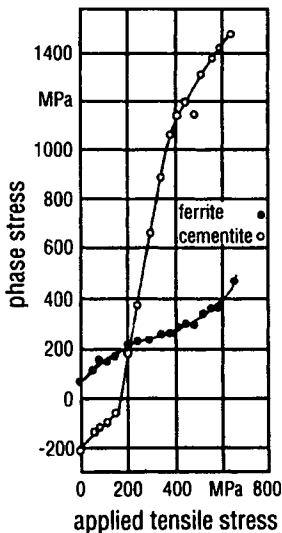


Figure 5. Phase stresses versus applied stresses in Fe-Fe₃C (C130 steel) /3/.

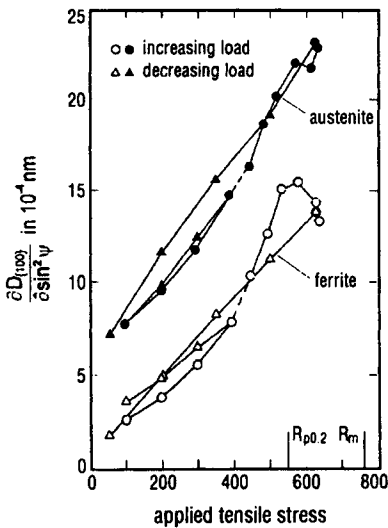


Figure 6. Phase Stresses versus applied stresses in ferrite-austenite /41/.

In Fig. 7, principally different possibilities for the variation of the ratio of the Young's moduli of both phases are displayed. Actual examples of phase stresses versus macrostress (LS + RS I) are plotted in Fig. 8.

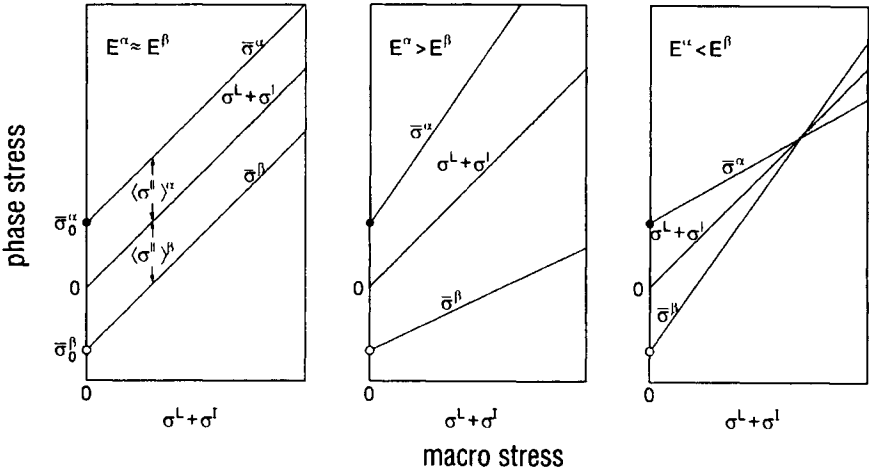


Figure 7. Phase stresses versus macrostress as a function of the variation of the ratio of the Young's moduli.

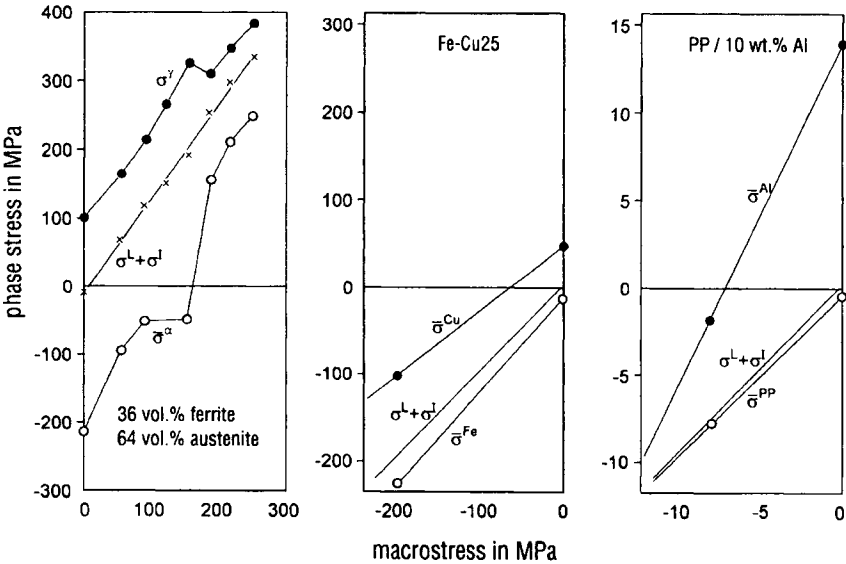


Figure 8. Phase stresses versus macrostress for different ratios of the Young's moduli.

The results of neutron diffraction studies on a duplex steel (ferrite and austenite phase each 50 vol.%) /42/ and on Al, reinforced with 20 vol.% SiC fibers /43/, which show the splitting of the loading capacities of the different $\{hkl\}$ -crystallites are of special interest, Fig. 9 and 10.

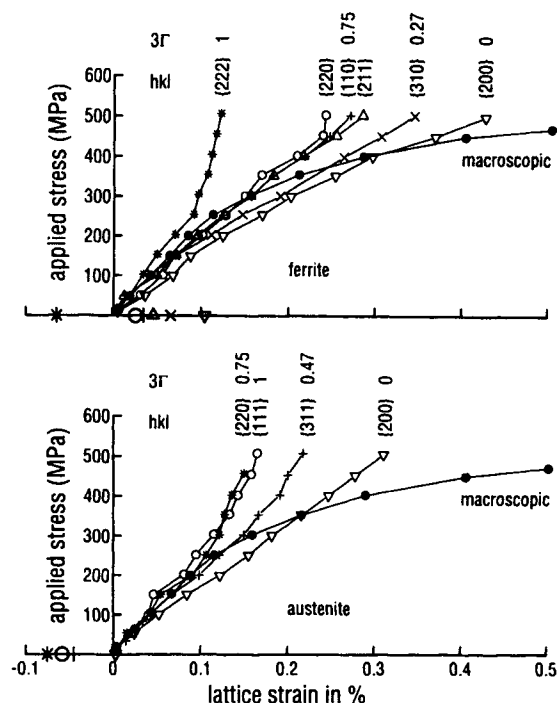


Figure 9. Applied stresses versus lattice strain for different reflections of a duplex alloy /42/, 3Γ orientation parameter.

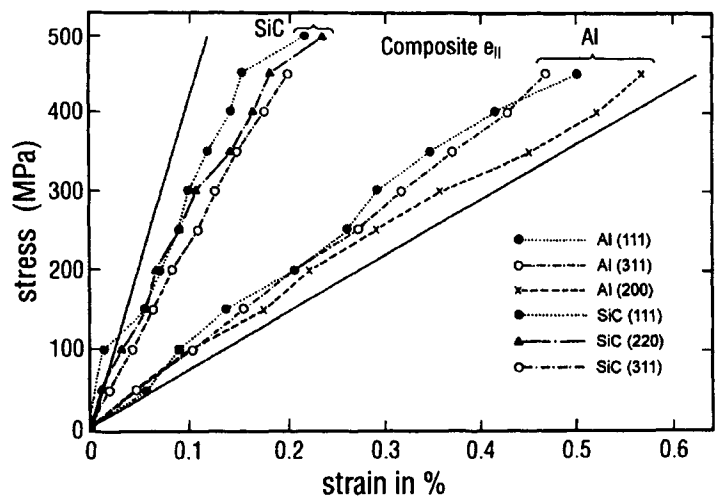


Figure 10. Al and SiC lattice strain response parallel to the applied stress, measured using the three Bragg's reflections indicated, in a composite sample of 20 vol.% SiC particulate in an Al (2014) matrix. The two solid lines are calculated using Young's moduli of $E(Al) = 72$ GPa and $E(SiC) = 420$ GPa /43/.

Combined measurements covering the outside surface by X-rays and the core by neutron rays, were performed on extruded and quenched Al bars reinforced by 25 vol.% SiC particles /44/, Fig. 11. Since the RS in all directions are equal they are hydrostatic RS, Fig. 12.

The effect of the size distribution of a second phase, graphite (medium to fine) in cast iron, on the load capacity of the matrix (ferrite) was studied on bending samples by the film method, Fig. 13 and 14. Fig. 14 shows the stress distribution in the matrix over the cross section measured on one side /45,46/.

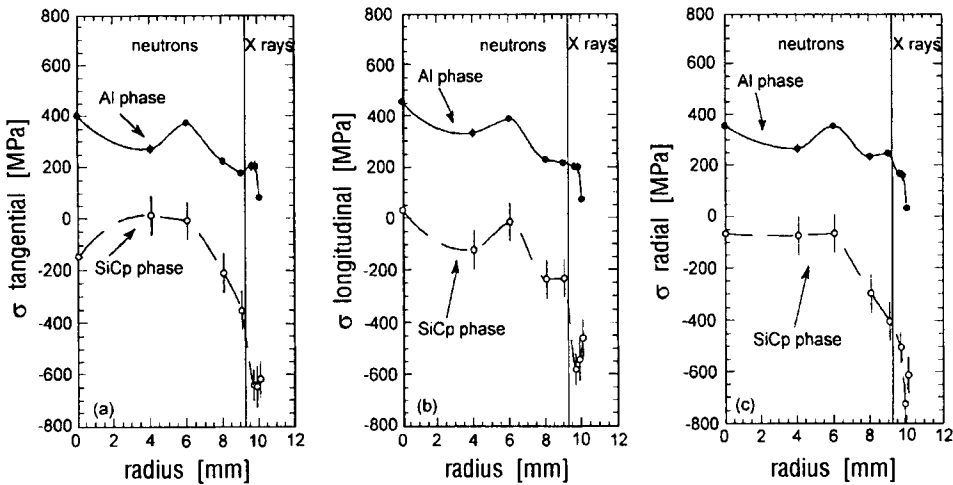


Figure 11. Evolution of residual stresses, in the Al 2024 matrix and the SiCp phase, in the tangential (a), longitudinal (b) and radial (c) directions /44/

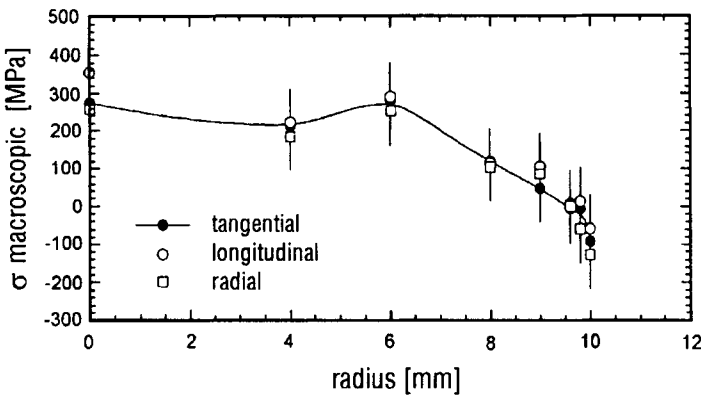


Figure 12. Evolution of the composite macrostresses derived from the measured microstresses according to the rule-of-mixture /44/.

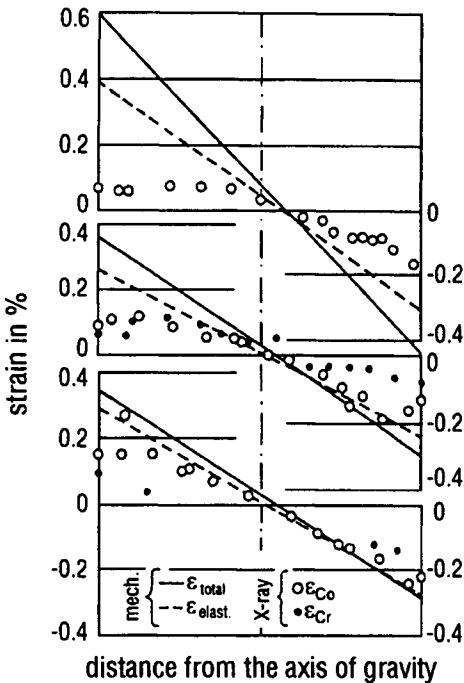


Figure 13. Influence of the size distribution of a second phase on the load capacity of the matrix, cast iron /45,46/.

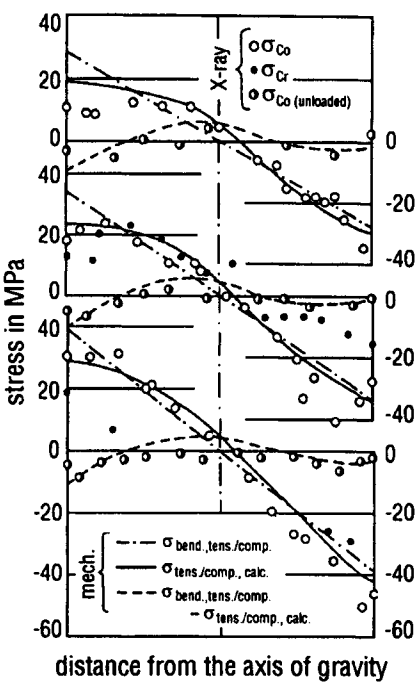


Figure 14. Stress distribution in the matrix over the cross section in a two-phase material /45,46/.

2.123b Polymeric unfilled and filled, reinforced materials

Polymeric materials are mostly multiphase materials, where only one phase is accessible to X-ray measurements. If fillers of fine crystalline metallic powder are used and mechanical deflection measurements are performed additionally, the macro-micro-RS state can be evaluated. Table 5 gives a summary of polymeric matrix-filler materials whose residual stresses have been measured for varied applied loads or versus the distance from the surface.

Fig. 15 shows the interesting behavior of the phases of α -PP and Al-particles within PP when a load is applied and after unloading. The above mentioned theoretical predictions with the stress factors $f_{11} = 2.0$ and $f_{12} = -0.3$ are excellently confirmed /40/. The triaxial stress state was studied on polymers with fiber reinforcement. Fig. 16 shows the big differences in the stress state between unfilled and filled PBT /5,4/. The filler geometry, glass sphere or glass fiber (non oriented) influences the stress state. Also, the effect of C-fibers was studied on the PEK + 20 wt.% C-fibers /47,48/, Fig. 17. Here, different residual stress states are found depending on the manufacturing methods (pressure and injection molding). Also the stress state development under applied stress is different.

Table 5. Studies on the influence of fillers on the stress state of polymeric materials.

polymer	phase in vol. %		filler	reference
	measurable	not measurable		
PP	α -PP, 45 monoclinic	amorphous, 45 β -PP, 10	—	/40/
			3.5 vol. % Al-powder	/40,19/
PBT	α -PBT, 27 triclinic	amorphous, 73 β -PBT	—	/5/
			22 vol. % glass spheres	/5/
			21 vol. % glass fibers	/4/
PEK	α -PEK, 30 orthorhombic	amorphous, 70	—	/47,48/
			15 vol. % C fibers	
			18 vol. % glass fibers	

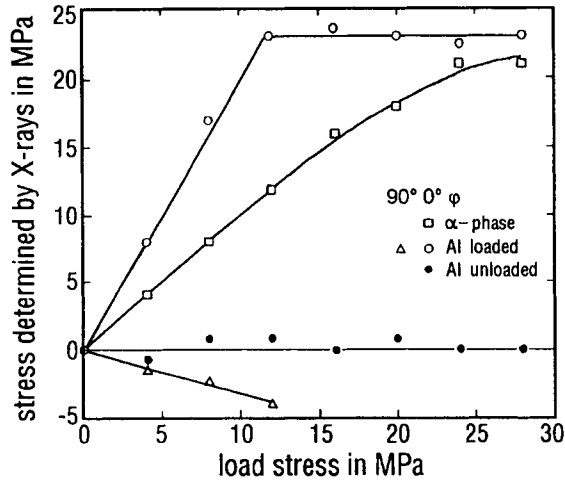


Figure 15. Stresses evaluated by X-rays in dependence on the mechanical load stress for PP filled with 10 m.% Al-powder. Al {511+333} peak with $\text{Cu-K}\alpha$ radiation, α -phase {130} peak with $\text{Cr-K}\alpha$ radiation /40/.

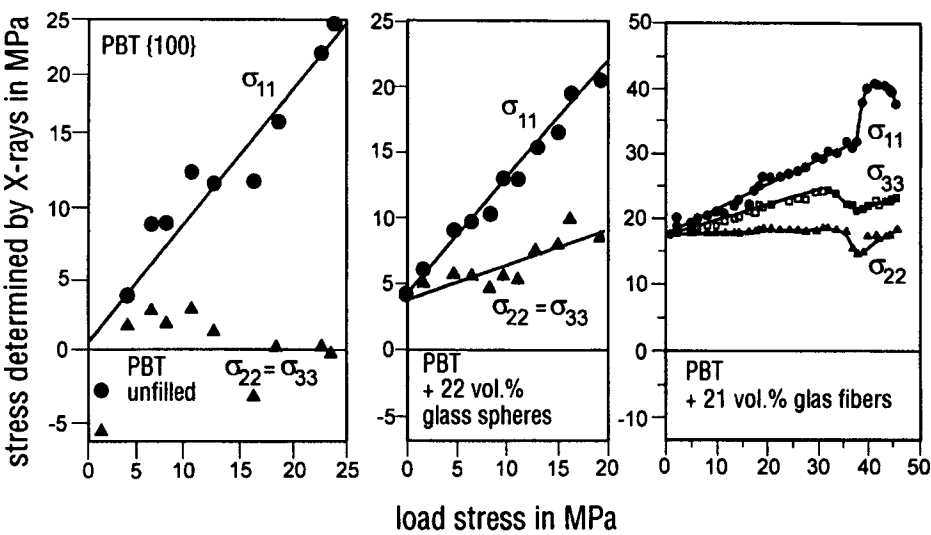


Figure 16. Stress states in unfilled and filled PBT /5,4/.

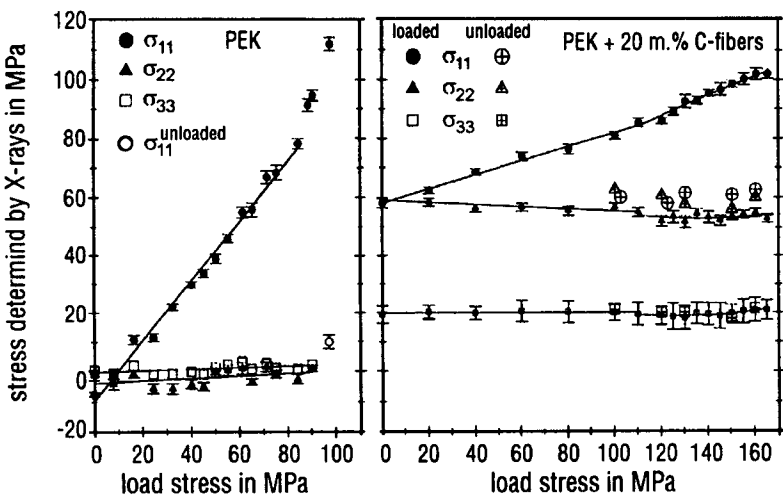


Figure 17. Stress states in unfilled and filled PEK /47,48/.

2.123c Thermally induced strains and stresses for two-phase materials

Thermally induced strains and stresses in two-phase materials were studied on Fe-Cu sintered materials /49/. Now this topic is of interest once again, when the influence of RS on the XEC and the texture of strongly elongated specimens is to be determined /50/. Fig. 18 shows the RS in both phases of softly ground samples versus the composition after the cited heat treatment. The RS in both phases are due to their different thermal expansion coefficients /49/.

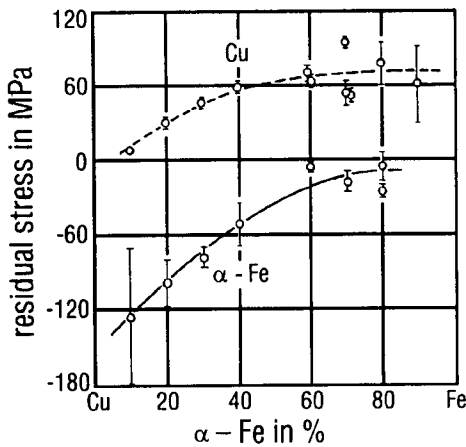


Figure 18. Residual stresses due to different thermal expansion coefficients after heat treatment 3h 550°C and 3h 650°C /49/.

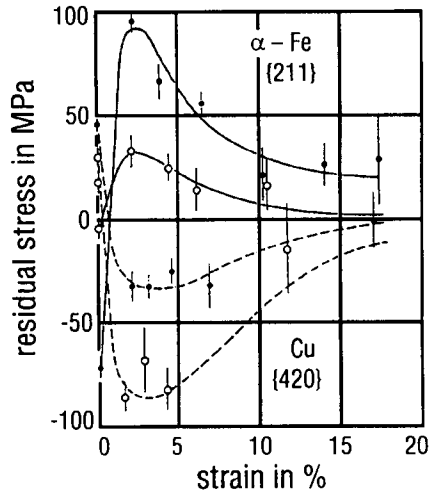


Figure 19. Residual stress in Cu- and α -Fe-phases versus strain ● 70/30, ○ 40/60 wt.% Cu/Fe /49/.

More detailed analyses of heating-cooling and deformation tests were made on reinforced ceramic materials. One example of a neutron ray investigation will be discussed /51/. The ceramic material has the composition 18 vol.% SiC whiskers embedded in an Al_2O_3 matrix /52/. The measured hydrostatic strain as a function of temperature for both phases is plotted in Fig. 20. The experimental results agree well with the calculated dependence. An excellent agreement was found between calculated and experimentally determined residual strain versus the angle to the c-axis of Si_3N_4 whiskers of hexagonal structure /52/, Fig. 21.

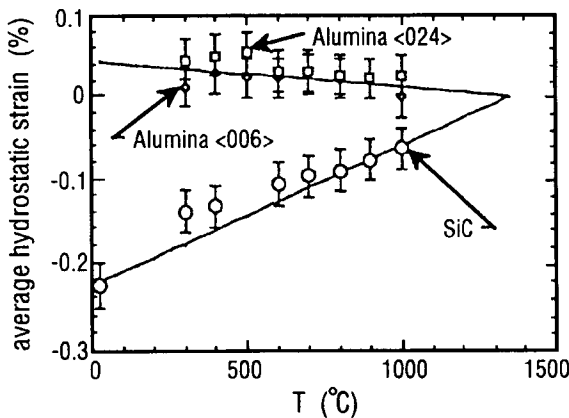


Figure 20. Measured (symbols) and predicted (solid line) residual average hydrostatic strain as a function of temperature for whiskers and a matrix of a ceramic composite, consisting of 18% volume fraction SiC whiskers embedded in an Al_2O_3 matrix /52/.

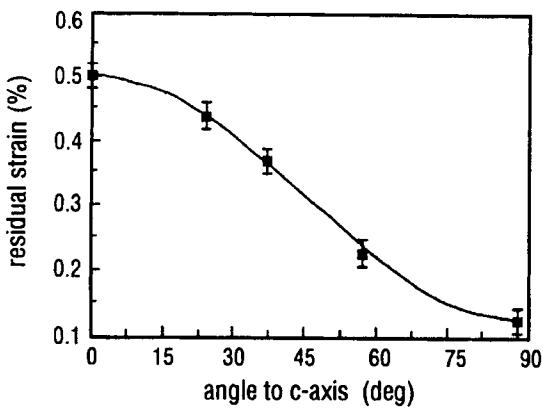


Figure 21. Calculated (solid line) and measured (symbols) strains as a function of angle relative to the c-axis of whiskers in a ceramic composite consisting of silicon-nitride whiskers embedded in an Al_2O_3 matrix /52/.

An interesting result was recently published /33,34/ about thermoelastic strains in white cast iron studied by X-rays, Fig. 22. The unexpected RS-temperature profiles of both phases, ferrite and cementite, were explained by considerations based on the Eshelby's theory.

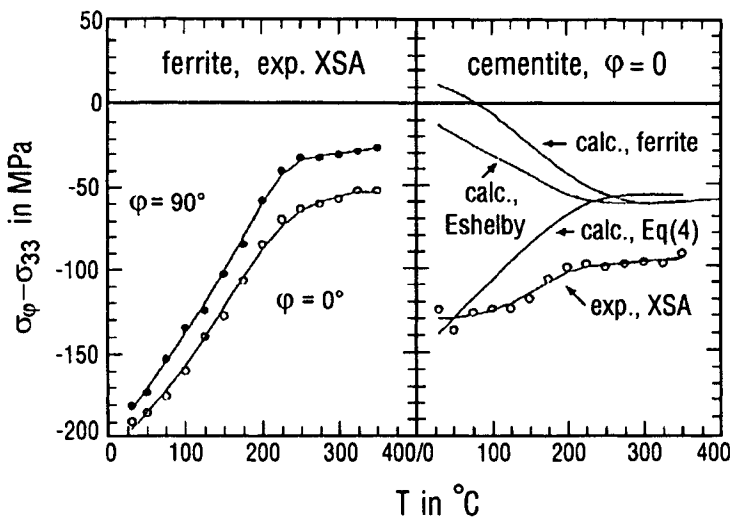


Figure 22. RS ($\sigma_\phi - \sigma_{33}$)-temperature profiles of ferrite and cementite, the two phases of white cast iron, experimentally and theoretically studied /34/.

2.124 Recommendations

- When studying multiphase materials, the stress state of all accessible phases should be analyzed.
- The influence of the shape and the volume concentration of a second phase in different metallic, ceramic, and polymeric materials should be studied systematically.
- Experimental studies should include the determination of the following parameters before, during and after the operations (production, heat treatment, elastic-plastic deformation, surface treatment): D_0^α , c^α microstructure, texture, stress state. The use of different radiations and measurements on different peaks increases the reliability of the results.
- Gradients of the measured parameters with the depth from the surface should be examined.
- The 3-dimensional strain-stress tensor should be evaluated for all phases of the material.
- Separation of macro- and microstresses and the evaluation of the hydrostatic pressure state should be done.
- The experimental procedure should include applying increasing and decreasing loads in several steps with measurements. This yields information about the stress capability and the transition of the yield limit of the phases as well as about the values of the XEC and RS of the phases.
- Heating and cooling experiments should also be performed to study the influence of the different thermal-expansion coefficients, and the stability of the phases, and the alterations of the RS-state.

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2.13 X-ray elastic constants (XEC)

2.131 Historical review

In the beginning of XSA the macromechanical elastic constants E Young's modulus and ν Poisson's ratio were used to evaluate LS and RS from the measured lattice strains. At the end of the thirties it was noticed that the determined LS by $\text{Co-K}\alpha$ radiation on the $\{310\}$ peak of iron base materials were approximately 15% higher, using the $\text{Cr-K}\alpha$ radiation on the $\{211\}$ 5% less than the mechanical applied stress /1-11/. This led to take into consideration the influence of the measuring method when applied to quasiisotropic polycrystalline materials consisting of anisotropic crystals.

Also in the thirties, studies were undertaken to calculate the physical properties of polycrystalline quasiisotropic materials starting from the respective data of the monocrystal. Different models of the aggregate of crystallites were used besides from those at this time already well known limit assumptions of Voigt /12/ (homogeneous strain of the crystallites and the same value as the polycrystalline material) and of Reuss /13/ (homogeneous stress).

By that time, the necessity to take the elastic anisotropy of a monocrystal into account for evaluating stresses from lattice strains measured on different peaks was obvious both from experimental results as well as from calculations of the elastic constants according to the models of Voigt and of Reuss. The calculations of the XEC (different for peaks with different values of the orientation parameter representing the measuring direction in the crystal system, and the average value for the group of crystals belonging to a respective wavelength and interference line) for cubic metals according to the assumption of Reuss were evaluated at the same time independently by Glocker /14/, Schiebold /15/, Möller and Martin /3/.

Neerfeld /10/ suggested using the average value of the calculated ones according to the models of Voigt and of Reuss as the best approximation to reality. To be mentioned is the proof of Hill /16/ that the calculated values of the mechanical elastic constants following any model must lie between the ones according to the border assumptions. The attempt to use the model of Bruggeman /17/ for calculating XEC and the XEC calculation of cubic metals using the monocrystal data and the shear modulus /18/ were unsuccessful. Also the paper of Stickforth /19/ will be mentioned in which the fundamentals of the lattice strain and the macro stresses are dealt with, the theoretical proof of the D -vs.- $\sin^2\psi$ linearity was stated, and the surface anisotropy was introduced.

The next and physically appropriate step was the assumption and evaluation of the model of a spherical anisotropic crystal in a homogeneous matrix. Eshelby /20/ calculated strains and stresses in and outside of ellipsoidal inclusions that had undergone a stress free strain. Kröner /21/ evaluated the polycrystalline elastic constants for cubic metals. Kneer /22/ extended the calculation to all other crystal systems and to textured materials. A detailed overview of the different models to calculate polycrystalline elastic data is given in /23/.

The adjustment of that model and the evaluated macroscopic elastic constants for the X-ray diffraction method was done by Bollenrath, Hauk and Müller /24/. Further steps of the development can be noticed in Table 1. One of the main advances was the calculation of the XEC for all crystal systems by Behnken and Hauk /25/ using the Eshelby-Kröner model supplemented by Kneer.

Table 1 contains models, crystal systems, and authors with the date of development of calculating XEC from the elastic data of the anisotropic monocrystal.

Besides the progress in metalphysics the experimental determination for XEC of all kinds of materials was pushed to check the calculation and to know the values of advanced materials where the data of monocrystals are not available. In the following the state-of-the-art of current calculations and tests, solved problems, and recommendations will be given.

Table 1. Existing calculations of the XEC for isotropic and textured materials /25/.

model		Voigt	Reuss	Eshelby-Kröner
authors	quasiisotropic material	cubic Voigt (1928)	Glocker (1938) Schiebold (1938) Möller, Martin (1939)	Bollenrath, Hauk, Müller (1967)
		hexagonal crystal symmetry : Evenschor, Hauk (1971, 1972) arbitrary crystal symmetry : Behnken, Hauk (1986)		
	textured material	I. O. – ODF Brakman (1987) Serruys et al. (1987)	Dölle, Hauk (1978) v. Baal (1983) Brakman (1983) Barral et al. (1983) Serruys et al. (1987)	– Behnken, Hauk (1988, 1991) Sprauel, Francois, Barral (1989)

2.132 Definitions

2.132a XEC of quasiisotropic materials

The X-ray elastic constants (XEC) s_1 and $\frac{1}{2}s_2$ of polycrystalline materials link the experimental, by means of diffraction methods, observed strains to the macroscopic and the homogeneous micro residual stresses or load stresses. They depend on the lattice plane under study and generally differ from the mechanical elastic constants according to the anisotropy of the crystals and due to the fact that only a specific part of all crystals contribute to the diffraction experiment, i.e. those crystallites the specific lattice plane of which are orientated perpendicular to the direction of measurement.

The average of the XEC, taken over all lattice planes, is connected to the macroscopic constants Young's modulus E, Poisson's ratio ν , shear modulus G and compression modulus K:

$$\langle s_1 \{hkl\} \rangle = s_1^m = - \frac{\nu}{E} = \frac{2G - E}{2EG}$$

(1)

$$\langle \frac{1}{2}s_2 \{hkl\} \rangle = \frac{1}{2}s_2^m = \frac{1 + \nu}{E} = \frac{1}{2G}$$

(2)

The elastic macroscopic constants are connected by the following equation:

$$E = 3K(1 - 2\nu) = 2G(1 + \nu)$$

(3)

For cubic materials the combination $3s_1 + \frac{1}{2}s_2$ is constant

$$3s_1\{hkl\} + \frac{1}{2}s_2\{hkl\} = 3s_1^m + \frac{1}{2}s_2^m = \frac{1}{3K} = \frac{1-2\nu}{E} \quad (4)$$

For given stresses the XEC determine the intercepts and the slopes of strain distributions versus $\sin^2\psi$. Equ. 6 describes the distribution in case of a planar stress state and $\varphi = 0^\circ$:

$$\varepsilon^{macro} = -\frac{\nu}{E}(\sigma_1 + \sigma_2) + \frac{1+\nu}{E}\sigma_1 \sin^2\psi \quad (5)$$

$$\varepsilon^{X-ray}(hkl) = s_1(hkl)(\sigma_1 + \sigma_2) + \frac{1}{2}s_2(hkl)\sigma_1 \sin^2\psi \quad (6)$$

Historically the XEC were first introduced empirically to describe the different behavior of the lattice planes. Stickforth proved the X-ray strain distribution (Equ. 6) to be formally identical to the macroscopic distribution (Equ. 5) if macroscopic isotropy and homogeneity are present only.

The XEC in general describe the strain response of the lattice planes of a phase to external loading or residual stresses. This response will depend on whether the respective crystals are part of a monophase material or of a multiphase material; therefore it will be distinguished between the phase-XEC (index α) and the compound-XEC (index C). The different phases are marked by greek letters.

The use of the phase- and the compound-XEC for stress evaluation depends on the material and on the kind of stresses to be determined. The different possibilities of stress evaluation in single-phase and multi-phase materials are discussed in detail in chapter 2.07.

2.132b X-ray stress factors of textured materials

The relationship between the lattice strains measureable by X-rays and the averaged stresses of the material phase cannot be described in the form of Equ. 6. The D-vs.- $\sin^2\psi$ dependences are usually nonlinear, they show more or less strong oscillations depending on the considered peak, except the peaks $\{h00\}$ and $\{hhh\}$ of cubic materials show, according to /31/, principally linear D-vs.- $\sin^2\psi$ dependences so far only the influence of the texture is present. Also materials consisting of elastically isotropic crystals show linear D-vs.- $\sin^2\psi$ dependences when they are caused by averaged stresses $\bar{\sigma}$. The strains determined by X-rays and the averaged stresses are related to each other by the X-ray stress factors F_{ij} /26/ which also may be called X-ray elastic factors XEF(φ, ψ, hkl), /26,27/, see also /28,29,30/.

$$\varepsilon_{\varphi, \psi} = F_{ij}(\varphi, \psi, hkl) \bar{\sigma}_{ij} \quad (7)$$

The $F_{ij}(\varphi, \psi, hkl)$ depend on the present texture, on the measuring direction, and on the peak, i.e. the plane group, but they are not the coordinates of a tensor. In case of a quasiisotropic material the F_{ij} correspond to combinations of the XEC s_1 and $\frac{1}{2}s_2$ which can be proved by comparison of Equ. 6 and 7. By the fictitious transition of a textured to a quasiisotropic material the factor $F_{11}(0^\circ, \psi, hkl)$, for example, becomes:

$$F_{11}(0^\circ, \psi, hkl) = s_1(hkl) + \frac{1}{2} s_2(hkl) \sin^2 \psi \quad (8)$$

For a given azimuth the F_{ij} can be arranged in a matrix. Equ. 9 shows the respective matrix for quasiisotropic materials and azimuth $\varphi=0^\circ/26^\circ$:

$$\begin{pmatrix} s_1(hkl) + \frac{1}{2} s_2(hkl) \sin^2 \psi & 0 & \left(\frac{1}{2}\right) \cdot \frac{1}{2} s_2(hkl) \sin 2\psi \\ 0 & s_1(hkl) & 0 \\ \left(\frac{1}{2}\right) \cdot \frac{1}{2} s_2(hkl) \sin 2\psi & 0 & s_1(hkl) + \frac{1}{2} s_2(hkl) \cos^2 \psi \end{pmatrix} \quad (9)$$

Because the F_{ij} vs. $\sin^2 \psi$ dependence settle D-vs.- $\sin^2 \psi$ distributions for a given $\bar{\sigma}_{ij}$ the F_{ij} can be determined experimentally by a tension- or a bending test or theoretically calculated using the ODF and an appropriate model of the elastic interaction of the grains.

Equ. 7 considers only the strains that are caused by the averaged stresses. Especially in deformed materials the strain distributions from the orientation dependent microstresses $\sigma_0(\Omega)$ are to be considered additionally.

2.133 Experimental determination of XEC and XEF

2.133a XEC of monophase materials

According to their definition the XEC relate Equ. 6 the lattice strains measured by a diffraction method to the averaged stresses $\bar{\sigma}$ which are present in the phase under concern of the material. In the same way, the stress factors XEF of textured materials are the proportionally factors between lattice strain and the averaged stress $\bar{\sigma}$ according to Equ. 7.

The experimental determination of both XEC and XEF in a mostly uniaxial tension- or bending-test should avoid influences of yielding and relaxation during the test. It will be assumed that the values of XEC are independent of plastic deformation. The influence of a mechanical surface treatment on the RS-state has to be considered. The most reliable values are to be expected if the evaluation method uses stress cycles and the strain measurement will be done at decreasing loads.

From the alterations/differences of the strains versus load the XEC or XEF are to be determined. In case of monophase materials it will be supposed that the alterations/differences of the load stresses are identical with the differences of the averaged phase stresses $\bar{\sigma}$. The equations to determine the XEC of quasi monophase materials from measured lattice-plane distances or lattice strains are the following:

$$\begin{aligned} \frac{1}{2} s_2 &= \frac{\partial}{\partial \sigma^L} \frac{\partial \varepsilon_\psi}{\partial \sin^2 \psi} = \frac{1}{D_0} \frac{\partial}{\partial \sigma^L} \frac{\partial D_\psi}{\partial \sin^2 \psi} = \frac{1}{D_0} \frac{\partial}{\partial \sin^2 \psi} \frac{\partial D_\psi}{\partial \sigma^L} = - \frac{\cot \Theta_0}{2} \frac{\partial}{\partial \sigma^L} \frac{\partial (2\Theta)}{\partial \sin^2 \psi} \\ s_1 &= \frac{\partial \varepsilon_{\psi=0}}{\partial \sigma^L} = \frac{1}{D_0} \frac{\partial D_{\psi=0}}{\partial \sigma^L} = - \frac{\cot \Theta_0}{2} \frac{\partial (2\Theta_{\psi=0})}{\partial \sigma^L} \end{aligned} \quad (10)$$

The experimental determination /32/ of the XEC is demonstrated in Fig. 1 as the graphical interpretation of Equ. 10 /33/. Fig. 2 gives an example of the XEC determination on the 54NiCrV6Mo steel /34/.

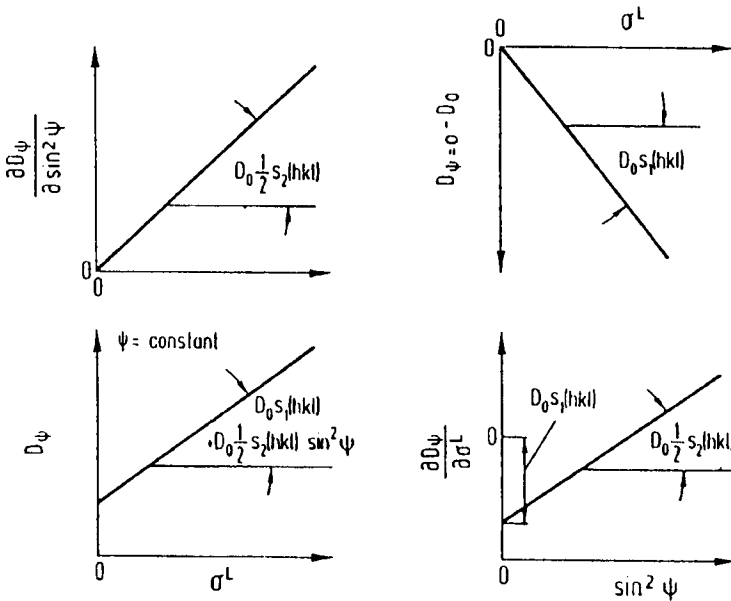


Figure 1. Two possibilities to evaluate the XEC s_1 and $\frac{1}{2}s_2$.

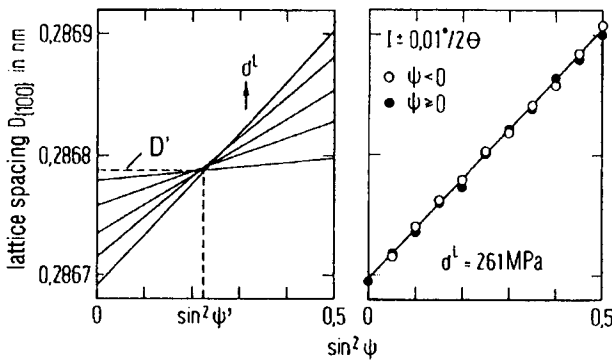


Figure 2. Example for a XEC determination on the 54NiCrV6Mo steel using Mo- K_{α} radiation and the $\{732 + 651\}$ lattice plane of ferrite. The error bar corresponds to the accuracy of the calibration of $\pm 0.01^\circ$ in 2θ . The D-vs.- $\sin^2\psi$ dependences were measured for decreasing load steps of 50 MPa (max. load 261 MPa) /34/.

A slight variation of the above mentioned usual experimental method was performed by /35/ working with polymeric materials in transmission technique. Taking the basic equation with

$$\sigma_2 = \sigma_3 = \sigma_{ik} = 0, \quad \psi = 90^\circ \quad (11)$$

and assuming no stress-dependent micro-RS are present, it follows

$$\varepsilon_{\varphi, \psi=90} = \frac{1}{2} s_2 \sigma^L \cos^2 \varphi + s_1 \sigma^L \quad (12)$$

$$s_1 = \frac{\partial \varepsilon_{\varphi=90, \psi=90}}{\partial \sigma^L} \quad (13)$$

$$\frac{1}{2} s_2 = \frac{\partial^2 \varepsilon_{\varphi, \psi=90}}{\partial \cos^2 \varphi \cdot \partial \sigma^L} \quad (14)$$

The graphical interpretation of Equ. 12 is shown in Fig. 3.

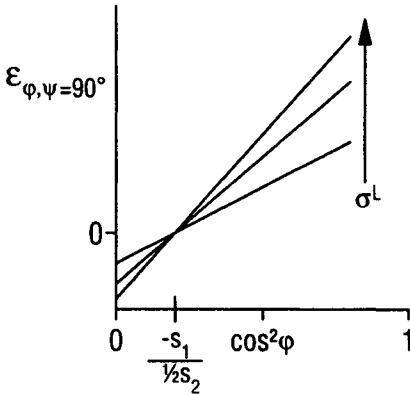


Figure 3. Graphical interpretation of the XEC determination of Equ. 12 .

2.133b XEC of multiphase materials

In case of multiphase materials alterations/differences of the load stresses σ^L are generally not identical with alterations/differences of the average phase stresses. Therefore, it must be distinguished between phase-(index α) and compound-(index C) XEC. The compound XEC describes the dependence of the strain of one phase within the multiphase material or multiphase polycrystal. The strain of the multiphase polycrystal is caused by load on the total material. The above described experimental methods describe in case of multiphase materials the compound-XEC or compound-XEF. The equations to determine the compound-XEC in the 1-direction ($\varphi = 0^\circ$) are:

$$\frac{1}{2} s_2^{C, \alpha} (\varphi = 0^\circ, hkl) = \frac{\partial}{\partial \sigma_{11}^L} \frac{\partial \frac{1}{2} (\varepsilon_{0,+ \psi} + \varepsilon_{0,- \psi})}{\partial \sin^2 \psi} = \frac{1}{2} s_2 \frac{\partial (\bar{\sigma}_{11} - \bar{\sigma}_{33})}{\partial \sigma_{11}^L} \quad (15)$$

for constant stress-independent micro-RS it follows

$$\frac{1}{2} s_2^{C, \alpha} (\varphi = 0^\circ, hkl) = \frac{1}{2} s_2 \left(1 + \frac{\partial (\langle \sigma_{11}'' \rangle - \langle \sigma_{33}'' \rangle)}{\partial \sigma_{11}^L} \right) \quad (16)$$

The same holds for $s_1(\psi = 0^\circ)$

$$s_1^{C,\alpha}(\varphi = 0^\circ, hkl) = \frac{\partial \varepsilon_{\varphi=0, \psi=0}}{\partial \sigma_{11}^L} = \frac{1}{2} s_2 \frac{\partial \bar{\sigma}_{33}}{\partial \sigma_{11}^L} + s_1 \frac{\partial (\bar{\sigma}_{11} + \bar{\sigma}_{22} + \bar{\sigma}_{33})}{\partial \sigma_{11}^L} \quad (17)$$

for constant stress-independent micro-RS follows

$$s_1^{C,\alpha}(\varphi = 0^\circ, hkl) = \frac{1}{2} s_2 \frac{\partial \langle \sigma_{33}^{\prime\prime} \rangle}{\partial \sigma_{11}^L} + s_1 \left(1 + \frac{\partial (\langle \sigma_{11}^{\prime\prime} \rangle + \langle \sigma_{22}^{\prime\prime} \rangle + \langle \sigma_{33}^{\prime\prime} \rangle)}{\partial \sigma_{11}^L} \right) \quad (18)$$

The elastically caused micro-RS in multiphase materials, their dependence on load stresses and on the content of the phases determine therefore the compound-XEC. Making the transition to the monophase material, $\frac{\partial}{\partial \sigma_{11}^L}$ will be replaced by $\frac{\partial}{\partial \bar{\sigma}_{11}^\alpha}$ and the compound-XEC are identical with the phase-XEC. The compound F_{11} will be respectively determined according to the formula

$$F_{11}^{C,\alpha}(\varphi = 0^\circ, \psi, hkl) = \frac{\partial \varepsilon_{0,\psi}^\alpha}{\partial \sigma_{11}^L} \quad (19)$$

To get the F_{ij} of the phase from the experimentally determined compound factor F_{ij}^C , the relation between the averaged stresses and the load-stresses, the transfer factor

$$f_{ijkl}^\alpha = \frac{\partial \bar{\sigma}_{ij}^\alpha}{\partial (\sigma^L + \sigma^I)_{kl}} \quad (\text{paragraph 2.037})$$

must be known. From the definition of F_{ij} , Equ. 8, follows

$$F_{11}^C(\varphi = 0^\circ, \psi, hkl) = \frac{\partial \varepsilon_{0,\psi}}{\partial \bar{\sigma}_{ij}} \frac{\partial \bar{\sigma}_{ij}}{\partial \sigma_{11}^L} = F_{ij}(\varphi = 0^\circ, \psi, hkl) \cdot f_{ij11} \quad (20)$$

On the right hand side of the equation the summation goes for i, j from 1 to 3. Analogous relationships exist for different azimuths φ and for the other compound stress factors F_{ij}^C . The following general formula holds

$$F_{mn}^C(\varphi, \psi, hkl) = F_{ij}(\varphi, \psi, hkl) \cdot f_{ijmn} \quad (21)$$

The above formula 20 will get simpler for a quasiisotropic material and azimuth $\varphi = 0^\circ$ if there are no alterations of the ψ -splitting of the D-vs- $\sin^2\psi$ distributions upon loading, i.e. no shear stresses are induced and one gets the compound-XEC from the intercept and slope of the

$F_{11}^C(\varphi = 0^\circ, \psi, hkl)$ vs. $\sin^2\psi$.

$$F_{11}^C(\varphi = 0^\circ, \psi, hkl) = s_1^C + \frac{1}{2} s_2^C \sin^2 \psi \quad (22)$$

with

$$s_1^C = s_1(f_{1111} + f_{2211}) + (s_1 + \frac{1}{2} s_2)f_{3311} \quad (23)$$

$$\frac{1}{2} s_2^C = \frac{1}{2} s_2(f_{1111} - f_{3311})$$

When the components of the transfer factor tensor f of a quasiisotropic material are known the determined compound-XEC can be related to the phase-XEC. If otherwise the phase-XEC are known the components of f can be calculated. Equ. 23 holds for $\varphi = 0^\circ$. From measurement at different azimuths the other components f_{ijmn} can be calculated.

$$F_{11}^C(\varphi = 0^\circ, \psi, hkl) = s_1(f_{1111} + f_{2211}) + \left(s_1 + \frac{1}{2}s_2\right)f_{3311} + \frac{1}{2}s_2(f_{1111} - f_{3311})\sin^2 \psi + \frac{1}{2}s_2(f_{1311} + f_{3111})\sin 2\psi \quad (24)$$

2.133c Strain-, stress-independent direction

The accomplishment of the condition that the D-vs.- $\sin^2\psi$ distributions of several loads have to meet in one point ($\sin^2\psi'$, D') or, in case of a textured material, in several points is a proof of the experimental accuracy. The equation for the determination of the mentioned point is the following:

$$\frac{\partial \frac{1}{2}(\varepsilon_{+ \psi} + \varepsilon_{- \psi})}{\partial \sigma_{11}^L} = \frac{\partial \frac{1}{2}(\varepsilon_{\varphi=0, + \psi} + \varepsilon_{\varphi=0, - \psi})}{\partial \sigma_{11}^L} = \frac{\partial \bar{\varepsilon}_{\varphi=0, \psi}}{\partial \sigma_{11}^L} = 0 \quad (25)$$

In general, the point ($\sin^2\psi'$, D') (strain-stress-independent direction) is different from the point ($\sin^2\psi^*$, D_0) (strain-stress-free direction $\varepsilon(\psi^*) = 0$), Fig.4.

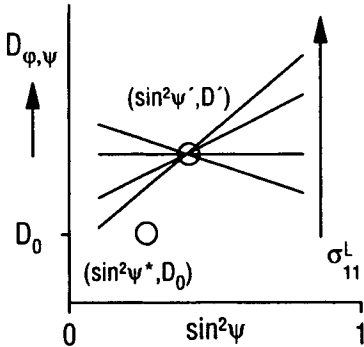


Figure 4. The stress-free direction ψ^* and the stress-independent direction ψ' in a D-vs.- $\sin^2\psi$ diagram for different applied-load stresses.

In general (multiphase material) Equ. 25 runs :

$$\frac{\partial \bar{\varepsilon}_{\varphi=0, \psi}}{\partial \sigma_{11}^L} = \frac{\partial \bar{\varepsilon}_{\varphi=0, \psi}}{\partial \bar{\sigma}_{11}} \frac{\partial \bar{\sigma}_{11}}{\partial \sigma_{11}^L} + \frac{\partial \bar{\varepsilon}_{\varphi=0, \psi}}{\partial \bar{\sigma}_{22}} \frac{\partial \bar{\sigma}_{22}}{\partial \sigma_{11}^L} + \frac{\partial \bar{\varepsilon}_{\varphi=0, \psi}}{\partial \bar{\sigma}_{33}} \frac{\partial \bar{\sigma}_{33}}{\partial \sigma_{11}^L} \stackrel{!}{=} 0 \quad (26)$$

Or with using the transfer factors f_{ij} (Voigt's notation):

$$\begin{aligned} \frac{\partial \bar{\varepsilon}_{\varphi=0, \psi}}{\partial \sigma_{11}^L} &= \left(s_1 + \frac{1}{2}s_2 \sin^2 \psi\right)f_{11} + s_1 f_{21} + \left(s_1 + \frac{1}{2}s_2 \cos^2 \psi\right)f_{31} \\ &= \left(s_1 + \frac{1}{2}s_2\right)f_{31} + s_1(f_{11} + f_{21}) + \frac{1}{2}s_2 \sin^2 \psi (f_{11} - f_{31}) \stackrel{!}{=} 0 \end{aligned} \quad (27)$$

$$\sin^2 \psi' = \frac{-s_1(f_{11} + f_{21}) - (s_1 + \frac{1}{2}s_2)f_{31}}{\frac{1}{2}s_2(f_{11} - f_{31})} \quad (28)$$

There will be one crossing point if $\langle \sigma_{ii}'' \rangle$ are linearly dependent on the load/applied stress.

For quasiisotropic, single-phase materials it follows from Equ. 25:

$$\frac{\partial \bar{\epsilon}_{\varphi=0, \psi}}{\partial \sigma_{11}^L} = s_1 + \frac{1}{2}s_2 \sin^2 \psi' = 0 \quad (29)$$

$$\sin^2 \psi' = \frac{-s_1}{\frac{1}{2}s_2} \quad D' = D_0 \left[1 + s_1 \bar{\sigma}_{22} + \left(2s_1 + \frac{1}{2}s_2 \right) \bar{\sigma}_{33} \right] \quad (30)$$

The formulae hold also for the assumption of a linear dependence of the micro-RS on the load stress. Some examples for different stress states are shown in Fig. 5, chapter 2.11 /36/.

In case of multiphase materials Equ. 28 has to be used

$$\text{with } f_{ij} = \frac{\partial \bar{\sigma}_i}{\partial \sigma_j^L} \quad (\text{Voigt's notation}) \quad (31)$$

If $f_{21} = f_{31} = 0$ (no alteration of microstresses in the transverse direction) the same formulae result as for homogeneous materials.

The formulae of the crystallite-group method for the $\{211\}\langle 01\bar{1} \rangle$ crystal group are as follows:

$$\sin^2 \psi' = - \frac{s_{11} + \frac{1}{6}s_0}{\frac{1}{2}(s_{44} + \frac{2}{3}s_0)} \quad (32)$$

$$D' = D_0 \left[1 + \left(s_{12} + \frac{1}{3}s_0 \right) \bar{\sigma}_{22} + \left(2s_{12} + \frac{1}{2}s_{44} + \frac{2}{3}s_0 \right) \bar{\sigma}_{33} \right] \quad (33)$$

The D-vs.- $\sin^2 \psi$ straight lines should cross in one point. However, no example of measurements showing the point ($\sin^2 \psi', D'$) could be found for crystallite-group evaluation.

For textured materials, however, of which intensity distributions are described by pole-figures and by ODF, no generally valid formulae of the stress-independent direction can be given. In case that the phase stresses in the transverse directions are not altered by the applied load, i. e. $f_{12} = f_{13} = 0$, the stress-independent directions for $\varphi = 0^\circ$ are given by the zero-points of $F_{11}(hkl, \sin^2 \psi)$ -dependences.

The nonlinear dependences for different applied stresses σ_{11}^L may cross in two or more points related to the type and the degree of the texture. An example is given in Fig. 6, chapter 2.11 /37/. The appropriate formulae are:

$$\epsilon_{\varphi, \psi} = F_{11}(\varphi, \psi, hkl) \bar{\sigma}_{11} + F_{22}(\varphi, \psi, hkl) \bar{\sigma}_{22} \quad (34)$$

$$\frac{\partial \bar{\epsilon}_{\varphi, \psi}}{\partial \sigma_{11}^L} = F_{11} \frac{\partial \bar{\sigma}_{11}}{\partial \sigma_{11}^L} + F_{22} \frac{\partial \bar{\sigma}_{22}}{\partial \sigma_{11}^L} \quad ; \quad \sin^2 \psi': F_{11}(\psi') = 0 \quad (35)$$

2.133d Determination of relative XEC and anisotropy

The relative XEC i.e. the anisotropy can also be evaluated without applying load stresses if there are residual stresses in the sample /38/. The RS should not be too small to get results with reasonable accuracy.

Orientation-dependent micro-RS should not be present or they must be taken into account. The macroscopic phase E^m must be known from experiment or the calculated value might be used if the compliances of the monocrystal of the phase in question are available. The strains of different $\{hkl\}$ -peaks must be measured. If using different radiations the assumption must be made that the RS will have no gradient with depth from the surface in the respective range.

In the case of rotationally symmetric stresses the method can be described as follows /39/. Several D-vs.- $\sin^2\psi$ measurements should be made on different peaks. The existence of a rotational symmetric stress state allows, as Fig. 5 shows, to evaluate $\nu\{hkl\}$ with the known D_0 (strain-stress-free lattice constant) according to the formula

$$\sin^2 \psi^* = - \frac{2s_1}{\frac{1}{2}s_2} = \frac{2\nu\{hkl\}}{1 + \nu\{hkl\}}. \tag{36}$$

The next step is to get ν^m at $3\Gamma = 0.6$ for cubic materials, Γ being the orientation parameter, Equ. 45. The relative XEC can be determined because the

$$XEC^m = \frac{1 + \nu^m}{E^m} \tag{37}$$

and the stress are known. Another way is to calculate $\frac{1}{2}s_2\{h00\}$ according to the Eshelby-Kröner model /24,25,40,41/ with the known compliances of the material and to get $\frac{1}{2}s_2\{hkl\}$ by the following formula, Fig. 6:

$$\frac{1}{2}s_2\{hkl\} = \frac{1}{2}s_2\{h00\}_{cal} \cdot \left(\frac{\partial D}{\partial \sin^2 \psi}\right)_{\{hkl\}} \cdot \left(\frac{\partial D}{\partial \sin^2 \psi}\right)^{-1}_{\{h00\}} \tag{38}$$

The $s_1\{hkl\}$ vs. 3Γ can be evaluated in a similar way using the above mentioned formulae.

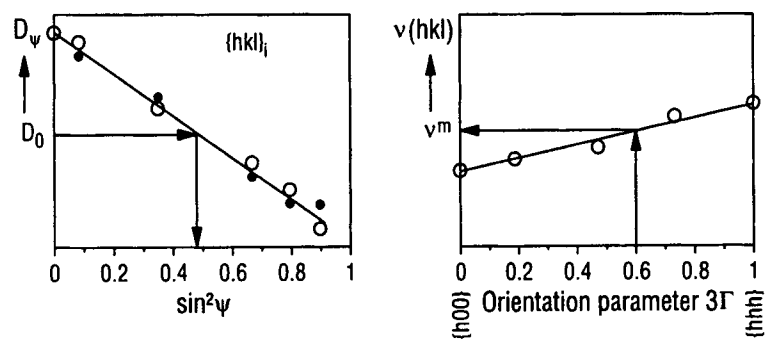


Figure 5. Determination of the stress-free direction ψ^* to evaluate the Poisson's ratio $\nu(hkl)$ and ν^m according to Equ. 36 in case of rotationalsymmetric stress states.

A real experimental result for PVD-TiN-layers is shown in Fig. 7 /39/.

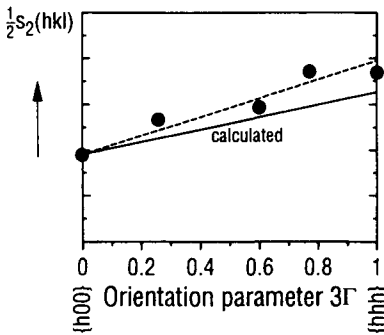


Figure 6. Comparison between experimentally and theoretically determined XEC /39/.

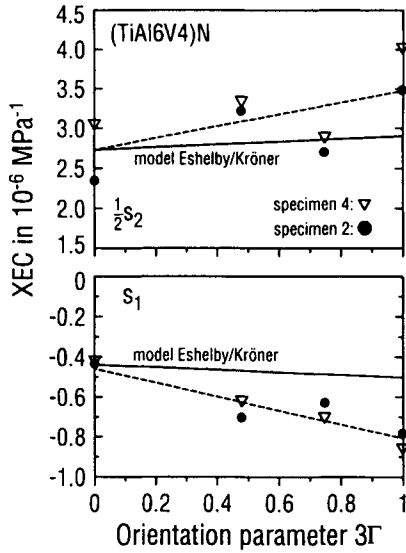


Figure 7. X-ray elastic constants, evaluated from lattice spacing measurements on two stressed samples; values refer to $3\Gamma = 0$ (calculated) /39/.

2.134 Calculation of XEC from the elastic data of monocrystal

2.134a Monophase materials

As was already discussed on the basis of Table 1 it follows that the development of the XEC calculation for materials of different crystallographic systems took quite a long time. Three main steps may be distinctly mentioned: The introduction of the Eshelby-Kröner model with the calculation of the polycrystalline elastic constants into X-ray stress analysis /24/, the extension of the calculation of XEC to all crystal systems /25/ and the application to textured materials, see 2.132b. The formulae to calculate the XEC for arbitrary crystal symmetry from the models of Voigt, Reuss and Eshelby-Kröner are developed in /25/ based on the work of /19/. The respective calculations for textured materials are given in /42/.

The calculation programs for the constants of quasiisotropic and textured materials are described and examples are given in /43/. The XEC are generally written /19, 25/:

$$s_1 = \frac{1}{2} \gamma_i \gamma_j (\delta_{mn} - \gamma_m \gamma_n) A_{ijmn}^0 \quad (39)$$

$$\frac{1}{2} s_2 = \frac{1}{2} \gamma_i \gamma_j (3\gamma_m \gamma_n - \delta_{mn}) A_{ijmn}^0 \quad (40)$$

γ_i represent the coordinates of the plane normales within the crystal system, δ is the Kronecker symbol. The relation between γ_i and the Miller's indices is given for all crystal symmetries in /44/.

According to the model of the choice the tensor components A_{ijmn}^0 are given by

$$A_{ijmn}^0 = \begin{cases} \left(\langle c(\Omega) \rangle^{-1} \right)_{ijmn} & \text{Voigt} \\ s_{ijmn} & \text{Reuss} \\ (S + t)_{ijmn} & \text{Kröner} \end{cases} \quad (41)$$

$$\text{with } t(\Omega) = \left[(c(\Omega) - C + Cw)^{-1} (c(\Omega) - C) \right] S,$$

c and s are the tensor coordinates of the stiffnesses (elastic moduli) and of the compliances (elastic coefficients) of the monocrystals. S is the macroscopic tensor of the compliances. For spherical grains the tensor w [20,21] is denoted in Equ. 62.

According to Kröner the tensor t describes the different strain behaviour of the single crystallites in relation to the total material (model Eshelby-Kröner [21]). It is dependent on the data of the monocrystal and on the macroscopic tensor S . According to Kröner it holds: $\langle S + t \rangle = \langle s \rangle \Rightarrow \langle t \rangle = 0$. The symbol $\langle \rangle$ means the averaging over all crystal orientations. S will be evaluated by an iteration program with the condition $\langle t \rangle = 0$ [22,25]. After S has been determined, t can be evaluated. For the assumption of homogeneous strain of the different crystallites, as Voigt suggested, the XEC are generally independent of the plane. They can be evaluated for all crystal symmetries from the monocrystal stiffnesses c_{ij} as follows:

$$s_1 = -\frac{3}{2} \frac{x + 4y - 2z}{(x - y + 3z)(x + 2y)} \quad \frac{1}{2} s_2 = \frac{15}{2x - 2y + 6z} \quad (42)$$

$$x = c_{11} + c_{22} + c_{33} \quad y = c_{12} + c_{23} + c_{13} \quad z = c_{44} + c_{55} + c_{66}$$

The graphical representation of the XEC is given versus the orientation parameters. The coordinates γ_i of the measuring direction within the crystal system can generally be given by the pole angle η and the azimuth ρ :

$$\gamma_1 = (1 - H^2)^{\frac{1}{2}} \cos \rho \quad \gamma_2 = (1 - H^2)^{\frac{1}{2}} \sin \rho \quad \gamma_3 = H \quad (\text{ETA}) \quad (43)$$

$$H = \cos \eta \quad -1 \leq H \leq 1 \quad \text{and} \quad 0 \leq \rho \leq 2\pi$$

The number of independent elastic coefficients of the monocrystals depends on the crystal symmetry as was outlined in chapter 2.03. For cubic symmetry only three components A_{ijmn}^0 are independent: A_{1111}^0 , A_{1122}^0 , A_{2323}^0 which are in Voigt's notation A_{11}^0 , A_{12}^0 , A_{44}^0 . The other components are dependent or zero. From Equ. 39, 40 and 41 it follows now:

$$\begin{aligned} \text{Reuss:} \quad s_1 &= s_{12} + s_0 \Gamma & \frac{1}{2} s_2 &= s_{11} - s_{12} - 3\Gamma s_0 \\ \text{Kröner:} \quad s_1 &= S_{12} + t_{12} + \Gamma t_0 & \frac{1}{2} s_2 &= S_{11} - S_{12} + t_{11} - t_{12} - 3\Gamma t_0 \end{aligned} \quad (44)$$

with the abbreviations

$$\Gamma = \gamma_1^2 \gamma_2^2 + \gamma_2^2 \gamma_3^2 + \gamma_3^2 \gamma_1^2 = \frac{h^2 k^2 + k^2 l^2 + l^2 h^2}{(h^2 + k^2 + l^2)^2} \quad (45)$$

$$s_0 = s_{11} - s_{12} - \frac{1}{2}s_{44} \quad t_0 = t_{11} - t_{12} - 2t_{44}$$

One gets a linear dependence of both constants and for all models versus the orientation parameter 3Γ . Therefore, the XEC of cubic materials will be plotted versus 3Γ .

The three linear dependences are crossing approximately in one point, but the crossing points of straight lines according to the models Reuss-Voigt and Kröner-Voigt are not exactly on the same point, which is due to the influence of the anisotropy. The respective consideration for hexagonal symmetry reveals that the plot versus the parameter

$$H^2 = \cos^2 \eta = \frac{l^2}{\frac{4}{3}(c/a)^2(h^2 + k^2 + hk) + l^2} \quad (46)$$

is advantageous, it gives parabola sections for both XEC.

The single-crystal data are listed in /45/. These data were used to calculate the XEC s_1 and $\frac{1}{2}s_2$ in dependence of the orientation parameter. For materials with lower crystal symmetry one needs two parameters. All orientation parameters and their relation to the Miller's indices of the planes are collected in Table 2 /43/.

Calculated XEC of cubic metals according to the three models of Voigt, of Reuss and of Eshelby-Kröner were first evaluated by /24/. The principal same procedure for hexagonal metals was done by /46,47/. In /25/ corrected data of the calculation /47/ are given. Behnken and Hauk /25/ have published the method to calculate the XEC of materials crystallizing in an arbitrary crystallographic system.

In Fig. 8 the XEC are given of some cubic and hexagonal metals. The mechanic value is marked with mech. or m. It is calculated as the average of all $\{hkl\}$ families of planes. For cubic metals the mechanical value can be found at $3\Gamma = 0.6$ /3,24/. However, there is no such specific value of the orientation parameter for all other crystallographic systems, where the mechanical value could be taken. The characteristics of the models of Voigt and Reuss are limiting assumptions /16/ and the values according to the Eshelby-Kröner model are within these limits, but not exactly fulfilled in the vicinity of the crossing point /18/. The precise values of XEC for the peaks $\{h00\}$ and $\{hhh\}$ as well as the mechanical ones are listed in Table 3.

The slope of $\frac{1}{2}s_2$ is generally negative, but there are some materials with a positive slope, for example Cr and V due to the elastic anisotropy of the monocrystal:

$$s_{11} - s_{12} - \frac{1}{2}s_{44} < 0 \quad (47)$$

The XEC of ceramic materials are of smaller absolute value and lie in the range $\frac{1}{2}s_2 \approx 3 \cdot 10^{-6} \text{ MPa}^{-1}$. The details can be found in the captions of the Fig. 9 to 13.

Table 2. Orientation parameter (o.p.) for different crystal symmetries /43,44/.
[x] means integer of x.

crystal symmetry	o.p.	$\gamma_1, \gamma_2, \gamma_3$	η, ρ	h, k, l
cubic	3Γ	$3 \cdot (\gamma_1^2 \gamma_2^2 + \gamma_2^2 \gamma_3^2 + \gamma_3^2 \gamma_1^2)$	/	$3 \cdot \frac{h^2 k^2 + k^2 l^2 + l^2 h^2}{(h^2 + k^2 + l^2)^2}$
hexagonal	H ²	γ_3^2	$\cos^2 \eta$	$\frac{l^2}{\frac{4}{3}(c/a)^2(h^2 + k^2 + hk) + l^2}$
trigonal hexagonal cell	H	γ_3^2	$\cos \eta$	$\frac{l}{\left(\frac{4}{3}(c/a)^2(h^2 + k^2 + hk) + l^2\right)^{\frac{1}{2}}}$
	P	/	$\frac{3\rho}{2\pi} - \left[\frac{3\rho}{2\pi}\right]$	$\frac{3}{2\pi} \arccot \cot \left(\sqrt{3} \frac{h}{2k+h} \right) - \left[\frac{3}{2\pi} \arccot \cot \left(\sqrt{3} \frac{h}{2k+h} \right) \right]$
tetragonal	H ²	γ_3^2	$\cos^2 \eta$	$\frac{l^2}{(c/a)^2(h^2 + k^2) + l^2}$
	K	$\frac{\gamma_2^2}{\gamma_1^2 + \gamma_2^2}$	$\sin^2 \rho$	$\frac{k^2}{h^2 + k^2}$
ortho- rhombic	H ²	γ_3^2	$\cos^2 \eta$	$\frac{(l/c)^2}{(h/a)^2 + (k/b)^2 + (l/c)^2}$
	K	$\frac{\gamma_2^2}{\gamma_1^2 + \gamma_2^2}$	$\sin^2 \rho$	$\frac{(k/b)^2}{(h/a)^2 + (k/b)^2}$
monoclinic triclinic	H	γ_3	$\cos \eta$	chapter 1.1 books on crystallography
	ρ	/	ρ	

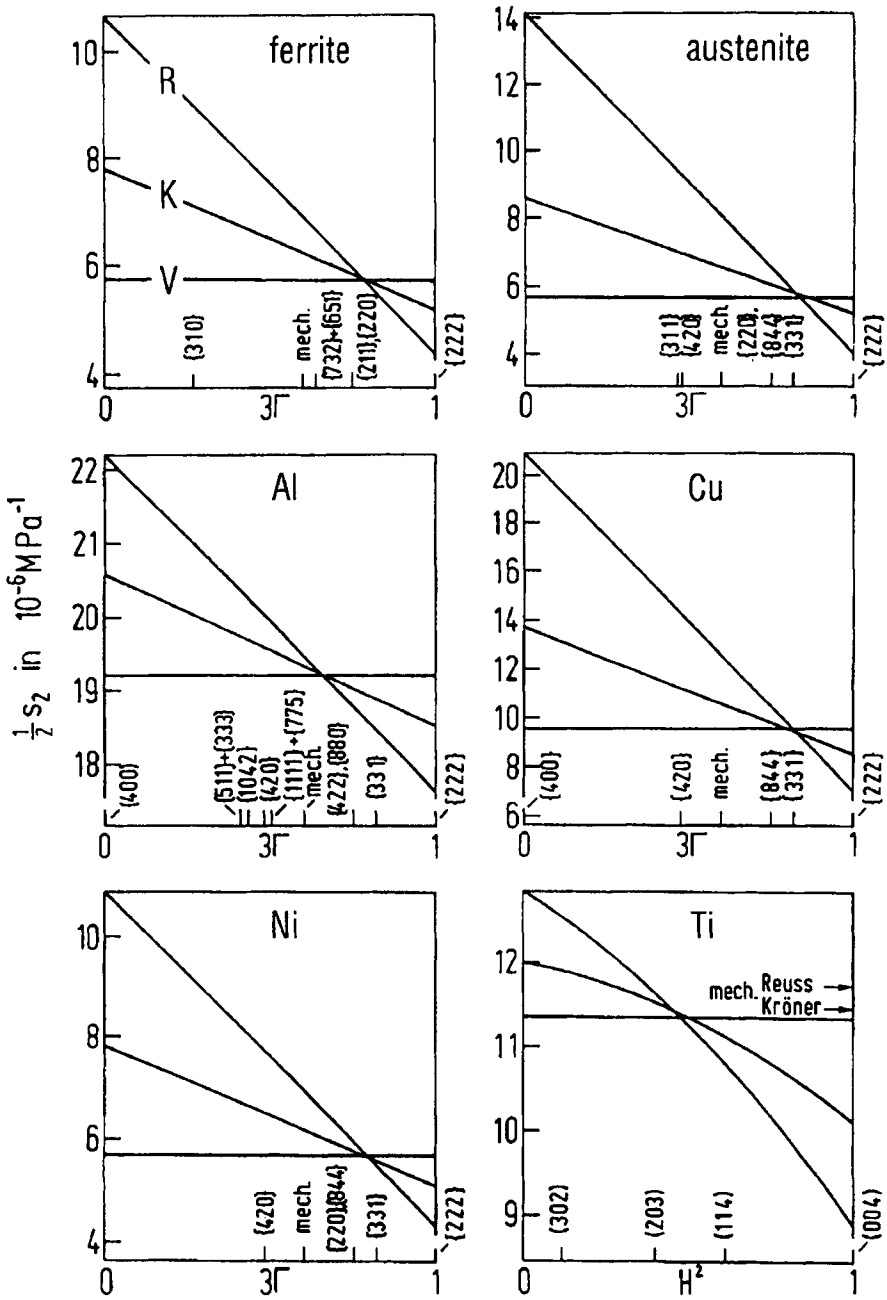


Figure 8. Examples of calculated XEC using elastic data of monocrystals /45/ and the three models according to Reuss, Eshelby-Kröner-(Kneer), Voigt.

Table 3. XEC according to the models of Voigt, Reuss, and Eshelby-Kröner. The elastic stiffnesses were taken from /45/. XEC in 10^{-6}MPa^{-1} , o.p. orientation parameter.

material (o.p.)	model	$\alpha\text{-Fe}$ (3 Γ)	$\gamma\text{-Fe}$ (3 Γ)	Cu (3 Γ)	Ni (3 Γ)	Al (3 Γ)	Ti (H ²)
$s_1(0)$	V	-1.20	-1.32	-2.25	-1.21	-4.93	-2.71
	R	-2.84	-4.17	-6.29	-2.94	-5.81	-3.24
	K	-1.88	-2.36	-3.73	-1.91	-5.33	-2.94
$s_1(1)$	V	-1.20	-1.32	-2.25	-1.21	-4.93	-2.71
	R	-0.76	-0.77	-1.41	-0.76	-4.45	-1.81
	K	-1.02	-1.12	-1.94	-1.03	-4.71	-2.25
$\frac{1}{2}s_2(0)$	V	5.61	5.73	9.16	5.43	19.10	11.31
	R	10.53	14.29	21.28	10.64	21.74	12.86
	K	7.63	8.86	13.62	7.53	20.29	11.98
$\frac{1}{2}s_2(1)$	V	5.61	5.73	9.16	5.43	19.10	11.31
	R	4.27	4.10	6.64	4.10	17.67	8.65
	K	5.06	5.14	8.23	4.90	18.45	9.96
s_1^{mech}	V	-1.20	-1.32	-2.25	-1.21	-4.93	-2.71
	R	1.59	-2.13	-3.36	-1.63	-4.99	-2.85
	K	1.36	-1.62	-2.66	-1.38	-4.96	-2.78
$\frac{1}{2}s_2^{mech}$	V	5.61	5.73	9.16	5.43	19.10	11.31
	R	6.77	8.18	12.50	6.72	19.30	11.74
	K	6.09	6.63	10.39	5.95	19.19	11.51

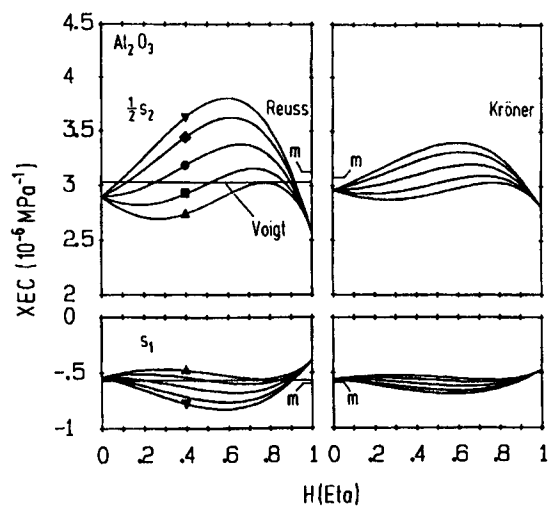


Figure 9. XEC of trigonal Al_2O_3 , calculated, /48/, P parameter of the dependences: $\blacktriangledown$ 0.25, $\blacklozenge$ 0.1 and 0.4, $\blacksquare$ 0 and 0.5, $\blacktriangle$ 0.75.

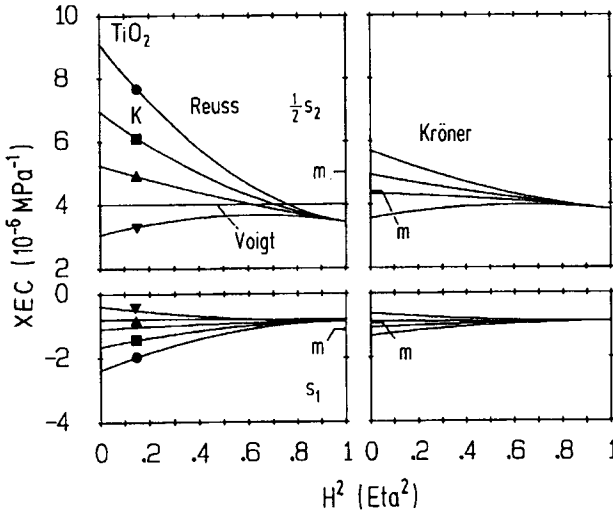


Figure 10. XEC of tetragonal TiO_2 , calculated, /48/, K parameter of the dependences: $\bullet$ 0, $\blacksquare$ 0.1, $\blacktriangle$ 0.2, $\blacktriangledown$ 0.5.

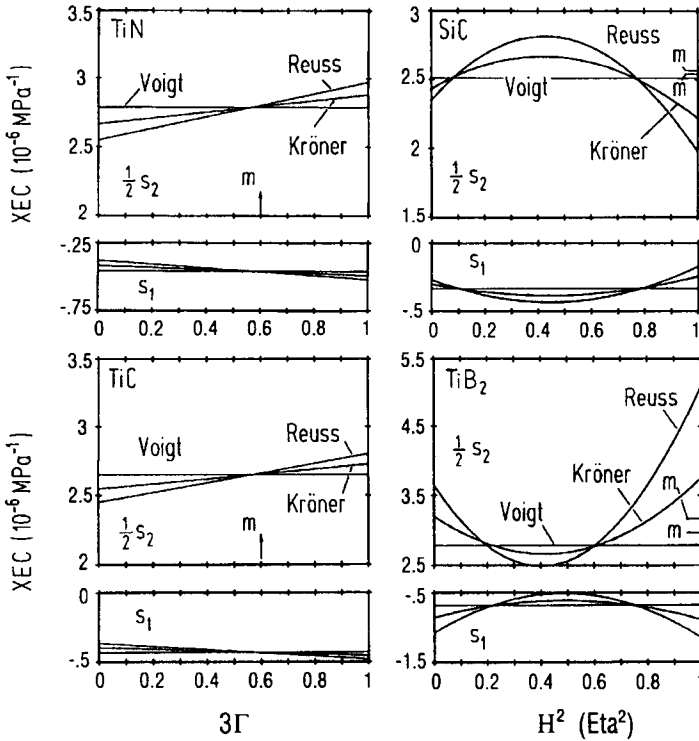


Figure 11. Calculated XEC of cubic TiN and TiC (left) and hexagonal SiC and TiB_2 (right). /48/.

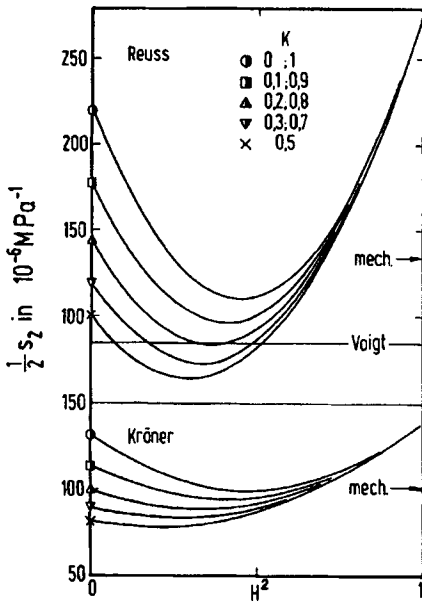


Figure 12. Calculated XEC of tetragonal Indium /25/.

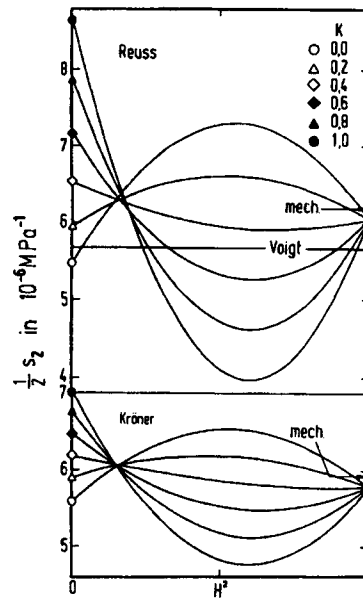


Figure 13. Calculated XEC of orthorhombic Uranium /25/.

2.134b Textured materials

The calculation of the stress factors F_{ij} of textured materials follows the paper /42/. The principal equations are given in the following. According to section 2.132b the stress factors are defined as

$$F_{ij}(\varphi, \psi, hkl) = \frac{\partial \varepsilon_{\varphi\psi}(hkl)}{\partial \bar{\sigma}_{ij}} \quad (48)$$

They connect the strain determinable by diffraction methods to the mean phase stress within a textured material. For their calculation, the strains in the measuring direction $\underline{m}$ have to be averaged considering all those crystals that contribute to the interference line, i.e. those crystals of which the lattice planes $\{hkl\}$ under study are oriented perpendicular to the direction $\underline{m}$, which is described by the angles (φ, ψ) . The strain of a single crystal is given by

$$\varepsilon_{\varphi, \psi}(\Omega) = \varepsilon_{ij}(\Omega) m_i m_j \quad (49)$$

with the unit vector $\underline{m}$ in the direction of measurement:

$$\underline{m} = \begin{pmatrix} \cos \varphi \sin \psi \\ \sin \varphi \sin \psi \\ \cos \psi \end{pmatrix} \quad (50)$$

The orientations of the crystals can be made congruent by rotation around $\underline{m}$ by an angle λ . For the calculation of the averaged strain of the lattice plane $\{hkl\}$ in direction $\underline{m}$ one has to express the orientation Ω and the strain $\epsilon(\Omega)$ of each contributing crystal in dependence of φ , ψ and λ , and integrate them over λ taking the frequency $f(\Omega)$ of the orientations (ODF) into account.

$$\epsilon_{\varphi,\psi} = \frac{\int_0^{2\pi} \epsilon_{ij}(\Omega) f(\Omega) m_i m_j d\lambda}{\int_0^{2\pi} f(\Omega) d\lambda} \quad (51)$$

and with Equ. 7 we get

$$F_{ij}(\varphi, \psi, hkl) = \frac{\int_0^{2\pi} \frac{\partial \epsilon_{kl}(\Omega)}{\partial \bar{\sigma}_{ij}} f(\Omega) m_k m_l d\lambda}{\int_0^{2\pi} f(\Omega) d\lambda} \quad (52)$$

This equation is described also in /49/ with other notation.

At this point a model assumption about the dependence of the crystal strains on the mean phase stress $\bar{\sigma}$ has to be introduced, i.e. for the Reuss model

$$\frac{\partial \epsilon_{kl}(\Omega)}{\partial \bar{\sigma}} = s_{ijkl}(\Omega) \quad (\text{Reuss}) \quad (53)$$

and for the Voigt model

$$\frac{\partial \epsilon_{kl}(\Omega)}{\partial \bar{\sigma}} = \langle c(\Omega) \rangle_{ijkl}^{-1} \quad (\text{Voigt}) \quad (54)$$

For the model according to Eshelby-Kröner we have to insert

$$\frac{\partial \epsilon_{kl}(\Omega)}{\partial \bar{\sigma}} = [S + t(\Omega)]_{ijkl} \quad (\text{Kröner}) \quad (55)$$

After inserting Equ. 53, 54 or 55 into Equ. 52 the anisotropic stress factors $F_{ij}(\varphi, \psi, hkl)$ for the three models can be calculated.

The tensor $[S + t]$ that has to be inserted for the Eshelby-Kröner model has to be calculated beforehand from the condition $\langle t \rangle = 0$ using an iteration program, analogously to the isotropic case. Also the ODF has to be considered. Examples are described in section 2.135e.

2.134c Two- and multiphase homogeneous materials

The equations to calculate the XEC according to the model Eshelby-Kröner are given in section 2.134a. The tensor S has to be calculated under the condition $\langle t \rangle = 0$. t depends on the macroscopic tensor S . The averaging over all orientations is to be made in case of isotropic materials. In case of textured materials additionally the ODF has to be considered. For multiphase materials the averaging must be made also over the phases in question with their contents. The macroscopic tensor of the compliances S of the multiphase material will be evaluated. This enables one to calculate t and $(S + t)$ of the equation for the XEC. The method described needs some effort. Another way is to use, if known, the experimentally determined tensor S .

As a third method in accordance with Hill, the averaged value of the macroscopic tensors due to the models of Voigt and Reuss will be taken. It has been shown /43/ that the resultant errors in XEC are small.

The formulae to evaluate XEC of two-phase materials are given in the following. Under a second phase the following will be understood: metallic, ceramic or polymeric phase, layer, fiber, pores. Examples of XEC demonstrate the influence of a second phase and the difference between phase-XEC and compound-XEC.

To calculate the compound-XEC from the phase XEC again models of the composite heterogeneous two-phase material have to be supposed. First, the two limiting assumptions will be dealt with. The Reuss model of homogeneous stress in all phases delivers a trivial result, namely that the compound and the phase-XEC are identical:

$$s_1^{C,\alpha} = s_1^\alpha \quad ; \quad \frac{1}{2} s_2^{C,\alpha} = \frac{1}{2} s_2^\alpha \quad (56)$$

The assumptions of homogeneous strain in these texture-free phases (Voigt), and of identical stress systems of the phases within the two-phase material, result in the following formulae /50/ :

$$s_1^{C,\alpha} = \frac{1}{2} s_2^\alpha \frac{E^\alpha}{EC} \frac{\nu^\alpha - \nu^C}{(1 + \nu^\alpha)(1 - 2\nu^\alpha)} + s_1^\alpha \frac{E^\alpha}{EC} \frac{1 - 2\nu^C}{1 - 2\nu^\alpha} \quad (57)$$

$$\frac{1}{2} s_2^{C,\alpha} = \frac{1}{2} s_2^\alpha \frac{E^\alpha}{EC} \frac{1 + \nu^C}{1 + \nu^\alpha} \quad (58)$$

The average values of the macro E and ν of the matrix (index M) of a two-phase material /8/ can be calculated by the following formulae:

Model Voigt

$$\begin{aligned} \lambda^M &= c^\alpha \lambda^\alpha + c^\beta \lambda^\beta & \mu^M &= c^\alpha \mu^\alpha + c^\beta \mu^\beta \\ E^M &= \frac{(3\lambda^M + 2\mu^M)\mu^M}{\lambda^M + \mu^M} & \nu^M &= \frac{\lambda^M}{2(\lambda^M + \mu^M)} \end{aligned} \quad (59)$$

Model Reuss

$$E^M = \frac{E^\alpha \cdot E^\beta}{c^\alpha E^\alpha + c^\beta E^\beta} \quad \nu^M = E^M \left(c^\alpha \frac{\nu^\alpha}{E^\alpha} + c^\beta \frac{\nu^\beta}{E^\beta} \right) \quad (60)$$

It is also possible to calculate the compound-XEC of multiphase materials according to the model of an ellipsoidal crystallite in a homogeneous matrix, the Eshelby-Kröner model /48/. The tensor S of the single-phase material in Equ. 41 has to be replaced by the respective tensor of the multiphase material, S^C . It can again be calculated by an iterative procedure using the condition $\langle t(\Omega) \rangle = 0$. This average has now to be taken over all crystallite orientations Ω and all present phases α . If the material is textured, the orientations have to be weighted by the ODF of the respective phase.

$$\langle t(\Omega) \rangle = \int \int_{\alpha \Omega} t^{\alpha}(\Omega) f^{\alpha}(\Omega) d\Omega = 0 \quad (61)$$

It is much easier to take the Hill approximation for the macroscopic data:

$$E^{M,Hill} = \frac{1}{2} (E^{M,Voigt} + E^{M,Reuss}) \quad ; \quad S_{1111} = \frac{1}{E} \quad (62)$$

$$\nu^{M,Hill} = \frac{1}{2} (\nu^{M,Voigt} + \nu^{M,Reuss}) \quad ; \quad S_{1212} = -\frac{\nu}{E}$$

The compound-XEC follows from Equ. 40 and 41.

The Fig. 14-17 show some compound-XEC of technically important materials calculated in dependence of the phase content. Details are to find in the captions. Applying the model Eshelby-Kröner, each phase is considered to be the inclusion in the homogeneous matrix built up by the two phases. As the macroscopic Young's modulus and the Poisson's ratio the average values of Voigt- and Reuss model are used. The compliances of cubic ZrO_2 were taken since those of the tetragonal and monoclinic ZrO_2 phases are not available.

The influence of the second phase on the compound-XEC of the other phase depends on the difference between the macroscopic constants of both phases. The following examples exhibit large and small variations of the XEC with the phase contents. It should be mentioned that the experimental XEC-values generally will have an accuracy of $\pm 5\%$.

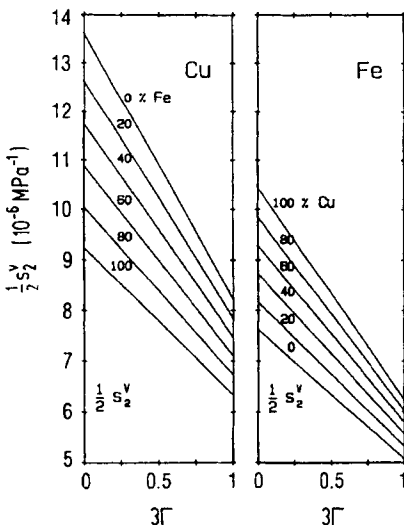


Figure 14. Compound-XEC of the Cu- and the Fe-phase of an Fe-Cu compound material /48/, dependence on the phase contents; calculation according to Eshelby-Kröner.

Another approach to the calculation of compound-XEC of multiphase materials was given in /51/. The comparison of the models will be discussed together with those of heterogeneous materials at the end of section 2.134g.

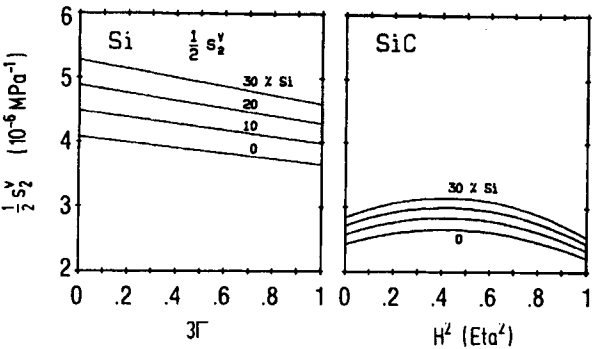


Figure 15. Compound-XEC of the Si- and the SiC-phase of a Si-SiC compound material /48/, dependence on the phase contents; calculation according to Eshelby-Kröner.

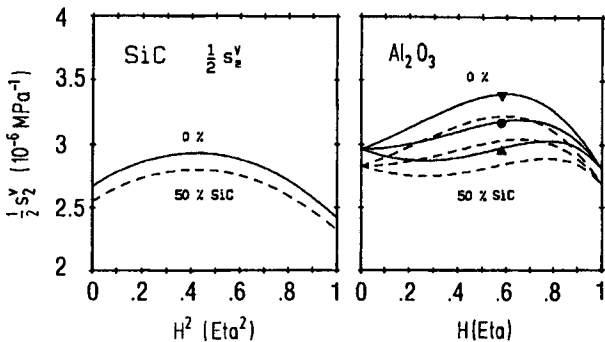


Figure 16. Compound-XEC of the SiC- and the Al_2O_3 -phase of a SiC- Al_2O_3 compound material /48/, dependence on the phase contents; calculation according to Eshelby-Kröner; parameter labels according to Fig. 9.

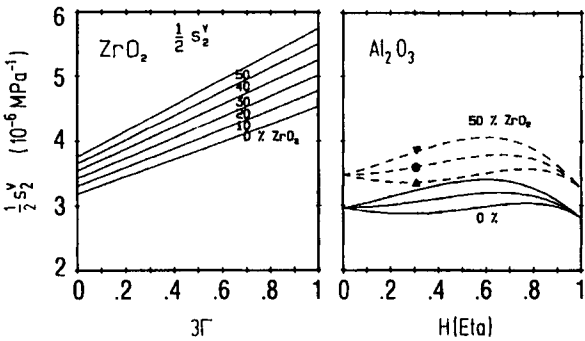


Figure 17. Compound-XEC of the ZrO_2 - and the Al_2O_3 -phase of a ZrO_2 - Al_2O_3 compound material /48/, dependence on the phase contents; calculation according to Eshelby-Kröner; parameter labels according to Fig. 9.

Hauk and Kockelmann /52,53/ have calculated the influences of the pores on the XEC on the basis of the papers of Hoffmann and Stroppe /54,55/. The shape of the pores is assumed to be ellipsoidal with different ratios of the main axis. The calculation is too lengthy to be reported here. The result is given in Fig. 18. The relative XEC, ratio XEC (material with pores) to XEC (material without pores), versus the formfactor of pores are plotted with the content of pores noted in vol.% as parameter.

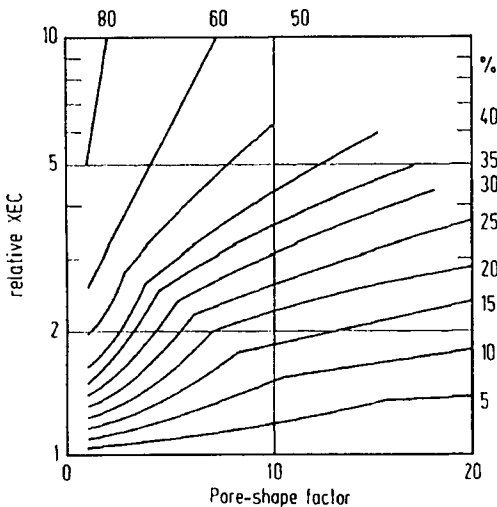


Figure 18. Dependence of the relative XEC of a porous material on the pore-shape factor /52,53/.

2.134d XEC-formulae for the application of the model of Eshelby-Kröner

The derivation of the formulae and the respective calculations of XEC for one- and two-phase materials are too lengthy for this publication. Therefore, the appropriate papers are cited: basic work /19/, one-phase material /24,25/, two-phase/multiphase material /56/.

2.134e XEC of heterogeneous layered composite materials

Hauk and Kockelmann /51/ have calculated the compound-XEC from the phase-XEC, Young's modulus and Poisson's ratio of the substrate and of the layer material. The following assumptions are made:

- The thickness of the layer is small relative to the thickness of the substrate
- The strains in the deformation (longitudinal) direction and in the transverse direction are homogeneous
- The strain in thickness direction is not constrained
- The load stress is acting parallel to the surface
- Layer and substrate materials are not textured.

Fig. 19 shows schematically the composite and the symbols /51/.

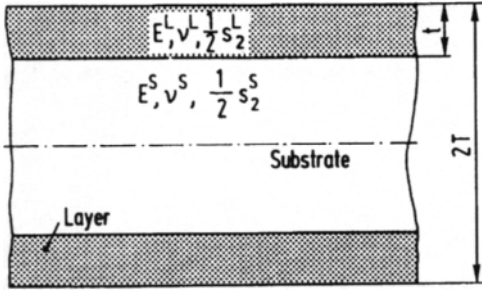


Figure 19. Layer - Substrate - Composite, schematically.

The stress state at the surface, according to the assumption that RS are not present or are negligible, is as follows:

$$\sigma_t = \frac{E^L}{E^C} \frac{1 - \nu^L \nu^C}{1 - (\nu^L)^2} \sigma_L \quad \sigma_t = \frac{E^L}{E^C} \frac{\nu^L - \nu^C}{1 - (\nu^L)^2} \sigma_L \quad \sigma_n = 0 \quad (63)$$

l, t, n longitudinal, transversal, normal direction.

The volume concentrations are noted

$$c^L = \frac{t}{T}, \quad c^S = \frac{2(T-t)}{2T} = 1 - c^L \quad (64)$$

and the composite ν^C and E^C are given by

$$\nu^C = \frac{c^L E^L \nu^L [1 - (\nu^S)^2] + c^S E^S \nu^S [1 - (\nu^L)^2]}{c^L E^L [1 - (\nu^S)^2] + c^S E^S [1 - (\nu^L)^2]} \quad (65)$$

$$E^C = c^L E^L \frac{1 - \nu^L \nu^C}{1 - (\nu^L)^2} + c^S E^S \frac{1 - \nu^S \nu^C}{1 - (\nu^S)^2} \quad (66)$$

The evaluated XEC are as follows

$$s_1^C = s_1^L \frac{E^L}{E^C} \frac{1 - \nu^C}{1 - \nu^L} \quad \frac{1}{2} s_2^C = \frac{1}{2} s_2^L \frac{E^L}{E^C} \frac{1 - \nu^L \nu^C}{1 - (\nu^L)^2} \quad (67)$$

The method can also be used when the substrate material and/or the layer material are multiphase materials.

2.134f XEC of fiber-reinforced composites

The effect of thin fibers of high-strength material oriented uniaxially in the matrix on the XEC of the composite was studied by /51,57/. The formulae derived in these papers are too voluminous to be published here.

Fibers, some μm thick and some mm in length, have a strong influence on the elastic and plastic properties and on the RS-state of materials. Anisotropic macroscopic properties are the consequence, especially if the fibers get aligned during the manufacturing process. The origin of plastic deformation and rupture processes take first place at the top of favourably oriented fibers.

The XEC of a matrix reinforced with randomly oriented fibers can be approximately handled as that of homogeneous materials, section 2.134c. The macroscopic elastic constants are needed. The compound-XEC of the fiber material can be calculated according to the models of Voigt and Reuss - homogeneous strain and stress. The shape of the fibers is irrelevant. As was proposed by /58/ the shape should be considered using the formulae of Eshelby-Kröner.

2.134g Calculation of compound-XEC of reinforced polymer materials

In the paper /59/ the XEC of polymeric materials without and with reinforcement by spheres and by fibers are experimentally determined and the results are discussed with respect to theoretical thoughts. According to the model and calculations of Eshelby /20/ and Kröner /21/ the XEC have been deduced /24,25/. Within this model, ellipsoidal crystallites are surrounded by a homogeneous matrix of the same elastic properties as the macroscopic material. If there are macroscopic stresses σ^m the homogeneous strain within the crystallites is

$$\varepsilon(\Omega) = [S + t(\Omega)]\sigma^m \quad (68)$$

S is the tensor of elastic coefficients, Ω the orientation of the crystallites and $t(\Omega)$ is a measure of the deviation of the crystallite strain from the average strain of the material /21/.

$$t(\Omega) = \left[(c(\Omega) - C + Cw)^{-1} (c(\Omega) - C) \right] S \quad (69)$$

$c(\Omega)$ and C are the elasticity tensors of the monocrystal and of the macroscopic material.

The tensor w is isotropic for spherical crystallites within an isotropic surrounding. There exist the following connections /20/:

$$w_{11} + 2w_{12} = \frac{3K + 4G}{3K} \quad w_{11} - w_{12} = \frac{5(3K + 4G)}{4(3K + 6G)} \quad (70)$$

The elastic stiffnesses and compliances C and S , necessary to calculate t , can be obtained from $\langle t \rangle = 0$ /21/. The averaging must contain all crystallite orientations and phases. Experimental values can also be used. The following relations exist

$$C_{11} = \frac{(1 - \nu)E}{(1 - 2\nu)(1 + \nu)} \quad C_{12} = \frac{\nu E}{(1 - 2\nu)(1 + \nu)} \quad (71)$$

$$S_{11} = \frac{1}{E} \qquad S_{12} = \frac{-\nu}{E}$$

(72)

The relation between the strain of the polymer and the stress within the surrounding matrix consisting of polymer and reinforcement (C-fiber, glass-fiber, glass-sphere) can be calculated with the use of the mechanical elasticity data of the polymer phases and of the macroscopic data of the total material. The average compound-XEC can be obtained from the formula /25,48/:

$$\langle s_1^C \{hkl\} \rangle = s_1^{C, mech.} = S_{12} + \langle t_{12} \rangle^P = -\frac{\nu}{E} + \langle t_{12} \rangle^P$$

(73)

$$\langle \frac{1}{2} s_2^C \{hkl\} \rangle = \frac{1}{2} s_2^{C, mech.} = S_{11} - S_{12} + \langle t_{11} - t_{12} \rangle^P = \frac{1+\nu}{E} + \langle t_{11} - t_{12} \rangle^P$$

(74)

Index P means the averaging over the phase in question. If there is only one phase, $\langle t \rangle^P = 0$ /21/ and one gets Equ. 1 and 2. $\langle t \rangle^P$ describes in case of multiphase materials the deviation of the mean strain of the phase from the macroscopic strain of the material. Fig. 20 demonstrates the different possible and necessary methods to calculate the XEC /59,56/.

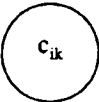
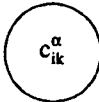
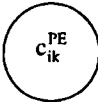
	onephase material	two-phase and multiphase material	PE	PEK PBT
inclusion				
matrix	E, ν calculated according to Kröner	E, ν calculated $E = \frac{1}{2} \left(\overline{E^K}^{Voigt} + \overline{E^K}^{Reuss} \right)$	E, ν varied	E, ν varied
XEC	$-s_1 \{hkl\}$ $\frac{1}{2} s_2 \{hkl\}$	$-s_1^c \{hkl\}$ $\frac{1}{2} s_2^c \{hkl\}$	$-s_1^c \{hkl\}$ $\frac{1}{2} s_2^c \{hkl\}$	$-s_1^{c, mech.}$ $\frac{1}{2} s_2^{c, mech.}$

Figure 20. XEC calculations according to the Eshelby-Kröner model /59,56/.

2.134h Comparison of XEC-calculations on multiphase materials using different model assumptions

A study on the XEC of two-phase materials was done early using different kinds of structures /51/. The details of the different assumptions, models and the calculation itself are handled at the appropriate places of this book. Here, as an example, the XEC of Cu-Fe materials will be discussed taking into account the XEC of Cu{331} and Fe{211}. Fig. 21 /51/ demonstrates the XEC versus the Cu-Fe composition with the structure or model as parameter. The margins are again the limiting assumptions of homogeneous strain and homogeneous stress, here for the interaction between the phases. Here, reference will be made to two similar diagrams but XEC versus orientation parameter are plotted with the Cu-Fe composition as parameter /48,56/. This figure is shown in section 2.134c.

The suppositions, models, and references of the XEC-calculations are summarized in Table 4.

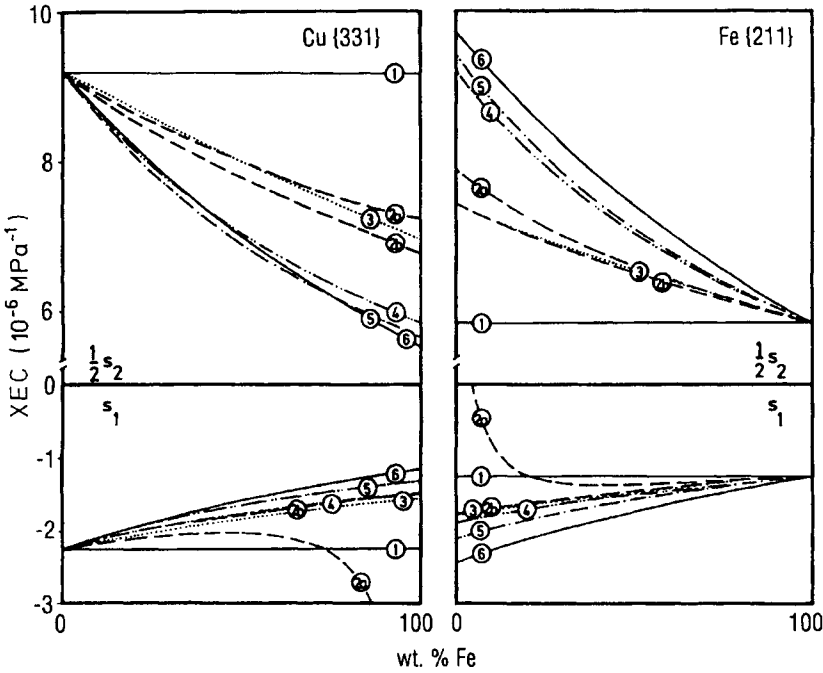


Figure 21. XEC of two-phase Cu-Fe materials /51/, — ① homogeneous stress, --- ② sintered material: a matrix, b inclusion; ③ monocrystal in two-phase matrix; - · - ④ layer-substrate material; - - - ⑤ fiber-reinforced composite (Cu matrix, Fe fiber), — ⑥ homogeneous strain.

Table 4. Publications on XEC-calculation of nontextured materials and materials with preferred orientation.

material	supposition	model	reference
nontextured; $s_1, \frac{1}{2}s_2$			
one phase	cubic	Voigt, Reuss, Eshelby-Kröner, shear modulus known	/14,15,3/ /24/ /18/
	hexagonal	V, R	/46/
	all systems	V, R, E+K+Kneer	/25/
two-/multiphase	phase-XEC known	V R	/50/ /51/
	macroscopic E known	E + K + Kn	/48,56/
two phase layer-substrate fiber reinforces pores	phase-XEC known	Oldroyd-Stroppe V V Oldroyd-Stroppe	/51/ /51/ /57/ /52/
textured; F_{ij}			
one phase	ODF	V R E + K + Kn	/60,61,37,42/ /28,29,30,61,37,42/ /42,58/
two-/multiphase	ODF, cubic, macroscopic, E known	E + K + Kn	/42/

2.134i Elastic surface anisotropy

Stickforth /19/ considered a surface anisotropy that should influence the determination of stresses in the surface region. The effect should be especially strong if the penetration depth of a radiation is small relatively to the grain size. The problem has been treated by /63/ in spite of the negative assessments by /64,65/.

The fundamental relation should read according to /19/ as follows, whereby at that time neither σ_3 nor the shear components were considered

$$\begin{aligned} \varepsilon_{\varphi,\psi} = & t_1(\psi,hkl)[\sigma_1 + \sigma_2] + \frac{1}{2}t_2(\psi,hkl)[\sigma_1 \cos^2 \varphi + \sigma_2 \sin^2 \varphi] \sin^2 \psi \\ & + t_3(\psi,hkl)[\sigma_1 - \sigma_2] \sin 2\varphi \end{aligned} \tag{75}$$

The XEC $t_1, \frac{1}{2}t_2$ correspond to $s_1, \frac{1}{2}s_2$ but here they are ψ -dependent. The term with t_3 which is also ψ -dependent, represents the influence of elastic surface anisotropy.

Bending tests /63/ on a through hardened 110MoCr4 steel and strain measurements with Cr-radiation on martensite {211} and austenite {220} prove that the modification of the fundamental strain-stress formula as postulated by Stickforth is unnecessary.

The problem of a possible influence of a surface-anisotropy on the XEC was later studied again /34/.

To estimate the influence of the stress state at the outermost surface on the XEC and on the strain distributions determined by means of X-rays the deviations caused by the condition $\sigma_{13} = \sigma_{23} = \sigma_{33} = 0$ at the surface were calculated in /66/.

The three-dimensional strains within each crystal in the volume V of the material is thought to be induced by the macroscopic stresses and strains. The stress tensor depends on the orientation of the crystal with respect to the coordinate system of the macrostresses. In this system all components of the stress tensor $\sigma(\Omega)$ of a crystal are generally finite.

The surface crystals additionally have to obey the above mentioned condition. This condition can be considered by introducing additional stress components $-\sigma_{13}$, $-\sigma_{23}$ and $-\sigma_{33}$. The strains induced by these additional stresses represent the difference between the crystal strain within the volume and the strains of the surface crystals. Two different assumptions were made:

- 1) The surface layer that is affected by this condition can be strained independently on the substrate material.
- 2) The additional strains parallel to the surface are constrained by the substrate, only the component normal to the surface is independent.

The X-ray mean value of the deviations of surface crystal's strains results from the averaging over all those crystals that are oriented in such a way that their respective lattice-plane normals coincide with the direction of measurement. It will be called ε_ψ . The profile of strain with the depth from the surface is now assumed to be described by

$$\varepsilon_\psi(z) = \varepsilon_\psi^V + \varepsilon_\psi^0 \exp\left\{-\frac{z}{d}\right\} \quad (76)$$

with z the depth, 0 means $z = 0$, d the affected surface layer thickness, and ε_ψ^V the X-ray average of the homogeneous strain of volume crystals.

Because of the attenuation of X-rays the information from the different depths has to be weighted to get the experimentally observable strain values $\langle \varepsilon \rangle$.

$$\langle \varepsilon_\psi \rangle = \frac{\int_0^D \varepsilon_\psi(z) \exp\left\{-\frac{z}{\tau}\right\} dz}{\int_0^D \exp\left\{-\frac{z}{\tau}\right\} dz} \quad (77)$$

Inserting Equ. 76 into Equ. 77 and assumig the specimen thickness D to be much larger than the penetration depth τ of the X-rays the following equation results:

$$\langle \varepsilon_\psi \rangle = \varepsilon_\psi^V + \varepsilon_\psi^0 \frac{1}{1 + \frac{\tau}{d}} \quad (78)$$

The possible influence of the surface condition on the X-ray result was calculated for the {100} lattice plane of materials with the elastic constants of α -iron. The ratio τ/d of the penetration depth and the thickness of the affected surface layer varied between 0.1 and 16. The calculated value $\frac{1}{2}s_2\{h00\}$ (model Eshelby-Kröner) is $7.63\cdot 10^{-6}\text{ MPa}^{-1}$, Fig. 22. The maximized resulting D-vs.- $\sin^2\psi$ distributions for assumed stresses of 100 MPa and 200 MPa are shown in Fig. 23: Two different models of a free and of a constrained affected surface layer give slightly different results, but both show a steeper slope than calculated for the crystals in the bulk, which are represented by the straight line in Fig. 23. The average of the two assumptions yields a more or less straight line from which the effective surface XEC can be deduced.

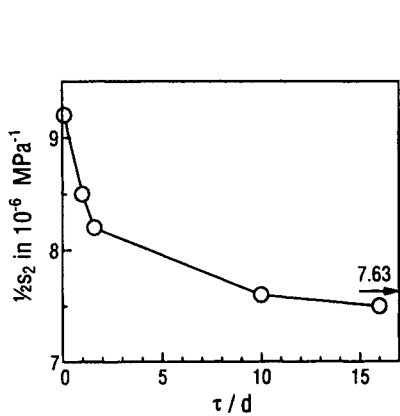


Figure 22. Influence of surface condition on XEC depending on relative penetration depth, α -Fe, Eshelby-Kröner model, {h00} peak /66/.

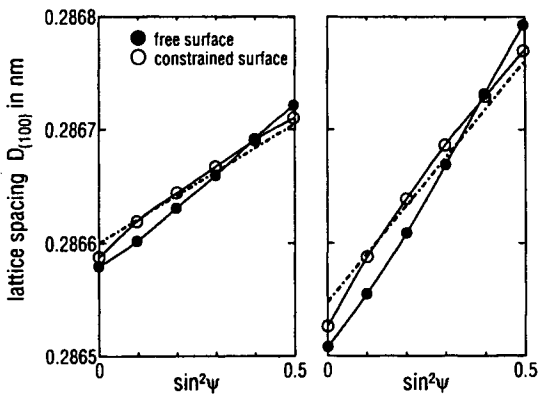


Figure 23. D{h00}-vs.- $\sin^2\psi$ distributions considering the influences of the models at the surface zone, α -Fe, Eshelby-Kröner model, $\tau/d = 1/10$, left $\bar{\sigma}_1 = 100\text{ MPa}$, right $\bar{\sigma}_1 = 200\text{ MPa}$ /66/.

According to the described model, the surface condition $\sigma_{33}(0) = 0$ should have a measurable effect on the X-ray result if the ratio τ/d is smaller than about 5. The experiments reported by /63/ and /66/ on steels using different radiations and thus penetration depths τ revealed no influence of the free surface. That means that the affected surface layer must be less than 1/5 of the smallest penetration depth used for the experiment which was about $\tau = 2\text{ }\mu\text{m}$ (Cu-radiation on steel) /66/. Therefore, the affected surface layer must be smaller than $0.4\text{ }\mu\text{m}$. Experiments with improved measuring techniques using grazing incidence or measuring the strain distribution up to $\sin^2\psi > 0.9$ can push the limit to even smaller values or perhaps determine a finite thickness of the affected surface layer. Practical stress analysis using one of the usual radiations and $\sin^2\psi < 0.9$, however, is not affected by the surface anisotropy.

It has to be stated that the above model describes the results of the condition $\sigma_{33}(0) = 0$, not the surface anisotropy effect predicted qualitatively by Stickfort /19/. Stickfort showed principally the effect of the less symmetric surrounding of surface crystals compared to those in the bulk material, but quantitative values are not available.

2.134j Determination of monocrystal data from mechanical and X-ray elastic constants of the polycrystal

In many practical cases the elastic data of monocrystals are not available because the stiffnesses or the compliances are not measured till now or the material cannot be grown in form of monocrystals of appropriate size. In these cases, a way round seems to be a solution. The XEC will be determined and from these values the elastic data of the monocrystal calculated. From these values XEC versus the orientation parameter can be calculated and the procedure checked. Hauk and Kockelmann /67/ introduced this method.

The procedure is the following: Due to section 2.134a the following basic formulae of the XEC hold, Equ. 39, 40, 41.

$$s_1 = \frac{1}{2} \gamma_i \gamma_j (\delta_{mn} - \gamma_m \gamma_n) A_{ijmn}^0 \quad \frac{1}{2} s_2 = \frac{1}{2} \gamma_i \gamma_j (3\delta_{mn} - \gamma_m \gamma_n) A_{ijmn}^0 \quad (79)$$

with γ_i components of the lattice-plane normals in the crystal lattice

δ_{mn} Kronecker symbol

A_{ijmn}^0 crystallite-coupling tensor

Further there are the three models of coupling

$$A_{ijmn}^0 = \begin{cases} \langle c(\Omega) \rangle_{ijmn}^{-1} & \text{Voigt} \\ s_{ijmn} & \text{Reuss} \\ (S - t)_{ijmn} & \text{Eshelby - Kröner} \end{cases} \quad (80)$$

There hold the following relations

Voigt model: The XEC s_1 and $\frac{1}{2} s_2$ are constant. Therefore no inverse calculation of the at least three independent s_{ij} is possible.

Reuss model: With linear equations, the s_{ij} of the crystal are calculable using the same number of determined XEC, also the mechanical ones are at disposal. The constants for different crystal systems can be found.

Eshelby-Kröner model: An iterative calculation results S and t with the assumption $\langle t \rangle = 0$. The macroscopic elastic coefficients should be determined, then S and t can be calculated

$$t = [c - C + Cw]^{-1} [c - C] S \quad (81)$$

with S, C macroscopic tensors

c monocrystal data

w^{-1} Eshelby tensor (isotropic for spherical grains in a homogeneous matrix) is only dependent on the macroscopic compression and shear modulus

$$w_{11} + 2w_{12} = \frac{3K + 4G}{3K} \quad w_{11} - w_{12} = \frac{5(3K + 4G)}{4(3K + 6G)} \quad (82)$$

Altogether these represent a nonlinear system of equations. Examples were handled /67,68/.

2.135 Examples of calculated XEC and comparison with experimental results

2.135a Accuracy of determination

If not otherwise stated the values of the compliances of monocrystals are taken from /45/. Many data of iron, steels and other alloys, among them the mechanical elastic constants versus the temperature, can be found in /69/.

An estimate of the errors was made taking into account the average set of values (1979) and the average values of the compliances published 1984 /45/. The difference of the calculated XEC $\frac{1}{2}s_2(h00)$ for iron according to the Reuss model was even not 2 %. An other estimate of the errors of XEC-calculations was made for hexagonal Ti. In /45/ there are the following data in GPa with the standard error.

$c_{11} = 160 \pm 4.8 \quad c_{33} = 181 \pm 1.8 \quad c_{44} = 46.5 \pm 0.4 \quad c_{12} = 90 \pm 3.6 \quad c_{13} = 66 \pm 3.3$

The error of the value of the calculated XEC depends naturally on the accuracy of the monocrystal elastic data. To estimate the errors of XEC from different values of the elastic constants, the compliances or the stiffnesses of the monocrystal were considered. The XEC $\frac{1}{2}s_2$ dependences versus the orientation parameter for the models of Voigt, Reuss and Eshelby-Kröner-Kneer are shown in Fig. 24. The maximal deviation from the averaged curve (Eshelby-Kröner-model) can be noticed by the error in c_{11} . If all values of the stiffnesses vary with the cited errors two boundary curves exist beside the middle one, Fig. 25. Also the quotient c/a of the lattice constants was varied by 1.6 ± 0.1 . This has no influence on the values of the XEC at $H^2 = 0$ and $H^2 = 1$ and the value of $\frac{1}{2}s_2\{114\} = (11.12 \pm 0.07) \cdot 10^{-6} \text{ MPa}^{-1}$.

It seems reasonable and careful to assume the error of XEC as $\pm 5 \%$. That fits in regard to the accuracy of the stress determination. Therefore an error bar of $\pm 5 \%$ of the calculated (Eshelby-Kröner model) XEC^m is usually plotted in the diagrams XEC versus orientation parameter.

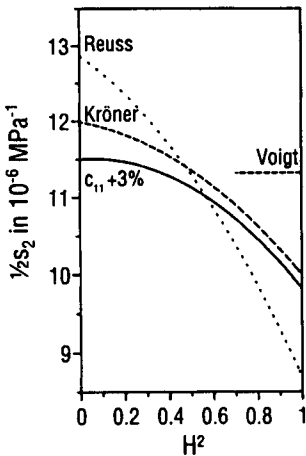


Figure 24. Calculated XEC of Ti using the three models, the stiffnesses cited in /45/ and an error in c_{11} of 3%.

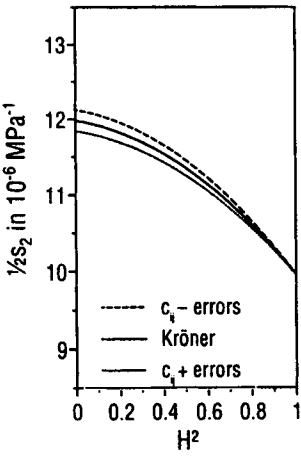


Figure 25. Calculated XEC of Ti using the Eshelby-Kröner model, stiffnesses with their errors cited in /45/.

2.135b Homogeneous materials

Beside quasi-single-phase materials, the two-phase materials Fe-Fe₃C, ferrite-austenite, martensite-austenite, Cu-Fe, Ag-W sintered materials (pores are encountered), semicrystalline polymers, ceramic materials and layer-substrate composites will be considered.

The most needed XEC are those of ferrite (α -Fe solid solution) and austenite (γ -Fe solid solution). Their XEC versus orientation parameter together with the XEC of nickel are represented in Fig. 26 for three models /70/. Experimentally determined XEC are compared below. The XEC of ferritic and low-alloy steels were often determined. Hauk and Kockelmann /71/ evaluated the averaged values depending on the orientation parameter for C-content zero and 1 wt.% as parameter using 364 experimental results of different authors. In Fig. 27 the calculated values according to the three models and the dependences from the experimental results are plotted. The agreement with the calculations based on the Eshelby-Kröner model is excellent. It is therefore to be expected that results of determinations with neutron-rays will also correspond with the calculations, Fig. 28 /72, 73/. The question of the surface anisotropy introduced by Stickforth /19/ (refer to section 2.134i) was again checked /34/, using different radiations, different penetration depths from approximately 2 μ m to 13 μ m with a low-alloy steel, Fig. 29. Here also the evaluated XEC correspond very well to the calculated ones and show no surface effect in the region considered.

Fig. 30 includes experimental and calculated results of XEC-determinations on copper /74,51/. Although the measurements were made at an early stage of the technique they fit well with the calculation using the Eshelby-Kröner model.

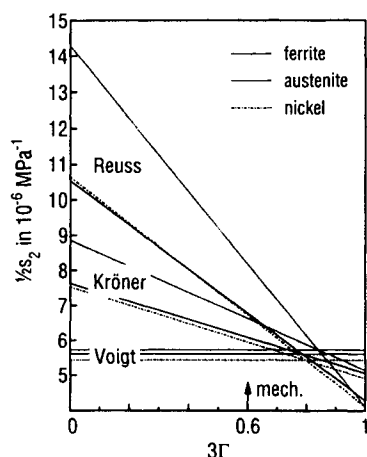


Figure 26. Calculated XEC of α -Fe, γ -Fe and Ni using the three models /70/.

Experimental XEC results on Ni-base alloys showing big differences were the reason to determine XEC on austenite- and Ni-base alloys with modern X-ray methods. The result was the confirmation of the calculated values according to the Eshelby-Kröner model, Fig. 31, /70/.

Looking at the area $3\Gamma = 0.75$ of Fig. 26 there could not be expected any surprising effect evaluating the compound-XEC of ferrite and of austenite in dualphase or duplex steels. X-ray and neutron-ray tests confirm the expectation: the XEC-values of the compound-XEC lie between the phase-XEC of both phases, Fig. 31. The experimental errors are relatively large.

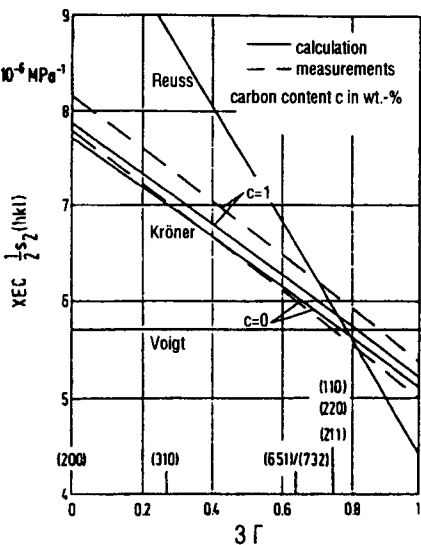


Figure 27. Dependence of the mean values of experimental XEC determinations reported in the literature as well as the calculated lines using different models /71/.

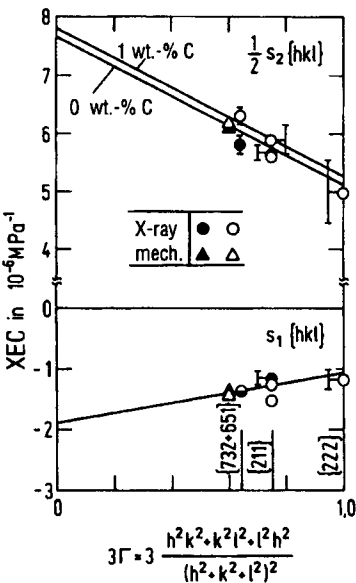


Figure 29. Experimental results for XEC of a quenched and at two temperatures tempered low-alloy steel /34/. Evaluated averaged values by /71/.

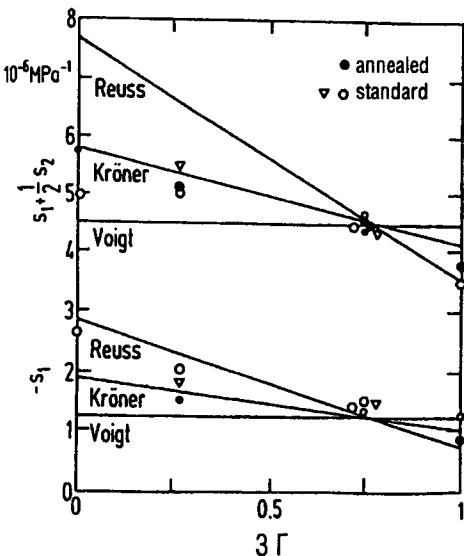


Figure 28. XEC determinations on different steels with neutron rays (/72/ ▽, /73/ ○●) compared to the calculated dependences.

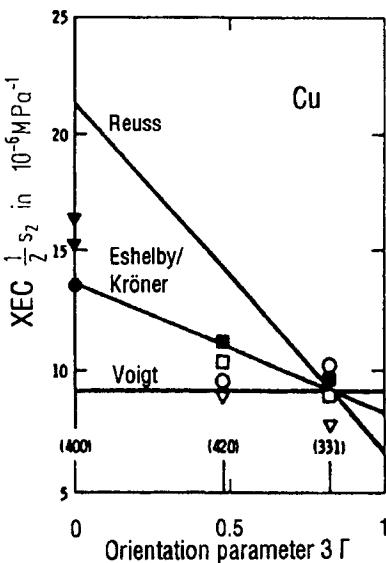


Figure 30. Experimental results of XEC for Cu, different authors besides /74/, for rolled Cu /51/.

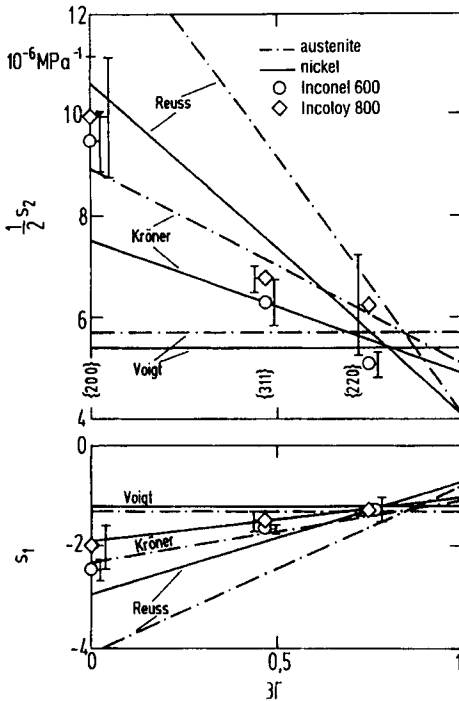


Figure 31. XEC versus the orientation parameter 3Γ determined on ground tension specimens of Incoloy 800 and Inconel 600 as well as the calculated values using monocrystal data and the three coupling models /70/.

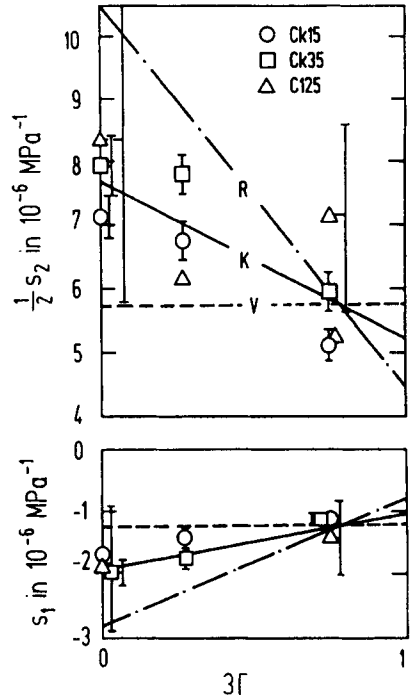


Figure 32. Results of the XEC-determinations on Ck15, Ck45, Ck35, and C125. XEC $\frac{1}{2}s_2$ and s_1 of the respective lattice planes $\{200\}+\{002\}$ ($3\Gamma=0$), $\{310\}+\{301\}+\{103\}$ ($3\Gamma=0.27$) and $\{211\}+\{112\}$ ($3\Gamma=0.75$) /75/.

A recently published study /75/ was dedicated to the problem of XEC of martensite and martensite – retained austenite steels. Experimentally there are difficulties in measuring the peaks of martensite with the very big half width and the tetragonal splitting of the peak. Reference is made to paragraph 2.045. In Fig. 32 the experimentally determined XEC of martensite of three steels versus the orientation parameter and the calculated data of ferrite are plotted. The previous recommendation is confirmed to use the values of ferrite according to the Eshelby-Kröner model.

The results of the compound-XEC martensite $\{211\}+\{112\}$ and austenite $\{220\}$ in dependence of the carbon content of martensitic-retained austenitic steels are drawn in Fig. 33. The calculated phase-XEC of ferrite and austenite due to the Eshelby-Kröner model are indicated by arrows. Possible reasons of the large scatter of the results are discussed in the paper, but no convincing conclusion could be drawn. Further systematic tests and experiments should be undertaken with variation of steels, peaks, radiations, tension and compression loads and intensive microstructure investigations. The compound-XEC of a ferritic-austenitic 50/50 vol.% duplex steel were also determined in the paper, Fig. 34, /75/. Results from neutron rays studies show practically no difference between ferrite and austenite of a duplex steel, Fig. 35 /73/. The consequences and recommendations of today for the use of compound-XEC of the two-phase steels ferrite/martensite and austenite are listed in the Table 5.

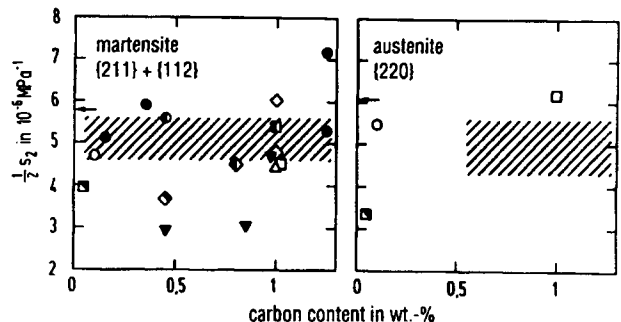


Figure 33. $\frac{1}{2} s_2$ -values of steels with martensitic-austenitic microstructure published by different authors. The regions of the numerous results of /63/ are hatched. Furthermore, the results of /75/ are drawn (●). The calculated values of ferrite and austenite, using the Eshelby-Kröner model, are indicated by arrows.

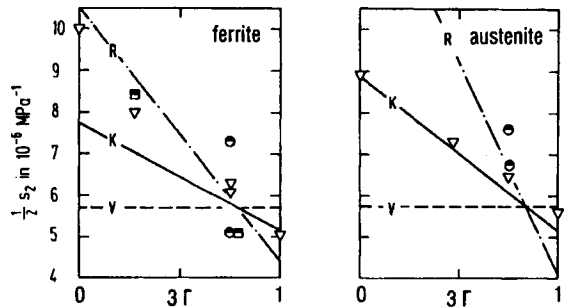


Figure 34. $\frac{1}{2} s_2$ -values of ferritic-austenitic duplex steels published by different authors. Also, the calculated linear $\frac{1}{2} s_2$ -vs.- 3Γ dependences according to the three models of Voigt, Reuss and Eshelby-Kröner are shown /75/.

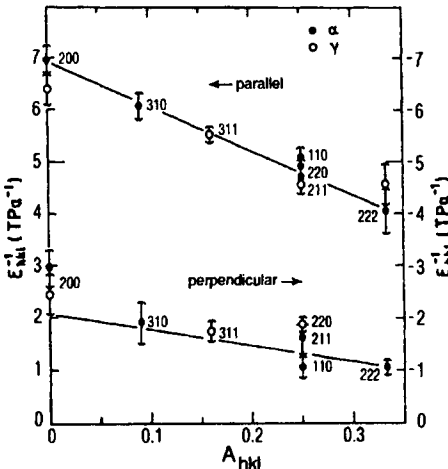


Figure 35. Anisotropic variation of elastic compliance constants for the α and γ phase of duplex alloys /73/.

Table 5. Recommended XEC /75/

material	phase-XEC $\frac{1}{2}s_2$ in 10^{-6} MPa^{-1}
dual/duplex steel, solution annealed	ferrite / martensite $\{211\} + \{112\}$: 5.8
martensitic steel + < 10% retained austenite	austenite / retained austenite $\{220\}$: 6.1
fine grained dual steel	both phases and $\{hkl\}$: 5.0
martensitic steel + > 10% retained austenite	

The XEC of the hexagonal Ti and of Ti-base alloys have been recently determined with X-rays and with neutron-rays /76,77/. The X-ray measurements were made on a peened surface evaluating the relative XEC. The calculated XEC (Eshelby-Kröner model) fit well to the experimental results. The neutron results show a larger anisotropy, maybe from an influence of texture /77/. Fig. 36 demonstrates the different results.

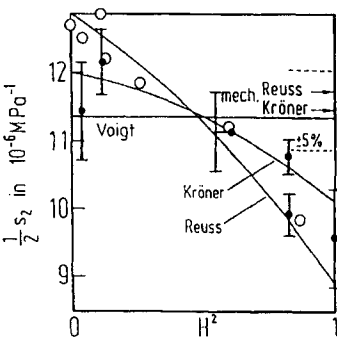


Figure 36. XEC versus orientation parameter of Ti, calculated from monocrystal data according to the three models. The mechanical value with 5% error bar is indicated. ○ neutron-ray measurement /78/, ● X-ray determination /77/.

Ceramic materials, single-phase or multiphase materials, show a rather small elastic anisotropy of the crystals /41,48/. Therefore, the mechanical XEC can be taken in most cases to evaluate LS and RS. The consequence is to look at the calculated XEC versus the orientation parameter, to check whether it is appropriate to use the mechanical values and if they do not exist take that from a tension test. The tedious experimental determination of XEC versus orientation parameter may not be necessary. Fig. 37 underlines this statement although it demonstrates the fact that the spread of the experimental results found by several authors is larger than the variation of the calculated ones versus the orientation caused by the anisotropy of the monocrystal. The calculated XEC of Al_2O_3 /41,48/ versus the orientation using the three models are shown together with experimental data of different authors /79/. The calculated mechanical values for the Eshelby-Kröner model (with a spread of $\pm 5\%$) are also plotted. Data of the XEC s_1 and $\frac{1}{2}s_2$ of various ceramic materials measured on different $\{hkl\}$ with different wavelengths by different authors are listed by /80/. The ceramics are α - Al_2O_3 (99%, 96%, 92% purity), $\text{ZrO}_2 + 3\text{mol}\% \text{Y}_2\text{O}_3$, β - Si_3N_4 (HP, HIP, sintered) and α -SiC. The experimental values should be assessed in relation to the measuring accuracy, the

relatively small anisotropy of ceramics, and the calculations using the three models /41,48/. To demonstrate the very big spread of the results a plot $\frac{1}{2}s_2\{hkl\}$ versus orientation parameter is preferable to a table, Fig. 38. The mechanical values $\frac{1}{2}s_2(\text{mech.})$ are also shown. The influence of the purity of Al_2O_3 can be estimated. The conclusion is to use the calculated mechanical values or the experimental mechanical ones.

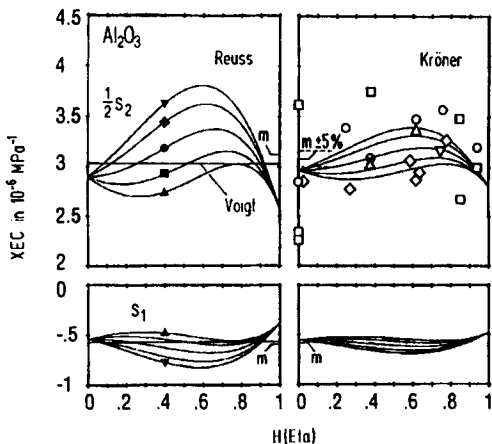


Figure 37. Calculated XEC's of Al_2O_3 /48/ compared to results of XEC-determinations by different authors, open symbols. Closed symbols indicate the orientation parameter P of the calculated curves /79/. H (Eta) second orientation parameter.

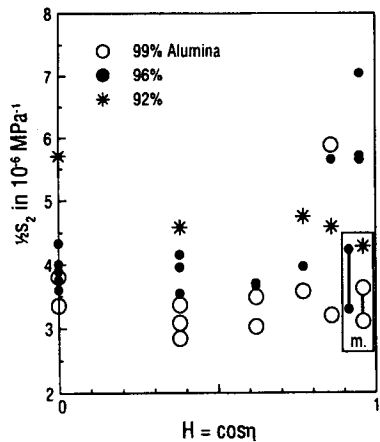


Figure 38. XEC of $\alpha\text{-Al}_2\text{O}_3$ of different purity determined by different authors listed by /80/.

XEC of two-phase sintered porous materials were tested and compared with the calculated XEC (Eshelby-Kröner-model) regarding the influence of pores /81,82/. Fig. 39 demonstrates the results of different ratios of the components and shows good agreement. Also Fig. 40 demonstrates determined and calculated XEC of Cu-Fe sintered porous materials /82/. The influence of uniaxial fibers of ferrite and of austenite in Cu was experimentally and theoretically studied /55/. Fig. 41 exhibits that they correspond very well.

XEC of $\text{Al}_2\text{O}_3 + \text{ZrO}_2$ have been determined for different compositions by /83-86/. The coupling of both phases versus the composition is dealt with in section 2.122b.

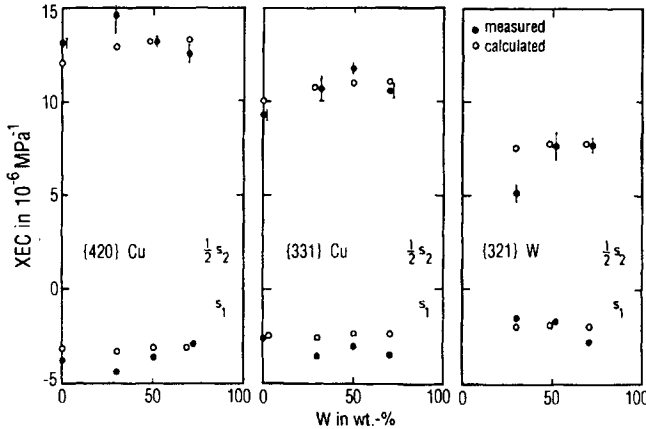


Figure 39. XEC of Cu-W two-phase sintered materials, experimentally determined and calculated /81/.

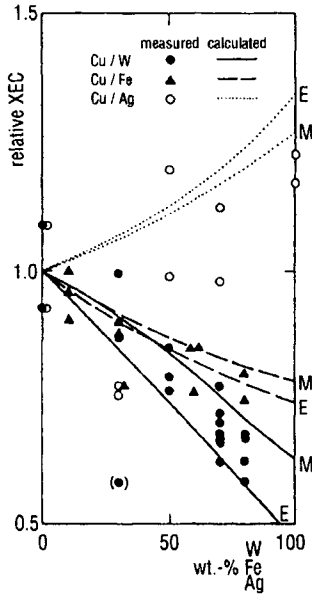


Figure 40. Relative XEC ($\frac{1}{2}s_2(\text{Cu})=1$) of Cu-based two-phase sintered materials, determined and calculated (model spherical inclusion) E and hollow sphere M /82/.

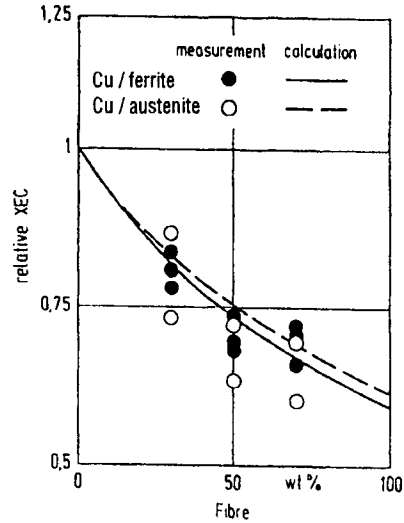


Figure 41. Relative XEC ($\frac{1}{2}s_2(\text{Cu})=1$) of both phases of fiber-reinforced (by ferrite, austenite) Cu materials /55/.

XEC of the two-phase Cu-Ni-alloys were determined and compared with the calculated ones concerning the three models, Fig. 42 /87/.

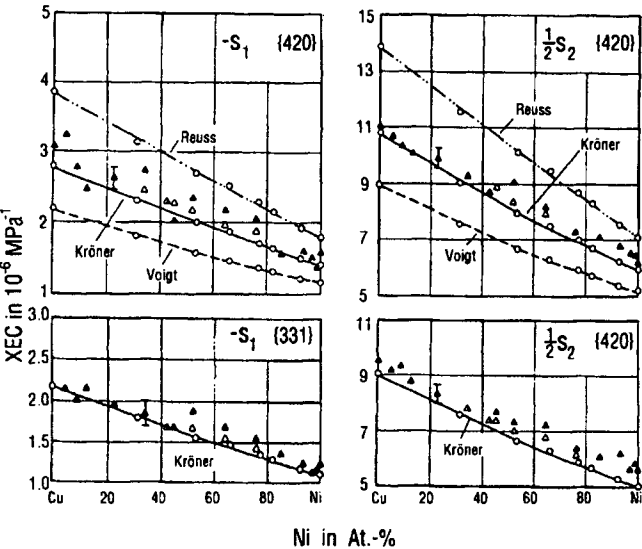


Figure 42. Measured and according to the three models calculated XEC of polycrystalline Cu-Ni-alloys /87/

2.135c Layers

Interesting results of comparisons of calculated and experimentally determined XEC of different layers on different substrates are shown in Fig. 43 /88/. The investigation demonstrates very well the validation of the theoretical studies of the layer-substrate composites, see section 2.134e. A further study of a seldom used but interesting composite Cr₇C₃ layer on steel furnished results for the XEC of the composite (compound-XEC) as well as of the phase-XEC of the layer Fig. 44, /89/. Of specific interest is the presence of macro-RS and oriented thermal micro-RS.

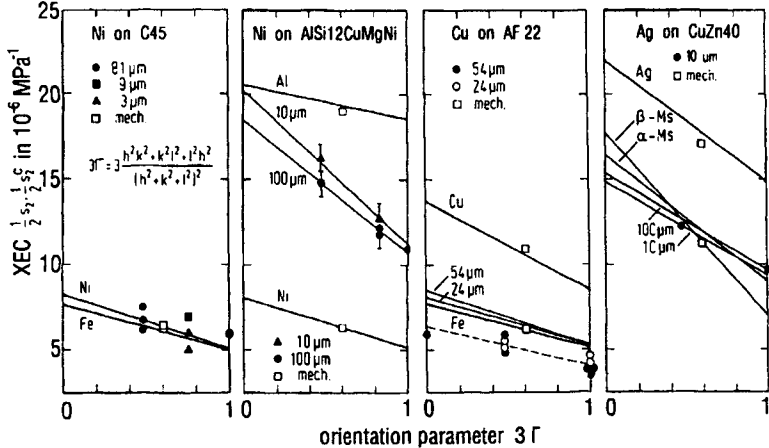


Figure 43. Compound-XEC of both phases of layer – substrate composites, from left to right: Ni-layer/C45 steel, Ni/Al alloy, Cu/duplex steel, Ag/CuZn40 /88/.

A further example of a layer-substrate composite was studied in the combination of Cr_2O_3 layer on Inconel substrate /91/. Loading and unloading experiments, Fig. 45, and compound-XEC determinations of layer and of substrate result in phase-XEC of the layer that agree well with the calculated ones.

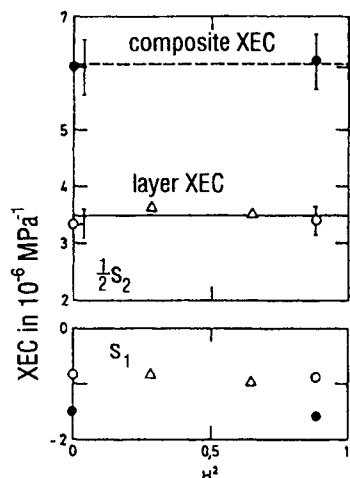


Figure 44. XEC data of Cr_7C_3 determined on the composite material Cr_7C_3 / steel ● and evaluated for the layer material ○. Measurement results of /90/ Δ /89/.

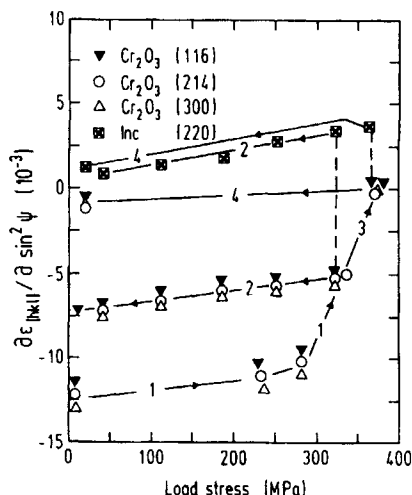


Figure 45. Slopes of the ϵ vs. $\sin^2\psi$ distributions of the Cr_2O_3 layer and the Inconel substrate versus the load stress. The numbers denote the chronological order /91/

2.135d Polymeric materials

A very interesting and new subject is the study of XEC of semicrystalline polymers. The first example concerns polyethylene /92/. Polyethylene (PE) consists of crystalline orthorhombic PE and amorphous PE. The XEC-vs.- H^2 were calculated according to the Eshelby-Kröner model from the compliances available /92/, Fig. 46. The orientation parameter H^2 equals to $\cos^2\eta$, η is the polar angle between the chain direction and the normal direction of $\{hkl\}$, under consideration; the parameter relates to azimuth ρ . Taking into account the real E-modulus of the matrix depending on the contents of crystalline-amorphous phases and the orientation of the chains the XEC for $H^2=1$ are of values typed of metallic materials, Fig. 47. Fig. 48 shows experimental results of three semicrystalline polymers. Fig. 49 demonstrates the connection between the mechanical and X-ray Young's moduli for two orientation parameters. The experimental values fit best to the Kröner dependence.

Besides the stress analysis on unfilled polymers, the strains and stresses in the phases of the polymers and in fillers like fibers in reinforced polymeric materials are of recent interest. In the following, the state-of-the-art will be described /59/. The determination of XEC of reinforced semicrystalline polymers was explained in section 2.134g. The elastic data of the polymers PEK and PBT that are necessary to calculate the XEC are not known. The averaged values, however, can be calculated, that describe the mutual relation between polymer and reinforcement (C-fibers, glass fibers, glass spheres). For the averaged compound-XEC it holds /25,48/:

$$\langle s_1^C \{hkl\} \rangle = s_1^{C,m} = S_{12} + \langle t_{12} \rangle^P = - \frac{\nu}{E} + \langle t_{12} \rangle^P \tag{83}$$

$$\langle \tfrac{1}{2} s_2^C \{hkl\} \rangle = \tfrac{1}{2} s_2^{C,m} = S_{11} - S_{12} + \langle t_{11} + t_{12} \rangle^P = \frac{1 + \nu}{E} - \langle t_{11} + t_{12} \rangle^P$$

Indices C indicate compound, m mechanical, P phase, $\langle t \rangle^P$ the deviations of the averaged strain of the phase from the macroscopic strain of the material.

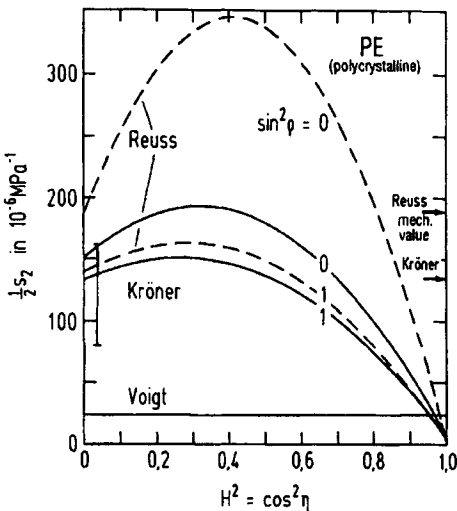


Figure 46. Calculated XEC of 100% crystalline PE using the three models and the following monocrystal stiffnesses (in 10³ MPa): c₁₁ = 6.77, c₂₂ = 9.48, c₃₃ = 2.87, c₂₃ = 4.05, c₁₃ = 1.90, c₁₂ = 2.48, c₄₄ = 3.04, c₅₅ = 1.23, c₆₆ = 3.07 /92/.

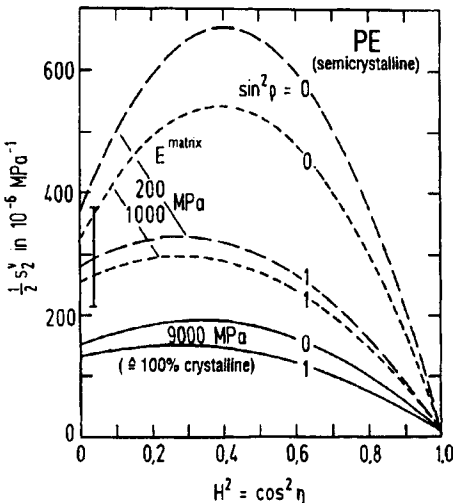


Figure 47. Calculated compound-XEC of PE using the model of Eshelby-Kröner and three values of Young's modulus /92/.

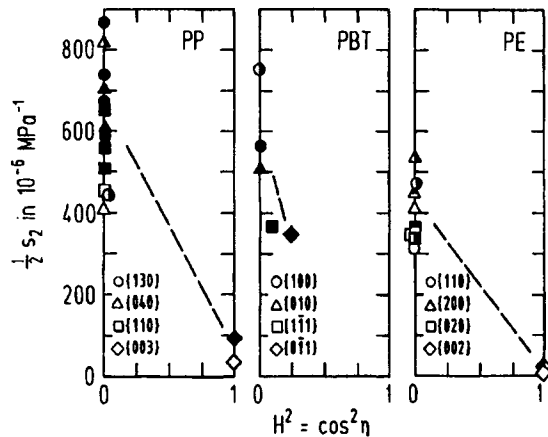


Figure 48. The published experimental XEC of PP, PBT, and PE versus the orientation parameter H² (ETA²). The data were taken from different papers /92/.

Fig. 50 and 51 demonstrate the compound-XEC versus the macroscopic Young's modulus of the polymers PEK and PBT each nonreinforced and reinforced by the named fillers /59/.

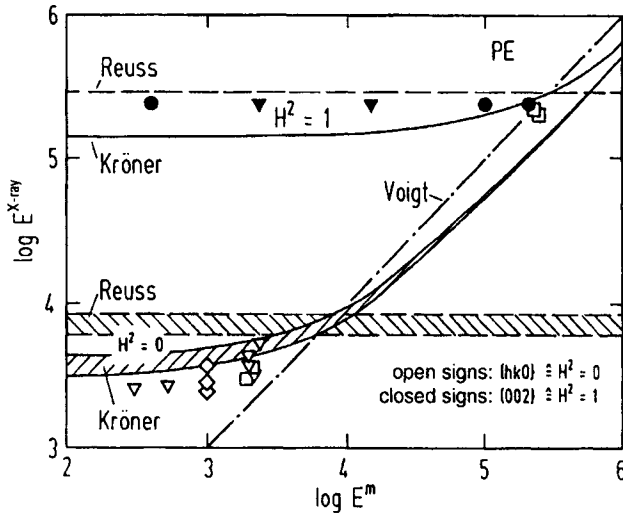


Figure 49. Relation of the $E^{X\text{-ray}}\{hkl\}=(s_1 + \frac{1}{2}s_2)^{-1}$ to Young's modulus E^m experimentally determined by different authors and calculated using the three models /92/.

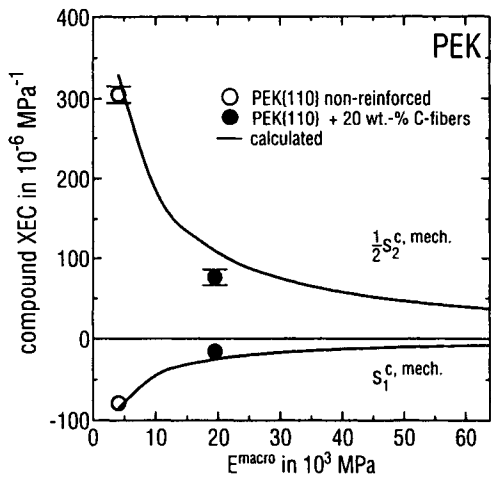


Figure 50. X-ray determined and calculated compound-XEC of nonreinforced and with C-fibers reinforced PEK composites, their dependence on the macroscopic Young's modulus /59/

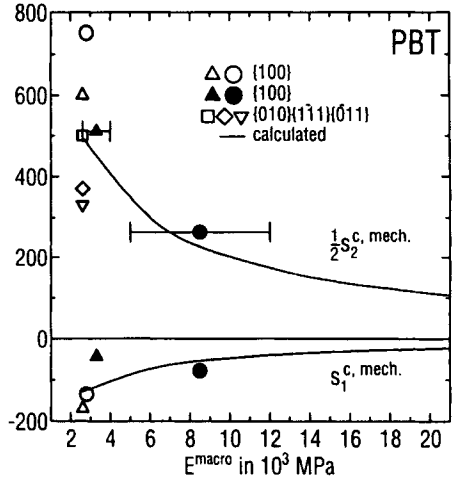


Figure 51. X-ray determined and calculated compound-XEC of nonreinforced and with glass reinforced PBT composites their dependence on the macroscopic Young's modulus ($\Delta \nabla \blacktriangle$ glass spheres, $\bullet \circ$ glass fibers) /59/.

2.135e XEC of textured materials

The comparison between calculated and experimentally in uniaxial tension test evaluated stress factors F_{11} is to be found in the following figures of ferrite, austenite, α - and β -phase of CuZn40. References are given in the captions, together with some details about the textured material and the measurement.

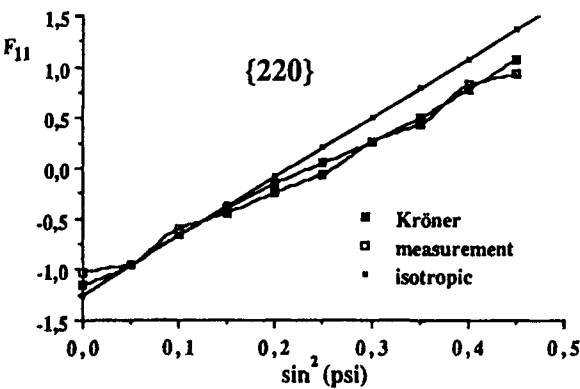


Figure 52. Comparison between experimental and calculated values of X-ray elastic constants, low carbon steel /58/.

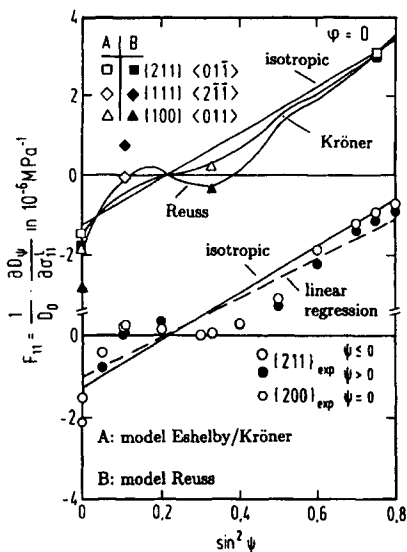


Figure 53. Calculated and experimentally determined stress factors $F_{11}(0^\circ, \psi)$ of the $\{211\}$ peak of a ferritic quasi-unalloyed steel strip. Also strains of the crystallite groups are drawn /37/

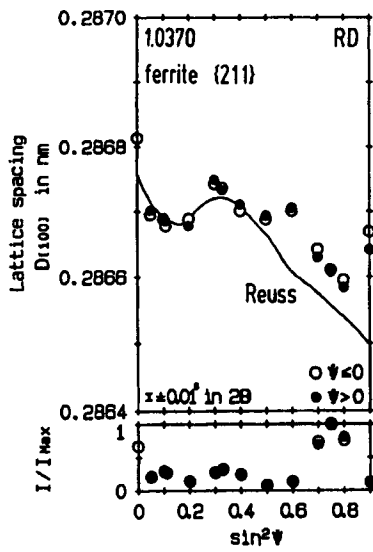


Figure 54. Lattice strain $\{211\}$ distribution versus $\sin^2 \psi$ of a cold-rolled ferritic quasi-unalloyed steel strip. The calculation considered the ODF /62,37/.

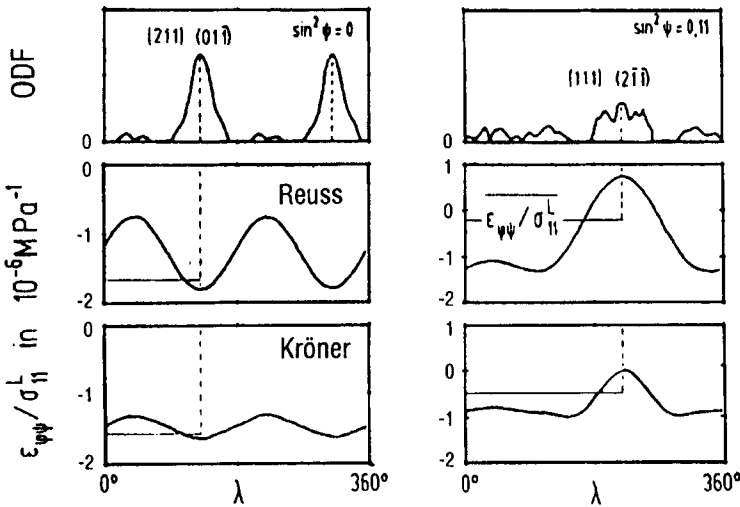


Figure 55. Intensity (ODF) and normalized strain $\epsilon_{0^\circ, \psi}(g)/\sigma_{11}^L$ of all those crystallites that contribute to the X-ray reflection in the poles of two crystallite groups in a ferritic steel strip; dependence on λ , i.e. the rotation angle of a crystallite around the lattice-plane normal. The X-ray mean values are indicated [42].

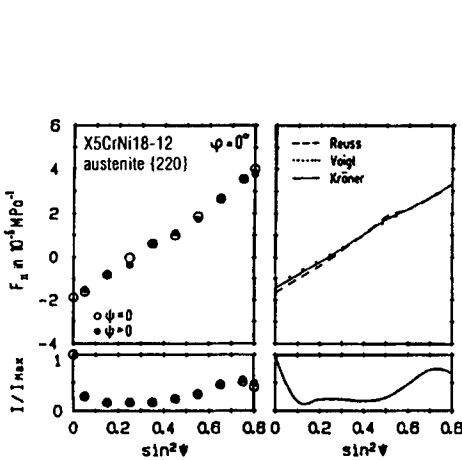


Figure 56. Measured and calculated stress factors F_{11} and intensities; austenitic steel, $\{220\}$, [42].

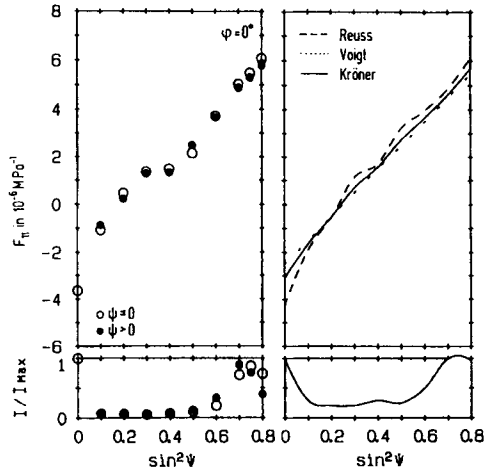


Figure 57. Measured and calculated stress factors F_{11} and intensities; α -phase of CuZn40, $\{220\}$, [42].

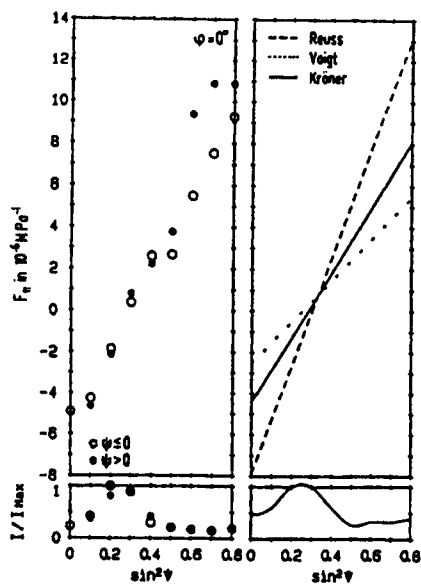


Figure 58. Measured and calculated stress factors F_{11} and intensities; α -phase of CuZn40, $\{200\}$, $/42/$.

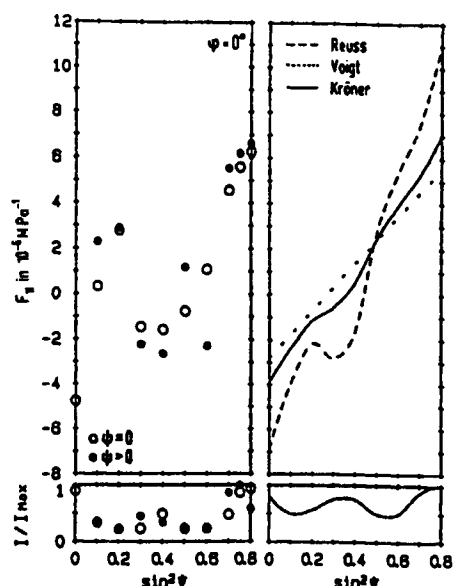


Figure 59. Measured and calculated stress factors F_{11} and intensities; β -phase of CuZn40, $\{211\}$, $/42/$.

2.135f Different influences on XEC

As stated above the experimental determination of the XEC will be done in a uniaxial tension or bending test. The question arose in the early stage of the XSA whether there are influences on the values of XEC with the type of load (tension, compression, torsion, uniaxial or biaxial loads, elastic or plastic deformation). The experimental investigations showed sometimes other values and some influence of the kind of elastic and plastic deformation. Different material-physical effects and interpretations were discussed. Here are some results that are known for several years.

Because of the microstructure of cast iron one expects differences of the XEC determined by applying tension or compression loads, Fig. 60 /93/.

XEC-determinations on steels with Co- and Cr-radiations on torsionally loaded specimens result in smaller values than those tensioned or calculated using the Eshelby-Kröner model, Table 6. It seems that further studies with torsional specimen should be made.

Cylinders of three steels were loaded inside with oil to determine the XEC in a test with biaxial elastic and elastic-plastic loads /96/. Fig. 61 proves that values of the XEC are as expected, but with the plastic deformation the XEC $\{211\}$ get smaller. Many tests were undertaken to check a possible influence of plastic strain on the XEC of steels. Fig. 62, /97/ demonstrates that there is no influence of the plastic strain on the value of XEC besides XEC $\{211\}$ which get smaller with the plastic strain. The same experiments were made with copper specimens with the result of no influence, Fig. 63, /74/. The reason of the diminishing of the XEC $\{211\}$ versus plastic strain may be explained by the easier deformability of the surface

region of the material. The measurement technique may have an additional effect in former times with the restriction in $\sin^2\psi \leq 0.6$.

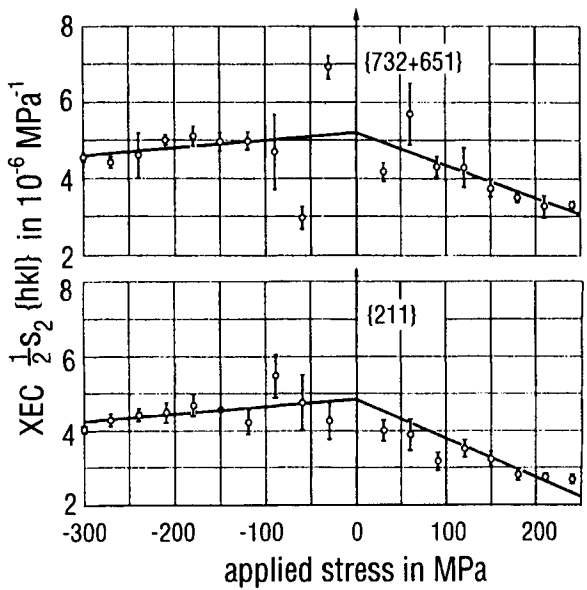


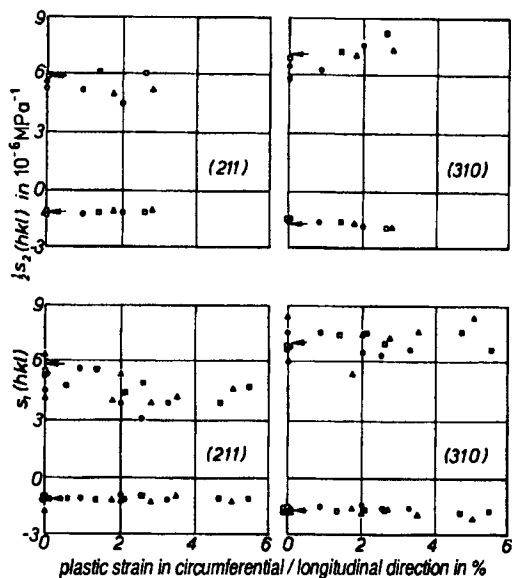
Figure 60. XEC $\frac{1}{2}s_2 \{hkl\}$ versus applied stress of GG22MoCr grey cast iron using different specimens for tensile and compressive loads /93/

Table 6. Comparison of XEC determined by torsional and tensile loads.

steel C in wt.%	load	$\frac{1}{2}s_2$ in 10^{-6} MPa^{-1}		author
		{310}	{211}	
0.6	torsion	6.0 ± 0.2	—	Glocker, Schaaber /94/ Hauk, Kockelmann /71/
	tension	7.3 ± 0.2	5.9 ± 0.1	
1.0	torsion	6.0 ± 0.2	5.3 ± 0.2	Bollenrath, Fröhlich, Hauk /95/ Hauk, Kockelmann /71/
	tension	7.4 ± 0.3	6.1 ± 0.2	

Results of a recently published study by neutron rays on the influence of plastic strain on the values of XEC of a low-alloy ferritic-pearlitic textured steel are plotted in Fig. 64 /98/. The conclusion seems correct that there is no influence of plastic strain. The values of the {200} peak are higher than predicted by the use of the Eshelby-Kröner model. The arrows on the left-hand axis mark the results of a XEC-calculation /24/. An influence of texture may be responsible for the high values of the {200} data.

A real surface effect is shown in Fig. 65, /97/. The XEC versus the ratio of the average penetration depth and crystallite size is constant when the measurement is made using {310} peaks and decreases using the {211} peaks.



steel	flat specimen	tube body
15Mo3	■	□
13CrMo4-4	▲	△
34CrMo4	●	○

Figure 61. XEC as a function of the plastic strain in the circumferential / longitudinal direction; the lower diagrams apply to measurements in strain-hardening direction, the upper diagrams are perpendicular to that direction /96/.

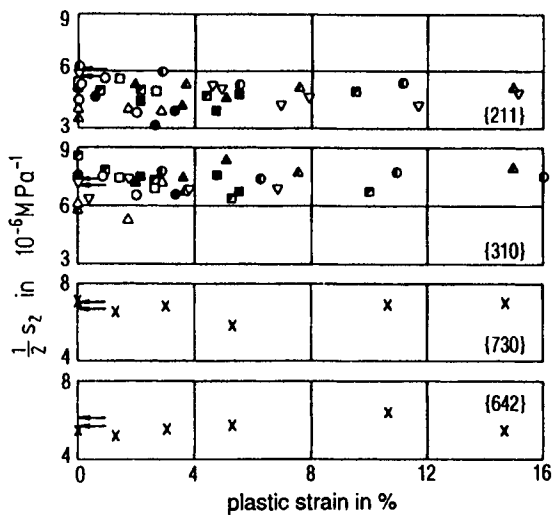


Figure 62. XEC $\frac{1}{2}s_2\{hkl\}$ versus plastic strain of ferritic pearlitic steel, different authors, the arrows indicate the calculated values /71/ for 0 and 1 wt.% C and no plastic strain /97/.

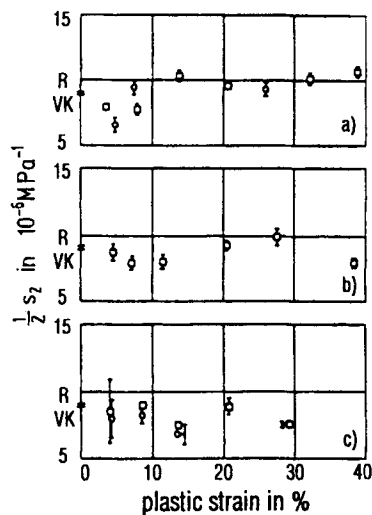


Figure 63. XEC $\frac{1}{2}s_2\{331\}$ of 0%, 50%, 90% rolled Cu versus plastic strain. The calculated values according to the three models are indicated /74/.

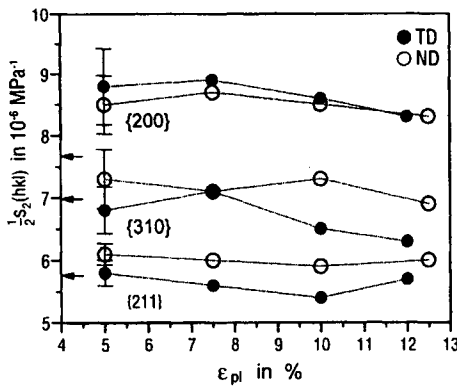


Figure 64. XEC versus plastic strain of a ferritic-pearlitic low-alloy textured steel evaluated by neutron rays /98/.

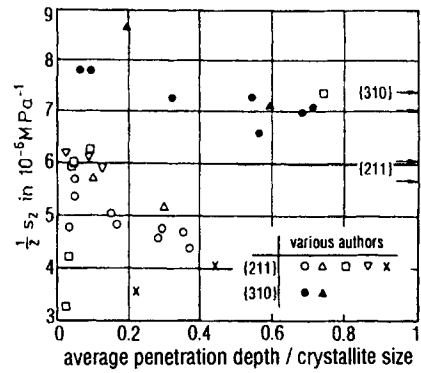


Figure 65. XEC $\frac{1}{2} s_2 \{hkl\}$ of steels versus the ratio averaged-penetration-depth / crystallite-size, different authors, the arrows indicate the values /71/ for 0% and 1 wt.% C /97/.

Temperature studies on different materials about the relaxation and development of RS increase. The determinations of the XEC have to take into account the different RS-states, macro- and microstresses, during heating, tempering, and cooling. In situ devices are in operation at some institutions. The following XEC-studies are known: /99-103/. Of practical interest but not very much used is XSA at elevated temperature. It is expected from the mechanical dependences of Young's moduli decreasing and Poisson's ratio being constant with temperature that XEC $\{hkl\}$ will increase with temperature. Results for different steels and for a Ni-alloy demonstrate the prediction. Again, XEC $\{211\}$ at 300°C makes an exception, Fig. 66, /97/. Fig. 67 shows results for a steel using a newly developed instrument /103/.

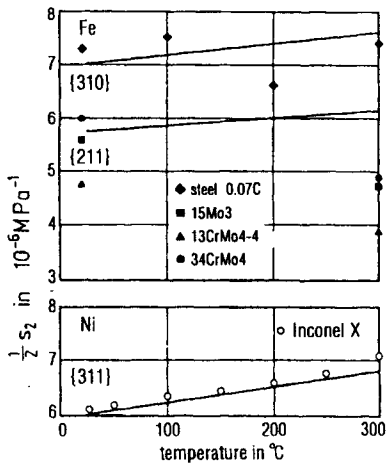


Figure 66. Temperature dependence of the XEC $\frac{1}{2} s_2 \{hkl\}$. Calculation from temperature-dependent monocrystal data as well as measured values /99,104,97/.

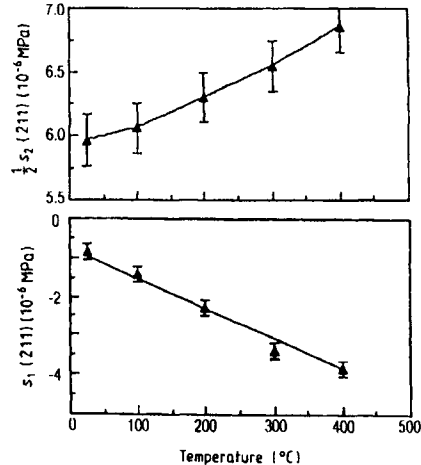


Figure 67. XEC $\{211\}$ as a function of temperature (XC75) steel /103/.

2.136 Recommendations

Calculated or experimentally determined XEC are used to evaluate stresses from strains, measured by X- or neutron-rays, caused by tension, compression, torsion, by applied or residual stresses, by uniaxial, biaxial or triaxial loads in elastically or elastically-plastically deformed material states. The procedure to determine and to use the XEC will be pointed out in the following. Several cases must be distinguished.

General remarks

- If the range of the XEC-values versus the orientation parameter calculated according to the Eshelby-Kröner model is small, the anisotropy influence can be neglected, and the calculated or even the actual mechanical XEC should be used. E^m the Young's modulus should be determined mechanically, if possible also the Poisson's ratio, or taken from the literature.
- A relatively small difference between one or two experimentally determined XEC and the calculated ones according to the Eshelby-Kröner model in dependence of the orientation parameter should not be the reason for a doubt on the assumptions of the theory. Influences of a second phase, of pores, of texture or of measurement inaccuracy may be the reason of the deviations.
- New experiments should be carefully planned. Otherwise the scientific knowledge will advance only by a very small step. Here are some thoughts for new studies. Check quantitatively the calculations according to the Eshelby-Kröner model by measurements on many peaks in the total range of the orientation parameter on materials with very high anisotropy, with low crystallographic symmetry, strongly textured, or strongly anisotropic reinforced heterogenous structures.
- In case that the micro-RS are constant during elastic straining of multiphase material, the phase-XEC will be determined, otherwise the composite-XEC.

Linear D-vs.- $\sin^2\psi$ dependence

- Calculated XEC versus orientation parameter according to the three models of Voigt, Reuss and Eshelby-Kröner are available or can be calculated from published data of compliances. The E^m should be determined and compared with the calculated one, the Poisson's constant should be taken from the literature. If there is an agreement between the experimental XEC and the calculated one (Eshelby-Kröner model) within about 5%, the calculated XEC should be taken.
- If there is a difference of more than 5% the calculated XEC should be corrected using the model of homogeneous strain or, in case of heterogeneous materials the model of Eshelby $-\log E^X - \log E^m$ curve - should be used for polymers.
- It should be checked carefully whether there exist calculated XEC or the compliances of the single crystal. If they are not available, an experimental determination of the XEC of the specific $\{hkl\}$ has to be made.
- Influences of the plastic deformation of the sample on the numerical quantity of XEC can be ignored if the deformation does not alter the D-vs.- $\sin^2\psi$ linear dependence.
- It is common practice to polish or to grind the surface of coarse-grained material to get closed with the respective accuracy measureable interference lines.

- XEC determinations should be done by decreasing loads to avoid creep and relaxation influences on the value of XEC. Measurements with increasing loads should be made to notice any deviation from a linear load-strain dependence.
- In case of multiphase materials the stresses in the transverse direction under load should be measured. If there are alterations with the load the stress transfer factors f_{11} and f_{12} must be taken into account. Details are to be found in section 2.122e.
- The compound-XEC of multiphase materials can be used to determine the LS and RS if the load-independent RS are known. If not, only the differences of LS and RS will be found. Some details are given in the Table 7 /76/.

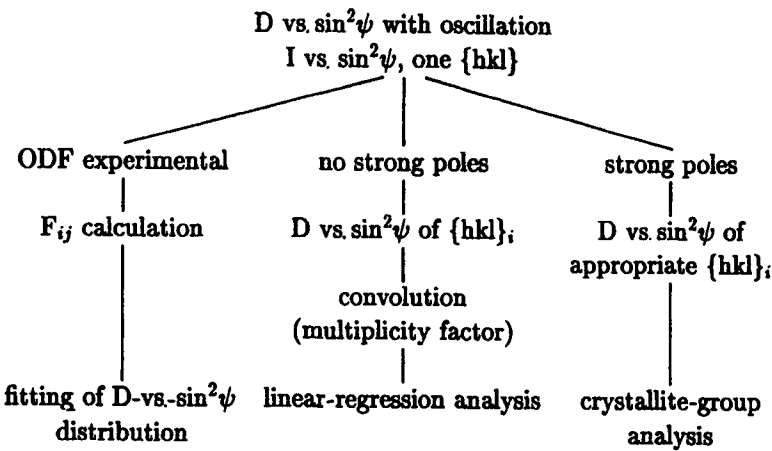
Table 7. As to the use of phase- and compound-XEC in stress evaluation /76/

structure of the material	phase-XEC		compound-XEC	
	experimentally determined on quasi-single phase material	calculated, model Eshelby/Kröner	experimentally determined on compound material	calculated using Eshelby model and Young's modulus layer- or fiber- compound model
	phase-RS		macro-RS	
quasi-single phase	$\sigma^I + \langle \sigma^{II} + \sigma^{III} \rangle$			
dual-/multiphase phase content	$\sigma^I + \langle \sigma^{II} + \sigma^{III} \rangle$		alteration of macrostress	
< 30 vol. %			$\sigma^I \pm 100 \text{ MPa}$	
≈ 50 vol. %			$\approx \sigma^I$	
> 80 vol. %				
layer-, fiber- composite			alteration of macrostress	

Nonlinear D-vs.- $\sin^2\psi$ dependences

- The procedure to select or to determine the stress factor should follow the rules which are given in Table 8; the chapter on the textured materials will deal with the details /76/.

Table 8. Stress evaluation in case of nonlinear D-vs.- $\sin^2\psi$ distributions /76/



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2.14 Shear components

2.141 Historical review

Studies of the RS after grinding of steel surfaces were the origin of the experimental discovery of the ψ -splitting. The interpretation as due to a shear component or an inclined normal stress state leads to today's commonly used method to determine the strain and stress tensor of the total RS-state. Years before, the method to measure the D-vs.- $\sin^2\psi$ dependence in both directions of $\psi \leq 0^\circ$ and $\psi > 0^\circ$ was used as a check of the exact alignment of the specimen within the diffractometer /1/. Evenschor and Hauk /2/ introduced the strain tensor of a volume-element in a triaxial-loaded component in which lattice strains with normal strains and with shears are transferred and showed the theoretical connection between shear components and the elliptical D-vs.- $\sin^2\psi$ dependences, Fig. 15, 2.072c. But Walburger /3,4/ was the first one who noticed that rotating a ground steel specimen in the diffractometer by 180° results in different D-vs.- $\sin^2\psi$ dependences, Fig. 1. Both branches $\psi < 0^\circ$ and $\psi > 0^\circ$ of the D-vs.- $\sin^2\psi$ dependence would correspond to different values of RS when evaluated separately. The azimuth and depth profiles were studied by /5/.

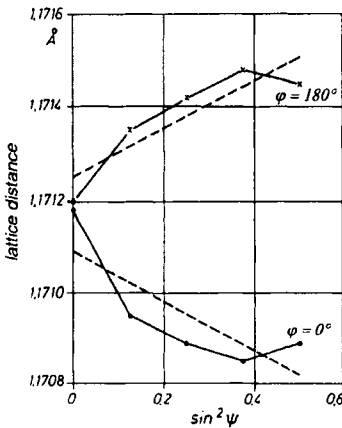


Figure 1. D-vs.- $\sin^2\psi$ distribution of a ground low-alloyed Cr-Ni-steel specimen before and after turning it in the diffractometer by 180° /3,4/.

Similar results with wear loaded samples /6/ were explained by Dölle and Hauk. They published the method to determine the total strain and stress tensor /7/. A different evaluation method was put forward by /8/. Many problems were studied in the following years: Is the ψ -splitting a consequence of a second phase in the material, are the σ_{ik} -components ($i \neq k$) of a RS-state macro- and micro-RS, how deep are surface regions affected by σ_{ik} -components after different mechanical surface treatments, what is the influence of elastic and plastic deformation on the ψ -splitting, are the shear components stable with dynamic loads? Most of these problems have been solved as the following explanations and examples will prove. However, a systematic investigation of the influence of plastic strain on the RS-profile and on the compensation of the micro-RS in multiphase materials is still needed.

Shear-RS and shear applied stresses are evidenced as ψ -splitting. Mechanical surface treatments with tangential forces result in ψ -splitting: It has been found after grinding, turning, roll-peening, inclined shot-peening and honing. Fig. 17 (section 2.072c) shows one example of the D-vs.- $\sin^2\psi$ dependence of a ground steel sample in the grinding and in the transverse direction. The ψ -splitting is pronounced in azimuth $\varphi = 0^\circ$ and none can be observed in azimuth $\varphi = 90^\circ$. These are the characteristics of grinding RS. The experimental studies of ground materials and the corresponding ψ -splitting were accompanied by theoretical aspects to the compensation and the separation of macro- and micro-RS /9/. Direct evidence of shear strains as the origin of a ψ -splitting was provided by Predecki and Barrett /10/ with an adhesive interphase of a single lap joint of Be and Al. Epoxy-glass-fiber laminates show under load ψ -splitting at the free edges. The embedded powder operates as the crystalline phase /11/.

2.142 Theoretical background

The fundamental complete connection between the experimentally determined $\langle \epsilon_{\varphi,\psi} \rangle$ and the ϵ_{ik} - or σ_{ik} -tensor is dealt with in section 2.072c and Fig. 16 explains the usually used method of Dölle and Hauk to evaluate these tensors. Here are some theoretical examples of D-vs.- $\sin^2\psi$ distributions for different azimuths of the specific stress tensors shown in the Fig. 2, /12/.

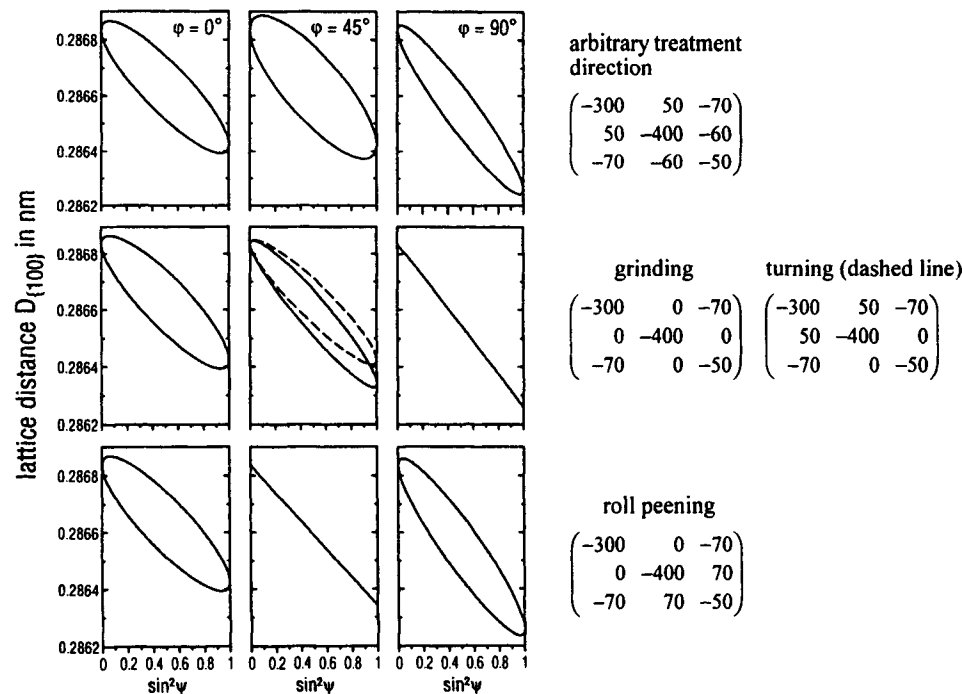


Figure 2. Calculated lattice-strain distributions using different stress tensors typical of the cited treatment; Cr-K α -radiation, {211}, $D_0 = 0.286650$ nm /12 supplemented/.

The basic formula simplifies for a grinding operation with the grinding direction in azimuth $\varphi = 0^\circ$ and $\varepsilon_{12} = \varepsilon_{23} = 0$ to

$$\varepsilon_{\varphi,\psi} = \varepsilon_{11} \cos^2 \varphi \sin^2 \psi + \varepsilon_{13} \cos \varphi \sin 2\psi + \varepsilon_{22} \sin^2 \varphi \sin^2 \psi + \varepsilon_{33} \cos^2 \varphi$$

Fig. 3 shows some experimental results, i.e. a symmetrical lattice-distance plot /13/. The principal strains and the tilt of the RS-state can easily be verified by the Mohr's circle, Fig. 4:

$$\Delta\psi = \frac{1}{2} \arctan \left(\frac{-2\varepsilon_{13}}{\varepsilon_{11} - \varepsilon_{33}} \right)$$

$$\varepsilon_1 = \frac{1}{2}(\varepsilon_{11} + \varepsilon_{33}) - r ; \quad \varepsilon_2 = \varepsilon_{22} ; \quad \varepsilon_3 = \frac{1}{2}(\varepsilon_{11} + \varepsilon_{33}) + r ; \quad r^2 = \frac{1}{4}(\varepsilon_{33} - \varepsilon_{11})^2 + \varepsilon_{13}^2$$

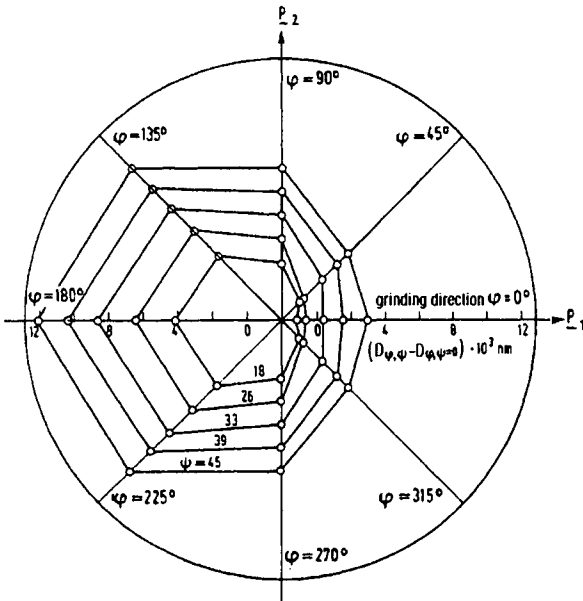


Figure 3. $D_{\varphi,\psi} - D_{\varphi,\psi=0}$ versus measuring direction (φ, ψ) , ground St52 steel, $\{211\}$, Cr-K α /13/.

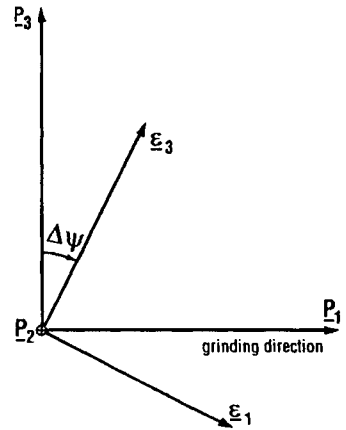


Figure 4. Orientation of the principal strains $\varepsilon_1, \varepsilon_2, \varepsilon_3$ after grinding with respect to the specimen system /13/.

In connection with the explanation of the RS in plastically uniaxially deformed specimens by a dislocation structure by Cullity /14/, Hanabusa and Fujiwara /15/ explained the σ_{13} -microstresses in the matrix and their compensation in the dislocations around the carbides of steels. Berveiller et al /16/ studied theoretically the ψ -splitting in the matrix of two-phase materials varying the volume fraction of the inclusion and the ratio of plastic global deformations $E_{13}^P / E_{33}^P = 0.3$. The second phase consists of spherical or oriented ellipsoidal inclusions. The ψ -splitting seems to be a heterogeneity property. The elastic calculations lead to overestimated stress values.

2.143 Experimental results

Influence of a second phase

Pure copper and pure silver materials showed after grinding no RS and no ψ -splitting, Fig. 5 and Fig. 6, /17/. Pure iron and the X5CrNi18-9 austenitic steel exhibit according to the used grinding conditions tension RS but no ψ -splitting, Fig. 7 and Fig. 8, /17/.

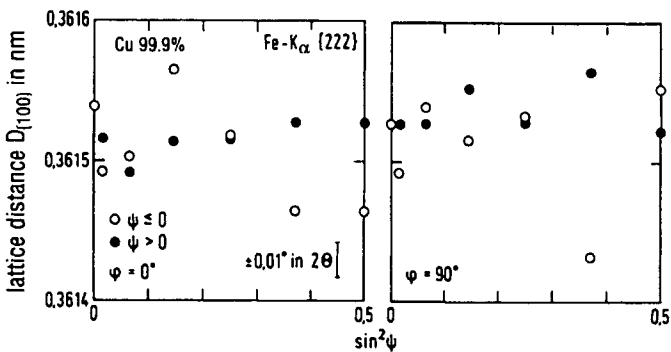


Figure 5. Lattice-strain distributions of Cu after grinding /17/.

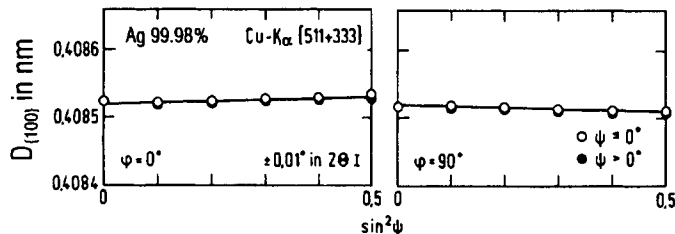


Figure 6. Lattice-strain distributions of Ag after grinding /17/.

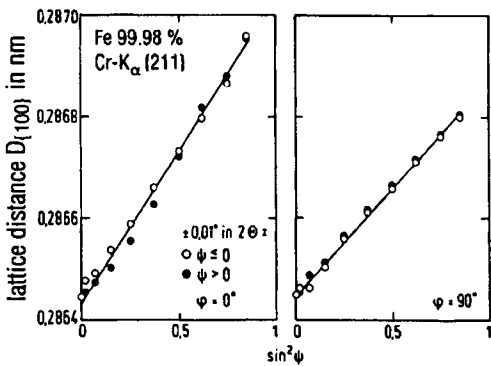


Figure 7. Lattice-strain distributions of Fe after grinding /17/.

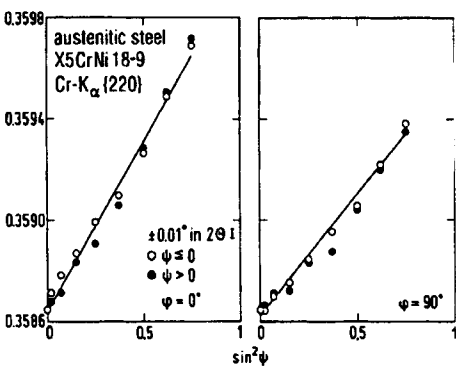


Figure 8. Lattice-strain distributions of austenitic steel after grinding /17/.

Multiphase materials even with only a small content of a second phase showed ψ -splitting after mechanical surface treatments with a tangential component. Japanese papers demonstrate very clearly the influence of the content of the second phase, Fig. 9 /18/ and Fig. 10 /15/.

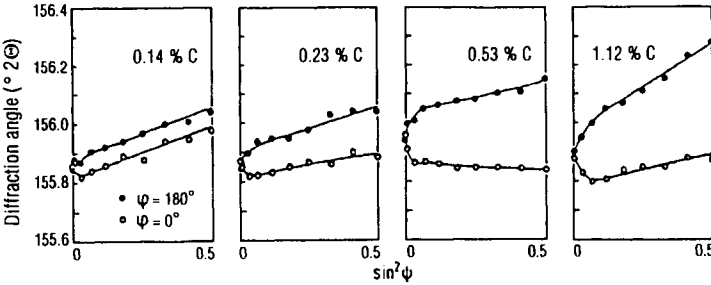


Figure 9. Influence of heterogeneity of steels (cementite content) on the ψ -splitting (shear stress) /18/.

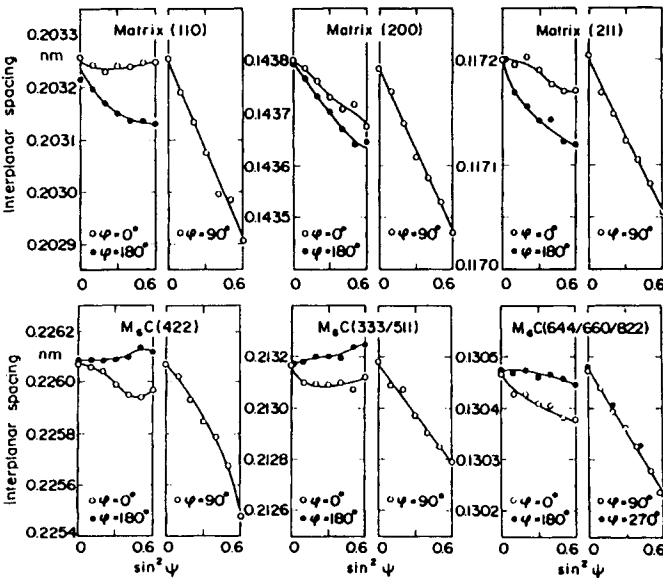


Figure 10. $\sin^2\psi$ diagrams obtained from different planes of the matrix phase (above) and the carbide phase, ground surface of a SKH2 steel specimen /15/.

A round-robin test was made to study the scatter of the data associated with different measurement techniques and with variations of the state of the materials /19/. The D-vs.- $\sin^2\psi$ dependences of ten specimens made of a ground 100Cr6 steel were determined by twelve institutions; three of them measured two specimens, Fig. 11. The average values of all measurements are: $\sigma_{11} = -460 \pm 40(11)$ MPa, $\sigma_{22} = -668 \pm 39(11)$ MPa, $\sigma_{13} = -58 \pm 17(4)$ MPa.

The errors of a single measurement are given and in brackets those of the average values. Three linear dependences of $\varphi = 90^\circ$ differ apparently from the majority of the results.

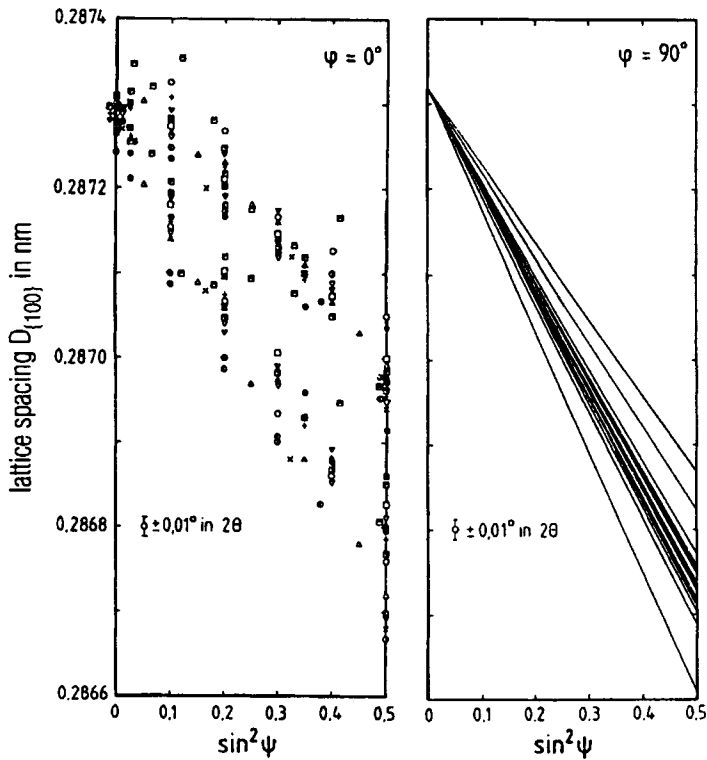


Figure 11. Result of a round-robin test on a ground 100Cr6 steel /19/.

2.143a Compensation of shear stresses

Ferrite and cementite phases were found to have shear stresses of opposite sign after grinding, Fig. 12, /20/.

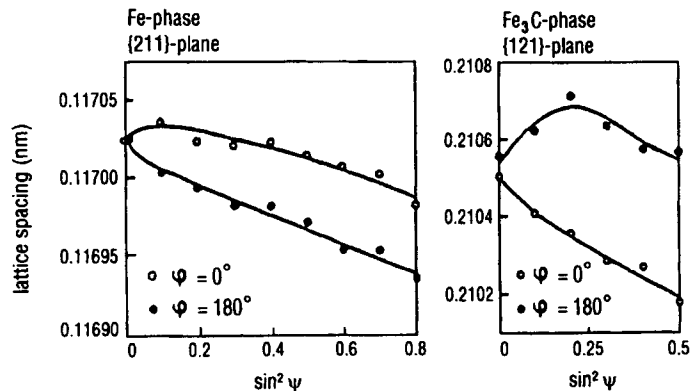


Figure 12. Ferrite and cementite phases exhibiting shear stresses of opposite signs in ground steel /20/.

Hanabusa and Fujiwara /15/ concluded that the compensation of the shear-RS is fulfilled when considering the volume fractions of the matrix, the second phase and the grain boundaries/dislocation walls.

But there are also examples known where compensation of the shear-RS in the phases of two-phase materials does not occur. For example the sintered materials Cu-Ag, Fig. 13 /17/ and Cu-Fe, Fig. 14 /17/. The effect of pores must be considered, too. However, unbalanced shear-RS have been also found in compact two-phase materials such as $(\alpha + \beta)$ -brass, ferrite-austenite and martensite steels after grinding, Fig. 15, 16 /21/.

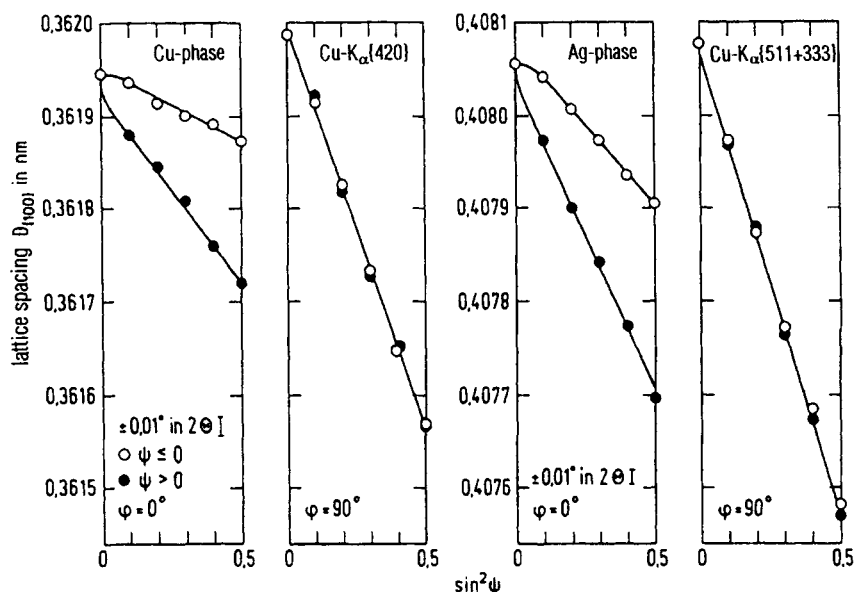


Figure 13. Lattice-strain distributions for the two phases of a ground sintered Cu-Ag 50/50 wt.% specimen in the grinding direction $\varphi = 0^\circ$ and in the transverse direction $\varphi = 90^\circ$ /17/.

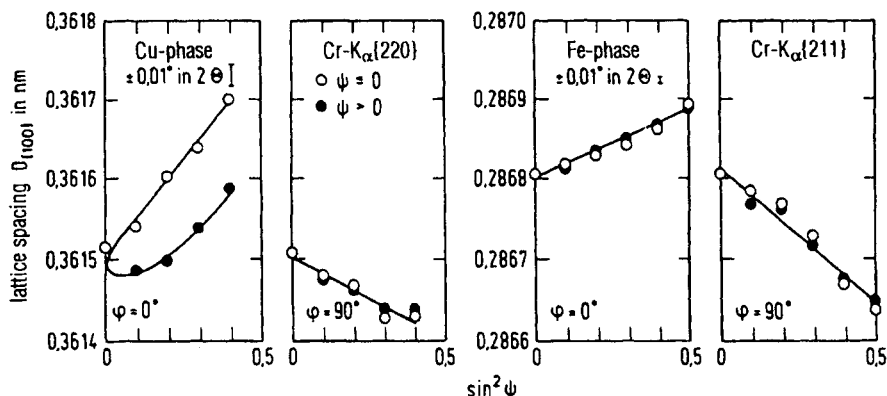


Figure 14. Lattice-strain distributions for the two phases of a ground sintered Cu-Fe 30/70 wt.% specimen, grinding direction $\varphi = 0^\circ$ /17/.

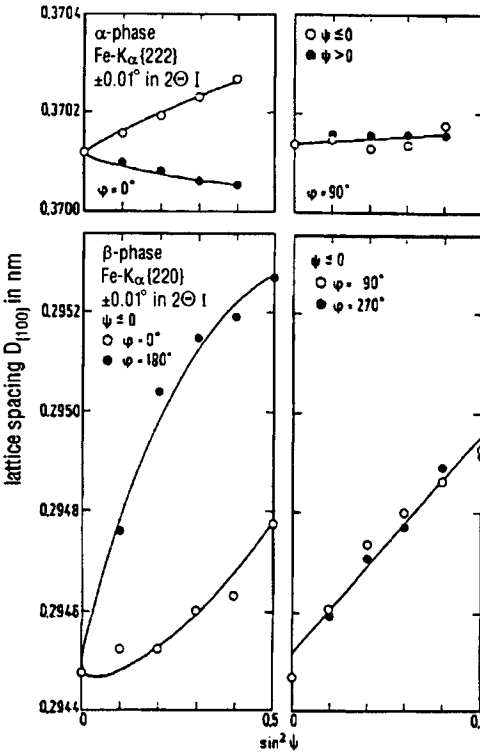


Figure 15. Lattice-strain distributions vs. $\sin^2\psi$ for a $(\alpha + \beta)$ -brass specimen after grinding; α -phase $\{222\}$ -reflections, β -phase $\{220\}$ -reflections $\{21\}$.

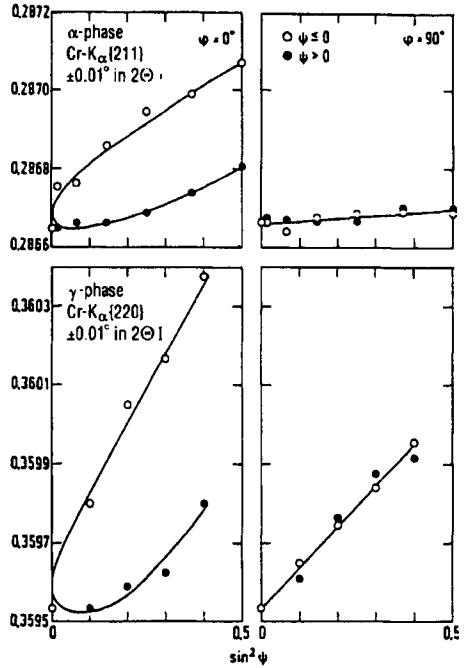


Figure 16. Lattice-strain distributions vs. $\sin^2\psi$ for a 100Cr6 specimen after grinding; martensite $\{211\}$ -reflections, retained austenite $\{220\}$ -reflections $\{21\}$.

Summarizing the following conclusions may be drawn. Single-phase materials show generally no ψ -splitting after surface treatment with a tangential force. Rather small amounts of a second phase with high-strength properties are sufficient for a ψ -splitting. The signs of the shear stress components are expected to be opposite but there exist examples where compensation does not occur; may be there exists a not-yet-known lattice-plane dependence.

2.143b Depth profiles of shear stresses

The profile of shear-RS can be evaluated by using different X-ray wavelengths or by etching surface layers and measuring the lattice strains on each new surface. Here are several examples. Fig. 17, $\{22,23\}$ shows the D -vs.- $\sin^2\psi$ dependence of a shot-peened steel sample where the operation was not done as usual but oblique at an angle of 60° to the normal of the sample. In the direction of the shot-peening there exists a ψ -splitting and none in the transverse direction. No ψ -splitting was observed when the same operation was done with a

sheet of an unalloyed iron. Fig. 18, /22,23/ demonstrates the depth profiles of the normal- and shear-RS. The sheet was shot-peened and also etched on both sides. The results demonstrate the small surface region with shear-RS compared with that with normal-RS. In another test series the depth profile was determined using different radiations, Fig. 19, /24/.

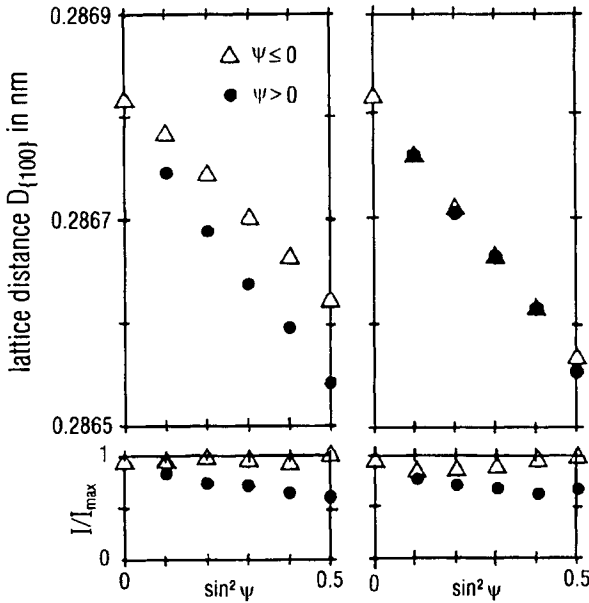


Figure 17. Lattice-strain and relative-intensity distributions of a shot-peened (60° incidence) steel specimen, $\{211\}$ -peak; left in the shot-peening direction, right transverse to the shot peening direction, etching depth $30 \mu\text{m}$ /22,23/.

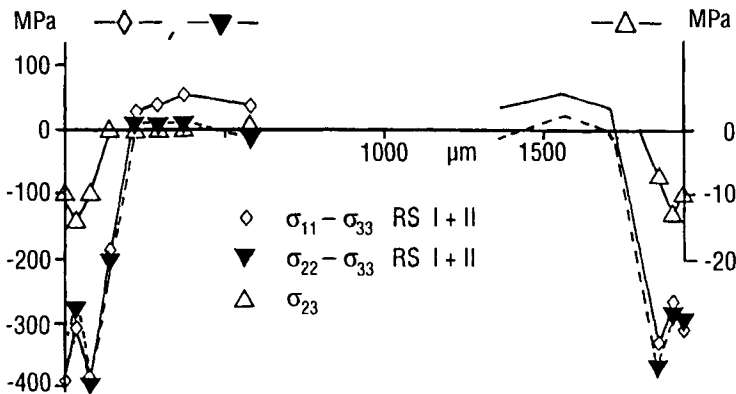


Figure 18. RS versus depth from the surface of an inclined shot-peened steel specimen /22,23/.

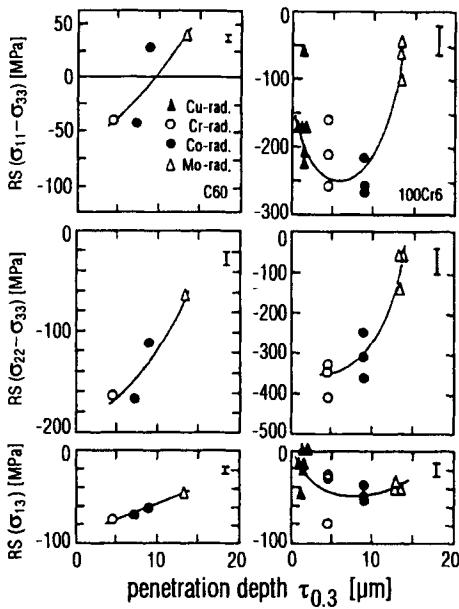


Figure 19. Residual stresses after grinding versus penetration depth of X-rays with different wavelengths /24/.

The normal- and the shear-RS-state after roll-peening depends among other parameters on the shape of the tool, cylindrical or elliptical. Fig. 20, /25/ shows the D-vs.- $\sin^2\psi$ dependences of measurements at different azimuths. There exist the shear-RS σ_{13} and σ_{23} of opposite sign when using an elliptical-shaped roll. Fig. 21 illustrates the RS depth profiles of steel specimens after rolling with different compressions and multi etchings /25/. Fig. 22 shows the D-vs.- $\sin^2\psi$ distributions of a roll-peened ferrite-austenite duplex-steel specimen /26/. Only σ_{23} shear-RS of a small amount can be observed. Fig. 23, demonstrates clearly the existence of a σ_{33} - and a σ_{13} -profile of a roll-peened steel sample /27/. The results were gained by a multi-etching process.

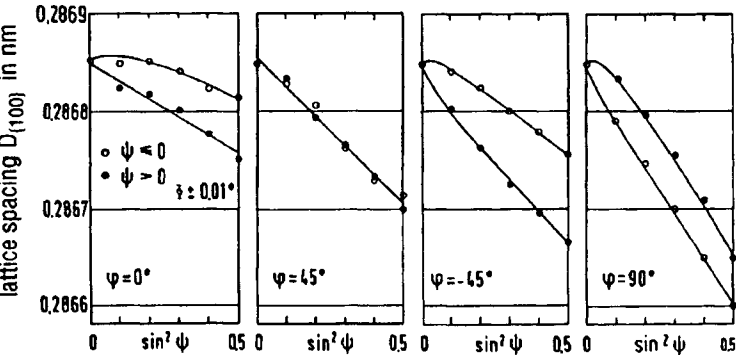


Figure 20. Lattice-strain distributions vs. $\sin^2\psi$ of a roll-peened surface; azimuths $0^\circ, \pm 45^\circ, 90^\circ$; Cr-K α , {211} /25/.

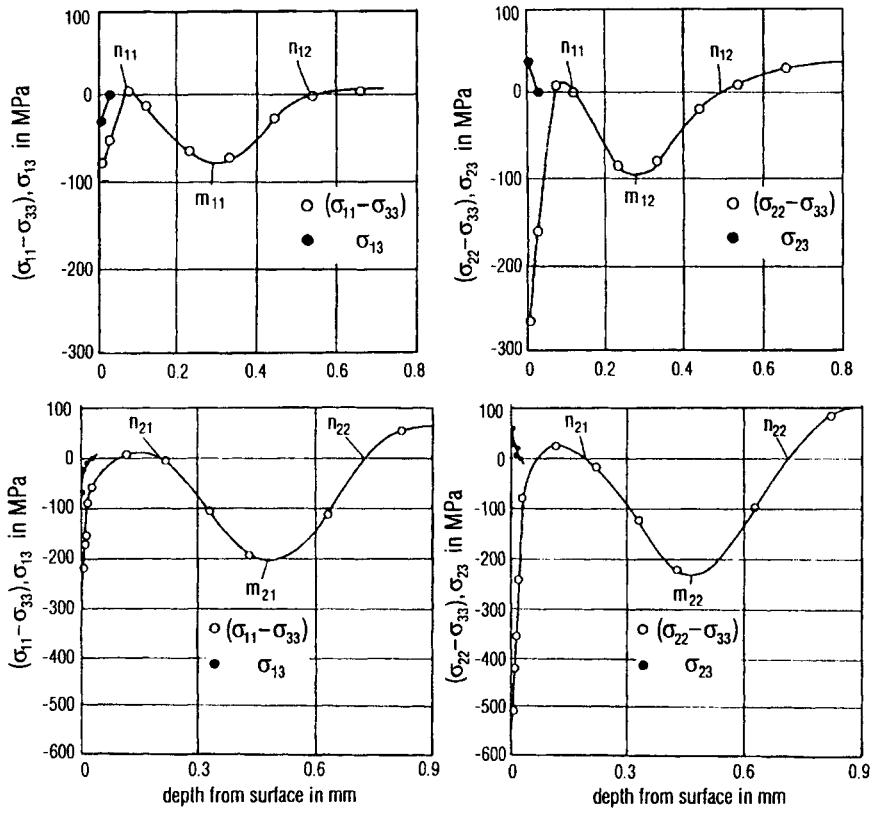


Figure 21. RS depth profiles of roll-peened steel specimens, compression 2000 MPa (upper figures) and 3000 MPa (lower figures) /25/.

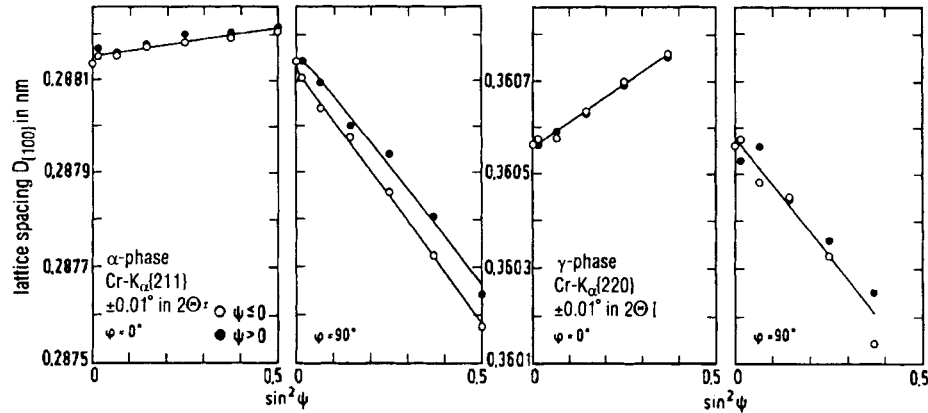


Figure 22. Lattice-strain distributions in the two phases of a duplex steel after roll-peening, rolling direction $\varphi = 0^\circ$ /26/.

Summarizing these results of determining shear-RS profiles of different surface-treated samples, the X-ray method proved to be the appropriate one, but the assessment of the value and depth of the shear-RS profiles should be evaluated by dynamic tests.

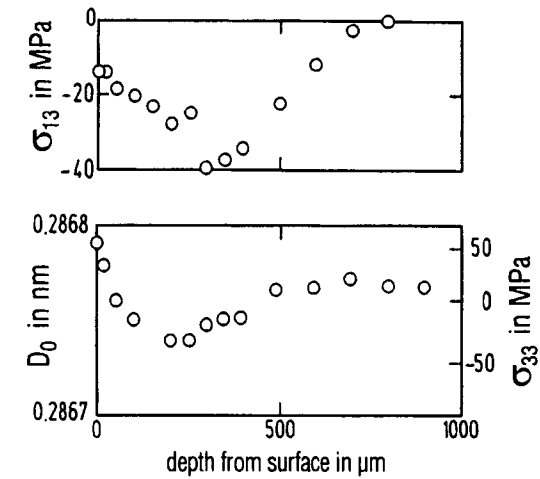


Figure 23. Shear- and normal-components versus the depth from the surface of a roll-peened flat specimen of the 37CrS4 steel; Cr-K α radiation, many etching steps /27/.

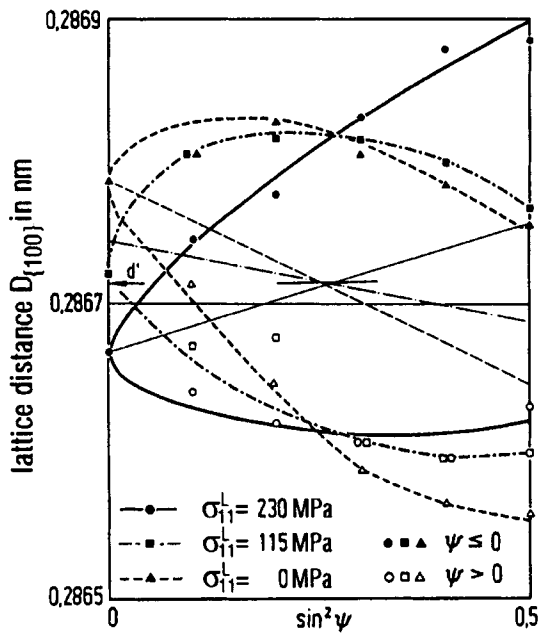


Figure 24. Lattice-strain distributions of a ground 100Cr6 steel specimen with superposed tensile load stress /28/.

2.143c Shear-RS state with additional elastic or plastic strain

As the basic formula reveals, elastic strain is an additive parameter. XEC determination on a specimen with shear-RS can be done neglecting the ψ -splitting, Fig. 24, /28/. Plastic strain applied to a specimen with shear-RS showed in many cases no more ψ -splitting. The diminishing of the ψ -splitting by plastic strain is shown in Fig. 25, /29/. Further studies of this problem are necessary.

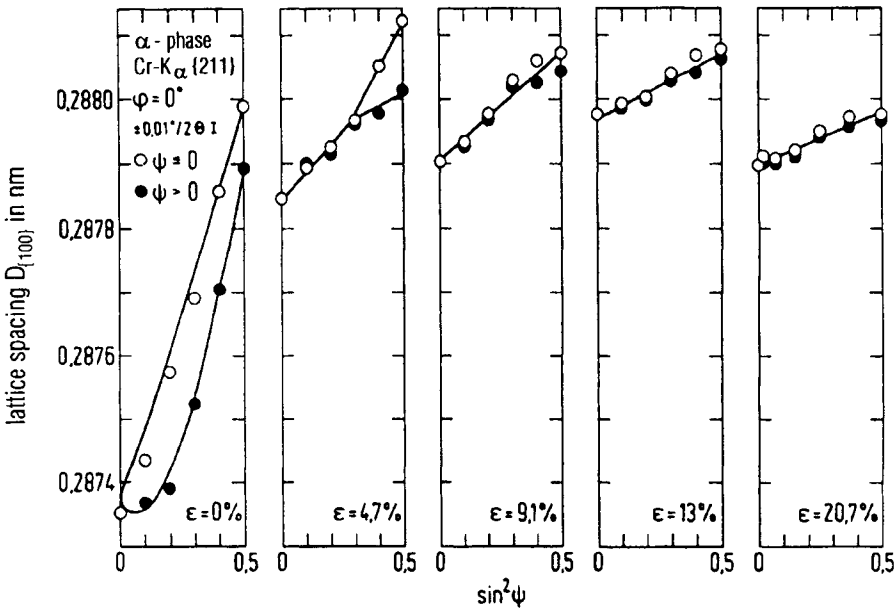


Figure 25. Lattice-strain distributions of the α -phase of a ground duplex-steel specimen superposed with plastic strain /29/.

2.144 Recommendations

All D-vs.- $\sin^2\psi$ distributions should be measured at positive and negative angles ψ or measurements should be made at azimuths $\varphi = 0^\circ$ and $\varphi = 180^\circ$.

If there are pronounced differences between $D_{\varphi, \psi > 0}$ and $D_{\varphi, \psi < 0}$, the reality of the ψ -splitting should be checked by rotating the specimen 180° and retesting. Should the ψ -splitting not be confirmed the alignment of the diffractometer should be improved.

If ψ -splitting is noticed at $\varphi = 0^\circ$, additional measurements at $\varphi = \pm 45^\circ$ and $\varphi = 90^\circ$ should be undertaken to evaluate the total stress tensor.

Taking into account the ψ -splitting of the lattice-strain components the evaluation of the stress tensor should follow the procedure of Dölle and Hauk /7/. If the shear components can be neglected the mean values of the lattice strains measured at $\psi < 0^\circ$ and $\psi > 0^\circ$ should be taken to evaluate the normal-stress components.

For multiphase materials the ψ -splitting of as many as possible lattice planes of all phases should be determined and the compensation of the shear components should be checked.

The origin of the ψ -splitting, the compensation of shear-RS in multiphase materials, the alteration of ψ -splitting after plastic deformations and the assessment of shear components in surface regions by tests using cyclic loads are problems that should be handled theoretically.

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2.15 The evaluation of strain-, stress- and D_0 -profiles or gradients with the depth from the surface

2.151 Historical review

Knowledge of the distribution of strain and stress over the cross section of plates, bars, tubes, components, especially after heat and mechanical treatments, has been a task from the beginning of residual-stress determination. Turning off, boring out, sectioning, all these mechanical methods were used to release stresses and to measure the ensuing deformations. Algorithms were developed to evaluate the original stress distribution. Reference will be made to books in which the experimental and theoretical methods are summarized, chapter 1.1. Early, the fact was discussed about the difference between the stress at the surface and the value determined by X-rays if a gradient is present /1,2/. Also the triaxiality of the RS-state below the very surface after plastic deformations and the modification of the basic equations for the biaxial stress state at the surface were considered /3,1/.

Since the X-ray method of RSA probes surface-regions the etching of thin layers was introduced to evaluate RS-profiles versus the depth from the surface in the late 1930s. The etching method was often used to decide whether there is a macro- or a micro-RS state. Also the sectioning of relatively thick metallic products was used to get knowledge of the RS-state therein by employing X-ray methods on the newly created surfaces and deconvoluting the original RS-state from these data. Lattice strain might be measured on one side of the cutout plates and thinning of the plate will be done from the other side by grinding and etching. The fundamental formula for evaluating the stress gradient in the thickness direction z from measurements in the penetration field τ was introduced by /4,5/. The authors evaluated steep stress gradients in ground steel specimens.

Systematic theoretical studies of the influence of stress gradients on the D -vs.- $\sin^2\psi$ dependences /6,7/ showed the optimal way to solve gradient problems. Starts were made to evaluate RS-profiles by power series /7,8,9/.

Alloying, heat treatment, cladding of surface layers are associated with gradients of micro-structure and gradients of the strain-stress-free lattice distance. They may give rise to apparent stresses /10/; but the exact, real D_0 -value for each point of measurement is necessary to establish the total stress tensor. Finally the modeling of stress- and D_0 -profiles must be used to explain experimentally determined D -vs.- $\sin^2\psi$ complex dependences /11/.

Further impacts were given by the studies with X-rays on surface treated materials, especially by grinding, on thin film substrate composites and on ceramic materials. Progress was achieved stepwise by using different wavelengths, by making measurements on different peaks with very high values of ψ with low incident angle, with synchrotron radiation and from tests with energy dispersive examination.

The application of neutron diffraction for stress determination opens the possibility to investigate metallic materials up to 20 mm thickness. The ultrasonic techniques which are also nondestructive, are able to test surface zones up to 3 mm thickness. Today's research programs studying the RS-state of near-surface regions are using different testing methods. In as far as the X-ray technique is concerned three kinds of methods are used to investigate the depth dependence of RS. They are based on the variation of the observed stresses with the penetration depth of X-rays with different wavelengths, on non-linearities of D -vs.- $\sin^2\psi$ distributions, especially for very high ψ -tilts, and on the asymmetry of reflection lines in case of the presence of high stress gradients. The choice of the method to evaluate strain-stress dis-

tributions, profiles, steep gradients as function of the depth from the surface is related to the extension of the profile itself. Mechanical, ultrasonic, micromagnetic and the diffraction methods with neutron- and with X-rays are able to solve the specific problem.

Recommendations are given how to find the appropriate method and how to use it to get the correct answer.

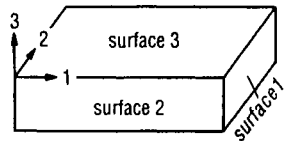
2.152 The influence of multiaxial RS-state gradients and of D_0 -gradients on lattice strain data

2.152a Existing and measurable stress components

In the following table the LS- and RS-components are listed that may exist in a component and that will be evaluated by X-rays and/or by neutron rays [12]. This is of importance for the choice of the method and the direction of the measurement.

Table 1. Load- and residual-stress components measured with X- and neutron(N)-rays for different triaxial load and residual-stress states in the interior and at the surfaces of a flat specimen with 3-direction as the thickness direction. $\langle \rangle$ measured weighted averaged value of the sum of the stresses over the penetration depth /12/ $\langle \sigma^{III} \rangle$ not considered.

stress state	bulk, surface	rays	evaluated LS, RS I, RS II
LS + RS $\sigma_3^I = 0$	bulk	N	$\langle \sigma_1^I + \sigma_1^I + \sigma_1^{II} \rangle - \langle \sigma_3^I + \sigma_3^{II} \rangle$ $\langle \sigma_2^I + \sigma_2^I + \sigma_2^{II} \rangle - \langle \sigma_3^I + \sigma_3^{II} \rangle$
	surface 3	X	$\langle \sigma_1^I + \sigma_1^I + \sigma_1^{II} \rangle - \langle \sigma_3^I + \sigma_3^{II} \rangle$ $\langle \sigma_2^I + \sigma_2^I + \sigma_2^{II} \rangle - \langle \sigma_3^I + \sigma_3^{II} \rangle$
RS $\sigma_3^I = 0$ σ_1 -gradient, σ_2 -gradient in 3-direction	bulk	N	$\langle \sigma_1^I + \sigma_1^{II} \rangle - \langle \sigma_3^{II} \rangle$ $\langle \sigma_2^I + \sigma_2^{II} \rangle - \langle \sigma_3^{II} \rangle$
	surface 3	X	$\langle \sigma_1^I + \sigma_1^{II} \rangle - \langle \sigma_3^{II} \rangle$ $\langle \sigma_2^I + \sigma_2^{II} \rangle - \langle \sigma_3^{II} \rangle$
	surface 1	X	$\langle \sigma_2^I + \sigma_2^{II} \rangle - \langle \sigma_1^{II} \rangle$ $\langle \sigma_3^{II} \rangle - \langle \sigma_1^{II} \rangle$
RS σ_1 -gradient, σ_2 -gradient in 1-, 2-direction	bulk	N	$\langle \sigma_1^I + \sigma_1^{II} \rangle - \langle \sigma_3^I + \sigma_3^{II} \rangle$ $\langle \sigma_2^I + \sigma_2^{II} \rangle - \langle \sigma_3^I + \sigma_3^{II} \rangle$
	surface 3	X	$\langle \sigma_1^I + \sigma_1^{II} \rangle - \langle \sigma_3^{II} \rangle$ $\langle \sigma_2^I + \sigma_2^{II} \rangle - \langle \sigma_3^{II} \rangle$
	surface 1	X	$\langle \sigma_2^I + \sigma_2^{II} \rangle - \langle \sigma_1^{II} \rangle$ $\langle \sigma_3^I + \sigma_3^{II} \rangle - \langle \sigma_1^{II} \rangle$



2.152b Basic formulae, stress gradient

If there are no gradients at all, that means a homogeneous stress state in the surface region of a homogeneous microstructure with a constant D_0 -value the lattice strain D-vs.- $\sin^2\psi$ is represented by a linear dependence. In case of the presence of shear stresses the average D-values for $\psi > 0^\circ$ and $\psi < 0^\circ$ should be taken. The question was how and to what amount do changes of the homogeneous state produce alterations of the D-vs.- $\sin^2\psi$ linearities.

The systematic treatment of the influence of stress gradients on the lattice strain D-vs.- $\sin^2\psi$ was published by /6,7/. Other papers /13,14,15/ also dealt with this subject.

The basic formula with V the illuminated volume of the specimen and μ the attenuation factor is

$$\varepsilon_{\varphi,\psi} = \frac{\iiint_V \varepsilon_{\varphi,\psi}(x,y,z) \exp\{-\mu z\} dx dy dz}{\iiint_V \exp\{-\mu z\} dx dy dz} \quad (1)$$

Further, the following propositions are made:

- the material is homogeneous, mechanically isotropic, and an elastic surface anisotropy can be neglected.
- there are only RS, they are dependent only on the distance from the surface z , all forces and moments are in equilibrium.
- at the surface $z = 0$: $\sigma_{13} = \sigma_{23} = \sigma_{33} = 0$.
- the lattice strain will be measured versus the penetration depth τ .
- the stress profile versus the penetration depth τ should be converted into the stress profile versus the distance from the surface z .

Here are the basic formulae, the formulae for the penetration depth τ for Ω - and Ψ -diffractometers were cited previously. By means of X- or neutron-rays an average strain is determined depending on the wavelength, the direction of the rays and the attenuation factor of the material

$$\langle \sigma_{ij}(\tau) \rangle = \frac{\int_0^P \sigma_{ij}(z) \exp\left\{-\frac{z}{\tau}\right\} dz}{\int_0^P \exp\left\{-\frac{z}{\tau}\right\} dz} \quad (2)$$

with the usual equation

$$\begin{aligned} \langle \varepsilon_{\varphi,\psi} \rangle = & \frac{1}{2} s_2 \left[\langle \sigma_{11}(\tau) \rangle \sin^2 \psi \cos^2 \varphi + \langle \sigma_{12}(\tau) \rangle \sin^2 \psi \sin 2\varphi + \langle \sigma_{13}(\tau) \rangle \sin 2\psi \cos \varphi \right. \\ & \left. + \langle \sigma_{22}(\tau) \rangle \sin^2 \psi \sin^2 \varphi + \langle \sigma_{23}(\tau) \rangle \sin 2\psi \sin \varphi + \langle \sigma_{33}(\tau) \rangle \cos^2 \psi \right] \\ & + s_1 \left[\langle \sigma_{11}(\tau) \rangle + \langle \sigma_{22}(\tau) \rangle + \langle \sigma_{33}(\tau) \rangle \right] \end{aligned} \quad (3)$$

The following examples of $\langle \varepsilon_{\varphi,\psi} \rangle$, $D_{\varphi,\psi}$ and $\frac{\langle \sigma_{ij} \rangle}{\sigma_{ij \max}}$ versus $\sin^2\psi$ are calculated for Fe and partially also for Al, a Ψ -diffractometer and the following stress profiles versus depth z .

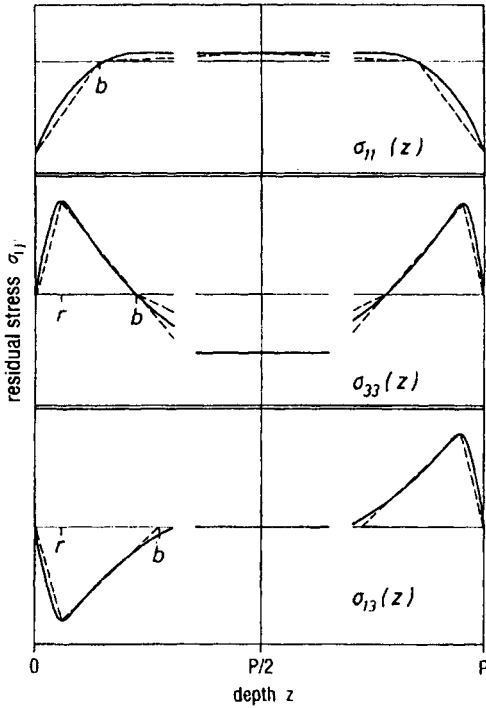


Figure 1. Assumed residual stress profiles of σ_{11} , σ_{33} and σ_{13} /6/.

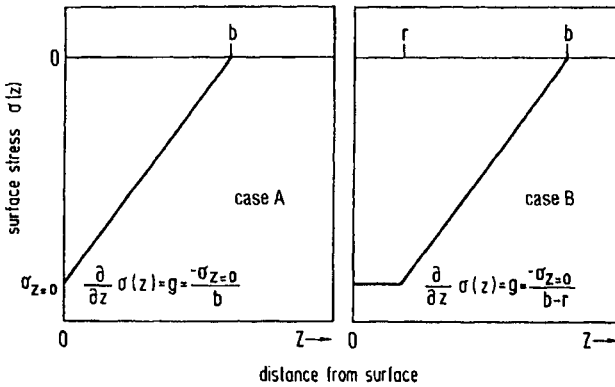


Figure 2. Assumed stress profiles, parameters: $\sigma(z = 0)$, r , g /7/.

The effects of a stress gradient are a curved D-vs.- $\sin^2\psi$ line and a stress value evaluated by X-ray which is smaller than the real one at the surface, the difference depending on the stress gradient. In the following figures examples of theoretical dependences $\langle \epsilon'_{33} \rangle$ or D-versus- $\sin^2\psi$ and stresses relative to $|\sigma_{11}(z = 0)|$ are plotted versus $\sin^2\psi$; details are described in the following sections /6,7/.

In the papers /6/, /7/ and also in /16/ several typical cases of stress gradients and the respective D-vs.-sin²ψ distributions are discussed; here are some examples. The captions and the descriptions of the figures should be sufficient for their understanding.

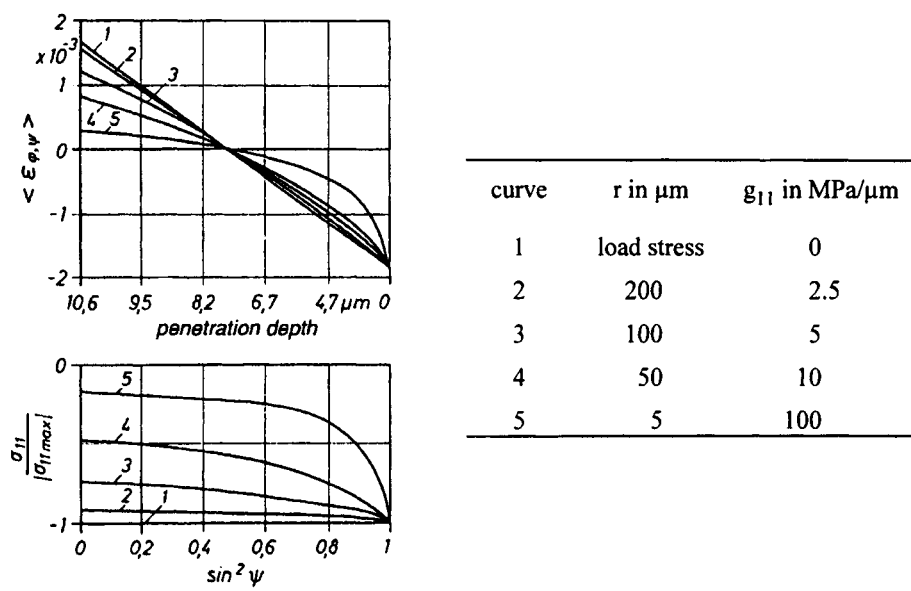


Figure 3. Lattice strain versus penetration depth and normalized-RS versus sin²ψ of a rotational stress state in Fe. The calculation used the following parameters σ₁₁(z) = σ₂₂(z), σ₁₁(z = 0) = -500 MPa, σ₃₃ = σ₁₃ = 0 and those in the table at the right-hand side (r,g see Figure 2) /6/.

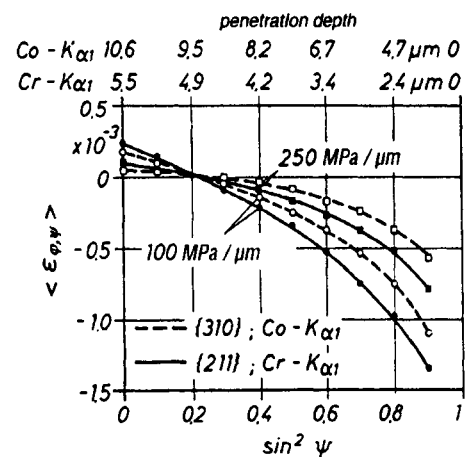
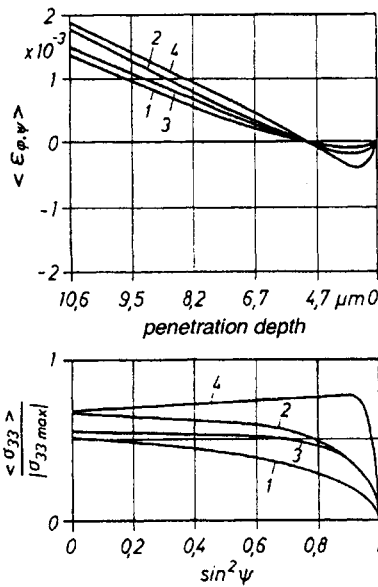
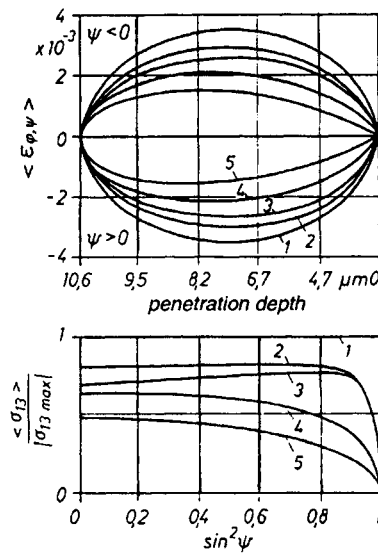


Figure 4. Lattice strain versus sin²ψ for two gradients and two radiations /6/.



curve	r in μm	b in μm	g_{33} in MPa/ μm for	
			$0 \leq z < r$	$r \leq z < b$
1	20	220	25	-2.5
2	10	210	50	-2.5
3	10	60	50	-10
4	2	52	250	-10

Figure 5. Lattice strain versus penetration depth and normalized-RS $\langle \sigma_{33} \rangle$ versus $\sin^2 \psi$ calculated using the following parameters: Fe, Ψ -diffractometer, max. stress $\sigma_{33}(z=0) = -500$ MPa, $\sigma_{11} = \sigma_{13} = 0$ and those in the table at the right-hand side (r,g,b see Figure 2) /6/.



curve	r in μm	b in μm	g_{13} in MPa/ μm for	
			$0 \leq z < r$	$r \leq z < b$
1	load stress		0	0
2	2	102	-250	5
3	2	52	-250	10
4	20	220	-25	2.5
5	20	70	-25	10

Figure 6. Lattice strain versus penetration depth and normalized-RS $\langle \sigma_{13} \rangle$ versus $\sin^2 \psi$ calculated using the following parameters: Fe, Ψ -diffractometer, max. stress $\sigma_{13}(z=0) = -500$ MPa, $\sigma_{11} = \sigma_{33} = 0$ and those in the table at the right-hand side /6/.

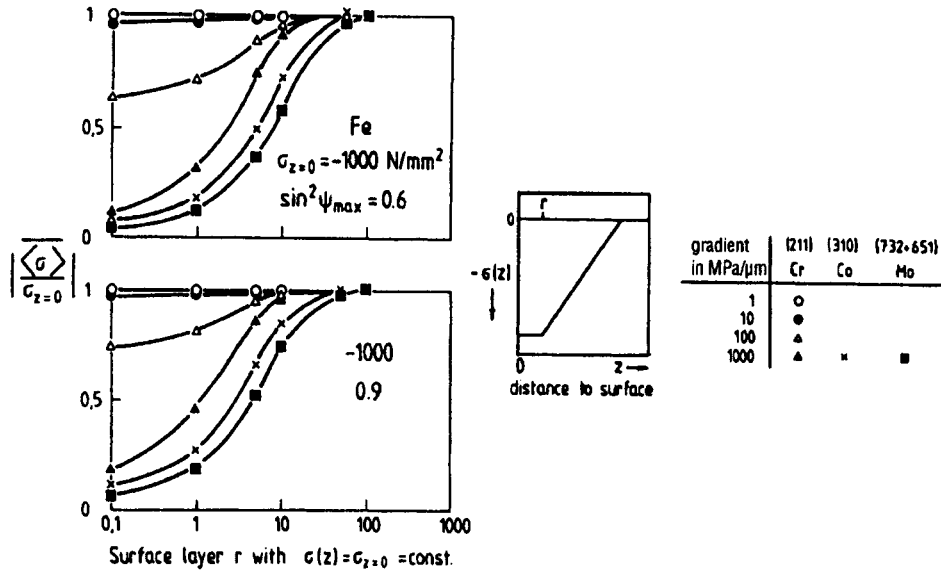


Figure 7. Normalized stress versus the depth r of a surface layer with $\sigma(z) = \sigma(z = 0) = \text{const.}$ Calculation for Fe, Ψ -diffractometer, different X-ray radiations and different gradients /7/.

2.152c Example of calculating D vs. $\sin^2 \psi$

In the following an example of a calculated D -vs.- $\sin^2 \psi$ distribution for a special stress profile will be explained, see Fig. 8. The suppositions are $\sigma_{22} = \sigma_{33} = 0$, $\sigma_{12} = \sigma_{13} = \sigma_{23} = 0$, $\sigma_{11}(z = 0) = -500 \text{ MPa}$, $r = 1 \mu\text{m}$, $g = 100 \text{ MPa}/\mu\text{m}$, $\text{Fe}\{211\}$, $D_0 = 0.286648 \text{ nm}$, $\text{Cr-K}\alpha$, Ψ -diffractometer, specimen thickness $P \gg \tau$ (see Fig. 2).

$$\langle \epsilon'_{33} \rangle = \left(\frac{1}{2} s_2 \sin^2 \psi + s_1 \right) \cdot \langle \sigma_{11}(\tau) \rangle \tag{4}$$

$$D \gg \tau$$

$$\begin{aligned} \langle \sigma_{11}(\tau) \rangle &= \frac{1}{\tau} \left(\int_0^r \sigma_{11}(z) \exp\left\{-\frac{z}{\tau}\right\} dz + \int_r^b \sigma_{11}(z) \exp\left\{-\frac{z}{\tau}\right\} dz \right) \\ &= \sigma(z = 0) + \tau g \left(\exp\left\{-\frac{r}{\tau}\right\} - \exp\left\{-\frac{b}{\tau}\right\} \right) \end{aligned} \tag{5}$$

$$\begin{aligned} D_{0^\circ, \psi=45^\circ} &= D_0 + D_0 \left(0.5 \cdot \frac{1}{2} s_2 + s_1 \right) \left[\sigma(z = 0) + 3.82 \cdot 10^2 \cdot \left(\exp\left\{-\frac{1}{3.82}\right\} - \exp\left\{-\frac{6}{3.82}\right\} \right) \right] \\ &= 0.286515 \text{ nm} \end{aligned}$$

This value can be verified in Fig. 8 left. The right hand side of Fig. 8 shows the corresponding D -vs.- $\sin^2 \psi$ distributions for an Al material /7/.

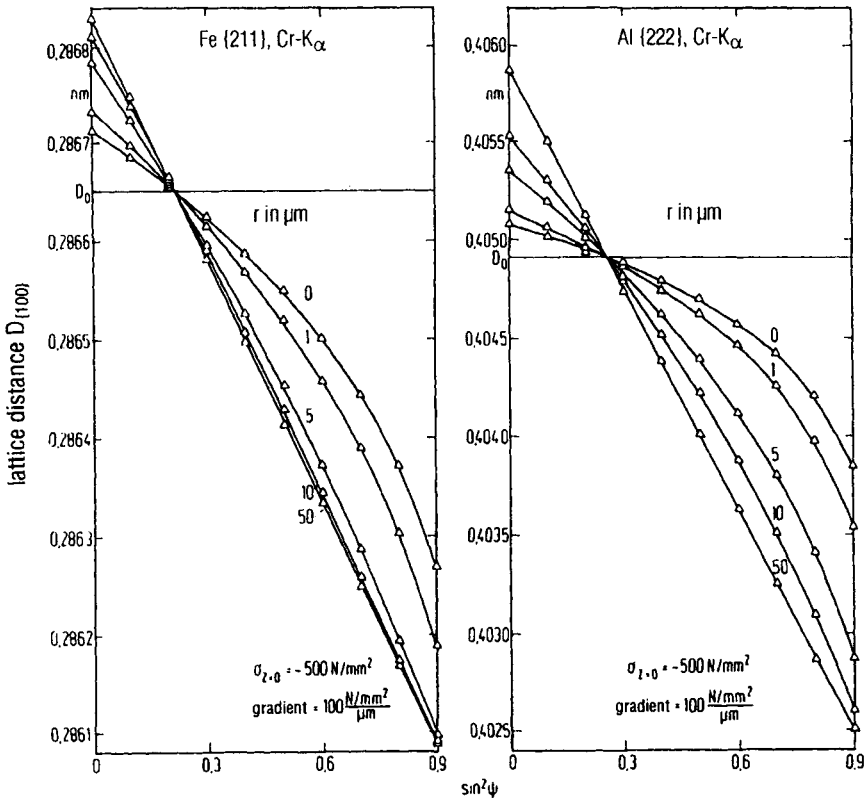


Figure 8. D-vs.- $\sin^2\psi$ distributions for the stress gradient with parameters are given in the figure, left Fe, right Al //.

2.152d Very high stress gradients, D-vs.- $\sin^2\psi$ distribution and asymmetry of peaks

After grinding of surfaces of ceramic materials, very high RS-gradients are observed. The limits of the measuring techniques had to be extended to find answers to fundamental problems. D-vs.- $\sin^2\psi$ distributions measured with Mo- or even Cu-radiation show no slope, therefore the penetration depth had to be reduced further either by using a longer wavelength or by extending the measurements up to very high angles ψ , additionally tilting the specimen in the ω -direction. To explain the different influences on the D-vs.- $\sin^2\psi$ dependences and to point out the necessary measuring accuracy the following examples are theoretically elaborated. It is recommended to reflect these or similar thoughts to the problems and to perform calculations before starting the experiments.

The propositions are: tetragonal ZrO_2 , rotational biaxial RS with a linear gradient or with stepwise gradient, use of an additional specimen tilt ω' in a Ψ -diffractometer. The strain $\epsilon_{\phi,\psi,\omega'} = \epsilon'_{33}$ was calculated using the formula and the data that are shown in the captions of the figures.

Fig. 9 shows the penetration depths of different radiations versus $\sin^2\psi$ for the $\{222\}$ peak of tetragonal ZrO_2 . Fig. 10 reveals the influence of the stress gradient. The differences between the curves will be detected more easily when the measured D-data are plotted versus ψ instead of $\sin^2\psi$ in the higher values. As the Fig. 10 demonstrates very clearly, the D-vs.- $\sin^2\psi$ dependences show practically no stress in the lower $\sin^2\psi$ range but exhibit strong curvatures in the higher range.

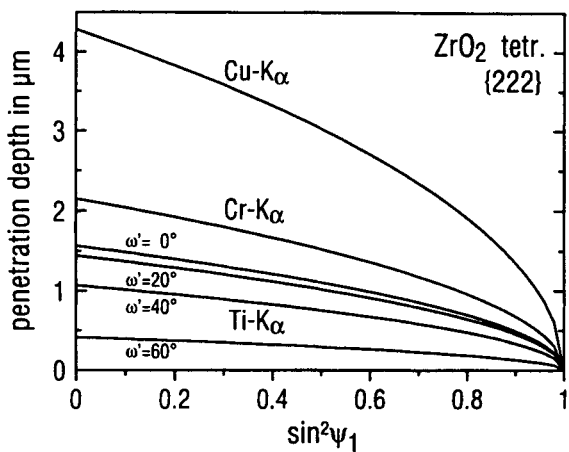


Figure 9. Penetration depth versus $\sin^2\psi_1$, tetragonal ZrO_2 , Ψ -diffractometer, ω' tilt, $\{222\}$, three radiations.

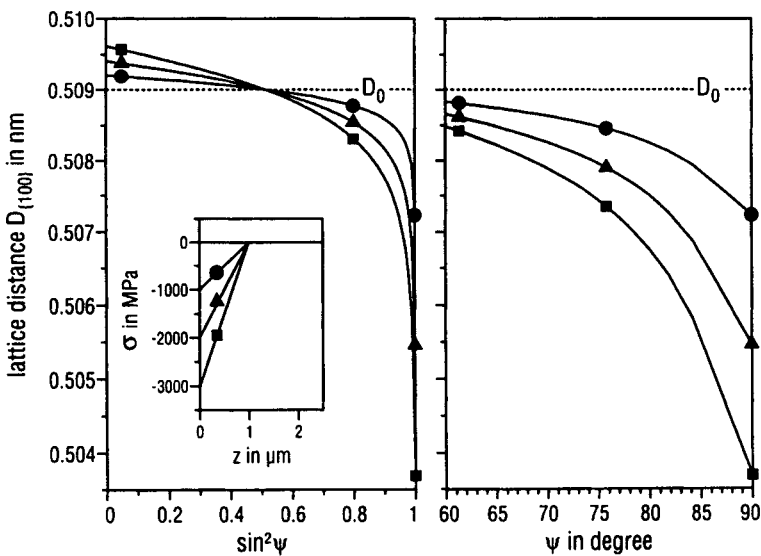


Figure 10. Lattice distance versus $\sin^2\psi$ (left), versus ψ (right) of tetragonal ZrO_2 in a rotational symmetrical stress state with gradients in the surface layer, Ψ -diffractometer, $\text{Cu-K}\alpha$, $\{222\}$, $2\Theta = 62.82^\circ$.

Fig. 11 demonstrates the influence of the kind of the gradient, linear or stepwise. The differences in the D -vs.- $\sin^2\psi$ dependences are relatively small as well as for that obtained by using $\text{Cu-K}\alpha$ or $\text{Ti-K}\alpha$ radiation, but tilting the specimen 60° offers the possibility to a better distinction of the two kinds of gradients. This effect is of certain importance; it offers discussion of the real form of the gradient.

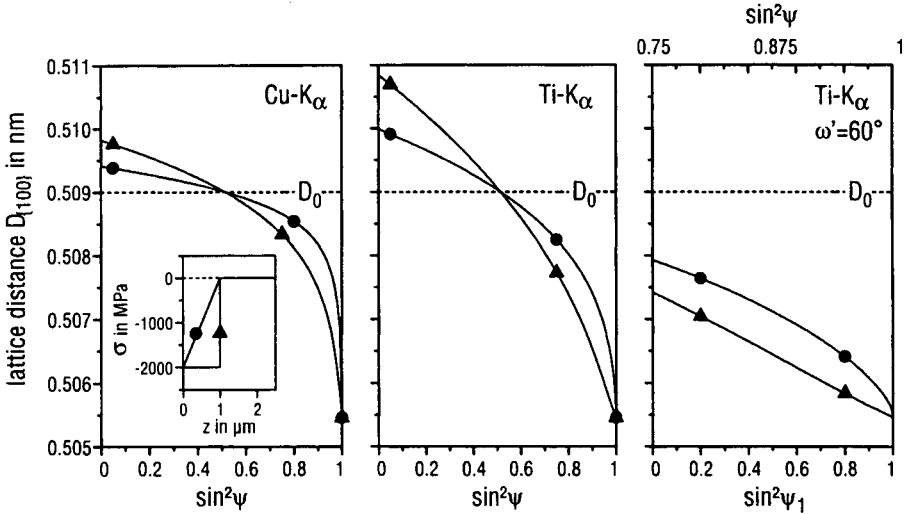


Figure 11. Lattice distance versus $\sin^2\psi$, tetragonal ZrO_2 with two kinds of surface gradient of rotational-symmetrical stress state, Ψ -diffractometer, right: additional tilt $\omega' = 60^\circ$, $\text{Cu-K}\alpha$, $\{222\}$.

It was demonstrated by [17] that stress values may have large uncertainties as the consequence of using different lower thresholds assessing the position of the distorted interference line which is caused by large stress- or texture-gradients. These results are based on strain-, stress-evaluation using the center of gravity method with various lower thresholds for the $\text{K}\alpha$ -doublet.

In the following it will be shown by means of two theoretical stress profiles with very high gradients what kind of distortions are present in the doublet, the symmetrized and the separated $\text{K}\alpha_1$ -interferences [18]. Recommendations will be given about how to correctly evaluate the position of the $\text{K}\alpha_1$ -peak.

Fig. 12 shows the two theoretical uniaxial stress profiles, the second one corresponding to the first one after a $2\text{ }\mu\text{m}$ thick layer was removed, see 2.072k. The data used to calculate the intensity distributions are the following: $\text{Fe}\{211\}$ -interference, $(\text{K}\alpha_1 + \text{K}\alpha_2)$ doublet symmetrized, $\text{K}\alpha_1$ -separated line, $\text{Cr-K}\alpha$ radiation, the lattice constant 0.28665 nm , $\mu = 905\text{ cm}^{-1}$, $\text{FWHM} = 1^\circ$ in 2Θ , Gaussian distribution, $s_1 = -1.25$, $\frac{1}{2}s_2 = 5.76 \cdot 10^{-6}\text{ MPa}^{-1}$. The intensity versus 2Θ at the surface $z = 0$ of the stress profiles holds:

$$I_{z=0}(2\Theta) = I_{z=0-S}(2\Theta) \left(1 - \exp\left\{-\frac{S}{\tau}\right\} \right) + I_{z=S-\infty}(2\Theta) \exp\left\{-\frac{S}{\tau}\right\} \quad (6)$$

$$\frac{S}{\tau} = \frac{2S\mu}{\sin\Theta \cos\psi} \quad S = 3 \text{ or } 1\text{ }\mu\text{m} \quad (7)$$

Fig. 13 demonstrates the calculated K_{α} -doublets, left $\sin^2\psi = 0.7$, right $\sin^2\psi = 0.9$, upper part 3 μm thick stress zone, lower part the 1 μm one. The distortion of the doublet is clearly seen. The symmetrized doublet and the separated $K_{\alpha 1}$ -line are distorted. Fig. 14 shows examples for the 1 μm thick stress zone at $\sin^2\psi = 0.7$ and $\sin^2\psi = 0.9$. Evaluating such distorted lines in the usual manner using the center-of-gravity method and variable lower threshold between 0.55 and 0.80 of the maximum intensity the following D_{τ} -vs.- $\sin^2\psi$ distributions will result, Fig. 15. The deviations from the correct dependence are marked, especially for the higher gradient using the K_{α} -doublet separation.

Consequences of this thinking are to use the symmetrization procedure as yet, but when using the K_{α} -separation method the lower threshold should be varied between 0.20 up to 0.70 of the maximum intensity. Line-fitting and parabola methods may cause errors.

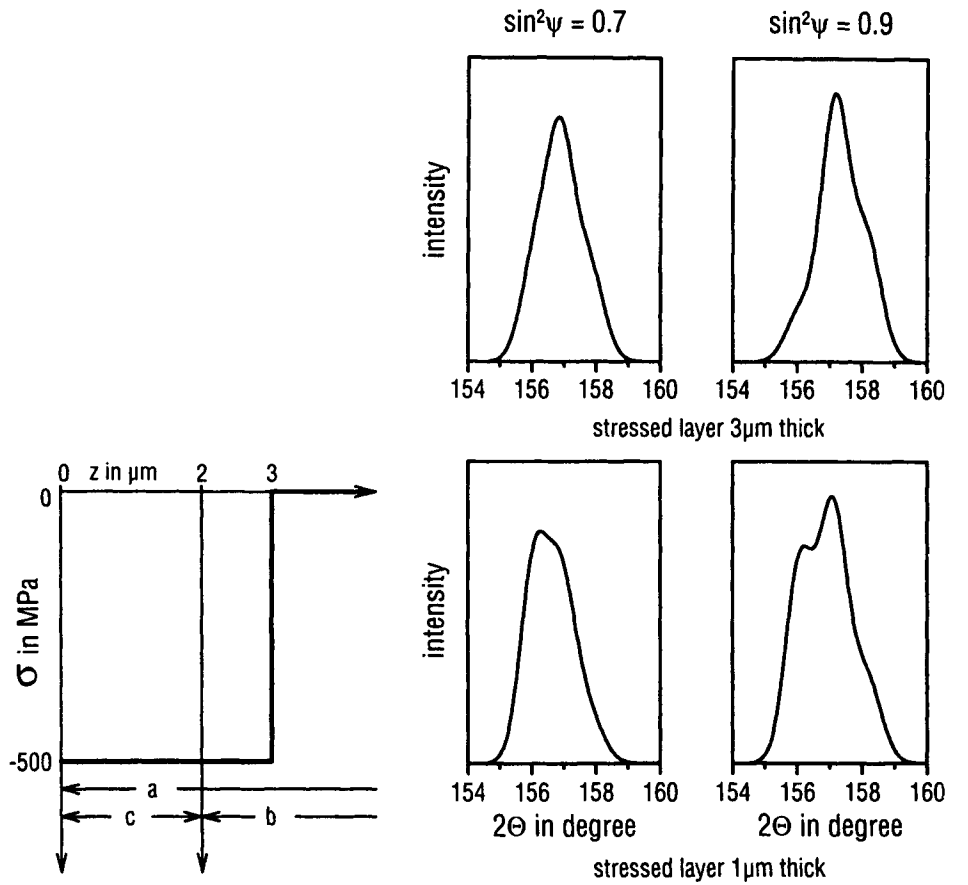


Figure 12. Two hypothetical stress profiles: $\sigma(z \leq 3 \mu\text{m}) = -500 \text{ MPa}$, $\sigma(z = 2 - 3 \mu\text{m}) = -500 \text{ MPa}$.

Figure 13. Distorted peaks as a consequence of a large stress gradient (Figure 12), Fe{211}, Cr- K_{α} .

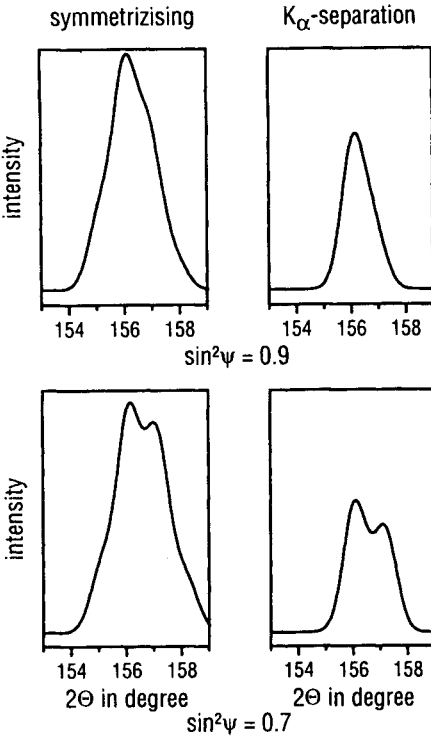


Figure 14. Distorted symmetrized K_{α} -doublet and $K_{\alpha 1}$ -peak after K_{α} -doublet separation expected for a large stress gradient in a region $1\text{ }\mu\text{m}$ thick (Fig. 12), Fe{211}, Cr- K_{α} .

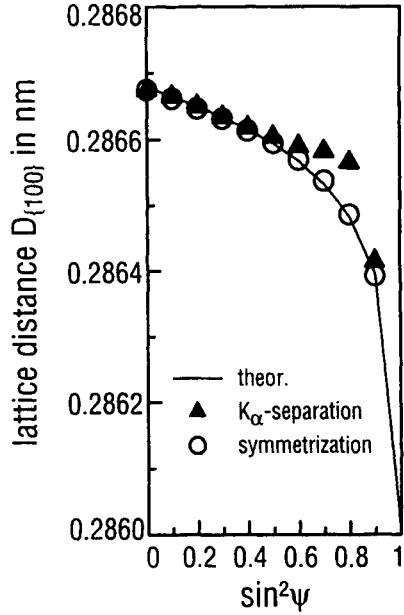


Figure 15. D-vs.- $\sin^2\psi$ distributions evaluated from distorted lines given by a stress-gradient region $1\text{ }\mu\text{m}$ thick (Fig. 12, Fig. 14) with the center-of-gravity method varying the threshold between 0.55 and 0.8 of the maximum intensity.

The following theoretical dependences should demonstrate the effect of a RS-gradient in a textured material. Here we have a superposition of oscillations in D-vs.- $\sin^2\psi$ distributions with the effect of a biaxial stress gradient.

For the theoretical study the following parameters were taken: iron, Cr- K_{α} radiation, $D_0 = 0.28665\text{ nm}$, $2\theta = 156.072^\circ$. In Fig. 16 the D-vs.- $\sin^2\psi$ are plotted for three peaks, a {211} peak with the usual oscillations and two with different shapes of D-vs.- $\sin^2\psi$ distributions.

Four types of the biaxial RS-gradient were taken, which are shown together with the D-vs.- $\sin^2\psi$ representation in Fig. 17a-d.

The superposed effect of the two parameters, texture and gradient, is demonstrated in Fig. 18a-d. For each set the three D-vs.- $\sin^2\psi$ distributions with oscillations according to Fig. 16A-C are taken. Generally speaking, the original shape of the lattice strain is in many cases not easy to recognize. If one assumes an error bar of $\pm 5 \cdot 10^{-6}\text{ nm}$, the different shapes may be difficult to distinguish. On the other side, the figures demonstrate very clearly that it is not easy to obtain besides the texture influence also the real stress gradient. Further studies

with measurements using different wavelengths, considerations of the influence of the experimental errors and tests with reconstruction of the D-vs.-sin²ψ distributions based on texture and gradient evaluation would help to clarify the real material state.

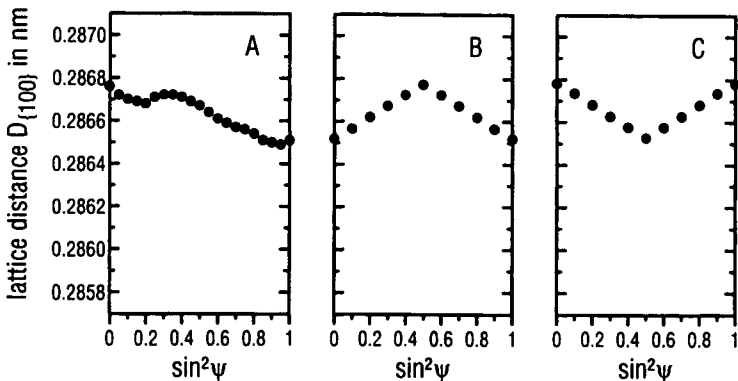


Figure 16. Three lattice-strain distributions of textured iron, α -Fe, Cr-K α , {211} and two arbitrary {hkl}, no gradient assumed.

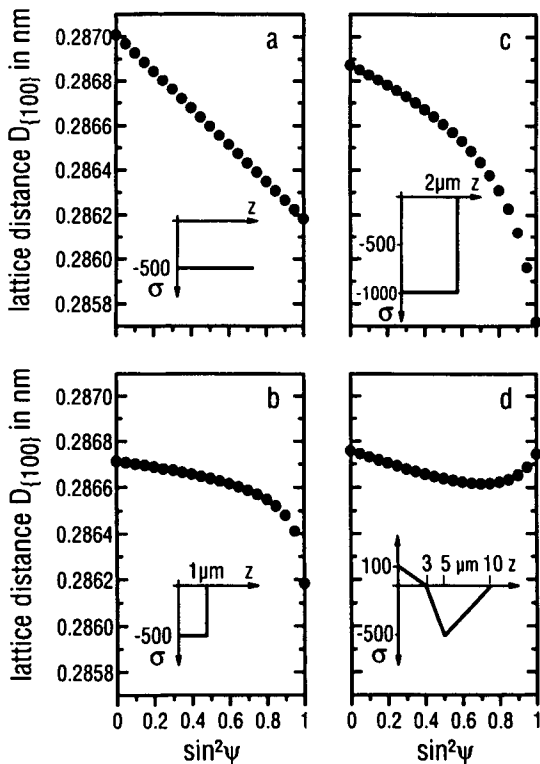


Figure 17: Lattice-strain distributions for four kinds of stress gradients and nontextured material.

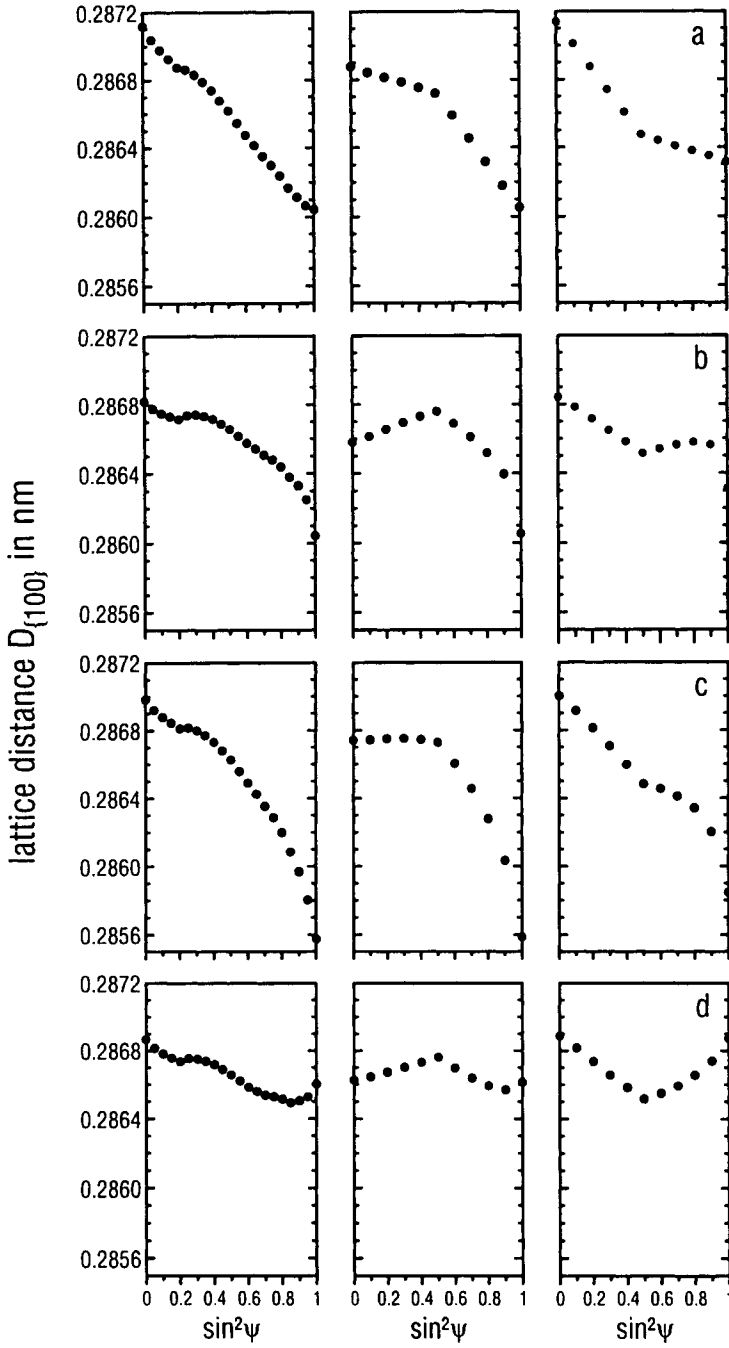


Figure 18. Superposed effect of four biaxial stress gradients (a-d Figure 17) on three textured states of iron (A, B, C, Figure 16).

2.152e Basic formulae, D_0 -gradient

Materials with a marked D_0 -gradient $\frac{dD_0}{dz}$ in near surface layers caused by a composition gradient as a consequence of alloying and/or heat treatment reveal ψ -dependent shifts of the interference lines /10/. Neglecting the influence of really existing residual stresses, the $\langle D_0 \rangle$ -values measured with particular X-rays in certain distances from the very surface can be estimated with the formula

$$\langle D_{(100)} \rangle = \frac{\int_0^P D_{(100)}(z) \exp\left\{-\frac{z}{\tau}\right\} dz}{\int_0^P \exp\left\{-\frac{z}{\tau}\right\} dz} \quad (8)$$

which considers the attenuation of the X-rays used. τ is the penetration depth and P is the thickness of the specimen.

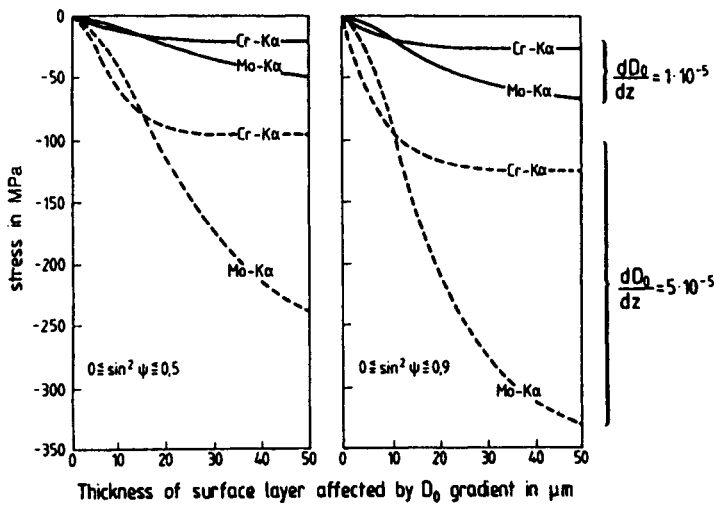


Figure 19. Fictitious stresses determined by Cr-K α and Mo-K α radiation caused by different D_0 -gradients and measured in different $\sin^2\psi$ -ranges /16/.

When Equ. 8 is applied to iron with specified linear $D_0(z)$ -distributions extending over different distances beneath the surface, fictitious stresses can be calculated, which additionally depend on the ψ -range for which the calculations are performed. Examples are shown in Fig. 19 for $\frac{dD_0}{dz} = 1 \cdot 10^{-5}$ nm/ μm and $5 \cdot 10^{-5}$ nm/ μm . As can be seen, gradients $\leq 1 \cdot 10^{-5}$ nm/ μm produce practically no fictitious stresses, if $\{211\}$ peaks are measured with Cr-K α radiation. Fictitious stresses can also be disregarded if D_0 -gradients are less than $5 \cdot 10^{-5}$ nm/ μm in surface layers smaller than 5 μm and measurements are carried out with other X-ray wavelengths. For a given X-ray wavelength, the fictitious stresses increase with the D_0 -gradient, the thickness of the affected surface layer and the covered ψ -range.

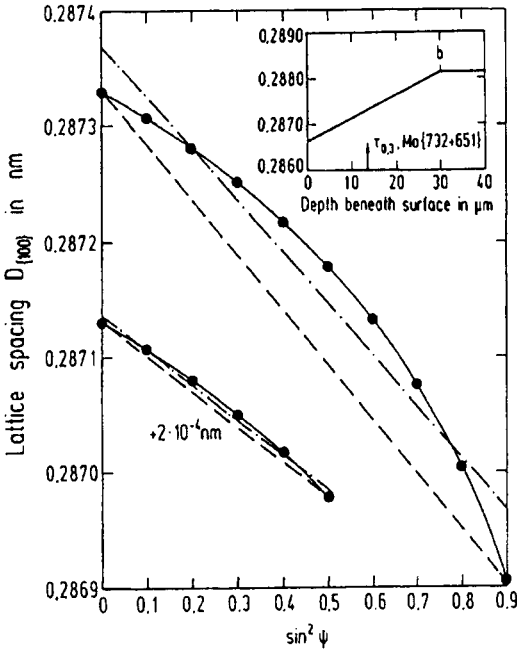


Figure 20. Curved D -vs.- $\sin^2\psi$ dependence resulting from a D_0 -gradient due to a composition gradient /19/.

The D -vs.- $\sin^2\psi$ dependences are shown in Fig. 20 for both measuring ranges, $\sin^2\psi \leq 0.5$ and $\sin^2\psi \leq 0.9$, using Mo- K_α {732+651} and a Ψ -diffractometer. The linear regression line and the chord between the points on the ends are drawn, the slopes of both are approximately the same.

The fictitious RS have been calculated for different D_0 -gradients, for the depth of the surface layer b influenced by the D_0 -gradient and for two radiations with the peaks Cr {211} and Mo {732+651} of iron. Using Mo-radiation D_0 -gradients $\leq 2 \cdot 10^{-6}$ nm/ μ m and Cr-radiation $\leq 10^{-5}$ nm/ μ m will have no influence on the result of RS. Higher gradients as stated will show up as fictitious RS.

2.152f Transformation of the τ - into the z -stress field

The principal way of determining RS in a stress field with gradients is to fix very exactly by strain measurements the RS versus penetration depth ($\tau_{0.3}$ values) dependences and to convert it into RS versus distance from the surface of the specimen z . The conversion will be done using the Laplace transformation, using computer programs or handbooks /20/.

According to /5/ the $\sigma_{ij}(z)$ profile will be written as a power series /21/:

$$\sigma_{ij}(z) = \sum_{n=0}^{\infty} a_n z^n = a_0 + a_1 z + a_2 z^2 + \dots \quad (9)$$

Using the Equ. 9 and 2 /22, 23, 6/ it follows

$$\langle \sigma_{ij} \rangle = \frac{1}{\tau} \sum_{n=0}^{\infty} \left(a_n \int_0^{\infty} \exp\left\{-\frac{z}{\tau}\right\} z^n dz \right) \quad (10)$$

$$\langle \sigma_{ij} \rangle = \frac{1}{\tau} \sum_{n=0}^{\infty} a_n n! \tau^{n+1} = a_0 + a_1 \tau + 2a_2 \tau^2 + \dots \quad (11)$$

τ is usually used as $\tau_{\sin^2\psi} = 0.3$; the a_n will be determined according to the last equation. With these a_n Equ. 9 gives the result $\sigma_{ij}(z)$; in case of an approximately linear dependence the procedure simplifies to:

$$\langle \sigma_{ij}(\tau) \rangle = a_0 + a_1 \tau = \sigma_{ij}(z) = a_0 + a_1 z \quad (12)$$

The following method is based on the same principles but was worked out in a somewhat different way /24/.

Because the D-vs.- $\sin^2\psi$ distributions measured with three wavelengths on three peaks were not linear, the normal-RS components were evaluated in the same operation for the LD and the TD. $\langle \sigma(\tau) \rangle$ is the transformed – by Laplace transformation – $\sigma(z)$ -profile. For the $\langle \sigma(\tau) \rangle$ -profile the following formula was used

$$\langle \sigma(\tau) \rangle = \frac{l_0 \tau^2 + l_1 \tau + l_2}{(1 + \alpha \tau)^3} \quad (13)$$

The coefficients l_i and α were computed using a nonlinear least-squares algorithm. The RS versus depth from the surface distribution in the LD and in the TD are derived from all D-measurements using different wavelengths according the following formula:

$$\sigma(z) = \left[l_2 + (l_1 - 2l_2\alpha)z + \left(l_0 - l_1\alpha + l_2\alpha^2 \right) \frac{z^2}{2} \right] \exp\{-\alpha z\} \quad (14)$$

The determination of the shear components can be separated from those of the normal components /8/. The condition at the surface is the following:

$$\sigma_{i3}(z=0) = 0 \quad i = 1, 2, 3; \quad \frac{\partial}{\partial z} \sigma_{ij}(z=0) = 0$$

The formulae used by /8/ are the following:

$$\langle \sigma_{13}(\tau) \rangle = \frac{l_0 \tau^3 + l_1 \tau^2}{(1 + \alpha \tau)^4} \quad (15)$$

$$\sigma_{13}(z) = \left(\frac{1}{2} l_1 z^2 + \frac{1}{6} (l_0 - l_1 \alpha) z^3 \right) \exp\{-\alpha z\} \quad (16)$$

$$\langle \sigma_{ii}(\tau) \rangle = \frac{l_0 \tau^2 + l_1 \tau + l_2}{(1 + \alpha \tau)^3} \quad i = 1, 2 \quad (17)$$

$$\sigma_{ii}(z) = \left(l_2 + (l_1 - 2\alpha l_2)z + \frac{1}{2} (l_0 - l_1 \alpha + l_2 \alpha^2) z^2 \right) \exp\{-\alpha z\} \quad (18)$$

A further modification of the principal method was brought forward by /25/. The proposition is a biaxial normal-RS state

$$\sigma_{1,2}(z) = \sum_{r=0}^g \sigma_{1,2}^{(r)} z^r \quad (19)$$

The details and the application to a practical example will be demonstrated in section 2.153b. The last mentioned methods require a modeling in so far as the evaluated RS-profile versus the depth from the surface will be converted into the measured D-vs.- $\sin^2\psi$ distribution and vice versa.

The stress-depth profile can be evaluated instead of one by several polynomials, sectionwise of lower order /26/. Fig. 21 shows schematically the principle.

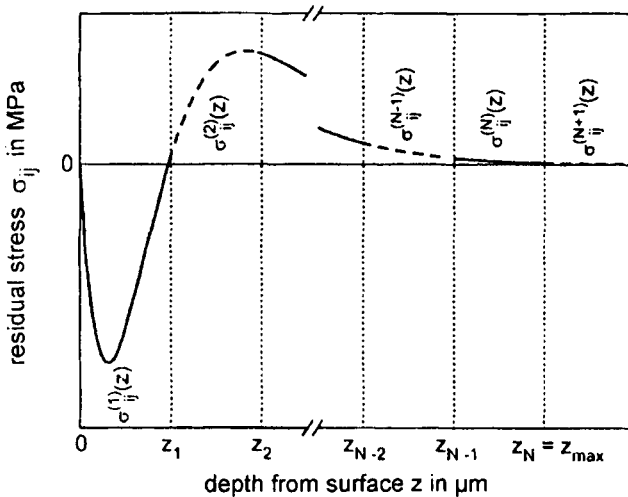


Figure 21. Sectionwise plot of a residual-stress depth profile /26/.

2.152g D, ϵ , σ versus τ , z diagram for $\sigma_{33} = 0$

In the previous chapters the results of measurement for different peaks are considered with the task to evaluate the RS-gradient in the surface region. Each D-vs.- $\sin^2\psi$ dependence - for one peak with one wavelength - was evaluated and the so-determined stresses plotted in a stress versus penetration depth diagram. For a good presentation the range of penetration can be shown instead of a special penetration depth, for example $\tau_{0.3}$. The average σ vs. τ curve can be transformed into the σ -vs.-z diagram.

Another approach is to average the $\bar{\epsilon}$ vs. τ distributions results of several to many peak measurements. This method was introduced by /9/. The assumption is made $\sigma_{33} = 0$ and D_0 must be known very accurately and has to be constant over the penetration depth. To get the correct D_0 -value causes difficulties for stress analysis on textured as well as on two- or multiphase materials. These items restrict the use of the method.

The basic XSA formula for a biaxial normal stress is given by

$$\varepsilon_{\varphi,\psi} = \frac{1}{2}s_2(\sigma_1 \cos^2 \varphi + \sigma_2 \sin^2 \varphi) \sin^2 \psi + s_1(\sigma_1 + \sigma_2) \quad (20)$$

The following operations are obvious:

$$\varepsilon_{0,\psi} \pm \varepsilon_{90,\psi} = \frac{1}{2}s_2(\sigma_1 \pm \sigma_2) \sin^2 \psi + (1 \pm 1)s_1(\sigma_1 + \sigma_2) \quad (21)$$

The sum is needed for the diagram, the difference to calculate each σ_1 and σ_2 :

$$\frac{\varepsilon_{0,\psi} + \varepsilon_{90,\psi}}{2s_1 + \frac{1}{2}s_2 \sin^2 \psi} = \frac{D_{0,\psi} + D_{90,\psi} - 2D_0}{D_0(2s_1 + \frac{1}{2}s_2 \sin^2 \psi)} = \sigma_1 + \sigma_2 \quad (22)$$

An example will be discussed in section 2.153c /27/.

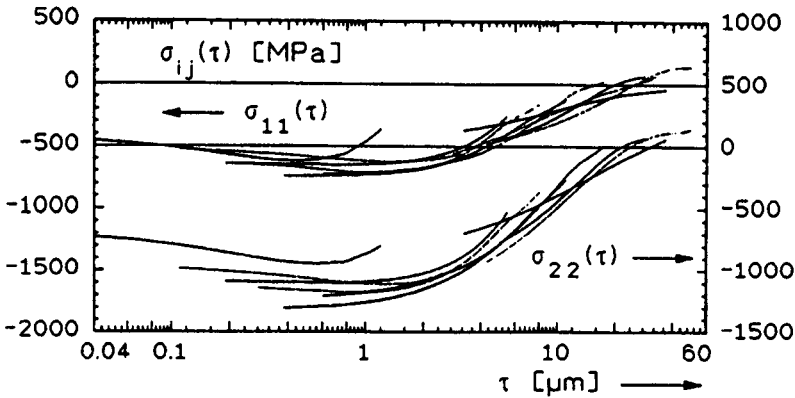


Figure 22. RS vs. penetration depth τ determined on different $\{hkl\}$ with the energy-dispersive method /27/.

2.152h An integral evaluation method

The evaluation of strain measurements according to the so-called RIM (Röntgen-Integral-Method) is based on the $2\Theta_{\varphi,\psi}$ -vs.- $\sin^2\psi$ measurement data. To demonstrate the results of such an evaluation reference is made to a paper published recently /28/. The specimen was made of $\text{Al}_2\text{O}_3 + 5 \text{ vol.}\% \text{ TiC}$ ground with a diamond abrasive wheel. Measurements were made with $\text{Ti-K}\alpha_1$ radiation on the peak $\{116\}$ in the azimuths $\varphi = 0^\circ$ and $\varphi = 90^\circ$, Fig. 23. The stress evaluation is given as a component matrix with the error matrix. The geometrical representation of the results is plotted in Fig. 24. The zero crossing of the linear dependence of σ_{11} and σ_{22} is approximately at $10 \mu\text{m}$ from the surface. The error of the σ_{33} -component is considerable. A modification of the evaluation procedure with regard to nonlinear gradients was presented at the 44th Denver X-Ray Conference. Fig. 25 shows the result of stress profiles. The zero crossing is now at approximately 4 to $5 \mu\text{m}$. Additional assumptions were not made and an error matrix is not available /29/.

The conclusion may be drawn that measurements on stress states with steep gradients allow different interpretations dependent on the particular RIM evaluation procedure.

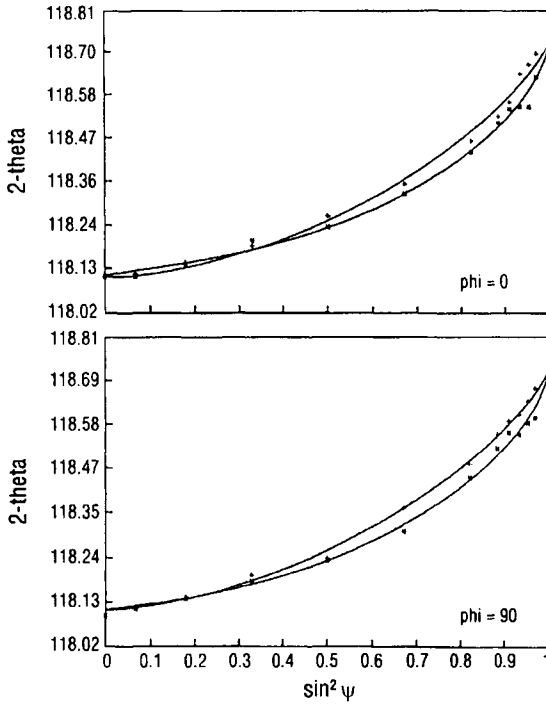


Figure 23. $2\Theta_{\phi,\psi}$ -vs.- $\sin^2\psi$ measurements with $\text{Ti-K}\alpha_1$ radiation on the $\{116\}$ peak of a ground $\text{Al}_2\text{O}_3 + 5 \text{ vol.}\% \text{ TiC}$ specimen; courtesy H.Wern.

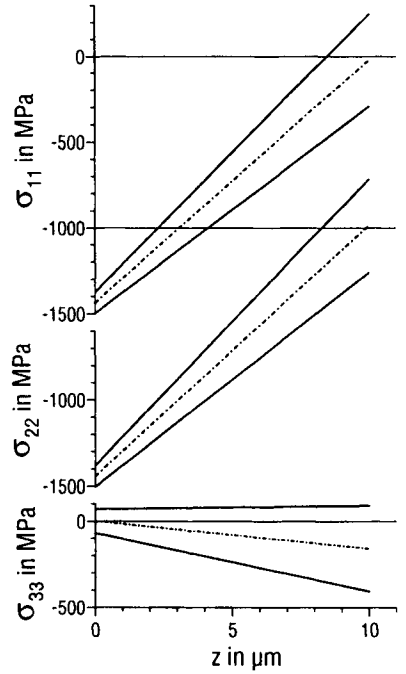


Figure 24. The evaluated stress components versus depth from the surface according to the measurement results /28/.

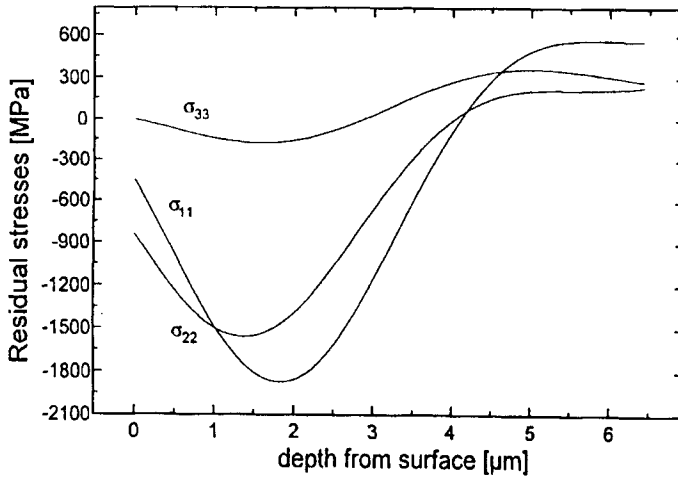


Figure 25. Evaluation of the $2\Theta_{\phi,\psi}$ -vs.- $\sin^2\psi$ measurement with the assumption of a nonlinear gradient /30/.

2.152i Stresses in removed layers

The following method can be used to determine a very steep gradient of microstructure and thereby a D_0 -gradient, a very strong stress gradient and the influence of overlapping of peaks of thin layers-substrate composites. The principle is to measure the peaks on each new surface after removal of thin layers in the surface region. The supposition is that the removal of thin layers by etching or other treatments does not introduce additional stresses. The specimen thickness P should be large in regard to the penetration depth τ , which is defined as the thickness in which $1/e$ of the initial intensity is attenuated. The idea and the first application to Cr-coatings on steel was introduced by [31]. The basic formulae used previously are the following:

$$\langle 2\Theta_{\varphi,\psi} \rangle = \frac{\int_0^t 2\Theta_{\varphi,\psi}(z) \exp\left\{\frac{-z}{\tau}\right\} dz}{\int_0^t \exp\left\{\frac{-z}{\tau}\right\} dz} \quad (23)$$

$$\langle I_{\varphi,\psi} \rangle = \frac{\int_0^t I_{\varphi,\psi}(z) \exp\left\{\frac{-z}{\tau}\right\} dz}{\int_0^t \exp\left\{\frac{-z}{\tau}\right\} dz} \quad (24)$$

We assume a composite of a thin film that will be divided into n layers and a substrate. The thicknesses of the layers are t_i , that of the whole composite P . The penetration depth after each removal (i) is τ_i . In total the following $(2n+1)$ steps are necessary:

- Measurement 1 of $\langle 2\Theta_{\varphi,\psi} \rangle^{(1)}$ vs. $\sin^2\psi$.
- Removal of layer 1.
- Measurement 2 of $\langle 2\Theta_{\varphi,\psi} \rangle^{(2)}$ vs. $\sin^2\psi$.
- Removal of layer 2.
- .
- .
- Measurement n of $\langle 2\Theta_{\varphi,\psi} \rangle^{(n)}$ vs. $\sin^2\psi$.
- Removal of layer n .
- Measurement $(n+1)$ of $\langle 2\Theta_{\varphi,\psi} \rangle^{(n+1)}$ vs. $\sin^2\psi$ of the substrate alone.

Assuming a homogeneous stress state and $\sigma_{33} = 0$ in each layer n , the following formulae with index (s) for substrate and the number (i) for measurement (i) are to be used:

$$\langle 2\Theta_{\varphi,\psi} \rangle = \frac{\sum_{i=1}^s \left(\prod_{v=1}^i \exp\left\{-\frac{t_{v-1}}{\tau_{v-1}}\right\} \right) \cdot 2\Theta_{\varphi,\psi}^{(i)} \cdot I_{\varphi,\psi}^{(i)} \cdot \tau_i \left(1 - \exp\left\{-\frac{t_i}{\tau_i}\right\} \right)}{\sum_{i=1}^s \left(\prod_{v=1}^i \exp\left\{-\frac{t_{v-1}}{\tau_{v-1}}\right\} \right) \cdot I_{\varphi,\psi}^{(i)} \cdot \tau_i \left(1 - \exp\left\{-\frac{t_i}{\tau_i}\right\} \right)} \quad (25)$$

with

$$\langle 2\Theta_{\varphi,\psi} \rangle^{(s)} = 2\Theta_{\varphi,\psi}^{(s)}, \quad t_{i=0} = 0 \quad (26)$$

$$\langle I_{\varphi,\psi} \rangle = \frac{\sum_{i=1}^s \left(\prod_{v=1}^i \exp \left\{ -\frac{t_{v-1}}{\tau_{v-1}} \right\} \right) \cdot I_{\varphi,\psi}^{(i)} \cdot \tau_i \left(1 - \exp \left\{ -\frac{t_i}{\tau_i} \right\} \right)}{\sum_{i=1}^s \left(\prod_{v=1}^i \exp \left\{ -\frac{t_{v-1}}{\tau_{v-1}} \right\} \right) \cdot \tau_i \left(1 - \exp \left\{ -\frac{t_i}{\tau_i} \right\} \right)} \quad (27)$$

with

$$\langle I_{\varphi,\psi} \rangle^{(s)} = I_{\varphi,\psi}^{(s)}, \quad t_{i=0} = 0$$

Difference in $\langle 2\Theta_{\varphi,\psi} \rangle$ after one removal:

$$\begin{aligned} \langle 2\Theta_{\varphi,\psi} \rangle &= \frac{\sum_{i=1}^s \left(\prod_{v=1}^i \exp \left\{ -\frac{t_{v-1}}{\tau_{v-1}} \right\} \right) \cdot 2\Theta_{\varphi,\psi}^{(i)} \cdot I_{\varphi,\psi}^{(i)} \cdot \tau_i \left(1 - \exp \left\{ -\frac{t_i}{\tau_i} \right\} \right)}{\sum_{i=1}^s \left(\prod_{v=1}^i \exp \left\{ -\frac{t_{v-1}}{\tau_{v-1}} \right\} \right) \cdot I_{\varphi,\psi}^{(i)} \cdot \tau_i \left(1 - \exp \left\{ -\frac{t_i}{\tau_i} \right\} \right)} \\ &= \frac{2\Theta_{\varphi,\psi}^{(1)} \cdot I_{\varphi,\psi}^{(1)} \cdot \tau_1 \left(1 - \exp \left\{ -\frac{t_1}{\tau_1} \right\} \right) + \sum_{i=2}^s \left(\prod_{v=1}^i \exp \left\{ -\frac{t_{v-1}}{\tau_{v-1}} \right\} \right) \cdot 2\Theta_{\varphi,\psi}^{(i)} \cdot I_{\varphi,\psi}^{(i)} \cdot \tau_i \left(1 - \exp \left\{ -\frac{t_i}{\tau_i} \right\} \right)}{I_{\varphi,\psi}^{(1)} \cdot \tau_1 \left(1 - \exp \left\{ -\frac{t_1}{\tau_1} \right\} \right) + \sum_{i=2}^s \left(\prod_{v=1}^i \exp \left\{ -\frac{t_{v-1}}{\tau_{v-1}} \right\} \right) \cdot I_{\varphi,\psi}^{(i)} \cdot \tau_i \left(1 - \exp \left\{ -\frac{t_i}{\tau_i} \right\} \right)} \quad (28) \end{aligned}$$

Abbreviations

$$A(1) = I_{\varphi,\psi}^{(1)} \tau_1 \left(1 - \exp \left\{ -\frac{t_1}{\tau_1} \right\} \right) \quad (29)$$

$$A(2)^{(i)} = \prod_{v=1}^i \exp \left\{ -\frac{t_{v-1}}{\tau_{v-1}} \right\} \cdot I_{\varphi,\psi}^{(i)} \cdot \tau_i \left(1 - \exp \left\{ -\frac{t_i}{\tau_i} \right\} \right)$$

$$\langle 2\Theta_{\varphi,\psi} \rangle = \frac{2\Theta_{\varphi,\psi}^{(1)} A(1) + \sum_{i=2}^s 2\Theta_{\varphi,\psi}^{(i)} A(2)^{(i)}}{A(1) + \sum_{i=2}^s A(2)^{(i)}}$$

$$2\Theta_{\varphi,\psi}^{(1)} = \frac{1}{A(1)} \left[\langle 2\Theta_{\varphi,\psi} \rangle \left(A(1) + \sum_{i=2}^s A(2)^{(i)} \right) - \sum_{i=2}^s 2\Theta_{\varphi,\psi}^{(i)} A(2)^{(i)} \right]$$

This is the correct Bragg's angle for the removed layer. The value of $A(2)^{(i)}$ must be evaluated with a further removal and measurement step. One must be aware of the fact that in this case the former layer with index $(i+1)$ transforms now into the layer with index (i) in the formula above. This procedure ends with the measurement of the substrate (s) , i.e. $(i+1) \rightarrow (s)$, where the measured peak has no overlapping component.

2.152j Stress- and D_0 -gradients

If gradients of both RS and D_0 are present the convoluted RS-profile has to be found by modeling. An example is given in section 2.073f.

Another example deals with γ -Fe₄N_{1-x} layers on α -Fe substrates /32,33,34,35/. Extensive studies have been dedicated to the separation of the composition- and the RS-profiles in the presence of pores.

2.152k Relaxation of stress components near a free surface

Triaxial stress evaluation requires the exact knowledge of the stress free lattice parameter D_0 as is outlined in paragraph 2.112. Because in many practical cases it is not possible or too tedious to determine this value, it is usual to assume the stress in normal direction σ_{33} of the studied surface to be zero which enables one to calculate the D_0 -value from measurement for two azimuths $\varphi=0^\circ$ and 90° . In most cases, this procedure is justified because σ_{33} is exactly zero at the surface and the penetration depth is small enough so that no appreciable σ_{33} -components will develop herein. This is valid if predominantly macrostresses are present whose period of lateral variation is large compared with the extension of the area and the depth illuminated by X-rays during the measurements.

To assess the applicability of the assumption $\sigma_{33}=0$ within the penetration depth of the X-rays it is necessary to get knowledge of the general behavior of σ_{33} in the vicinity of the surface. The length scale on which a non-zero σ_{33} will develop depends on the microstructure of the material, in particular on the length scale of quasi-periodicity. Calculations were performed in /36/ for a structure with spherical precipitates which are arranged like a cubic lattice of constant a . Besides the σ_{33} -component, also σ_{11} and σ_{22} relax near the surface. The conclusions of this fact for the XSA were firstly discussed in /37/ where the formulae of /36/ were used to calculate the profiles of all stress components in the surface region of a pearlitic structure (Fig. 26). Another approach was outlined in /38/ by applying the solution of Boussinesq /39,40/ for the stress induced by a point force on an infinite half space. The results of the three papers are similar in respect to the σ_{33} -component. Fig. 27 shows the profiles of the phase stresses in a pearlitic structure /38/ in the bulk material and at the surface. They are plotted versus z/a with z the depth beneath the surface and " a " the period of the structure. The lateral variations of the stresses within the phases depend on the volume fractions. The macrostresses $\sigma_{33}^{\text{macro}}$ relax at the surface as expected. The microstresses do relax, too, at the very surface, but, in a depth less than the period, they reach the level of that of the bulk material. The period may be the thickness of the lamella in the pearlitic structure, it may also be the mean distance between precipitates, grains of a second phase, whiskers etc.. Because, for a lot of materials, this period is much smaller than the penetration depth of X-rays, the effect of the

decrease of microstresses in the z -direction at the surface may not be detected. Experimental proofs of the fact, that the gradient $\sigma_{33}^{\text{micro}}$ at the surface may be too steep to be detected and, therefore, a triaxial microstress state is determined, were given in /38,41,42/.

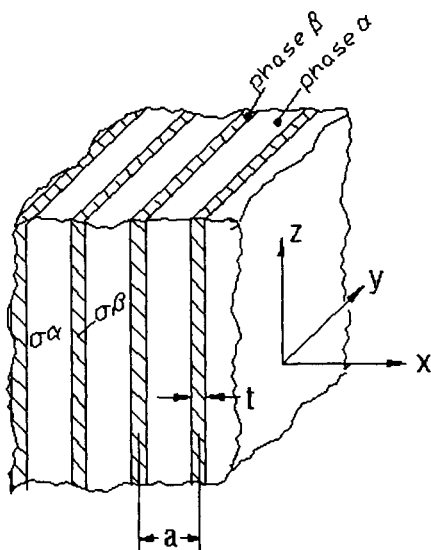


Figure 26. Periodical structure of a two-phase material. $\sigma_z = \sigma^\alpha$ in the phase α and $\sigma_z = \sigma^\beta$ in the phase β . The volume content c_β of the phase β is given by the thickness t of the β -lamellae and the period a : $c_\beta = t/a$.

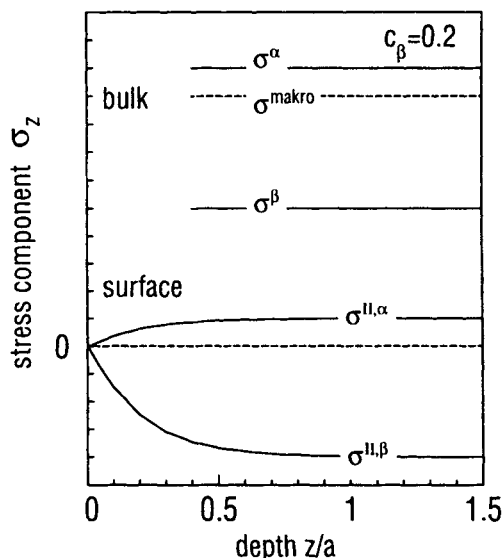


Figure 27. Profile of the stress component σ_z near the surface, dependent on the relative depth z/a with a the period of the phase distribution at the surface /38/.

The following conclusions can be drawn:

- the profile of stresses in the z -direction near the surface depends predominantly on the period " a " of the microstructure
- details of the microstructure are of minor importance
- macrostresses in the direction normal to the surface relax totally near the surface
- the range of the surface influence on the microstresses is less than one period, which is about $0.1 \mu\text{m}$ in pearlitic structures, i.e. it is small compared to the penetration depth of X-rays
- X-ray stress measurements will, therefore, generally reveal a biaxial macrostress state and a triaxial microstress state
- relaxation of the σ_{33} -component will affect the other components, too /36,37/. The effect on the interpretation of X-ray results has still to be elaborated.

2.153 Experimental methods and results

2.153a Use of different radiations and peaks

The X-ray method to determine a steep strain-stress gradient or a stress profile should be selected according to the gradient problem itself. The material thickness affected by the gradient, the RS-state and the steepness of the gradient determine the experimental method to use as many as necessary wavelengths and peaks to get the stress profile. Measurements up to $\sin^2\psi \leq 0.95$ or $\psi \leq 87^\circ$ are used in recent papers. In order to evaluate the stress at the very surface one needs to use a low penetrating wavelength-peak combination and if possible tilting the specimen in the Ψ -mode by the angle ω' . The stress versus penetration depth must be converted into the stress versus the distance from the surface in the thickness direction. Programs with Laplace transformation and regression-balancing least-squares method are used commonly.

There are different methods to determine the $\sigma(\tau)$ -profile. If the D-vs.- $\sin^2\psi$ dependences for different peaks obtained with different wavelengths are linear or if the curvature is small and may be neglected, as many peaks as possible should be evaluated. To reach the very surface, the specimen may or should be tilted. D-vs.- $\sin^2\psi$ distributions with marked curvature may be as an approximation stepwise-linear evaluated. The correct way is to consider the total curved D-vs.- $\sin^2\psi$ distributions of peaks from several radiations. It should be mentioned that evaluating a single D-vs.- $\sin^2\psi$ dependence may not be sufficient to determine the $\sigma(z)$ -profile in unique way.

Here are examples from the literature; details may be found in the short descriptions and in the captions. Interesting studies deal with the evaluation of the real RS at the very surface of surface-treated material (ceramics), thin-film-substrate composites, and cold-rolled materials. These problems relate with the influence of very strong RS-gradients on the positions of the peaks to be evaluated. Two developments in XSA made more detailed information of the RS-state in the near-surface region possible: The use of radiation of longer wavelengths and the possibility to measure to higher values of ψ , say up to $\sin^2\psi \leq 0.95$. Fig. 28 shows the penetration depth of different radiation-peak combinations for experiments with the following materials /43/.

Fig 29 shows RS-profiles of ground ceramics evaluated with different wavelengths on different peaks (left) /44/, the influence of an additional tilt ω' (center) /45/ and on the right-hand side results of measurements with Ti-K_α radiation on the $\{116\}$ interference line up to $\psi \leq 80^\circ$ /25/. Fig. 30 demonstrates the effect of very steep stress gradients on the outmost surface zone in rolled Ni /46/ and rolled Cu alloy /47/. An influence of surface anisotropy (section 2.134h) is indicated.

The RS-state after grinding chiefly of steels was several times studied by Hauk et al., using different wavelengths and measurements on several peaks /21,7,24,8/. The evaluation of the RS-gradient was done by use of exponential functions. The scatter of the results due to experimental errors was discussed in section 2.152f. The following formulae were used for the Laplace transformation from the τ -space (penetration depth) into the z -space (depth from the surface):

$$\sigma_{i3}(z=0) = 0 \quad i = 1, 2, 3 \quad \text{and} \quad \frac{\partial}{\partial z} \sigma_{ij}(z=0) = 0$$

$$\sigma_{ii}(z) = \left[l_2 + (l_1 - 2\alpha l_2) z + \frac{1}{2} (l_0 - l_1 \alpha + l_2 \alpha^2) z^2 \right] \exp\{-\alpha z\} \quad (30)$$

$$\sigma_{13}(z) = \left[\frac{1}{2} l_1 z^2 + \frac{1}{6} (l_0 - l_1 \alpha) z^3 \right] \exp\{-\alpha z\} \quad (31)$$

Data and evaluation results are shown in the following figures, some details are given in the captions /8/.

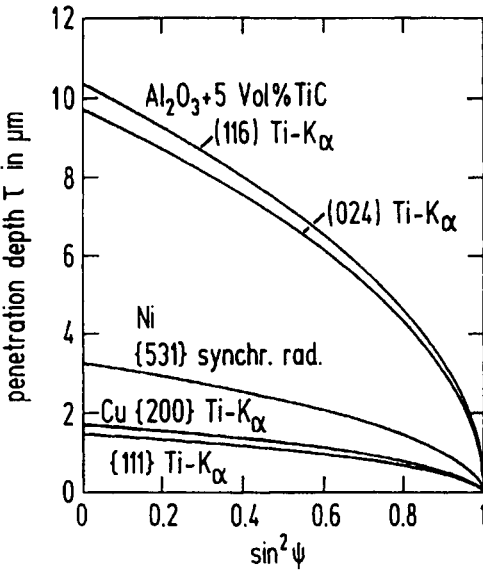


Figure 28. Penetration depths for different radiations in different materials, Ψ -diffractometer /43/.

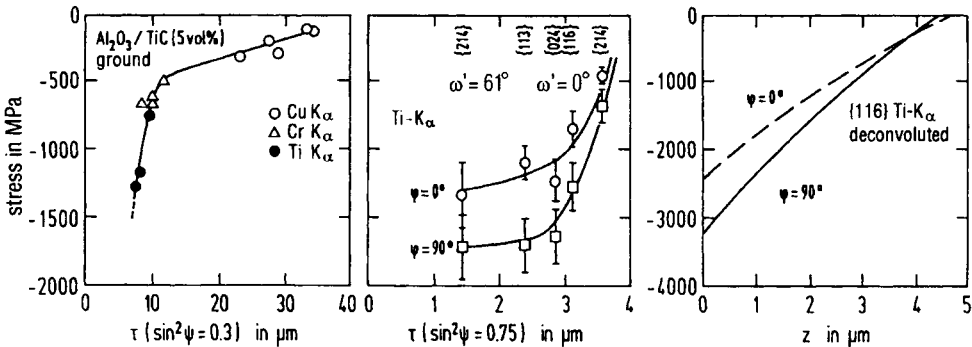


Figure 29. RS-profiles of ground ceramics evaluated from data for different radiations and peaks /43/.

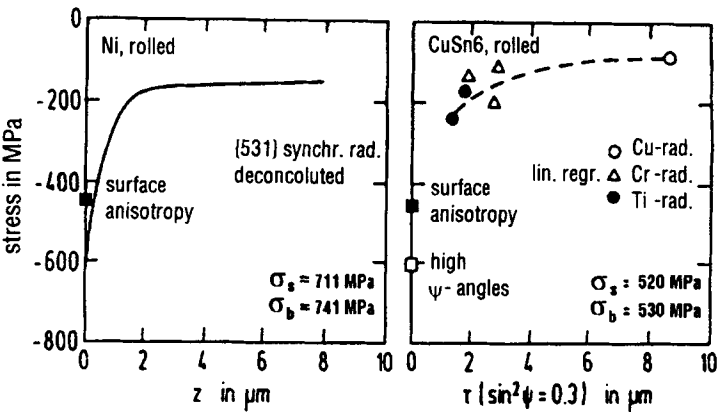


Figure 30. RS-profiles of rolled Ni and CuSn6 strips /43/.

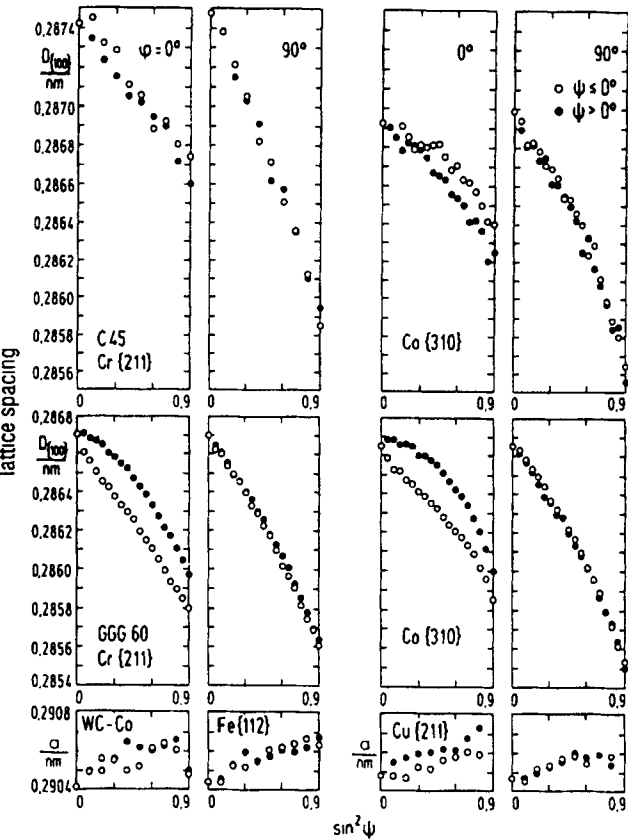


Figure 31. Lattice-strain distributions measured on different specimens with ground surfaces; each specimen was measured with two X-ray radiations of different penetration depth, material, radiation, reflection and azimuth as indicated /8/.

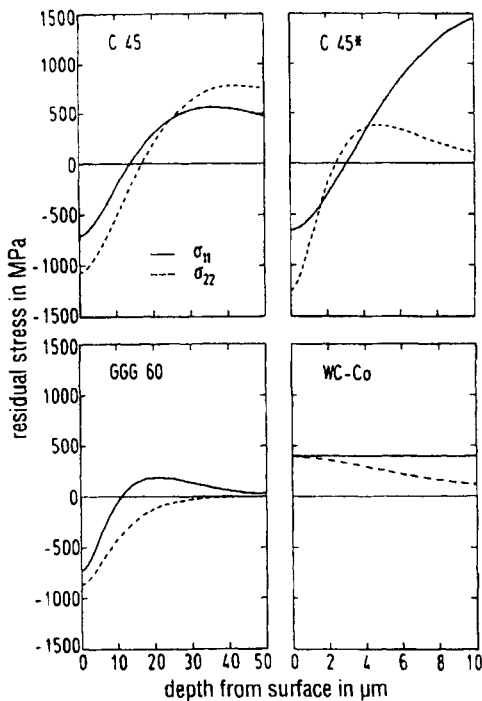


Figure 32. Depth dependencies of the residual-stress components σ_{11} and σ_{22} of the C45 specimens ground without and with (*) sparking out; the results for ground GGG-60 and WC-Co specimens are also represented /8/.

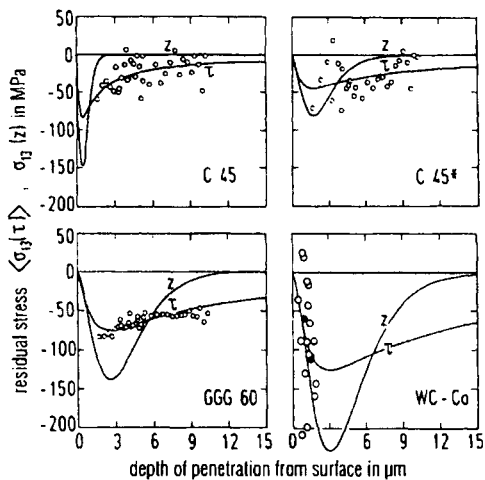


Figure 33. $\langle \sigma_{13} \rangle$ -values evaluated from the ψ -splitting and the fitting curve depending on the penetration depth, additionally the evaluated depth dependency of the σ_{13} residual-stress component of the C45 specimens ground without and with (*) sparking out, and of the ground GGG-60 and WC-Co specimens, resp. /8/.

2.153b Use of one peak

The D-vs.- $\sin^2\psi$ distribution for one peak may be sufficient to elaborate the RS-profile from the surface if a precise measuring and evaluating system is established. This was done by /25/ for a machined ceramic surface layer with a thickness of only some μm . The presuppositions are as follows: A biaxial RS-state parallel to the surface, $\sigma_{33}=0$, azimuth $\varphi=0^\circ$ machining, say the grinding direction and $\varphi=90^\circ$ the transverse direction. The penetration depth of the radiation τ_0 should be of the same order of magnitude as the region of the surface that is influenced by the machining finish. The measuring range should be as high as possible i.e. $|\psi| \leq 80^\circ$, with synchrotron radiation up to $|\psi| \leq 88^\circ$. The computerized evaluation should involve polynomial functions of increasing degrees. A stable function RS-state versus distance from the surface will then be the result.

The development of the formulae follows the previously mentioned ones. $\sigma_i(z)$ will be developed by polynomial functions up to a chosen order

$$\sigma_{1,2}(z) = \sum_{r=0}^g \sigma_{1,2}^{(r)} z^r \quad (19')$$

and inserted into the weighting integral

$$\langle \sigma_{1,2} \rangle = \frac{\int_0^\infty \sigma_{1,2}(z) \exp\left\{-\frac{z}{\tau}\right\} dz}{\int_0^\infty \exp\left\{-\frac{z}{\tau}\right\} dz}$$

This expression can be solved by Laplace transformation. The resulting equations for the Bragg's angles are:

$$\begin{aligned} 2\Theta_{\varphi,\psi} = 2\Theta_0 - 2 \tan \Theta_0 \sum_{r=0}^g \left[r! \left(\frac{\sin \Theta_0}{2\mu} \right)^r \cos^r \psi \left(\frac{1}{2} s_2 \cos^2 \varphi \sin^2 \psi + s_1 \right) \sigma_1^r \right] \\ - 2 \tan \Theta_0 \sum_{r=0}^g \left[r! \left(\frac{\sin \Theta_0}{2\mu} \right)^r \cos^r \psi \left(\frac{1}{2} s_2 \cos^2 \varphi \sin^2 \psi + s_1 \right) \sigma_2^r \right] \end{aligned} \quad (32)$$

Very exact $D_{0,\psi}$, $D_{90,\psi}$ - vs.- $\sin^2\psi$ measurements must be done. σ_1 , σ_2 and D_0 can be evaluated.

Here is given an example /25/ of a ground specimen $\text{Al}_2\text{O}_3 + 5 \text{ vol.}\% \text{ TiC}$, Fig. 34. The biaxial RS-state versus the depth is plotted for several calculated functions. The measured values of $D_{\varphi,\psi}$ vs. $\sin^2\psi$ are very well verified by the calculated function, see also /48/.

In cases in which a D-vs.- $\sin^2\psi$ dependence shows a narrow curvature in the very high ψ -range the plot D-vs.- ψ allows a better representation of the experimental data. Fig. 35 shows an example /49/.

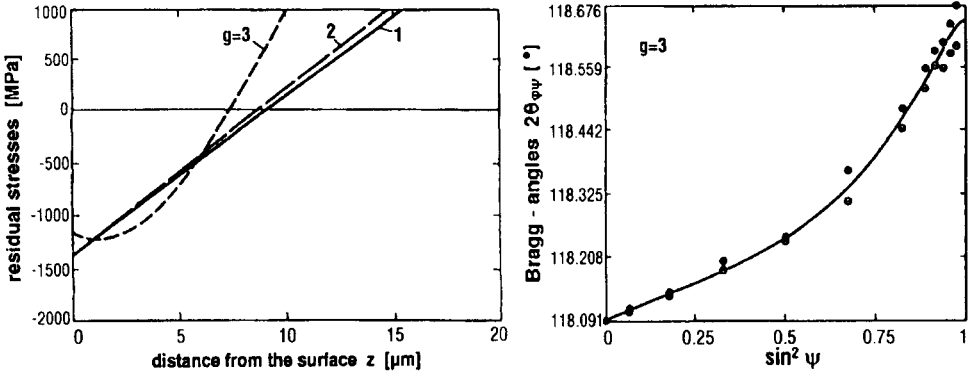


Figure 34. Residual-stress state and $2\theta_{\varphi,\psi}(\sin^2\psi)$ of ultrasonically machined $\text{Al}_2\text{O}_3/\text{TiC}$ (5 vol.%), $\text{Ti-K}\alpha$ radiation, $\{116\}$ -planes, evaluated with $g = 1\dots3/25$.

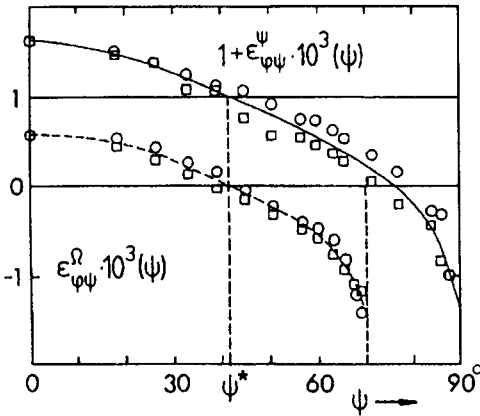


Figure 35. Data obtained from the $\{531\}$ reflections of a cold-rolled nickel plate: $1 + \epsilon_{0,90,\psi}^{\psi} \cdot 10^3$ obtained with Ψ -goniometry: full curve. $\epsilon_{0,90,\psi}^{\Omega} \cdot 10^3$ obtained with Ω -goniometry for $2\theta = 140^\circ$: dashed curve. Experimental points corresponding to $\epsilon_{0,\psi} \cdot 10^3$ and $\epsilon_{90,\psi} \cdot 10^3$: circles and squares, respectively /49/.

2.153c Energy-dispersive method

The RS-state was determined on a ground steel sample using the energy-dispersive X-ray method /50/. The method itself was shortly described in the respective paragraphs of 2.04 and 2.07. Here are reported the only existing results for the RS-state of a ground steel plate. The same specimen was tested earlier with X-rays on the peaks $\{211\}$ and $\{310\}$ and the RS-profile versus penetration depth converted by Laplace transformation into RS-versus-depth from the surface dependence /24,8/. Details are given in section 2.153a.

Here is given the evaluation of a lot of measurements with the energy-dispersive method for a biaxial RS-state /27,50,51/. Fig. 36 and 37 demonstrate the results for eleven peaks from {110} till {521} measured over the respective penetration depth. The RS vs. τ and RS vs. z are shown in Fig. 38. Additional evaluations on a cold-rolled Ni plate and a water-jet-peened steel plate demonstrate the ability of the method to give the RS-gradients in the surface-near regions of a few μm thickness.

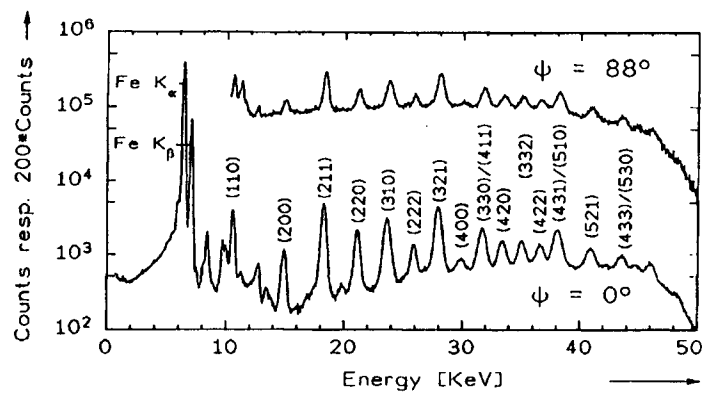


Figure 36. EDXRD spectra of a ground steel plate (W tube; 50 kV; 40 mA; counting time 3 h; $2\Theta = 33.5^\circ$). The specimen was tilted by $\psi = 0^\circ$ and $\psi = 88^\circ$. The number of counts for $\psi = 88^\circ$ was multiplied by a factor of 200 /27/.

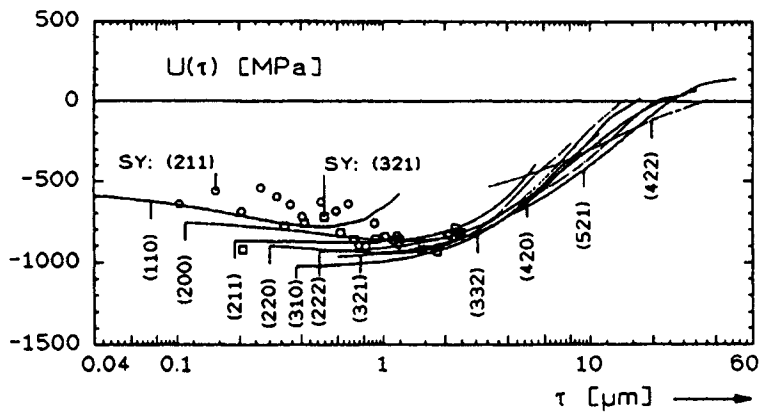


Figure 37. Master plots $U(\tau) = \frac{1}{2}[\sigma_{11}(\tau) + \sigma_{22}(\tau)]$ calculated from individually normalized $e_{\phi,\psi}^h(\tau)$ -curves: O, □ obtained with synchrotron radiation experiments /27/.

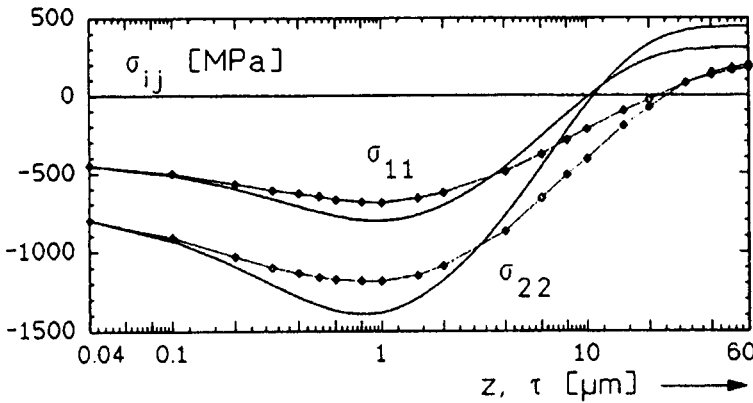


Figure 38. Normal residual stresses in a steel plate that was ground in the x-direction: $\diamond$, $\sigma_{ii}(\tau)$; —, $\sigma_{ii}(z)$. The two $\sigma_{ii}(z)$ -curves were obtained by inverse Laplace transformation of the $\sigma_{ii}(\tau)$ analytical fits /27/.

2.153d Grazing-incidence method

To investigate experimentally the outmost surface layer of a material or a film-substrate-composite Predecki et al. /52,53,54,55/ used the grazing-incidence X-ray diffraction method (see Fig. 26 in paragraph 2.042). Under the assumption $\sigma_{33} = 0$, determined stresses versus penetration-depth dependence will be fitted; functions are listed in Table 2. The transformation to the stress-vs.-depth from the surface profiles will be done using the inverse Laplace function, Table 2. Examples of ground $\text{Al}_2\text{O}_3 + 25 \text{ wt.}\% \text{ SiC}$ whiskers and sputtered-Mo films on glass illustrate the method.

Table 2: Equations obtained for τ and z -profiles /53/

τ -profile	z -profile
$\langle \sigma_{ii}(\tau) \rangle = m_1 + m_2 \exp(-m_3/\tau)$	$\sigma_{ii}(z) = m_1 + m_2 \cdot U(z-m_3)$
$\langle \sigma_{ii}(\tau) \rangle = m_1 + [m_2/\tau^{1/2}] \exp(-m_3\tau)$	$\sigma_{ii}(z) = m_1 + [m_2/(\pi z)^{1/2}] \cos(2[m_3 z]^{1/2})$
$\langle \sigma_{11}(\tau) \rangle = m_1 + m_2/[1-m_3\tau]$	$\sigma_{11}(z) = m_1 + m_2 \exp(m_3 z)$
$\langle \sigma_{11}(\tau) \rangle = m_1 + m_2 [1-\exp(-m_3/\tau^{1/2})]$	$\sigma_{11}(z) = m_1 + m_2 \operatorname{erf}(m_3/[2 z^{1/2}])$
$\langle w_i(\tau) \rangle = m_1 + [m_2/\tau] \exp(-m_3/\tau^{1/2})$	$w_i(z) = m_1 + [m_2 m_3/(4\pi z^3)^{1/2}] \exp(-m_3^2/4z)$

U = Heaviside unit function, erf = error function, w_i = FWHM, m_1 , m_2 and m_3 = fitted constants

2.153e The scattering vector method

Ch. Genzel

X-ray residual stress-gradient analysis in polycrystalline materials is based on the measurement of the lattice spacing $D_{\{hkl\}}$ for different values of the penetration depth τ . If angle dispersive diffraction methods are applied, the variation of τ may be achieved, in principle, in two ways which differ by whether the pole-angle ψ between the measuring direction $\underline{m}$ and the surface normal is kept fixed or not. In the second case, the measurements are performed in the Ω - or the Ψ -mode on the basis of the $\sin^2\psi$ -method. Approaches for evaluating the stress profiles $\sigma_{ik}(z)$ from the "Laplace"-profiles $D_{\{hkl\}}[\tau(\psi)]$ are given in section 2.152f.

An alternative formalism for the evaluation of strongly non-linear near-surface stress fields is proposed in /56/. It is based on the idea that the penetration depth may be varied independently of the pole-angle ψ , if the sample is rotated around the scattering vector $\underline{g}_{\varphi,\psi}$, which is parallel to the normal of the reflecting lattice planes, $\underline{N}^{hkl}$ (Fig. 39). Denoting the angle of rotation by η , the penetration depth τ becomes

$$\tau = \frac{\sin^2 \theta - \sin^2 \psi + \cos^2 \theta \sin^2 \psi \sin^2 \eta}{2\mu \sin \theta \cos \psi}.$$

(33)

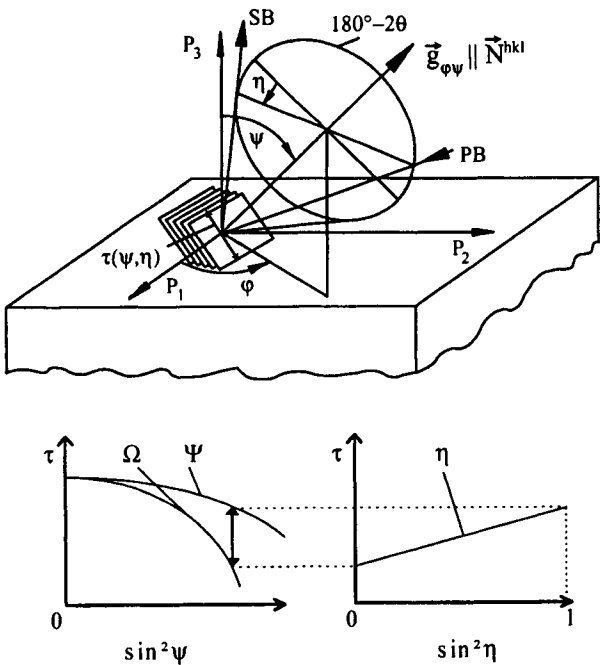


Figure 39. Schematic view of the diffraction geometry in XSA and the correlation of the penetration depth τ in Ψ -, Ω - and η -goniometry; PB, SB - primary and secondary (diffracted) beam, respectively.

Inserting for η the values $0, \pi$ and $\pi/2, 3\pi/2$, Equ. 33 yields the well-known expressions for the Ω - and the Ψ -mode in the conventional XSA, respectively. In the scattering vector goniometry, the absorption factor depends additionally on η :

$$A_{\psi, \eta}(\theta) = 1 + \tan|\psi| \cot \theta \cos \eta. \quad (34)$$

By means of Equ. 33 the penetration depth τ can be varied without changing ψ . Thus, if the sample is rotated by a certain amount of η around $\mathbf{g}_{\varphi, \psi}$, depth profiles of the lattice spacing $D_{\{hkl\}}(\tau)$ are obtained even for the same orientation with respect to the sample system. In the basic equation of XSA, the angle ψ now becomes a usual parameter in the same sense as the azimuth angle φ . Therefore, the scattering vector method permits the separation of individual components of $\sigma_{ik}(\tau)$ from the basic equation, if the depth scans $D_{\varphi, \psi}(\tau)$ performed in the η -goniometry at different positions of φ and ψ are combined in a suitable way. In order to minimise the errors due to an insufficient knowledge of the exact lattice parameter D_0 of the stress-free material, it is recommended to perform η -scans in at least two positions of ψ and taking the differences. In the case of a biaxial stress state (i.e. assuming $\sigma_{i3} = 0$, $i = 1, 2, 3$ within the penetration depth) this procedure yields for the azimuth $\varphi = 0^\circ$

$$\sigma_{11}(\tau_\eta) = \frac{D_{0, \psi_1}(\tau_\eta) - D_{0, \psi_2}(\tau_\eta)}{D_0 [F_{11}(0, \psi_1) - F_{11}(0, \psi_2)]} \quad (35)$$

In Equ. 35 τ_η denotes that τ is varied by η . F_{11} is the stress factor of the σ_{11} -component, for quasiisotropic materials one has formally $F_{11}(\varphi, \psi) = \frac{1}{2} s_2 \cos^2 \varphi \sin^2 \psi + s_1$. For $\varphi = 90^\circ$, an equivalent relation is obtained for $\sigma_{22}(\tau_\eta)$. From the conventional point of view, Equ. 35 may be considered as a "depth resolved" $\sin^2\psi$ -method performed at two discrete ψ -positions.

For finite values of σ_{i3} ($z \neq 0$), $i = 1, 2, 3$, the situation becomes more complicated. Considering the evaluation of $\sigma_{33}(\tau)$ -gradients in more detail, η -scans have to be performed in the azimuths $\varphi = 0^\circ, 90^\circ, 180^\circ$ and 270° . Denoting the average by

$$D_\psi^+(\tau_\eta) = \frac{[D_{0, \psi} + D_{90, \psi} + D_{180, \psi} + D_{270, \psi}]\tau_\eta}{4}$$

and taking the differences of two ψ -positions again, one obtains

$$\sigma_{33}(\tau_\eta) = \frac{F_{\parallel}(\psi_1) D_{\psi_2}^+(\tau_\eta) - F_{\parallel}(\psi_2) D_{\psi_1}^+(\tau_\eta) - D_0 [F_{\parallel}(\psi_1) - F_{\parallel}(\psi_2)]}{D_0 [F_{\parallel}(\psi_1) F_{33}(\psi_2) - F_{\parallel}(\psi_2) F_{33}(\psi_1)]} \quad (36)$$

where $F_{\parallel}$ and F_{33} are the stress factors of the in-plane and the normal stress component, respectively. For the quasiisotropic case, they are given by $F_{\parallel}(\psi) = \frac{1}{2} s_2 \sin^2 \psi + 2s_1$ and $F_{33}(\psi) = \frac{1}{2} s_2 \cos^2 \psi + s_1$. It should be noted that Equ. 36 yields $\sigma_{33}(\tau)$ from the difference of the lattice spacing distributions and therefore, errors due to an uncertainty in the exact D_0 -value will become of minor importance (Equ. 35). In Equ. 35 and 36, the "Laplace"-profiles of the stresses, $\sigma_{ik}(\tau) = \frac{1}{\tau} \mathcal{L}\left[\sigma_{ik}(z); \frac{1}{\tau}\right]$ are directly related to the measured strain profiles and, therefore, there is no need for describing them by means of analytical expressions.

A formalism for calculating the actual stress profiles in the (real) z -space, $\sigma_{ik}(z)$, from their discrete Laplace-transforms, $\sigma_{ik}(\tau_\eta)$, is given in /57/.

By means of the scattering vector method, depth profiles of the strains and stresses may be obtained even at grazing incidence. Therefore, the method is particularly suited for stress gradient analysis within coatings having a strong texture, if the deformation depth profiles are detected at its intensity poles. In this case, the isotropic stress factors $F_{ij}(\varphi, \psi, s_1, \frac{1}{2}s_2, hkl)$ have to be replaced with regard to the preferred crystallite orientation by the corresponding anisotropic stress factors (section 2.036c). A theoretical comparison of several approaches in X-ray stress gradient analysis with respect to their applicability to the thin layer problem is reported in /58/.

The investigation of a strongly $\langle 111 \rangle$ -textured $\text{Ti}_{0.85}\text{Cr}_{0.15}\text{N}$ -arc PVD layer on a high speed steel substrate (German grade M42) by means of the scattering vector method is demonstrated in Fig. 40, for details see /59/. A preliminary $\sin^2\psi$ -analysis revealed strictly linear D_ψ -vs.- $\sin^2\psi$ distributions up to $\sin^2\psi = 0.93$ and no ψ -splitting was observed. Further, the in-plane stress state was found to be of rotational symmetry by measurements at various azimuths φ .

The η -rotation was realised on a HUBER Ψ -diffractometer with integrated φ -table by means of a combined ω - φ - ψ -rotation. A parallel beam unit consisting of a horizontal Soller-slit followed by a LiF-analyser crystal was used to suppress the large horizontal divergence of the diffracted beam for measurements near the grazing incidence. For evaluating the depth distribution of the in-plane component of the intrinsic stresses, $\sigma_{\parallel}(\tau)$, diffraction profiles were recorded using $\text{Co-K}\alpha$ radiation after stepwise rotation of the sample around the intensity poles (311) ($\psi = 29.5^\circ$), ($3\bar{1}1$) ($\psi = 58.5^\circ$) and ($3\bar{1}\bar{1}$) ($\psi = 80^\circ$) themselves, as well as for neighbouring ψ -positions (35° , 53° , 75°). The stress profiles were determined by means of Equ. 35, for details of the procedure, see for example /56,57,58/. The texture was taken into account by calculating weighted anisotropic stress factors $F_{ij}(\psi, s_{ijkl}, hkl)$ in the Reuss approximation from the single crystal elastic compliances and the orientation distribution function.

The $D_\psi(\tau_\eta)$ -profiles obtained at the (311) and the ($3\bar{1}1$)-pole are shown in Fig. 40a, b. It should be noted that a uniform stress distribution, i.e. $\sigma_{\parallel} \neq f(\tau)$, would result in straight horizontal $D_\psi(\tau_\eta)$ -profiles, because ψ was kept fixed for the individual η -scans. In the present case, however, finite slopes are observed for the individual curves, the signs of which depend on whether the scans were performed at the low- or the large-angle side with respect to the strain-free direction of the plane-stress state within the layer. For the (311)-reflection of TiN, this direction corresponds to a ψ -value of approximately 35° . Therefore, the clear increase of the lattice spacing towards the interface, which was found for the ($3\bar{1}1$)-pole (Fig. 40b), indicates a decrease of the compressive stresses in the same direction. On the other hand, one realises from Fig. 40a that the same stress gradient leads to the opposite run of the lattice spacing in the (311)-pole at $\psi = 29.5^\circ$, whereas a nearly horizontal distribution is observed at $\psi = 35^\circ$, which corresponds to the strain-free direction of the plane-stress state.

What is striking too, is that the values of the lattice parameter at $\psi = 35^\circ$ are significantly larger than the D_0 of 0.4227 nm calculated from the corresponding values of TiN and CrN assuming the validity of Vegard's law. A triaxial stress evaluation by means of Equ. 36, however, revealed only tensile stresses up to a maximum of 200 MPa for σ_{33} , which are only

of minor importance in the present case. Therefore, the large lattice spacing should mainly be attributed to the widening effect of interstitial atoms within the crystal lattice.

Information on the stress profile with respect to the whole layer thickness is achieved by combining the $D_{\psi}(\tau_{\eta})$ -distributions in the distinct texture poles. Therefore, they are approximated by straight lines within the τ -range where they were measured (cf. Fig. 40a, b) and then, $\sigma_{\parallel}(\tau)$ is calculated using Equ. 35 for the respective depth (Fig. 40c).

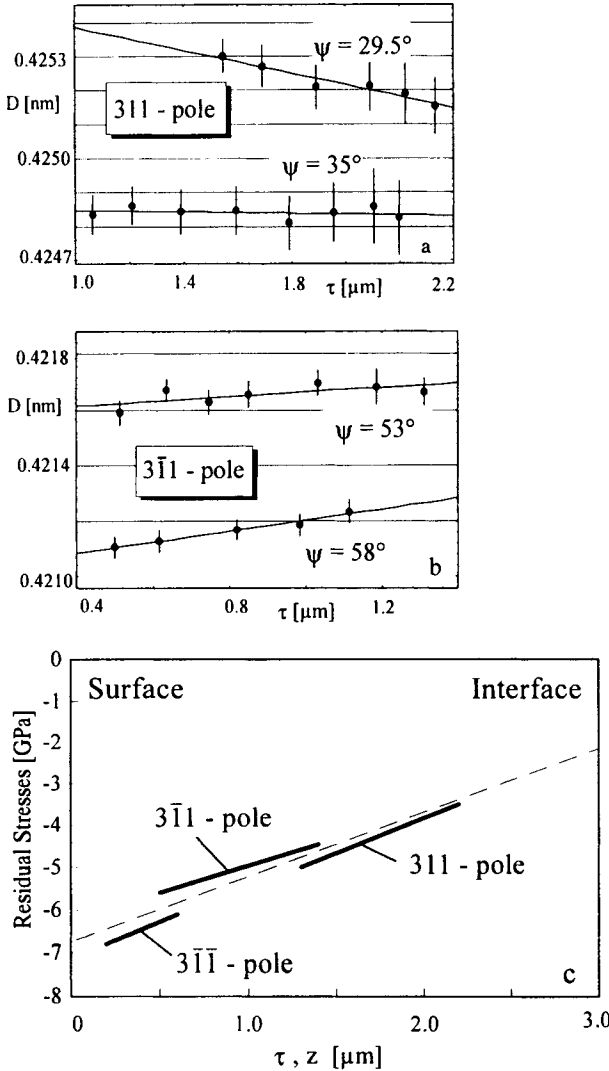


Figure 40. XSA performed for a $\text{Ti}_{0.85}\text{Cr}_{0.15}\text{N}$ -arc PVD layer (thickness $3\text{ }\mu\text{m}$) in the scattering vector mode. a) and b) depth distribution of the lattice spacing obtained at different intensity poles, c) Depth profiles of the residual stresses. Note that the linear approximation $\sigma_{\parallel}(z) = A + B \cdot z$ yields $\sigma_{\parallel}(z) = \sigma_{\parallel}(\tau)$.

2.153f Stress profiles requiring removal of surface layers or sectioning of the specimen

Measuring on one peak with one radiation and etching surface layers, is a frequently used method to evaluate RS-profiles with a not-too-steep gradient. Steel parts have been often tested in this way using the relatively low penetrating Cr-K α radiation measuring the {211} peak. Etching of surface layers and measuring on each new surface has to be done until the RS-profile reaches zero level. In many cases, the relaxed part of the RS has to be encountered. A transformation of the τ -into z-profiles is not necessary. Here, an example is given for a ground C60 steel specimen, Fig. 41 /21/.

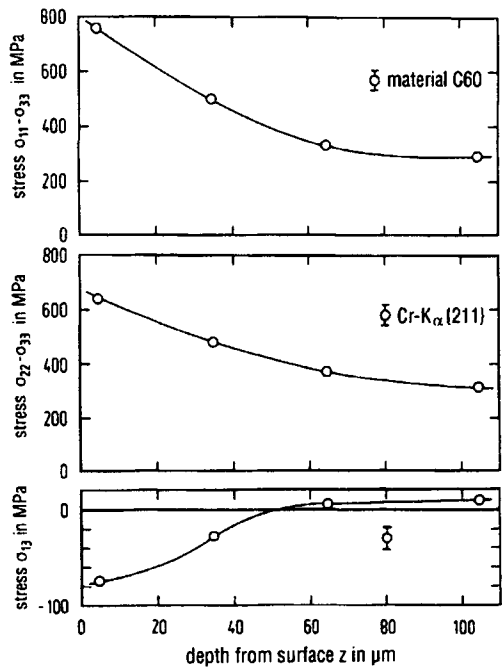


Figure 41. Residual stress distribution vs. depth from the surface of a ground C60 steel specimen showing tensile residual stresses, Cr-K α , {211} /21/.

Oblique etching shows up the RS-distribution on a slope. This was used by /60,61/ and in recent times by /62/.

In many practical cases of mechanically treated surfaces, it is necessary to do the unfolding twice. First in respect to separating RS I and RS II, and second in regard to relaxation by etching off the surface regions /63,64/.

2.153g Correction of released RS after removal of surface layers

Strain and deflection measurements after removal of surface layers.

If the stress profile has no sharp gradients, a bigger part of the surface or even the main part of the cross section has to be removed stepwise by etching or by mechanical means. The

released stresses alter the strain distribution in the remaining cross section. This will be measured by X-rays, by strain gages or by the curvature of specimen.

The mechanical methods to determine the RS-profile are numerous and well established. They are often used for comparative tests to diffraction methods because they evaluate macrostresses only. In the following, the mostly used methods on specimens with rectangular cross section will be discussed, and examples of RS-states over the cross section are given. Table 3 shows the methods to evaluate the macro-RS profiles. If the removal of surface regions is done on one side, a curvature of the specimen may be noticed; its extent depends on the ratio of removed part over the remaining part of the specimen.

Fig. 42 explains the coordinates of the specimens. Table 4 contains the details of the different methods, equations for stress evaluation and corrections as well as the authors. In Table 5 the applications, their tasks and results are collected.

Table 3. Method and location of strain measurement of RS-states. The numbers correspond to those in Table 4.

measurement		removal	
parameter	place	one side	both sides
strain	removal side	X-ray, 1	X-ray, 3
	opposite side	X-ray, 2 strain gages, 4	
curvature	inside	curvature, 5	-----

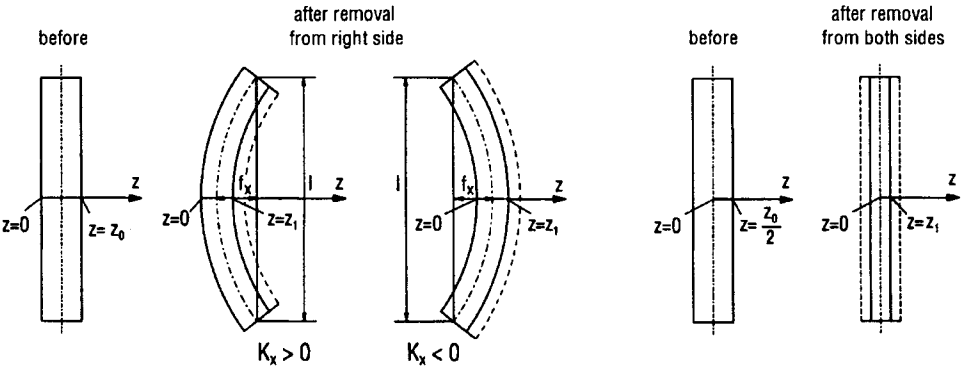
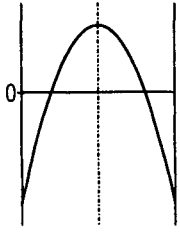
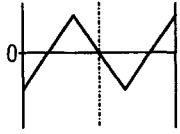
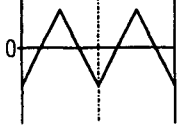


Figure 42. Coordinates of specimen for removal of one side (left) or both sides (right).

Table 4. Quantity to be determined, one or both sides removal, measuring technique, correction formula, reference. σ_x existent stress before removal, σ_{xm} measured stress after removal.

one side removal	
1 stress removal side X-ray	$\sigma_{xm}(z_1) = \sigma_x(z_1) - \frac{6}{z_1^2} \int_0^{z_1} \sigma_x z \, dz + \frac{2}{z_1} \int_0^{z_1} \sigma_x \, dz$ $\sigma_x(z_1) = \sigma_{xm}(z_1) + 2 \int_{z_1}^{z_0} \frac{\sigma_{xm}}{z} \, dz - 6z_1 \int_{z_1}^{z_0} \frac{\sigma_{xm}}{z^2} \, dz \quad /65/$
2 stress untreated side X-ray $\sigma_{xm,0} = \sigma_x(z=0)$	$\sigma_{xm} = \sigma_x(z=0) + \frac{6}{z_1^2} \int_0^{z_1} \sigma_x z \, dz - \frac{4}{z_1} \int_0^{z_1} \sigma_x \, dz$ $\sigma_x = \frac{1}{2} \left[z_1 \frac{d\sigma_{xm}}{dz} + 4(\sigma_{xm} - \sigma_{xm,0}) - 6z_1 \int_{z_1}^{z_0} \frac{(\sigma_{xm} - \sigma_{xm,0})}{z^2} \, dz \right] \quad /66/$
both side removal	
3 stress removal from both sides X-ray symmetric profile	$\sigma_{xm}(z_1) = \sigma_x(z_1) - \frac{1}{z_1} \int_0^{z_1} \sigma_x \, dz$ $\sigma_x(z_1) = \sigma_{xm}(z_1) - \int_{z_1}^{\frac{1}{2}z_0} \frac{\sigma_{xm}}{z} \, dz \quad /65/$
one side removal	
4 relative strain untreated side strain gage	$\epsilon_{xm} = \frac{1}{E} \left[\frac{6}{z_1^2} \int_0^{z_1} \sigma_x z \, dz - \frac{4}{z_1} \int_0^{z_1} \sigma_x \, dz \right]$ $\sigma_x = \frac{E}{2} \left[z_1 \frac{d\epsilon_{xm}}{dz} + 4\epsilon_{xm} - 6z_1 \int_{z_1}^{z_0} \frac{\epsilon_{xm}}{z^2} \, dz \right] \quad /66/$
5 deflection gage $k_x = \frac{2f_x}{f_x^2 + \left(\frac{l}{2}\right)^2}$ k_x curvature, f_x deflection, l chord length	$k_x = \frac{2}{z_1 E} \left[\frac{6}{z_1^2} \int_0^{z_1} \sigma_x z \, dz - \frac{3}{z_1} \int_0^{z_1} \sigma_x \, dz \right]$ $\sigma_x = \frac{E}{6} \left[z_1^2 \frac{dk_x}{dz} + 4z_1 k_x - 2 \int_{z_1}^{z_0} k_x \, dz \right] \quad /67/$

Table 5. Examples of RS-profiles and their evaluation.

RS-profiles	examples	layer removal	measurement	remarks
	polymers	one side	on removal side	in addition deflection measurements
		both sides	on both sides	specimen remains straight
		one side, thin plate	on back side	determination of micro RS and D_0
	bending bar	one side	on removal side	in addition deflection measurements
	round rod	on total circumference	on each new surface	specimen remains straight

The examples for using the formulae are taken from the RSA on the polymeric material PBT. Plates of 2.8 mm thickness were injection molded. Several samples (30×25 mm²) were carefully cut out and milled from one side to remove RS. The X-ray lattice strain measurements were done on each new surface. The RS were evaluated on the basis of linear regression and using the XEC determined in additional tests /68/. The formula 1 of Table 4 is used /65/. For n removal steps it follows:

$$\begin{aligned}
 \sigma_x(z_n) = & \sigma_{xm}(z_n) \\
 & + 2 \left[(z_0 - z_1) \frac{1}{2} \left(\frac{\sigma_{xm,0}}{z_0} + \frac{\sigma_{xm,1}}{z_1} \right) + \dots + (z_{n-1} - z_n) \frac{1}{2} \left(\frac{\sigma_{xm,n-1}}{z_{n-1}} + \frac{\sigma_{xm,n}}{z_n} \right) \right] \\
 & - 6z_n \left[(z_0 - z_1) \frac{1}{2} \left(\frac{\sigma_{xm,0}}{z_0^2} + \frac{\sigma_{xm,1}}{z_1^2} \right) + \dots + (z_{n-1} - z_n) \frac{1}{2} \left(\frac{\sigma_{xm,n-1}}{z_{n-1}^2} + \frac{\sigma_{xm,n}}{z_n^2} \right) \right]
 \end{aligned} \quad (37)$$

with

$z_1, z_2, \dots, z_{n-1}, z_n$ thickness of specimen after each removal of surface layers

$\sigma_{xm,0}, \sigma_{xm,1}, \dots, \sigma_{xm,n-1}, \sigma_{xm,n}$ residual stress measured after each removal

Fig. 43 shows the raw data and the evaluated results according to the above mentioned method /68/. To get a comparison between the X-ray results with those of mechanical tests the deflection method was also used. For this purpose twelve pieces (100 × 5 mm²) of a 2.8 mm thick plate were carefully cut out and milled to different remaining thickness. The curvatures

of the pieces were measured and the RS at this point of the cross section calculated according to formula 5 of Table 4 /67/. For n removal steps it follows:

$$\sigma_x(z_n) = \frac{E}{6} \left[\frac{z_n^2}{2} \left(\frac{k_{x,n-2} - k_{x,n-1}}{z_{n-2} - z_{n-1}} + \frac{k_{x,n-1} - k_{x,n}}{z_{n-1} - z_n} \right) + 4z_n k_{x,n} - 2 \left((z_1 - z_2) \frac{1}{2} (k_{x,1} + k_{x,2}) + (z_2 - z_3) \frac{1}{2} (k_{x,2} + k_{x,3}) + \dots + (z_{n-1} - z_n) \frac{1}{2} (k_{x,n-1} + k_{x,n}) \right) \right] \tag{38}$$

whit

E Young's modulus

$z_1, z_2, \dots, z_{n-1}, z_n$ thickness of specimen after each removal

$k_{x,1}, k_{x,2}, \dots, k_{x,n-1}, k_{x,n}$ curvature of specimen after each removal,

which is defined as (see Fig. 42):

$$\pm k_x(z) = \frac{1}{r} = \frac{2f_x}{f_x^2 + \left(\frac{l}{2}\right)^2} \tag{39}$$

r curvature radius, l chord length, f_x deflection

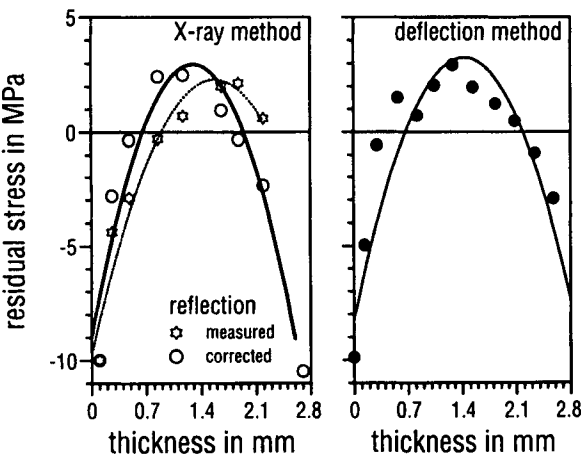


Figure 43. RS-distribution over the cross section of injection-molded PBT-plates /68/.

2.153h Correlation of different methods to determine RS-profiles

One of the largest round-robin tests to determine RS-profiles was undertaken on railway rods using X-ray, neutron-rays, ultrasonic waves, micromagnetic parameters, mechanical methods as bore-hole, ring-core and sectioning (groove). The procedure and the results were published /69/ and lead to the following conclusions: Very important for the practical usage is the knowledge of the RS in railway bars especially in daily use. The RS-state after production, joining, montage, rolling loads, abrasion, thermal loads is one of the main influences and parameters, which besides the material strength determine the lifetime. The principal RS-state of new rail bars has been often tested by mechanical sectioning methods, Fig. 44 /69/.

A round-robin test was made to study the RS-state of the running surface region of the head of a rail bar that underwent severe rolling loads in a test series. It was expected that the surface region was in a strongly inhomogeneous RS-state. To determine the RS-profile of the near-surface region it was necessary to use almost all methods of determining RS. The conclusions of this test which was elaborated by a great number of experts of different research and development institutions were drawn for the applications of all methods.

The results of the RS-values determined by the different methods for the different depths from the surface are drawn in Fig. 45 for the LD and TD. The agreement between the different measuring methods is very good.

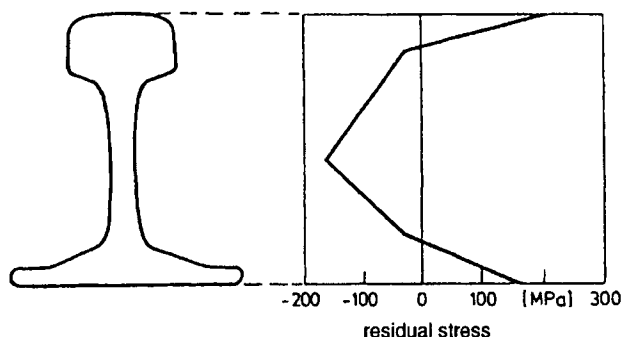


Figure 44. Longitudinal residual stresses (RS) after roll straightening of a rail UIC 60, quality A; results of mechanical tests (strain gauges, rods) /70/.

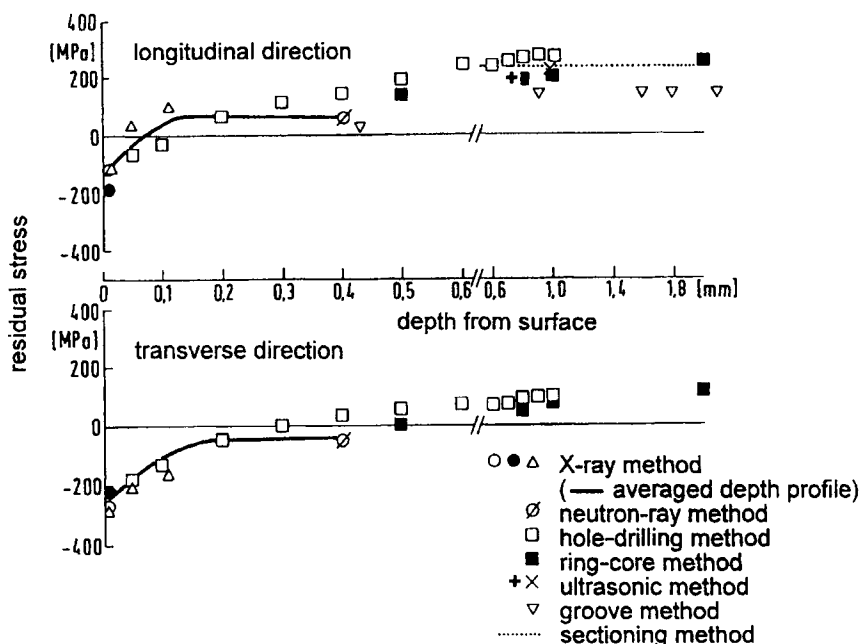


Figure 45. Comparison of the results of all used measuring methods on rail head /69/.

2.154 Recommendations

- The method for RS-evaluation with gradients should be selected with respect to the specific problem. In principle, mechanical, ultrasonic and diffraction methods can be employed.
- The choice of the method should also consider the material, the stress state and the depth zone to be analyzed.
- The selection of the method should also regard the question of proving micro-stresses. Diffraction methods probably in connection with US- or mechanical measurements offer the answer to many problems.
- In case of using X-ray methods several wavelengths and peaks should be used to get the strain/stress profile versus the penetration depth. In addition the ψ -range should be used as wide as possible ($\sin^2\psi \leq 0.9$ or even 0.95). To reach the outmost surface the specimen can be ω -tilted or grazing-incident method may be applied. The conversion $\sigma(\tau)$ into $\sigma(z)$, z the thickness direction, should be evaluated by using the Laplace transformation.
- The result of a steep stress gradient study should be analyzed by metal-physics proof. Maybe there exists a D_0 -gradient, maybe the grain size has an influence, or it is appropriate to assume a linear gradient or the stress in the very near-surface region is constant followed by a very steep gradient.
- In case of reduced measuring procedure the RS-profile may be not distinctly evaluated.
- Macro-RS have to compensate over the cross section, micro-RS within or between the phases.
- The evaluated steep RS-profiles should be verified by error consideration and modeling.
- In complicated cases, modeling of stress- and D_0 -profiles must be taken into account. Using X-ray methods as the method of the choice one wavelength should be taken in case of very steep gradients with the penetration depth in the range of the stress profile. The measured Θ -values should be converted in $D_{\{100\}}$ (in case of cubic materials and likewise for tetragonal and hexagonal materials stating the c/a value used for conversion). The $D_{\{100\}}$ values experimentally determined should be balanced versus ψ (not $\sin^2\psi$).
- If the gradient region has a thickness of 0.1 mm magnitude, measurements of the lattice strain with one wavelength at one peak can be done on the surface and after etching several layers with retesting on each new surface.
- For specimens of some mm thickness the thinning can be done in bigger steps from one or from both sides. In case of micro-RS profiles very thin plates (macro-RS small or zero) can be used. In very thick parts, sectioning is the way of choice and testing as shown above. In all these cases the relaxed stresses should be considered.
- The stress profile should be evaluated as a triaxial state, especially in case of very steep gradients on mechanically treated surfaces.
- A correction of the evaluated stress profile is necessary if a major part of the depth profile has been removed.
- A conversion of the τ -profile into the z -profile is necessary if a major part of the depth profile is radiated by the rays.
- The separation of macro-micro RS should be done by measurements on all phases or by using thinned plates.
- The additional recommendations in Table 6 and Table 7 are useful:

Table 6. Recommended X-ray method.

stress profile	used {hkl} and λ	measured from side
$\frac{z}{\tau_0} < 1$	several	one side
$\frac{z}{\tau_0} > 1$	one, etching	one or both
$\frac{z}{\tau_0} \gg 1$	sectioning, one or several	one or both

Table 7. Recommended method.

gradient, depth from surface	X-ray method	n-rays	US	mech.
1 μm	at least one λ and one {hkl}, ψ up to 88°			
10 μm	at least two λ and two {hkl}, ψ up to 70°			
100 μm	at least one λ and one {hkl}, ψ up to 70° , etching	×		
1 mm		×	×	(×)
10 mm		×	(×)	×

2.155 References

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2.16 Residual stresses after plastic deformation of mechanically isotropic and of textured materials

2.161 Historical review

The first X-ray stress analyses on specimens after plastic deformation were made at the end of the 1930s and the beginning of the 1940s: Bending /1/, tension /2/, compression /3/ tests were performed. The surprising effect was the existence of RS after plastic deformations in quasi-mono phase metals of opposite sign to the applied stress, compression after applied tension and vice versa. This was the very first sign that the X-ray method is a tool to find effects in the material characteristics and behavior other than only average LS and macro-RS. The main experimental work was done in Germany and in England/Australia. The effect is demonstrated by the very first results on a plastically elongated sample of an unalloyed steel /2/, Fig.1, first picture. Up to the elastic limit, the LS evaluated by X-rays correspond (besides the difference due to the at-that-time unknown influence of the elastic anisotropy) with the values of the specific load and Hooke's law. After passing the yield limit, the stresses determined with X-rays are smaller than the mechanical ones, and after unloading RS of opposite sign (compression) remain /4,5/. In the following, this effect was found in mono- and multiphase materials, bcc and fcc metals, Fig.1.

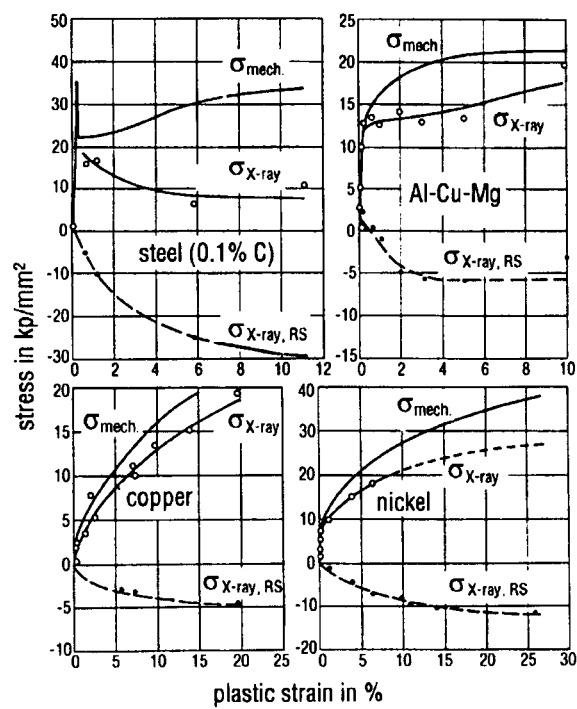


Figure 1. LS and RS of plastically elongated specimens of iron /2/, Al-Cu-Mg alloy /7/, copper /8/ and nickel /9/, summarized in /10/.

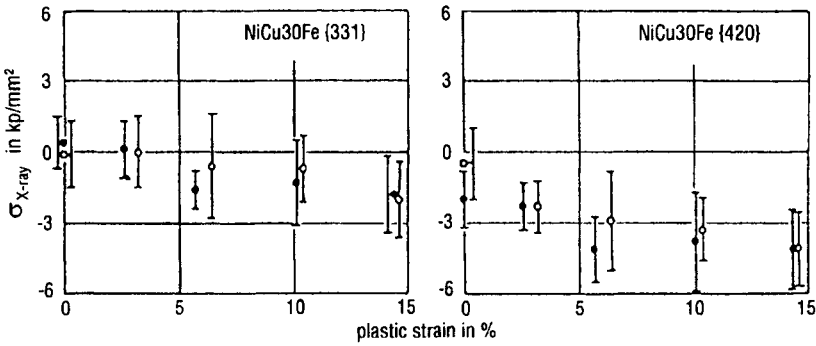


Figure 2. Residual stresses in two NiCu30Fe specimens after plastic straining. Cu-K α radiation, {331} and {420} lattice planes, /6/.

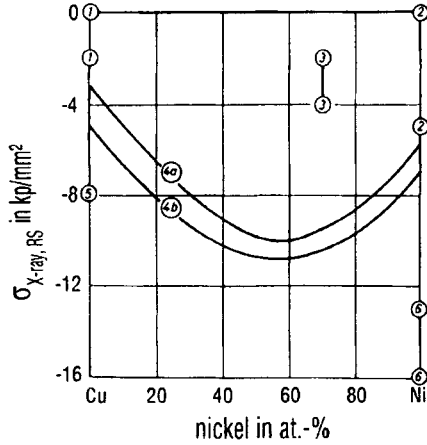


Figure 3. Residual stresses in Cu, Ni and Cu-Ni alloys after plastic straining. Results of several authors, summarized in /6/.

In most cases, the sum of the measured stresses during loading and the absolute value of the RS after unloading equals the applied load stress. Fig. 2 shows an example of a thorough study on the stress development caused by plastic straining /6/. The results of different authors on fcc Ni-Cu materials, including film and goniometer measurements, are summarized in Fig. 3, /6/. There is a big scatter of the experimental data, but it can be stated that the stresses in the surface region of fcc metals after plastic deformation are relatively small. In /6/ they were proven to be macrostresses.

It is not the intention to report and discuss all details of the enormous number of ideas and experiments that were undertaken in the following years. Here, only an outline is given with the main arguments for and against special thoughts and explanations. To decide whether the found effect is caused by macro-RS or by micro-RS, two possibilities exist: etching surface layers and measuring the lattice strain at each new surface and/or to measure the lattice strain on different lattice planes and all present phases. If the effect is a surface phenomenon the

compressive stress at the surface should be compensated by tensile RS in the core of the specimen. If only micro-RS between the crystals of different orientations are present, the RS determined on different lattice planes should compensate each other to zero.

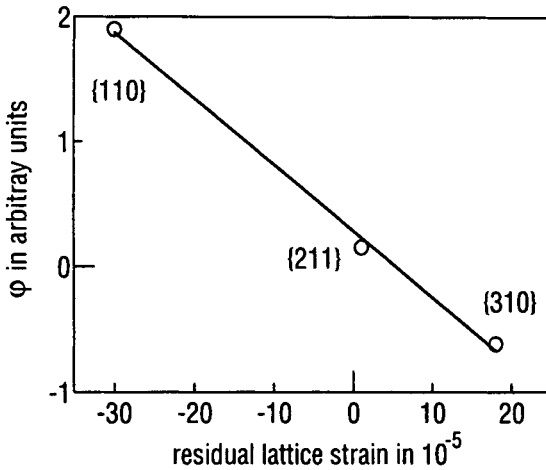


Figure 4. Lattice strains in the direction of the surface normal of a plastically elongated steel wire, dependence on the lattice plane, /16/. ϕ is the excess of the average yield tension for the grains contributing to the X-ray reflection over the mean value for all the grains in the aggregate.

In the discussion of the result for an unalloyed steel /2/, besides the easier surface deformability, a further idea should be taken into account, i.e. the reported profile of D_0 and the fact that the study was finished before reaching the final profile. The authors noticed that an alteration of the microstructure in the surface region should be taken into consideration to explain the D_0 and the very high compressive RS.

Wood et al. /11-14/ made tests with plastically deformed metals. Besides broadening of the Debye rings at perpendicular incidence of the X-ray beam, they reported permanent expansions of the lattice distance exceeding the yield limit of the materials.

Besides the macrostresses caused by surface effects and the microstresses between the present phases, the plastic deformation may create microstresses between the differently oriented crystals of the phases.

The compression-RS in plastically uniaxially elongated iron was supposed to be micro-RS firstly by Smith and Wood /15/. Greenough /16/ was the first who demonstrated the existence of microstresses between the grains of different orientations by X-ray strain measurements on different peaks ($\psi=0^\circ$) of plastically elongated iron and magnesium. Fig. 4 shows the strains of the different lattice planes perpendicular to the surface. He compared the strain values measured, for example on the lattice planes {310}, {211} and {110} of iron with the yield-stress anisotropy of the crystals, and pointed out that a system of Heyn intergranular stresses /17/ is present after plastic elongation. Calculations of the RS II were based on the Taylor theory /18-20/.

Kappler and Reimer /21/ as well as Hauk /4/ developed relations between the RS and the respective D-vs.- $\sin^2\psi$ dependences. Different further papers were published by Greenough /21a/,

Bateman /22/, Wood et al., Kappler and Reimer, Hauk, Macherauch on different bcc and fcc materials. But the experimental results did not fit in magnitude and not in the exact distribution over $\sin^2\psi$ by a factor 5 to 10 to the calculations /4/.

On two-phase materials, different RS in both phases after plastic deformation were expected and verified /23/. Ideas and proposals in the early stage of development can be found in /4,24-29/. The summary of the results and the explanation of the compensation are shown in the following Fig. 5, 6 and 7.

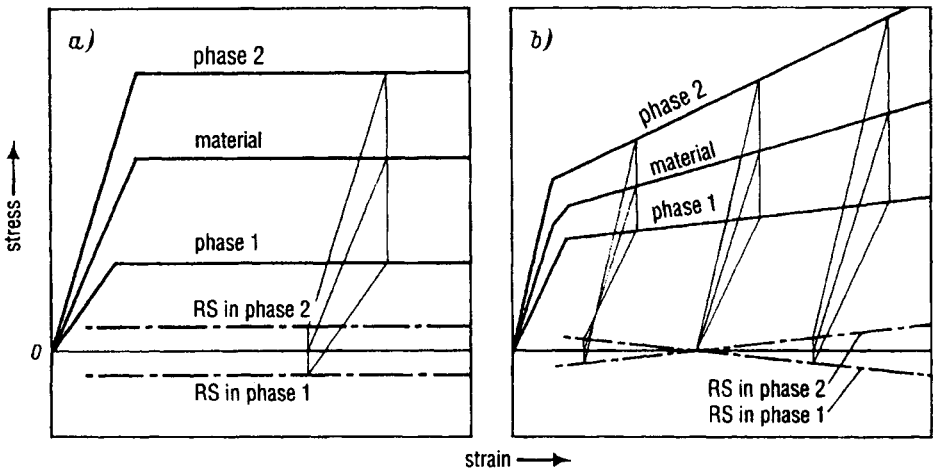


Figure 5. Origin of deformation-RS, micro-RS in the phases of a two-phase material; Influences of Young's modulus, yield point and strain hardening /23/.

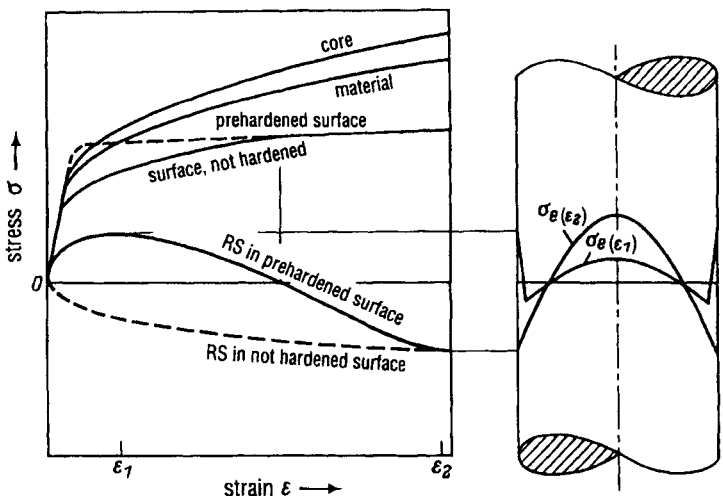


Figure 6. Origin of macro-RS by differences of strain hardening between surface and core; the influence of a pretreated surface zone /23,23a/.

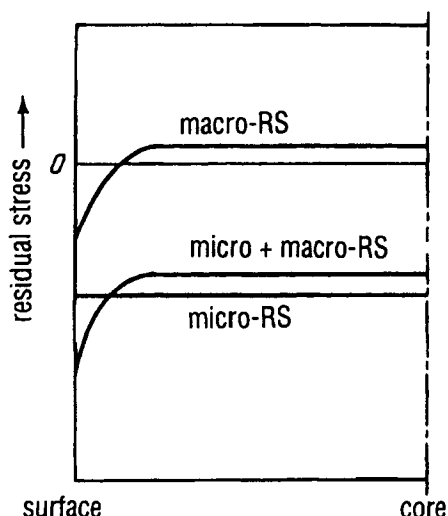


Figure 7. RS in a plastically elongated material, superposition of micro- and macro-RS. Influence of a compressive stress state at the surface region caused by a mechanical treatment or a relatively weak strength state. The micro-RS is supposed as essentially constant /23/.

In the following years a lot of experiments, now using diffractometers and counters, were made especially on different steels, after different amounts of elongations and after etching different thick surface layers. As explanation of the origin of the RS, the following were discussed: Gradient of RS, micro-RS, lattice expansion, yield stress, macro-anisotropy, influence of a second phase, subgrains, grain boundary, dislocation-structure (cells and walls), phase-stress, microstructure stress, texture. The summary of all attempts up to now is: There is a great deal of experimental knowledge on this problem of appearance, origin and development of micro-RS in single- and multiphase materials, but there is no entire theory to calculate the $\{hkl\}$ dependence and the depth profile and the compensation of the micro-RS in the surface regions of materials.

The registration of the texture state was done relatively late. That is the reason why studies of plastic deformation of textured materials as well as the development of texture and RS with plastic deformation are not often found in the literature. Some effects are known, but further tests and theoretical studies are needed. The idea that texture may be connected with the observed oscillations of $D_{\varphi\psi}$ -vs.- $\sin^2\psi$ distributions, which superimpose on the linear dependences caused by the microstresses between the phases of the material, was put forward by Hauk et al. /30-32/.

This opens a series of experiments and the connection of the two branches of X-ray studies: Stresses and texture, /33/. The results of the experiments profited from the improvement of the experimental possibilities: more precise measurements and an enlarged $\sin^2\psi$ -range up to 0.9 and using neutron rays which are able to measure also at $\sin^2\psi=1$. The development of the theoretical ideas and studies is listed in Table 1. Using the ODF, it is possible to calculate the D-vs.- $\sin^2\psi$ distribution of textured materials subjected to load stresses.

Table 1. Calculated XEC and lattice strain distributions caused by phase- and macrostresses in textured materials considering different descriptions of texture and models of crystallite coupling.

calculation	assumptions	necessary data	kind of texture	stress state	authors
lattice strain in intensity poles	homogeneous stress (Reuss)	ideal orientations		phase stress σ	/32,45/
		ideal orientations + isotropic fraction		σ	/46/
lattice-strain distributions ε -vs.- $\sin^2\psi$	Reuss	inverse pole figure		σ	/47,48, 49,50/
	homogeneous strain (Voigt)	inverse pole figure		σ	/49/
D-vs.- $\sin^2\psi$ of $\{hkl\}$, XEF(φ, ψ)	Reuss	ODF, monocrystal data	rolling texture	σ	/51-58/
	Voigt			σ	/59,54, 57,58/
	anisotropic spheres in a homogeneous matrix (Eshelby/Kröner)			σ	/60,57/
D-vs.- $\sin^2\psi$ of crystallite group, XEC(φ, ψ)	the texture is very sharp and can be described by a few crystallite groups or fiber axes	monocrystal data, description of the ideal orientations	rolling texture	crystal-lite-	/35,61, 47,62/
			arbitrary	group	/47,63/
			fiber texture	stress $\sigma(\Omega)$	/64,65/
linear alteration of the effective XEC (linear regression)	/	/	arbitrary	load stress	/66/
D-vs.- $\sin^2\psi$ being linear for $\{h00\}$ and $\{hhh\}$ lattice planes of textured cubic materials	/	/	arbitrary	σ	/67/
D-vs.- $\sin^2\psi$, XEC(φ, ψ) multiphase material	Voigt, Reuss, Eshelby/Kröner (Young's modulus can be averaged from Voigt and Reuss values)	ODF, monocrystal data	rolling texture	σ_{macro}	/68/
ε -vs.- $\sin^2\psi$ after plastic deformation, orientation dependent $\sigma(\Omega)$	Eshelby	finite number of crystal orientations	/	$\sigma(\Omega)$	/69/
	homogeneous stress, FE-analysis				/70/

Another handling of these problems was based on the idea to describe the texture by ideal orientations, oriented crystallite groups, and to determine the stresses of these groups separately /34-36/. The evaluation of LS and RS by this method was very successful and will be described in detail in the next paragraphs.

The compensation of compressive RS after uniaxial plastic elongation in quasi single phase materials, pure or unalloyed iron or steel, pure aluminium, copper, and so on, was early explained qualitatively, besides the above discussed possible origins by grain boundary areas and by dislocation walls /37-39/, Fig.8. In recent times, the authors of /40-44/ have studied this problem in great detail, especially on Cu, and showed that these RS III type are the cause of peak shifts.

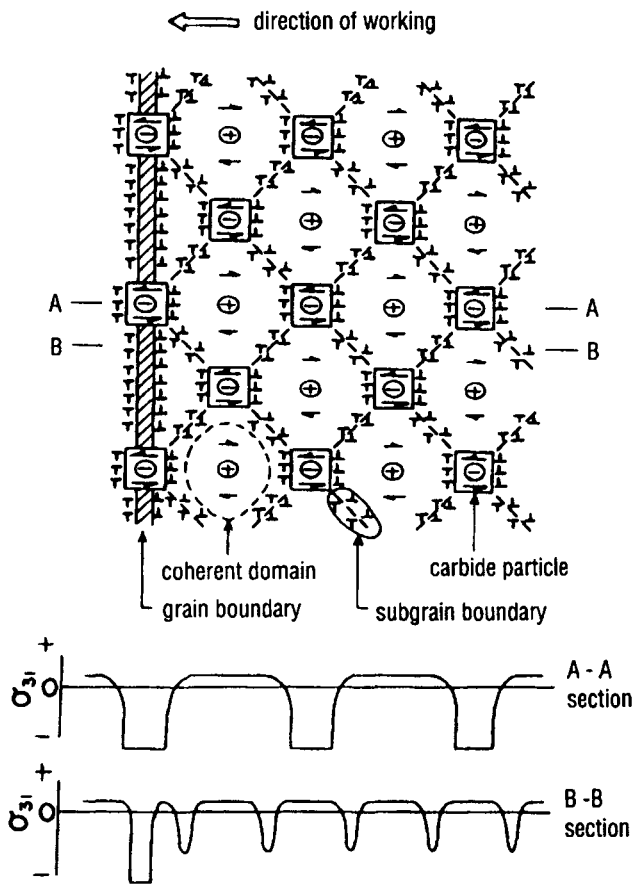


Figure 8. A model of a dislocation structure in the unidirectionally deformed sub-surface layer /38/.

2.162 Experimental results

The experimental results of studies on plastically deformed materials are numerous. In the following, the state of the art will be discussed. In former times, the state of the material was not always defined with sufficient accuracy. It should be distinguished between materials without preferred orientations and textured material, between stress free materials and those with RS. The plastic deformations were performed mostly by tension or rolled materials were tested. The texture state should be known before and after the plastic deformation.

2.162a Influence of the measuring technique on the RS-value

A special but important item in the study of the influences on the origin and development of phase specific micro-RS is the measuring technique. In early times, the $\sin^2\psi$ -range of the film method was restricted to 0.6. The extension of this range to 0.8, 0.9 and even 0.95 was only possible after the introduction of advanced designs of diffractometers. Fig. 9 and 10 as well as Fig. 40, 41 in paragraph 2.073 show D-vs.- $\sin^2\psi$ distributions of plastically deformed materials with pronounced differences between the lattice planes, oscillations and nonlinearities. It is obvious that evaluation of RS taking into account different $\sin^2\psi$ ranges will result in different stress values.

Neutron diffraction offers in this context the further advantage of measuring the D-values at $\sin^2\psi = 1$. Also the X-ray method can get D-vs.- $\sin^2\psi$ distributions over the total range by measuring at different cuts of a plastically strained bar and transforming the results into the same specimen system. Fig. 10 shows the D-vs.- $\sin^2\psi$ distributions of a practically not textured but plastically elongated duplex steel, left ferritic phase {211} and right the austenitic phase {220}.

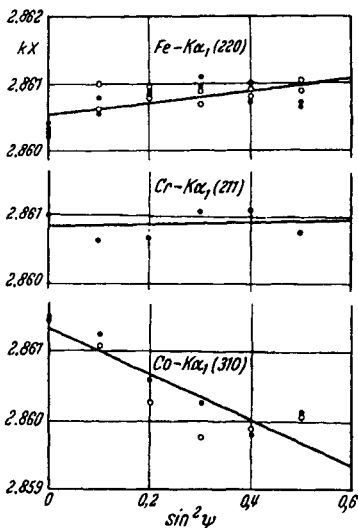


Figure 9. D-vs.- $\sin^2\psi$ distributions for three lattice planes of an unalloyed steel after 25% plastic strain [71]. The straight lines are drawn as an approximation. As it is pointed out in the publication, these distributions should not be evaluated by linear-regression analysis.

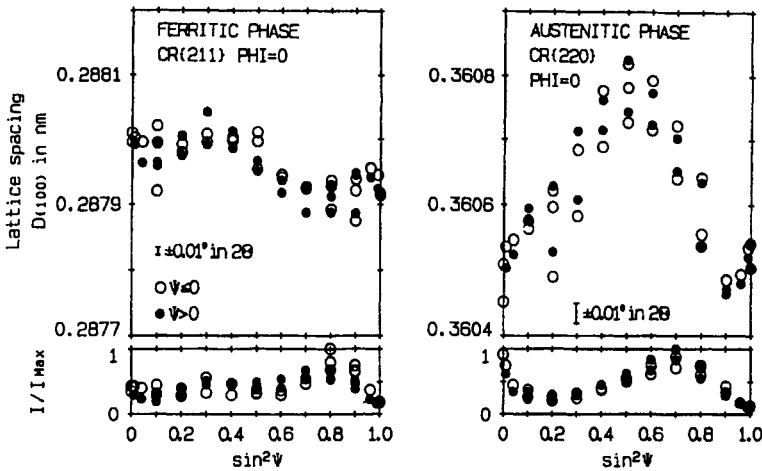


Figure 10. Lattice parameter and relative intensities versus $\sin^2\psi$, determined by X-rays on a 12% plastically elongated duplex steel (X2CrNiMoN22-5). The measurements were performed on different cuts of the specimen and the results transformed to the specimen system /72/.

2.162b The RS-state over the cross section, the compensation problem

The questions of the nature of the RS and the compensation over the cross section and/or between the phases were discussed already years ago /71,73/. From today's standpoint of research the following can be explained using Fig. 7 /23/.

We distinguish between quasi-single-phase and multiphase materials. Besides influences of the outmost surface by its lower strength compared to the bulk material, which results in compressive macro-RS, the micro-RS in the weaker phase are compressive and tensile in the stronger one. Generally no gradient is observed but the presence should be studied.

Besides effects like the mentioned weaker surface region there may be remaining RS from mechanical surface treatments or altered microstructure of diffusion origin.

Searching for compensation of the observed RS one has to take the volume concentration of the phases into account. This compensation holds for the average RS; no specific relation between lattice planes or crystal orientations are yet known. The amount of the RS is in most cases proportional to the heterogeneity, Fig. 11 /74/, Fig. 12 /75/.

The RS-state of uniaxially elongated materials, quenched and tempered steel and a duplex steel were determined on different cuts of the plastically strained specimens /72/. The results are that the deformation RS-state is in the core of the quasi-single-phase specimen compressive in the strain direction with a very small tensile component in the transverse and the thickness direction, Fig. 13, /72/. The amount of RS in transverse and normal direction may be larger in materials with very fine grained structure, for example at the ground surface of a steel sample, Fig. 14 /72/ or in pearlite, as reported by /75a/. The D-vs.- $\sin^2\psi$ distributions for the ferritic and austenitic phases of a duplex steel measured on different cuts through the core agree very well after transformation into the specimen system of co-ordinates, Fig. 10.

The micro-RS profile (average, measured on different peaks {hkl}) of fcc materials is in most cases characterized by a steep gradient of compressive stresses followed by a constant level of a RS of small size. The niveau of RS is different for different crystallite groups, Fig. 14a. The respective tensile stresses necessary for the compensation of the observed compressive RS were supposed /37-39/ and now proved /40-44/ to be at least partly located in the dislocation cell walls.

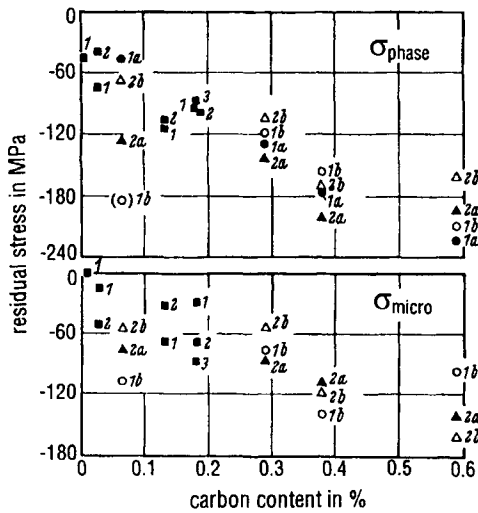


Figure 11. Dependence of macro- and micro-residual stresses on the carbon content of about 8% plastically deformed steels; results of several authors /74/.

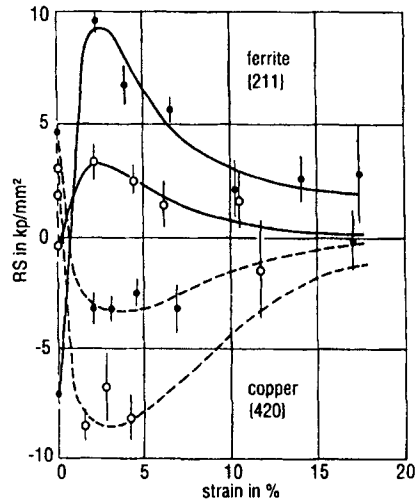
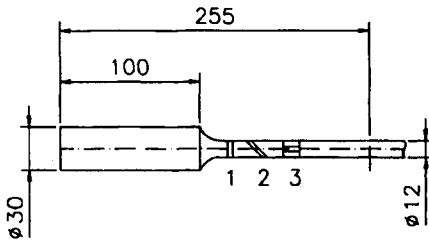


Figure 12. Phase stresses in two Cu-Fe sintered materials, dependent on the plastic deformation. Determination with X-rays on the Fe{211} and the Cu{420} lattice planes /75/.



specimen	stress tensor in MPa		
1	$\begin{pmatrix} -197 \pm 15 & 11 \pm 12 & -3 \pm 5 \\ & 8 \pm 31 & 0 \\ & & 7 \pm 25 \end{pmatrix}$		
2	$\begin{pmatrix} -160 \pm 20 & 1 \pm 4 & -37 \pm 4 \\ & 22 \pm 16 & -1 \pm 4 \\ & & 15 \pm 6 \end{pmatrix}$		
3	$\begin{pmatrix} -159 \pm 24 & 0 & 3 \pm 7 \\ & 15 \pm 24 & -5 \pm 7 \\ & & 15 \pm 13 \end{pmatrix}$		

Figure 13. Stress tensors determined on specimens 1, 2 and 3 of a 13.8% plastically deformed round tensile-test specimen of 25CrMo4 steel. The respective measured values are converted into the specimen system. Mo-K α , {732+651} lattice plane, /72/.

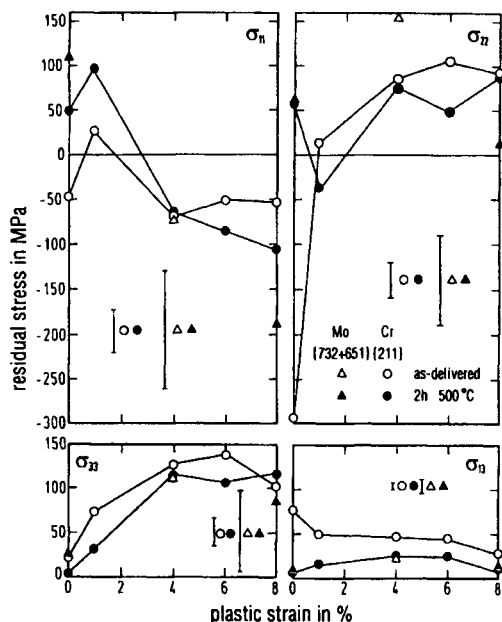


Figure 14. Residual stress components σ_{11} , σ_{22} , σ_{33} and σ_{13} determined on 54NiCrMoV6 samples for different plastic strains. The data were measured at the ground surface of the as-delivered material and after annealing it for 2 h at 500°C using Mo- K_{α} radiation ($\{732+651\}$ peaks) and Cr- K_{α} radiation ($\{211\}$ peaks) /72/

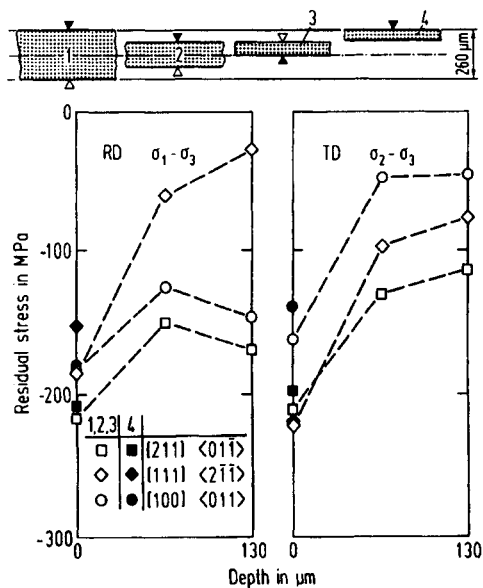


Figure 14a. Residual stress distribution versus the depth for three crystallite groups of a 88%-cold-rolled unalloyed steel in the rolling (RD) and the transverse (TD) direction, /36/.

2.162c Compensation of the phase-RS in multiphase materials quantitatively

The micro-RS between the phases of a plastically elongated material was quantitatively studied on Fe-Cu-sintered specimens /75/. The RS in both phases were determined after fabrication and their depth profiles after plastic uniaxial deformation, Fig. 15. Plastic elongation causes the RS to change from their initial values in the as-delivered state associated with different thermal expansion coefficients. With increasing plastic strain, the deformation-RS first increase, then reach a maximum and finally decrease again. The observed RS compensate each other.

The explanation of the origin of the RS will follow a two-crystal model /26/ and is based on the effect of yield strength, strain hardening, elasticity moduli /74/. The RS will be calculated according to the formulae

$$\sigma_{RS,Fe} = \sigma_{\epsilon,Fe} - \frac{E_{Fe}}{E_m} \sigma_{\epsilon,m} \qquad \sigma_{RS,Cu} = \sigma_{\epsilon,Cu} - \frac{E_{Cu}}{E_m} \sigma_{\epsilon,m}$$

As the specific model demonstrates /75/, the RS in both phases diminish with plastic elongation. The equations describing the effect and the compensation are the basis of the separation of the macro- and micro-RS.

According to the importance of the steels and here, the Fe-Fe₃C system, the search for the RS in the cementite phase was undertaken early /4/. The small amount of Fe₃C made it difficult to measure the strain, especially with the necessary accuracy. This was possible later after introduction of the diffractometer for measuring strains /45/, Fig. 16. The compensation of the compressive RS in the ferrite phase by the tensile RS in the cementite phase was sometimes examined /76, 77/.

It is obvious after all what has been said, that the compressive RS in the ferrite and consequently the tensile RS in the cementite phase depend on the content and shape of the cementite particles.

2.162d Peak dependencies

Plastic deformation of multiphase materials causes constraints between the phases that would result in linear D-vs.-sin²ψ dependences of the lattice planes in each non-textured phase. But the constraints between the crystals within the phases may superimpose nonlinearities that depend on the lattice plane under investigation.

Table 8 in section 2.073a shows schematically the nonlinearities - oscillations of the D-vs.-sin²ψ distributions for different lattice planes from experience.

In most cases the distributions are different for textured materials with superimposed elastic strains, in elastic isotropic materials after plastic uniaxial or two-dimensional deformed (predominantly rolled) materials.

The results for bcc and fcc materials resemble one another qualitatively and the differences between single-phase and multiphase materials are small. The specific distributions in relation to the lattice plane may be understood by calculations if the texture is known and no influence of plastic deformation is present. An explanation of the nonlinearities caused by plastic straining could be given for special distributions but is still missing for the whole variety of observed examples. But the gathering of experimental studies and bringing the typical appearances into the systematic order should be completed as early as possible.

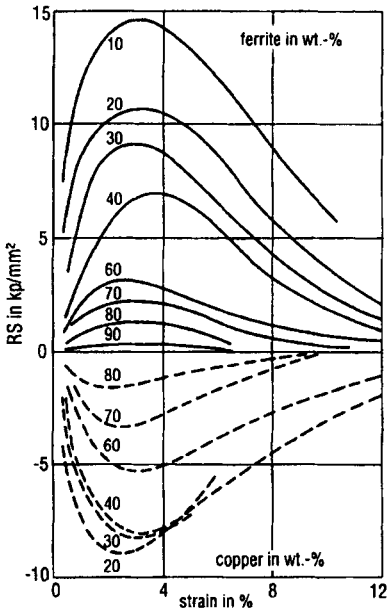


Figure 15. Stresses in the two phases of Fe-Cu sintered material after plastic elongations, determined with X-ray diffraction /75/.

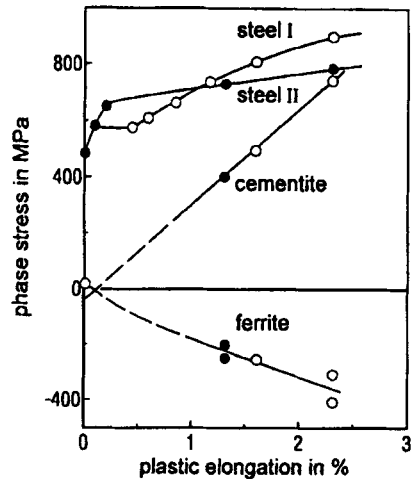


Figure 16. Development of stresses in the cementite and the ferrite phase with plastic deformation; measurements on two steels mechanically under load and by X-rays after unloading, /45/.

2.162e Strain hardening – RS

The questions about the value of RS after plastic deformation and the relation to characteristics of the material were early put forward. It should be stated again that the problems are not solved in general. Here are some experimental results.

Already in the very first test /2/ the authors demonstrated that $\sigma^{X-ray} - \sigma^{RS} = \sigma^{mech.}$ is valid or the absolute value of RS equals the difference $\sigma^{mech.} - \sigma^{X-ray}$ (the strain hardening). Results of a joint test on several unalloyed stress-free heat-treated steel samples showed that after plastic elongation, compressive micro-RS are present which increase with increasing C-Fe₃C-content. Surface effects are of minor influence /74/.

Studies on fcc-metals showed small values of RS after plastic deformation /6/ and no indication of surface effects as long as no second phase is present with a certain content /79/.

A systematic thesis of this problem-circle was performed by /78/ using different C-steels as well as tensile, compressive and bending plastic deformation. Some of the essential results are displayed in Fig. 17, 18, 19. The figures in conjunction with the captions are self-explaining. They demonstrate the differences between tensile and compressive loads but show the above observed relationship $\sigma^{X-ray} - \sigma^{RS} = \sigma^{mech.}$ for the total plastic deformation of approximately 5 %. It is an open question, whether this will be true for other steels and for larger plastic deformation.

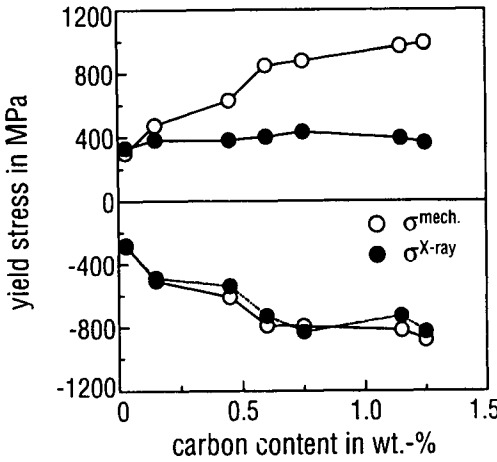


Figure 17. Yield stress $\sigma_{\text{eff}}^{\text{mech.}}$ and $\sigma^{\text{X-ray}}$ for tensile and compressive loading ($|\epsilon_{\text{pl.}}| \approx 5\%$), as a function of the carbon content, /78/.

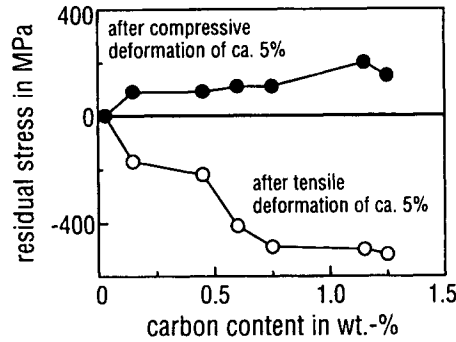


Figure 18. Residual stresses determined by X-rays after tensile and compressive loading ($|\epsilon_{\text{pl.}}| \approx 5\%$), as a function of the carbon content, /78/.

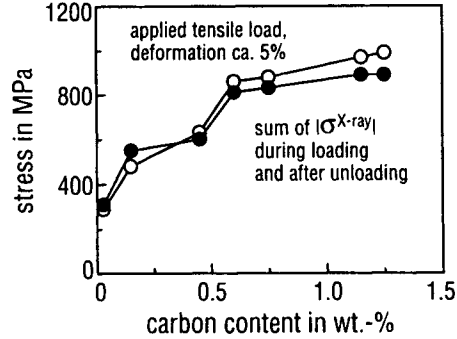


Figure 19. Yield stress $\sigma_{\text{eff}}^{\text{mech.}}$ as well as the sum of the X-ray yield stress $\sigma^{\text{X-ray}}$ and of the residual stress $|\sigma^{\text{RS}}|$ for tensile loading ($|\epsilon_{\text{pl.}}| \approx 5\%$), versus the carbon content, /78/.

2.162f Further experimental results

The RS after plastic elongations of the specimen of an unalloyed steel will have the opposite sign to the previous applied load: compression after tensile straining and vice versa. Another effect is the fact that the oscillations of D vs. $\sin^2\psi$ of a textured and plastically strained steel are different for different azimuth. Fig. 20 /80/ shows the D -distributions for a St52 specimen that was 75 % cold rolled and 4.4 % plastically elongated. The D -vs.- $\sin^2\psi$ distributions depend on the azimuth and show oscillations and ψ -splitting.

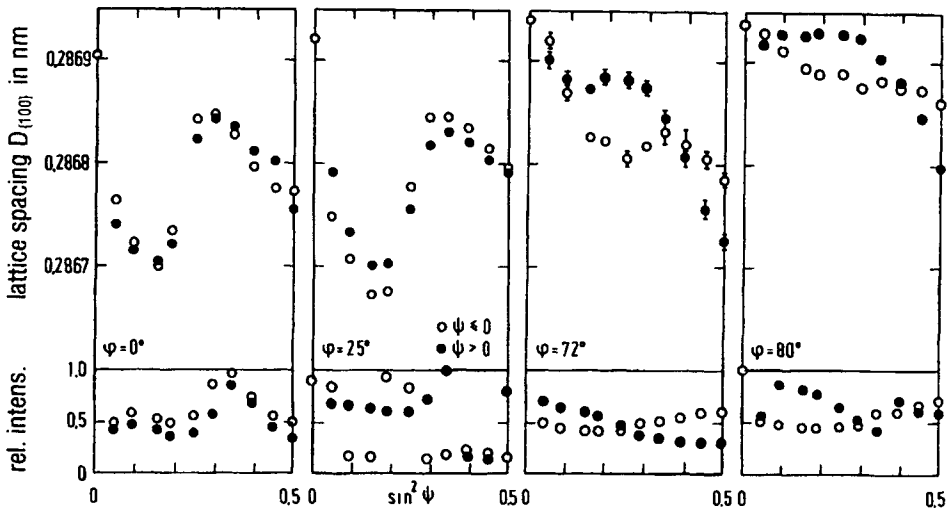


Figure 20. Distributions of the intensity and the lattice distances for a 4.4% uniaxially deformed steel, dependence on the azimuth φ and of the measuring direction, Cr-K α_1 , $\{211\} / 80^\circ$.

Theory demands that $\{h00\}$ and $\{hhh\}$ peaks will show no oscillations in textured materials /67/. This is true only if textured materials are loaded with elastic strain only. However when plastic elongation is applied, especially the $\{h00\}$ peaks show strong oscillations, see Table 8 in section 2.073a. One example is given in Fig. 40 /72/ of paragraph 2.073, where the D-vs.- $\sin^2\psi$ distribution for the $\{200\}$ peak of a plastically strained heat treated 25CrMo4 steel shows a nonlinear dependence measured by X-rays and by neutron rays. Another example is taken from /81/ for the $\{200\}$ peak of α -brass. The oscillations in the RD are pronounced and get very obvious when the differences ΔD -vs.- $\sin^2\psi$ are displayed, Fig. 21. The reason for that is the influence of the plastic anisotropy.

In /82/ the strain distributions of the $\{211\}$ lattice plane of an unalloyed steel after tensile and compressive deformations were studied. The deviations from the initial distributions are opposite in sign but very small, Fig. 22. Note that the ΔD -scale is magnified by a factor 10.

Another example is plotted in Fig. 53 of section 2.073f as a comparison of calculated and measured RS in a rolled unalloyed steel /83/. The calculation was done based on the biaxial RS-state, the texture state and the Reuss model. The experimental findings show additional oscillations. It is today not possible to separate quantitatively the influences of texture and of deformation on D-vs.- $\sin^2\psi$ distributions.

A further surprising effect is that elastically isotropic or nearly isotropic materials (W, Mo, Al) show oscillations on a high level, Fig. 23, /83,61/.

The transition region between elastic and plastic strain and their representation in lattice strain under applied loads has been studied by /84/ using neutron-rays. The material was a duplex steel with a ferritic- and austenitic-phase, 50 vol.% each. The Fig. 9, section 2.123a explains the results: In the elastic range the measured lattice strain demonstrates the influence of anisotropy. With increasing load, the strain of some peaks deviate from the linear response. However, at relatively high loads the strains deduced from different peaks still split according to the dependences given by the influence of elastic anisotropy (orientation parameter Γ).

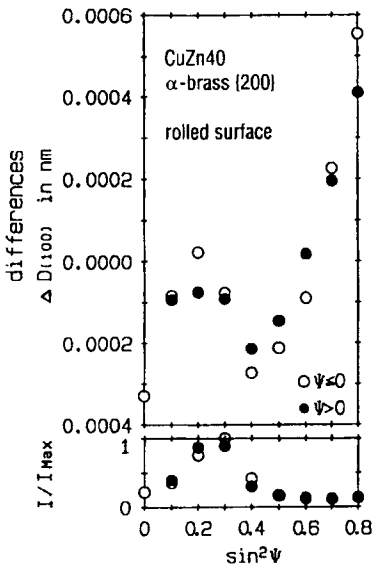


Figure 21. Differences ΔD vs. $\sin^2\psi$ for α -brass after and before a uniaxial elongation of about 0.2%, {200} lattice plane, /81/.

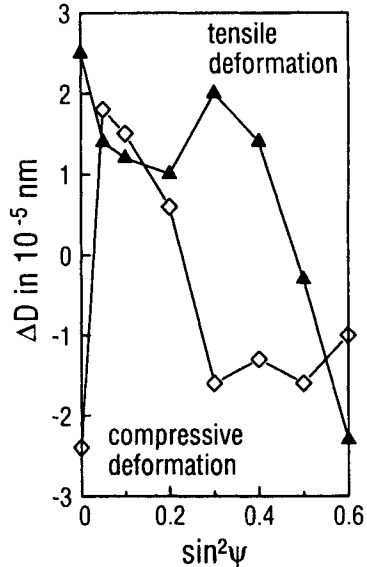


Figure 22. Alterations of the {211}-lattice-distance distributions of an unalloyed steel caused by tensile and compressive plastic deformation. Note that the ΔD increments are only $1 \cdot 10^{-5}$ nm, e.g. the effect on the {211} planes is small. Experimental data were taken from /82/.

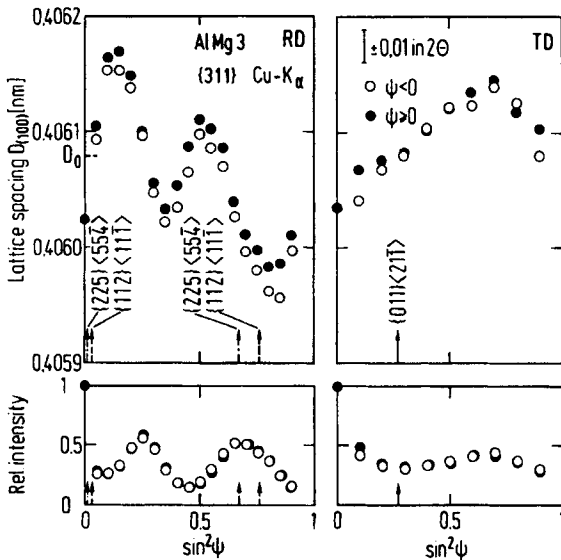


Figure 23. $D_{\{311\}}$ -vs.- $\sin^2\psi$ distribution of cold rolled AlMg3 after reduction of the thickness by chemical etching (50%). The poles of the main crystallite groups are marked /83,61/.

2.162g Systematic tests

Table 2 summarizes schematically the various shapes of D-vs.-sin²ψ distributions originated in nontextured materials by macro- and micro-RS in differently deformed regions of the material /75a/.

Table 2. Deformed regions of nontextured materials and their D-vs.-sin²ψ distributions by macro- or micro-RS.

differently deformed regions	retained residual stress	shape of D-vs.-sin²ψ distribution
treated surface / bulk	macro-RS σ^I	linear
phases	micro-RS $\langle \sigma^{II} \rangle$	linear
differently oriented grains	micro-RS $\langle \sigma^{II}(g) \rangle$	nonlinear, dependent on {hkl}
dislocation-cell structures	micro-RS $\langle \sigma^{III} \rangle$	linear

The appearance of linear and oscillatory D-vs.-sin²ψ distributions should be related to the different material treatments, to the LS in the elastic and in the plastic range, and to the RS after plastic elongation. Differences ΔD between two material states are characteristic for the respective treatment.

Table 3 shows the material on which the specific effects were demonstrated, see also /80/. The single-phase material should be understood as a material with practically only one phase and not a material with 3 or 5 nine purity. The dual or multiphase materials are divided into those where the second phase could not be measured and those where the stresses of both phases can be evaluated. Examples are disposed in the following figures.

Table 3. Examples of textured and nontextured materials on which the different elastic and plastic deformations were tested and evaluated.

1	2	3	4	5	6	7
material structure	texture	stress state as delivered	+LS	+LS	unloaded after 5	6-3
		RS	+ε _{el.}	+ε _{pl.}	RS	ΔD
single phase	nontextured	XEC determination (numerous publications)				
	textured	unalloyed steel 1.0370, X2CrNi18-12 /57,55/				
dual phase, <10% second phase	nontextured				25CrMo4 /72/	
	textured				54NiMoCr /85/	
>10% second phase	nontextured	X22CrNiMoN22-5 /86,87/				/88/
	textured	CuZn40 /86,87,57/				/88/

Fig. 24 and 25 show the development of the D-vs.- $\sin^2\psi$ distribution of two-phase brass and a duplex steel with the applied load. The differences between the state after about 0.2 % deformation and the initial state is plotted in Fig. 26. Especially the behavior of the $\{220\}$ lattice planes of the fcc phases resemble each other but also the $\{220\}$ planes of the bcc ferrite phase, Fig. 27.

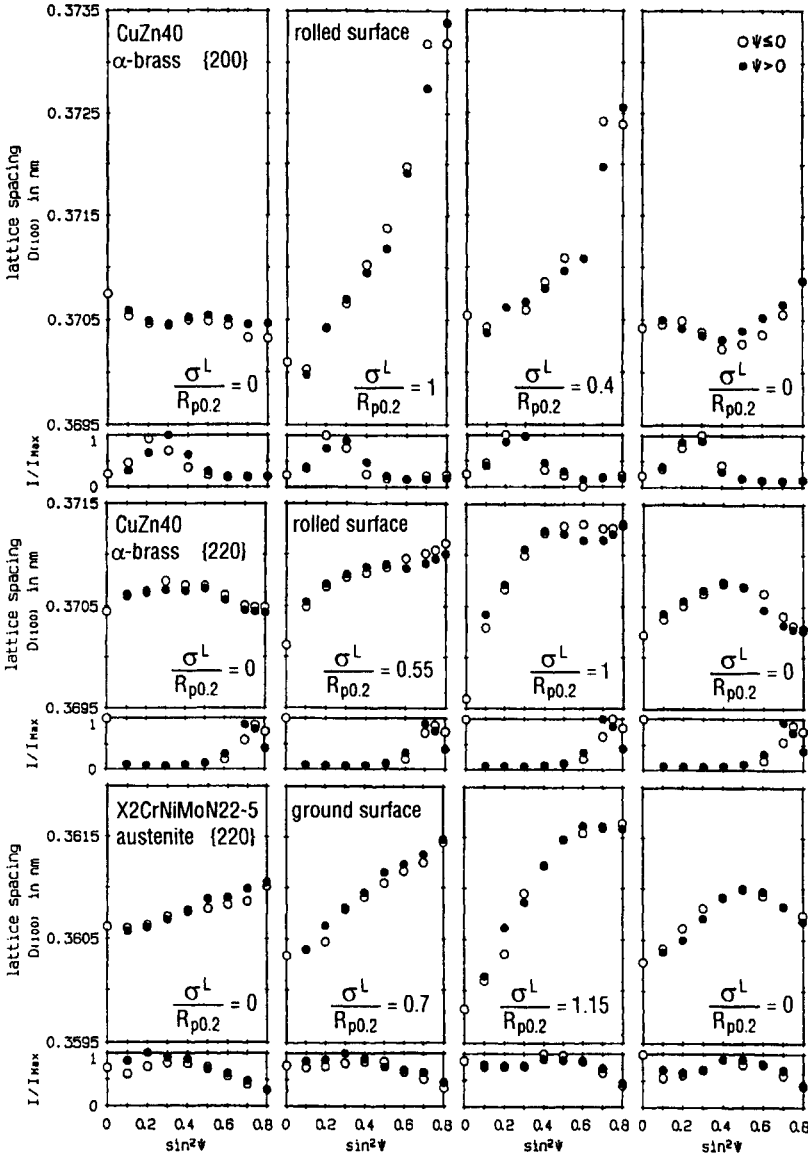


Figure 24. D-vs.- $\sin^2\psi$ distributions for different lattice planes and materials before and after uniaxial plastic straining (left and right pictures, respectively) as well as during loading, /88/.

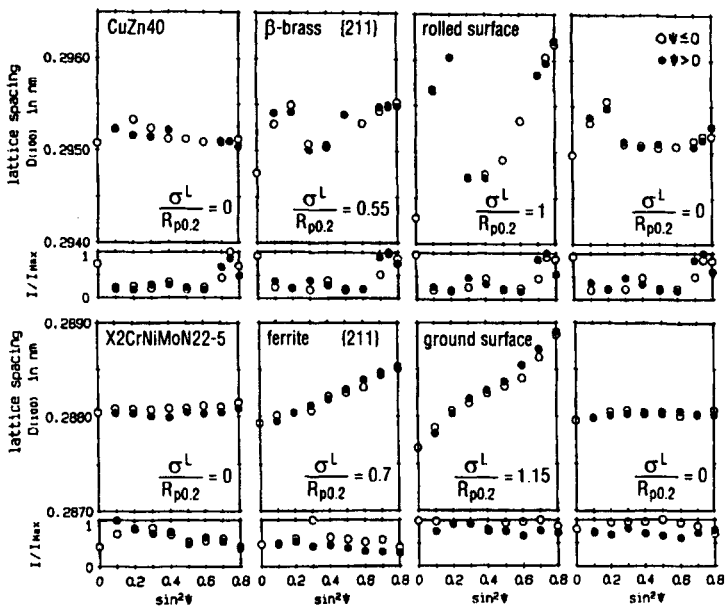


Figure 25. D-vs.- $\sin^2\psi$ distributions for different lattice planes and materials before and after uniaxial plastic straining (left and right pictures, respectively) as well as during loading, /88/.

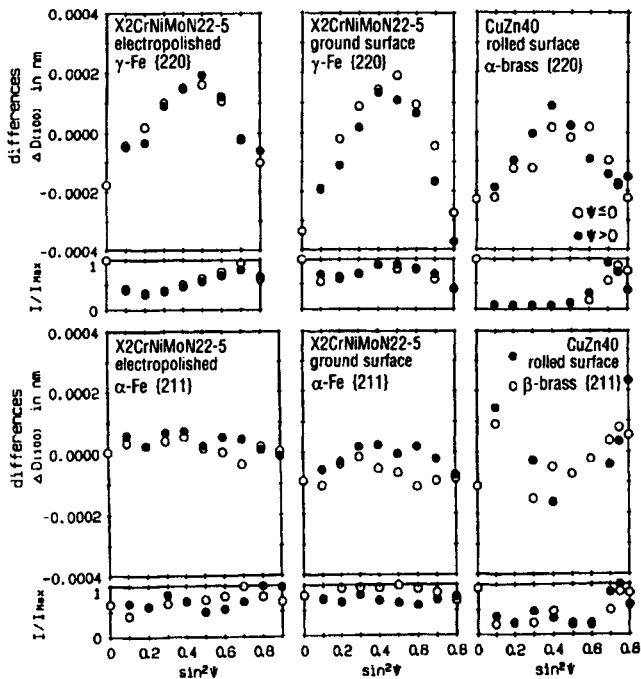


Figure 26. Differences between the D-vs.- $\sin^2\psi$ distributions after and before plastic strain, /88/.

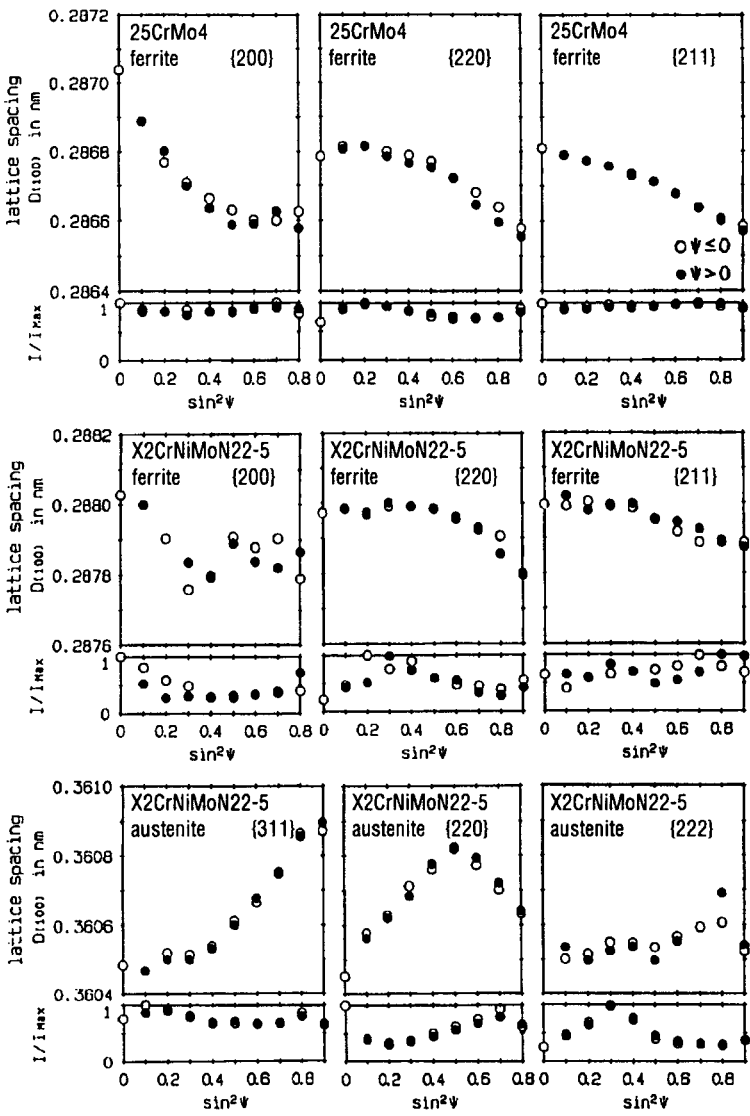


Figure 27. D-vs.- $\sin^2\psi$ distributions for different lattice planes of the ferritic steel 25CrMo4 and the ferritic-austenitic steel X2CrNiMoN22-5 after uniaxial plastic deformation of 8% and 12%, respectively, /72,88/.

2.162h Deformation stresses in polymeric materials

Stress evaluation on polymeric materials is a new branch in the field of load stresses and residual stresses determined by X-rays. This is because many polymeric materials are amorphous. Furthermore, their strengths are very low in comparison with metallic materials, and the necessary accuracy of lattice-strain measurements seems not adequate. Both problems were solved. In /89/ metallic powder in epoxy-carbon laminates was used as the crystalline phase. The peaks in the back-reflection zone have the necessary sensitivity. The peaks of α -Polypropylene (PP) in the front-reflection zone were used for lattice-strain measurements in /90/. The low Young's moduli of polymers allow one to achieve an accuracy of the RS better than that for metallic materials. Table 4 contains the polymeric materials that have been mostly tested in the form of plates, pipes or laminates /91/. All the polymeric phases as well as the added phases for the measurements are indicated there.

To study the effect of fillers, Al powder was added to the PP granules before injection molding of plates. Specimens were submitted to external loads and the lattice strains of the crystalline α -PP phase as well as of the Al powder were measured, Fig. 28 /92/. Unloading left no RS in the PP. The powder takes two times the external stress in the load direction and -0.3 times perpendicular to that direction. As a further effect on the embedded metallic powder in the polymeric material, a considerable part of micro-RS is observed, Fig. 29 /93/. This has consequences for using powder as filler or as a crystalline phase in polymeric products.

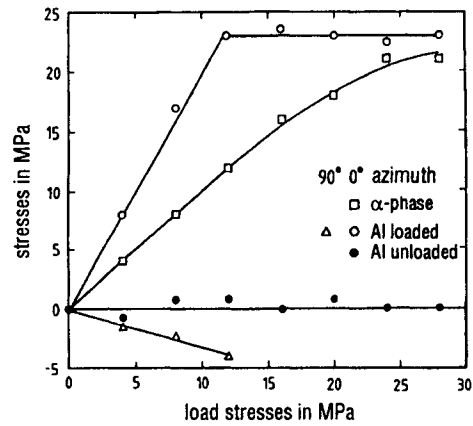


Figure 28. Dependence of the stress determined by X-rays on the applied load stress; injection molded plate of Polypropylene filled with 10 m% Al, /92/.

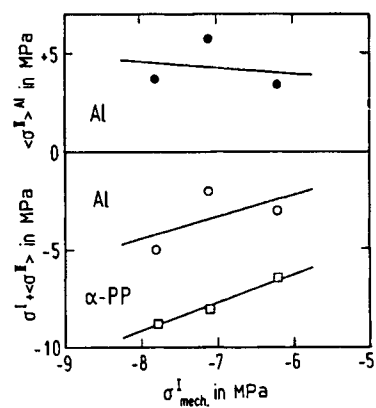


Figure 29. Stress determined by X-rays and evaluated micro-RS; injection-molded plate of Polypropylene filled with 10 m% Al, /93/.

In /94,95/ the behavior of PBT, filled with glass spheres as well as short glass fibers, during external loading was studied. Fig. 30 shows the dependence of the strain on the applied load. The strains alter dramatically at LS of more than 30 MPa in the case of fiber-filled PBT. The reason for that is the plastic flow in the matrix, probably combined with debonding effects. Fig. 17, left, in section 2.123b shows the development of the stress in the crystalline part of PEK-material with the applied load /96/. The respective dependence for a carbon-fiber-

reinforced PEK is given in Fig. 17, right, section 2.123b. Fig. 31 shows the RS-distribution of fiber-reinforced polyetherketone with large amount of micro-RS /96/.

Further experimental tests should be performed with filled semicrystalline polymers in which the particles are crystalline to measure their strains and to be able to study the compensation.

Table 4. Residual- and load-stress studies in polymers, measured and not measureable phases.

material	X-ray method		mechanical method	manufacture and structure-parameters	load stress	references
	measured phase	not measurable phase				
Polymethylmethacrylat (PMMA) + Al, Ag	Al, Ag	100 vol.% amorph.			×	/97,98/
Epoxy C-laminate + Al, Ag	Al, Ag	100 vol.% amorph.			×	/98,89,99,100/
Epoxy C-laminate + Nb, CdO	Nb, CdO	100 vol.% amorph.			×	/100,101,102,103/
Polystyrol (PS) + Al HI-PS + Al	Al	100 vol.% amorph.	×	×	×	/104/
Polypropylene (PP)	α-PP	55 vol.% amorph.	×	×	×	/90,105,92,91,106,107/
PP + Al	α-PP, Al	55 vol.% amorph.	×		×	/93/
PP + CaCO ₃	α-PP, CaCO ₃	55 vol.% amorph.			×	/108/
Polyethylene (PE)	PE	30 vol.% amorph.			×	/106,107/
PE + Al	PE, Al	30 vol.% amorph.			×	/108/
Polybutylenterephthalate (PBT)	PBT	70 vol.% amorph.	×	×	×	/109,94/
PBT + glass sphere	PBT	70 vol.% amorph., glass sphere			×	/94/
PBT + glass fiber	PBT	70 vol.% amorph., glass fiber			×	/95/
PEEK + C-fiber	PEEK	70 vol.% amorph., C-fiber			×	/110/
Polyetherketone (PEK)	PEK	70 vol.% amorph.			×	/111,112,96/
PEK + C-fiber	PEK	70 vol.% amorph., C-fiber			×	/111,112,96/
PEK + glass fiber	PEK	70 vol.% amorph., glass fiber			×	/96/

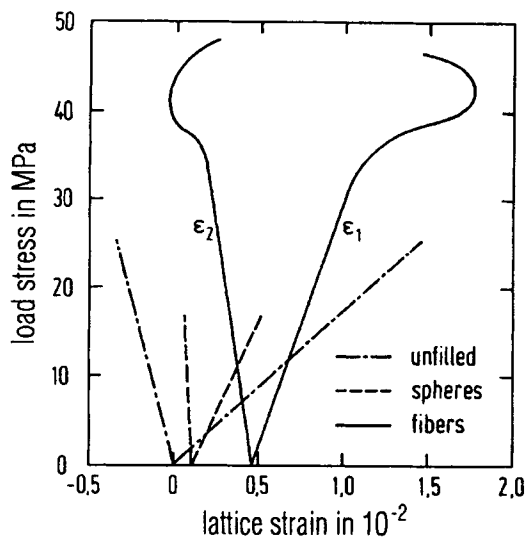


Figure 30. Lattice strain versus load stress; pressed PBT plates unfilled and filled with 22/21 vol.% glass spheres / glass fibers, /94,95/

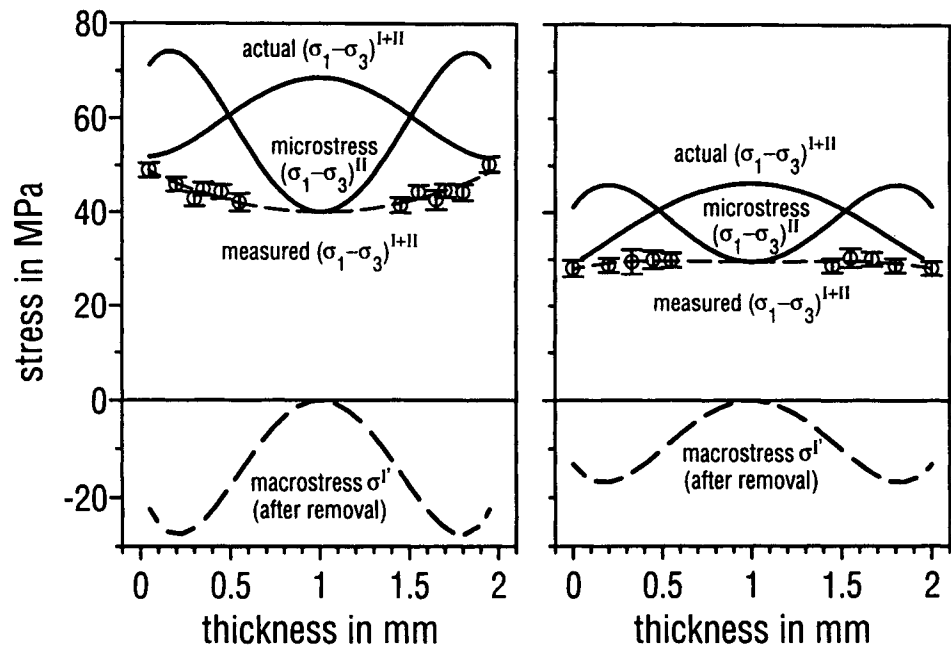


Figure 31. Distribution of residual stresses over the cross section of an injection-molded PEK plate evaluated by mechanical and X-ray methods. Left: reinforced by 20 m.% C-fibers, right: reinforced by 30 m.% glass fibers /96/.

2.163 Theoretical studies

The first attempt to calculate the deformation RS of kind II was done by Greenough /16, 20/. The basic idea was to elaborate the stressed microstructure after plastic deformation according to the orientation dependent yield stresses of the different crystallites. Experimentally, it was demonstrated for iron, magnesium, aluminium, copper and nickel /19/. The details of theoretical thoughts were put forward for $\epsilon_{\psi=0}$, the residual strain perpendicular to the applied stress, in /19/. Following the last mentioned paper some formulae are repeated here. The residual strain

$$\epsilon_{\psi=0} = -\frac{\nu}{E}(T_n - T_{mean})$$

T_n stress of a crystal n in axial or deformation direction, T_{mean} average value of all crystals

$$\tau_c = T \sin \chi \cos \lambda$$

τ_c critical shearing stress, χ angle between the applied stress and the most favourably oriented glide plane, λ angle between the applied stress and glide direction.

Table 5. Studies for the calculation of deformation-RS.

material		non-linearities, oscillations,		model
phases	texture	assumptions	reference	
single-phase material	quasi-isotropic	anisotropy of yield stress, self-consistent modeling, Taylor, Eshelby, FEM-analysis	/16,113, 116,119, 120/	dislocation structure: RS III, /37,40/
	textured	anisotropy of yield stress, self-consistent modeling, Taylor, hardening, ODF	/117,121/	
multiphase material	quasi-isotropic	anisotropy of yield stress, self-consistent modeling, Eshelby	/118,120/	two-phases-material model: yield stress, Young's modulus, strain hardening /23,75/
	textured		/121/	

The basic formula is extended for inclined incidence of X-rays with different wavelengths for measurements on different peaks /113,114,21,115/ :

$$D_{\psi} - D_0 = \frac{\sigma_L D_0}{E} \left[\nu - (1 + \nu) \sin^2 \psi \right] \cdot \left[1 - \frac{(\sum s)_{hkl}}{(\sum s)_m} \right]$$

Σs : minimal sum of slip of the five necessary independent slip systems calculated according to Taylor /18/ for fcc crystallites. The index hkl means averaging over all crystallites that contribute to the peak $\{hkl\}$ in the direction ψ . Index m means averaging over all orientations.

If Σs is replaced by $2 / \sin 2\chi$ the formula represents the assumption of free deformability of the crystallites. The angle χ lies between the tension- and the slip direction [111]. For iron, there is a qualitatively good correlation between theory and experiment, but not quantitatively since the calculated strains had to be multiplied by a factor of 5 to 10 /115/. Also superpositions of the predicted oscillations with linear D-vs.- $\sin^2\psi$ dependences were experimentally checked.

All these efforts can be checked by means of results of calculations and of tests on different materials, Fig. 32 /10/. With the exception of the AlCuMg alloy, the displayed examples are single-phase materials, where no influences of a second phase are present. The conclusion was and is still valid that the anisotropy of yield stress of different oriented crystallites cannot quantitatively explain the origin of deformation stresses.

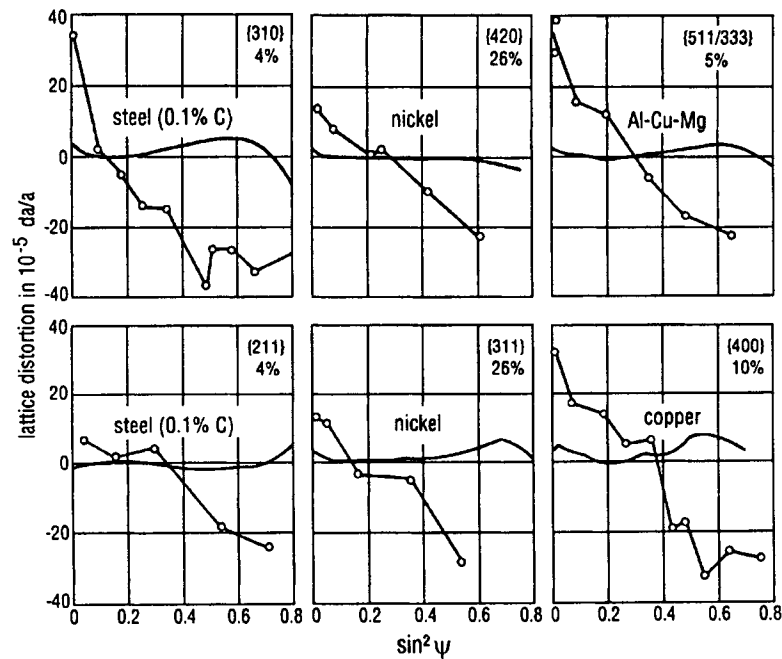


Figure 32. Experimentally determined and according to Greenough /16,19,20/ calculated lattice-strain distributions of several metals after plastic deformation. The authors are cited in /10/.

Another aspect of many years of studies on plastically deformed materials are the different theoretical approaches to predict the D-vs.- $\sin^2\psi$ distributions that are created by applied or RS on textured materials. Table 5 lists the assumptions and the models that are used. It is impossible to discuss here all the different publications and their different merits to the understanding of this important problem (see also Table 1 in paragraph 2.161).

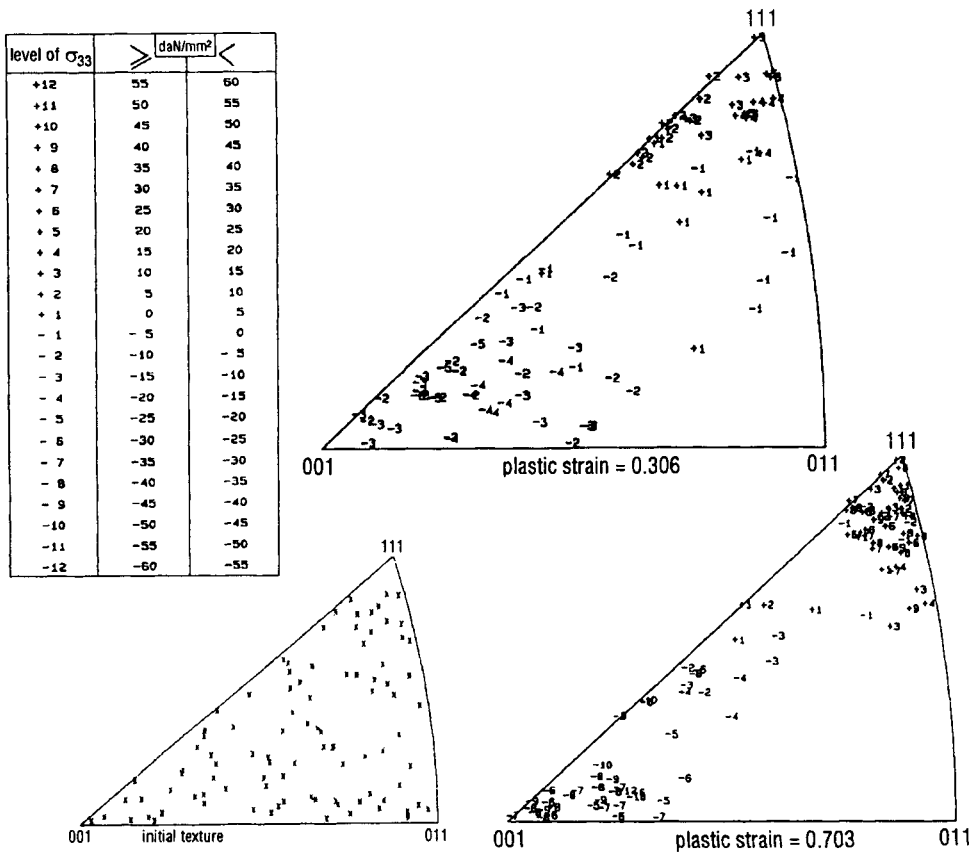


Figure 33. Crystallographic texture and residual stresses of the crystallites induced by plastic deformation //116/.

In //116/ the development of the stresses of an ensemble of differently oriented crystallites during plastic deformation were calculated using elasto-plastic models, Fig. 33. The Taylor theory was combined with the correct description of the texture (ODF) and the strain hardening of the material //117/, Fig. 34. Fig. 35 shows, for macroscopic compressive stress states, the mere elastic response of several crystallite groups with poles within the D-vs.- $\sin^2\psi$ distribution of the $\{211\}$ lattice plane of rolled steel /47/ (Reuss model). The resulting nonlinearities superimpose on those caused by plastic deformation and the respective microstresses. However also the recent publications cannot explain all the details of experimental studies. The current status is compiled in Table 5. A conclusive theory to explain quantitatively the D-vs.- $\sin^2\psi$ distributions with the specific oscillations after different kinds of plastic deformations is still missing.

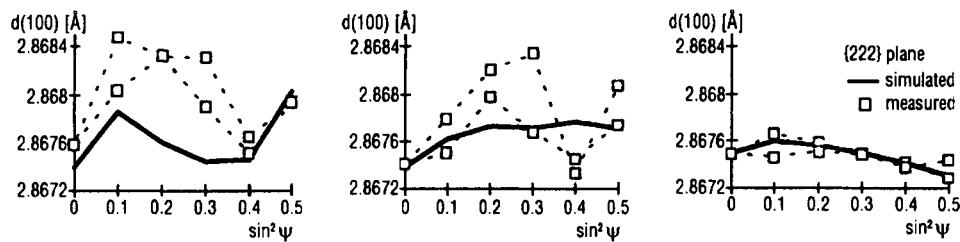


Figure 34. Experimental and simulated D-vs.- $\sin^2 \psi$ curves for the {222} diffraction plane of a cold rolled steel sheet. The data were simulated taking the averaged diffraction intergranular strain into account /117/.

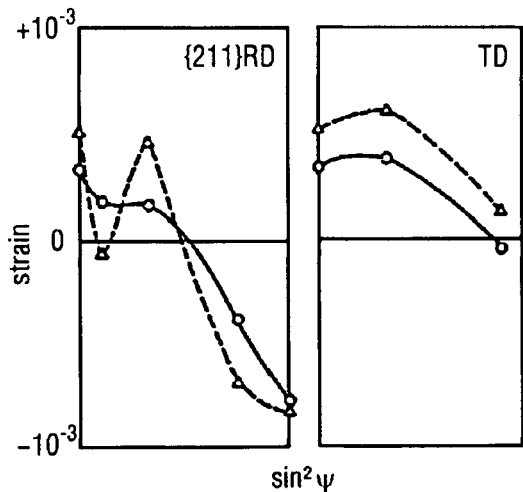


Figure 35. Strain of the crystallite groups with poles within the D-vs.- $\sin^2 \psi$ distribution of the {211} lattice plane of rolled steel /47/, calculated for two compressive macrostress states using the Reuss model.

2.164 Stress evaluation of lattice strain with oscillations

In paragraph 2.073 the different methods to evaluate LS and RS from lattice strain distributions with oscillations have been treated. Table 6 summarizes the methods and the suppositions to evaluate stresses from lattice strains measured by X- or neutron-rays /83/.

The kind of the stresses obtained depends on the method used. All mentioned methods are more or less approximative according to the investigated material state. A very sharp texture allows the stresses of the mainly present crystallite groups to be exactly calculated. The same is valid for the regression analysis, if orientation dependent microstresses are absent and the material is quasiisotropic. Because real stress states in technical materials usually are caused by a combination of surface- and heat-treatments, further investigations of the contribution of the single steps of production on the X- and neutron-ray lattice-strain distributions and especially on the strain of the differently oriented crystals should be made to improve the methods of stress evaluation.

Table 6. Methods of stress evaluation on textured and deformed materials /57/.

method of evaluation	fitting, regression			crystallite-group method
	stress factor F_{ij}	linearization		
		arbitrary peaks	multiple peaks	
suppositions and necessary data	no influence of ori- entation dependent micro-RS $\sigma_0(g)$, F_{ij} experimental or calculated using ODF	many D-meas- urements for $\psi \leq 0^\circ$ and $\psi > 0^\circ$ up to 90°	linear dependence D vs. $\sin^2\psi$	peak position in poles predomi- nantly determined by the crystallite group, D in poles
control test	comparison of cal- culated and experi- mental D-vs.- $\sin^2\psi$ distributions	comparison with other evaluation methods	comparison with other evaluation methods	calculation of the orientation distribution in the poles using ODF
evaluated stress	$\bar{\sigma} = \sigma^L + \sigma^I + \langle \sigma^{II} \rangle$	$\bar{\sigma}$	$\bar{\sigma}$	$\sigma(g)$

2.165 Recommendations

- The evaluation of LS and RS from lattice-strain distributions with oscillations requires a special measuring effort, and thorough considerations about the selection of the evaluating method.
- All methods result in values with larger scatter than the usual linear D vs. $\sin^2\psi$ method.
- Regard should be taken to the micro-RS $\sigma_0(g)$.
- Oscillations must be carefully checked whether they are true and reproducible or feigned by influences of coarse-grained material or measuring errors.
- Lattice distances should be determined by measurements in both directions $\psi \geq 0$, $\psi \leq 0$ and up to $\sin^2\psi \leq 0.8$, 0.9 or even 0.95. The steps in $\Delta\sin^2\psi$ should be 0.05.
- Since the shape of D-vs.- $\sin^2\psi$ dependences for different peaks may/will be different, interference lines as many as possible should be measured, especially in fundamental studies.
- The use of different wavelengths involves different penetration depths and hence in most cases strain/stress gradients have to be considered.
- Determination of the texture is indispensable, at least in the form of pole figures. In many cases, the ODF is a necessary basis for the stress evaluation. For texture research studies the notation of the model that is used to evaluate the ODF is valuable.
- Repeat- or routine-measurements can be done with less effort after thorough consideration.
- For the evaluation of stresses, use the methods in Table 6.
- Care should be taken when stress evaluation is made by linearization.

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2.17 Line broadening by non-oriented micro RS

Ch. Genzel

2.171 Historical review¹

Today, broadening of X-ray diffraction patterns of cold-worked metals and alloys is well-known to be caused by one or more of at least three effects, namely small size of coherently diffracting domains, lattice strains and stacking faults. *Scherrer* [1] first showed that the mean dimension, $\langle N \rangle$, of the crystallites composing a powder is related to the structural broadening, β , of the X-ray diffraction line profile by the equation

$$\langle N \rangle = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

where k is a constant approximately equal to unity. In his original work [1], *Scherrer* further proposed that β can be obtained from the experimental width, B , of the diffraction line by subtracting from it the instrumental width, b , of a line produced by a well-defined standard material by

$$\beta = B - b \quad (2)$$

The quantity β was sometimes defined as the full angular width at half-maximum intensity (FWHM) which is easy to determine and relatively insensitive to experimental errors. However, because there is no simple relationship between the FWHM and the quantities responsible for the broadening, *Laue* [2] defined β in a more physical manner as the integral breadth, $(\int I(2\theta) d2\theta) / [I(2\theta)]_{\max}$. For this reason, the quantities b , B and β in the following denote the integral breadth of the corresponding profiles. The mathematical law underlying the superposition of the intrinsic (physical) profile, $f(x)$, and the instrumental profile, $g(x)$, was first realised by *Spencer* [3]. According to *Jones* [4], the observed profile, $h(x)$, can be expressed in terms of the convolution

$$h(x) = \int_{-\infty}^{\infty} f(y) g(x-y) dy \quad (3)$$

¹ For list of symbols see end of this chapter

where $x, y = 2(\theta - \theta_{\max})$ are variables on the experimental 2θ - scale. A brief survey of the various methods for eliminating the instrumental influence on the base of Eq. (3) is given in Table 1. Of these, the methods proposed by *Berry* [12], *Stokes* [13] and *Paterson* [14] are the most general, because they do not make any assumption on the shape of the profiles. Therefore, they are suitable for detecting asymmetrical effects of instrumental broadening due to sample flatness, vertical divergence and absorption [15,16]. Owing to the fundamental investigations of *Bertaut* [17,18] and *Warren and Averbach* [19-23], who showed that detailed information on domain size and strain distribution within the material can be obtained from Fourier series expansions of the physical profile, $f(x)$, the correction method of *Stokes* [13] has become generally accepted in X-ray line profile analysis.

Below, starting with a brief survey of the line profile parameters related to microstructural effects and the procedures involved in line profile analysis, the most important methods that have been developed so far for analysing X-ray as well as neutron line broadening will be discussed. The attention is focused on the analysis of those lattice defects giving rise to microstrains that fluctuate on an atomic scale and, therefore, to non-oriented residual microstresses within the material.

2.172 Line profile parameters related to microstructure analysis

X-ray diffraction line profiles are ordinarily of little use themselves, but they are measured in order to derive from them various parameters of physical interest. According to *Wilson* [24], these parameters may be divided into four groups:

I. Measures of intensity

1. peak maximum intensity, $I_{\max}$
2. integrated intensity, $I_{\text{int}} = \int_{-\infty}^{\infty} f(2\theta) d2\theta$

II. Measures of location

3. peak maximum position, $2\theta_{\max}$
4. centroid position, $\langle 2\theta_c \rangle = \left(\int I(2\theta) 2\theta d2\theta \right) / \int I(2\theta) d2\theta$

III. Measures of dispersion

5. full width at half-maximum intensity (FWHM), $2w$
6. integral breadth, $\beta = \left(\int I(2\theta) d2\theta \right) / I_{\max}$
7. variance, $v = \left(\int (2\theta - \langle 2\theta_c \rangle)^2 I(2\theta) d2\theta \right) / \int I(2\theta) d2\theta$
8. Fourier coefficients, $F_L = A_L + i B_L$

IV. Measures of coherence

9. particle (domain) size distribution, $p(N_3)$
10. strain distribution, $q(e_L)$
11. distribution of lattice defects (dislocations, stacking faults, inclusions etc.)

Table 1
Methods for separation of instrumental and physical X-ray line broadening.

Assumptions on the profiles involved in Eq. (3)	Expressions for the corrected profile and/or integral breadth	References
$f(x): \exp(-k^2x^2)$ $g(x): \exp(-l^2x^2)$	$\beta = (B^2 - b^2)^{\frac{1}{2}}$	Warren and Biscoe (1938) [5]
Arbitrary mean of Scherrer [1] and Warren [5] correction formulae	$\beta = \left\{ (B^2 - b^2)^{\frac{1}{2}} - (B - b) \right\}^{\frac{1}{2}}$	Taylor (1941) [6]
$f(x): (1 + k^2x^2)^{-1}; \exp(-k^2x^2);$ $\sin^2(kx) / (kx)^2$ $g(x): \text{measured profile of standard substance}$	Correction functions in form of $\frac{\beta}{B} = f\left(\frac{b}{B}\right)$	Jones (1938) [4] Kochendörfer (1944) [7] Alexander (1950) [8]
$g(x): \exp(-k^2x^2)$ $h(x): \exp(-p^2x^2) + \tau \exp(-q^2x^2)$	Correction formulae for β in terms of p , τ and q	Shull (1946) [9]
$g(x): (1 + a^2x^2)^{-1}$ $h(x): (1 + c^2x^2)^{-1}$	$\beta = B - b$	Wood and Rachinger (1949) [10]
$g(x): c_1 / (1 + k_1^2x^2)^2$ $h(x): c_2 / (1 + k_2^2x^2)^2$	$f(x): \text{found by solving equation (3) by the Fourier-transform method.}$ $\frac{\beta}{B} = f\left(\frac{b}{B}\right)$	Schoening et al. (1952) [11]
$g(x), h(x): \text{represented by a Hermitian polynomial series}$	$f(x); \beta = \frac{\int f(x) dx}{f(0)}$	Berry (1947) [12]
$g(x), h(x): \text{represented by a Fourier series}$		Stokes (1948) [13]
$g(x), h(x): \text{experimental profiles are used directly}$	$f(x): \text{found by solving eq. (3) by approximating it by a set of linear equations}$	Paterson (1950) [14]

Fig. 1 shows for the example of cold-working of metals, how the coherence parameters are related to the line profile parameters of dispersion as well as those of location. Obviously, a separation of the individual effects that contribute to physical broadening requires a careful line profile analysis which can be divided into three steps: (I) measurements, (II) corrections of the line profiles and calculation of the parameters characterising the *f* profile, and (III) evaluation of the structural parameters [25].

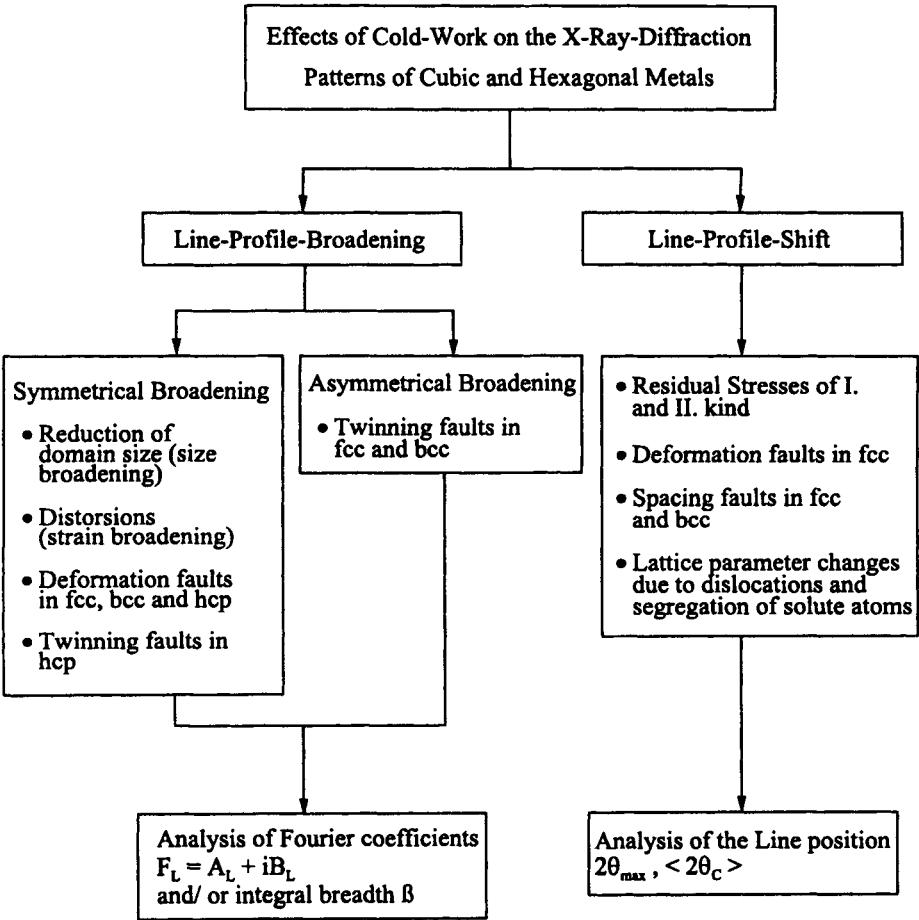


Figure 1. Effects of the microstructure on X-ray diffraction patterns.

For a determination of the microstructural parameters on an absolute scale, a *g* profile is necessary that comprises exactly the same instrumental broadening being present in the *h* profile. Some practical recommendations for choosing suitable experimental conditions, standard specimens etc. are given at the end of this Chapter; for detailed information see, for example, [16, 25-27].

A brief survey of the procedures involved in step II of X-ray line profile analysis is shown in Fig. 2. Since $I(2\theta)$ is a random variable, all quantities obtained by manipulating it are also random variables. The errors related to counting statistics in the normalised Fourier coefficients and breadth parameters of the f profile depend on the counting time, the shapes of the h and g profiles and on the background [25], for a detailed discussion see [24,27,28]. Because all physical quantities are related to the net intensities, background correction must be carried out very carefully. The most common errors made in evaluating the background are related to horizontal and vertical profile truncation and to overlapping of neighbouring lines. Concepts for handling and avoiding these errors were developed in [29-32].

In the theory of X-ray diffraction, the intensity distribution is usually within the reciprocal lattice [33]. Therefore, the variable $x = 2(\theta - \theta_{\max})$ must be transformed on a reciprocal scale according to

$$s = \frac{2}{\lambda} (\sin \theta - \sin \theta_{\max}) \quad (4)$$

[20]. For $2\theta \leq 120^\circ$, Eq. (4) can be approximated by $s = (\cos \theta / \lambda) x$ and further calculations can be performed on the experimental 2θ - scale [34].

After having corrected the line profiles for the θ - dependent factors (Lorentz-, polarisation and absorption correction, cf. Chapter 2.04), two different ways for deconvoluting the h profile into its physical and instrumental parts, f and g , respectively, are possible. If the micro-structure is to be studied in more detail, the complete information of the diffraction pattern in form of its Fourier coefficients obtained by *Stokes* method [13] should be used. All methods making use of the Fourier coefficients, are called 'Fourier-space methods'. For a rather rapid analysis, only the knowledge of the integral breadth of the profile is necessary, which can be obtained by approximating the measured h and g profiles by simple analytical functions. These methods are called 'Real-space methods'.

Because the Fourier-space methods do not make any assumptions on the shape of the profiles, a removal of the α_2 part is not necessary but may be done. The Real-space methods, however, are based on the assumption that the line profiles can be described by symmetrical Voigt functions which represent the mathematical convolution of Cauchy (Lorentzian) and Gaussian functions. Therefore, if no monochromator is used, the experimental profiles have to be corrected for α_2 which can be achieved, for example, by the methods employed in [35,36].

2.173 Fundamental methods in line profile analysis

(a) Fourier-space methods

According to *Stokes* [13], the intensity distributions of the broadened and instrumental (standard) profiles, $h(x)$ and $g(x)$, respectively, can be expressed in terms of Fourier series:

$$h(x) = \sum_{L=-\infty}^{\infty} H_L \exp(2\pi i L x / a) \quad (5a)$$

$$g(x) = \sum_{L=-\infty}^{\infty} G_L \exp(2\pi i L x / a) \quad (5b)$$

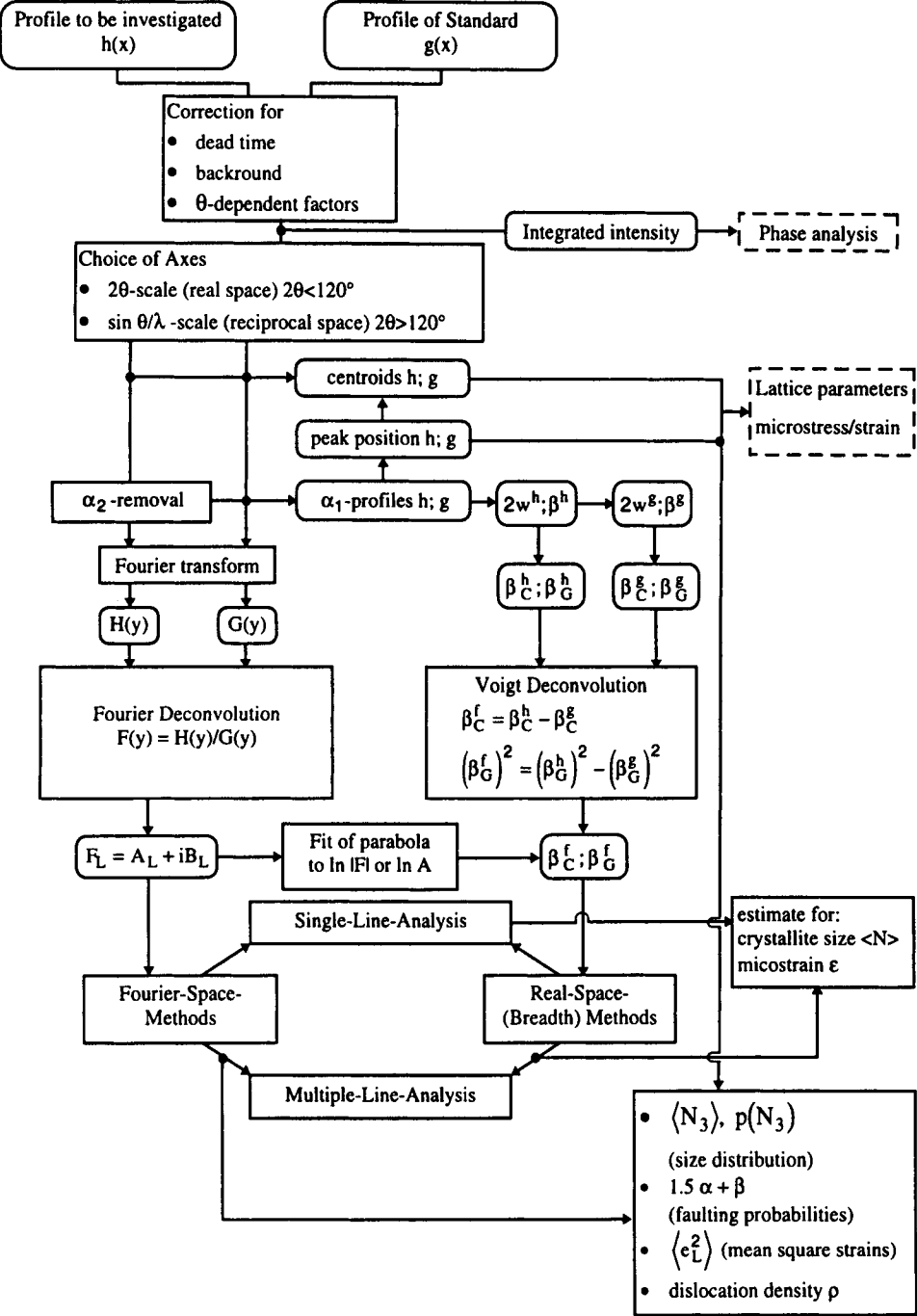


Figure 2. Line-profile processing procedures corresponding to [25]

The Fourier coefficients are given by

$$H_L = \frac{1}{a} \int_{-\frac{a}{2}}^{\frac{a}{2}} h(x) \exp(-2\pi i L x / a) dx \approx \frac{1}{a} \sum_{-\frac{a}{2}}^{\frac{a}{2}} h(x_v) \exp(-2\pi i L x_v / a) \quad \text{etc.} \quad (6)$$

The approximation applies to intensity distributions taken at discrete values of x within the range $[-a/2 ; a/2]$. Due to a fundamental theorem in the theory of Fourier transforms, the convolution product (3) in real space corresponds to an ordinary product in Fourier space and the physical line profile $f(x)$ can be expressed in terms of the Fourier coefficients of the g and h profiles:

$$f(x) = \sum_{-\infty}^{\infty} \frac{H_L}{a G_L} \exp(2\pi i L x / a) \quad (7)$$

with $H_L / (a G_L) = F_L$. Generally, F_L is complex, i.e. $F_L = A_L + i B_L$, and (7) may be written in form of a sine and a cosine series. Introducing the reciprocal space variable s defined by Eq. (4) yields

$$f(s) = \sum_L [A_L \cos(2\pi L s) + B_L \sin(2\pi L s)] \quad (8)$$

In (8), the cosine terms represent the symmetrical part of the profile, whereas the sine terms produce peak asymmetry which is mainly caused by twinning (growth) faults in fcc and bcc structures [23,37], for details see Chapter 2.09. Assuming that the sine coefficients B_L are small enough to be negligible, the line profile is completely determined by its cosine coefficients. Fig. 3 gives a survey of the possibilities for separating the contributions of size and strain components to the line broadening by means of the Fourier coefficients.

Most detailed information is obtained by the method of *Warren and Averbach* (WA- analysis) [20-23] (Method I in Fig. 3), which is based on the factorisation of the cosine coefficients A_L into particle size and distortion terms, A_L^S and A_L^D , respectively:

$$A_L(h_0) = A_L^S \cdot A_L^D(h_0) \quad (9)$$

where $h_0 = (h^2 + k^2 + l^2)^{\frac{1}{2}}$ is the order of the reflection. $L = n D_{hkl}$ represents a distance normal to the reflecting lattice planes hkl with interplanar spacings $D_{hkl} = a_0 / h_0$ and n the harmonic number of the Fourier coefficients. The following relations hold:

$$A_L^S = \frac{1}{\langle N_3 \rangle} \int_{|L|}^{\infty} (N_3 - |L|) p(N_3) dN_3 \quad (10)$$

$$A_L^D(h_0) = \int_{-\infty}^{\infty} q(e_L) \cos(2\pi L h_0 e_L / a_0) de_L \quad (11)$$

Eq.s (9) to (11) are based on the assumption that the coherently diffracting domains within the crystallites can be divided into columns of orthogonal unit cells normal to the reflecting lattice planes, each of them having an individual length N_3 [17,20]. $p(N_3)$ is the distribution function of the column length and $q(e_L)$ represents the distribution of average strain in a column between two unit cells which are $n = L / D_{hkl}$ cells apart. $\langle N_3 \rangle$ stands for the average domain size normal to the reflecting planes. For small values of L and e_L , (9) and (10) can be expanded into Taylor series and the following approximations hold [23,38,39]:

$$A_L^S \approx 1 - L \left[\frac{1}{\langle N_3 \rangle} + \frac{(1.5\alpha + \beta)}{a_0} V_{hkl} \right] \quad (12)$$

$$A_L^D(h_0) \approx 1 - 2\pi^2 L^2 h_0^2 \langle e_L^2 \rangle / a_0^2 \approx \exp \left(-2\pi^2 L^2 h_0^2 \langle e_L^2 \rangle / a_0^2 \right) \quad (13)$$

where $\langle e_L^2 \rangle$ is the mean square strain component in the direction of the normal to the reflecting planes. From Eq. (12) it is realised that size broadening is also influenced by the deformation and twin fault probabilities, α and β , respectively. The coefficient V_{hkl} depends on the structure as well as the reflection investigated [38]. For a detailed discussion of faulting, its influence on the diffraction patterns (cf. Fig. 1) and its analysis which is mainly based on the approaches of *Paterson* [37], *Wagner* et al. [38,39], *Wilson* [40] and *Warren* and coworkers [23,41,42], the reader is referred to Chapter 2.09 in this book.

The separation of the coefficients in (9) can be performed using the h_0 dependence of the strain term A_L^D . Hence, if different (at least two) orders h_0 of a reflection hkl have been measured, Eq. (9) can be rewritten with regard to (13) in one of the following forms:

$$\ln[A_L(h_0)] = \ln(A_L^S) - 2\pi^2 L^2 h_0^2 \langle e_L^2 \rangle / a_0^2 \quad (14a)$$

$$A_L(h_0) = A_L^S - A_L^S \cdot 2\pi^2 L^2 h_0^2 \langle e_L^2 \rangle / a_0^2 \quad (14b)$$

Eq. (14b) represents a "linear version" of the classical separation method (14a) and was first suggested in [43] in order to avoid the errors due to a truncated Taylor series of $\ln[A_L(h_0)]$. Following the classical approach (14a) of the WA- method [23] and plotting $\ln[A_L(h_0)]$ for fixed values of L against some function of h_0^2 , the intercept at $h_0^2 = 0$ obtained by extrapolation, gives $\ln(A_L^S)$, whereas the mean square strains $\langle e_L^2 \rangle$ are found from the slopes of the curves (Fig. 4).

Next page:

Figure 3. Fourier methods for separating size and strain broadening (for details see text).

Stokes corrected physical line-profiles

$$I(s) = \sum_L A_L \cos 2\pi Ls$$

Fourier coefficients A_L

Warren-Averbach (WA) Method (Method I)

$$-\left(\frac{dA_L^S}{dL}\right)_{L=0} = \frac{1}{\langle N_3 \rangle}$$

$$\left(\frac{d^2 A_L^S}{dL^2}\right) = \frac{p(N_3)}{\langle N_3 \rangle}$$

$\langle e_L^2 \rangle$ from slope of $A_L^D(h_0)$ versus h_0^2

Separation of β_{St} into size and strain term using Gauss or Cauchy relation (Method III)

$$\beta_{St} = \frac{\lambda}{\left(\cos \theta_0 d_{hkl} \sum_L A_L\right)}$$

$$\left(\beta_{St} \frac{\cos \theta}{\lambda}\right)^2 = \frac{1}{\langle N \rangle_G^2} + 16\epsilon_G^2 \left(\frac{\sin \theta_0}{\lambda}\right)^2$$

$$\left(\beta_{St} \frac{\cos \theta}{\lambda}\right) = \frac{1}{\langle N \rangle_C} + 4\epsilon_C \left(\frac{\sin \theta_0}{\lambda}\right)$$

Summation of the A_L^{SF} and A_L^D (Method II)

$$\beta_{WA}^S = \lambda \left(\cos \theta_0 d_{hkl} \sum_L A_L^{SF} \right)^{-1}$$

$$T = d_{hkl} \sum_L A_L^{SF} = \frac{\langle N_3^2 \rangle}{\langle N_3 \rangle} \geq \langle N_3 \rangle$$

$$\beta_{WA}^E = \lambda \left(\cos \theta_0 d_{hkl} \sum_L A_L^D \right)^{-1}$$

$$\epsilon = \beta_{WA}^E (2\sqrt{2\pi} \tan \theta_0)^{-1} = \sqrt[3]{\frac{1}{2} \left(\langle e_L^2 \rangle^{\frac{1}{2}} \right)}$$

Modified Voigt-Analysis (Method IV)

$$\ln(A_L) \approx -2\beta_C L - \pi\beta_G^2 L^2$$

$$\beta_C = \lambda \langle N \rangle \cos \theta_0)^{-1}$$

$$\beta_G = 4\epsilon \tan \theta_0$$

No separation of the coefficients

Separation into

$$A_L = A_L^S \cdot A_L^D$$

If the particle size coefficients A_L^S are plotted versus L (Fig. 5a), the initial slope of the curve gives directly the average column length $\langle N_3 \rangle$, whereas its distribution is obtained from the second derivative:

$$-\left(\frac{dA_L^S}{dL}\right)_{L=0} = \frac{1}{\langle N_3 \rangle} \quad \text{and} \quad \frac{d^2 A_L^S}{dL^2} = \frac{p(N_3)}{\langle N_3 \rangle} \quad (15)$$

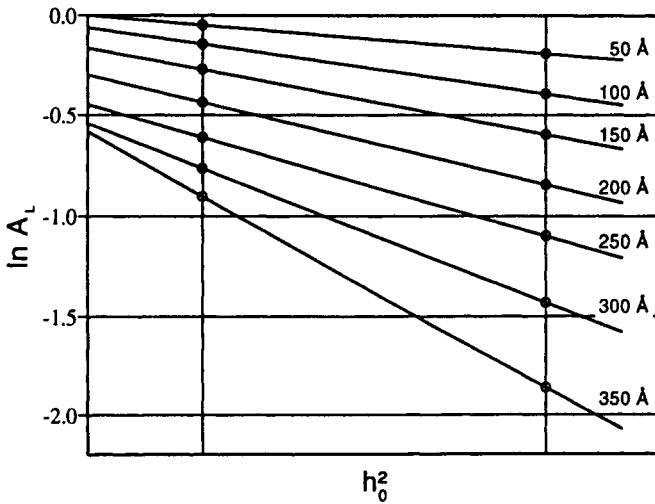


Figure 4. Plot of $\ln A_L$ against $h_0^2 = h^2 + k^2 + l^2$ for a 90% cold-rolled alloy of NiCo 50, profile (111). For each $L = n a_3$, the different hkl reflections fall upon a single curve (after [34]).

This result follows immediately from Eq.s (10) and (12) [20], if α and β are assumed to be negligibly. An additional estimation for $\langle N_3 \rangle$ is obtained by extrapolating of the A_L^S versus L curve on the abscissa. Since $(d^2 A_L^S / dL^2) \geq 0$ (cf. Eq. (15)) the A_L^S versus L curve can never be concave downward. However, for small values of L , the size coefficients often do reveal a concave curvature (cf. Fig. 5a) which is 'forbidden' within the framework of the WA-analysis. This so-called 'hook effect' was sometimes explained as due to the uncertainty in determining A_L^S for small values of L , because they are related to the long tails of the line profiles which are hardly recorded experimentally [23,44]. As shown by Wilkens et al. [45-47], the 'hook effect' can be interpreted in a more physical manner, if the assumption of discrete 'particles' with defined boundaries, is replaced by that of 'transparent' dislocation walls forming small-angle grain boundaries.

A second difficulty in deriving physical parameters from the WA-plots arises from the assumptions made on the strains within the columns. The approximation of the distortion coefficients A_L^D by a truncated Taylor series (Eq. (13)) and, therefore, the separation of the co-

efficients (Eq.s (14a,b)), are only valid, if the frequency distribution $q(e_L)$ of the strains is approximately Gaussian. However, as shown in various investigations (see, for example [44-50]), this assumption is not well justified, if dislocations with strain fields of $1/r$ divergences are the prevailing type of lattice defects. For this reason, an interpretation of the $\langle e_L^2 \rangle^{\frac{1}{2}}$ distributions often becomes difficult, especially in the range of small L , where the curves are strongly affected by experimental errors (cf. Fig. 5b).

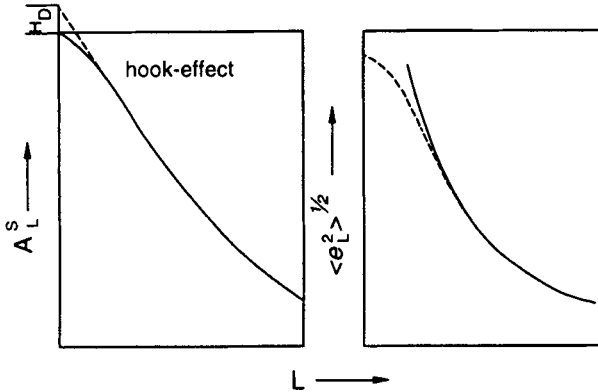


Figure 5. Particle size coefficients A_L^S (a) and mean square strains $\langle e_L^2 \rangle^{\frac{1}{2}}$ (b) as functions of L ; full lines - observed distributions, dashed lines - theoretically expected curves (after [45]).

Assuming that the WA- method can be used to separate particle size and strain effects to such high values of L that the A_L^S and A_L^D curves can be extrapolated to $L = \infty$, values for the particle size T and the microstrain ϵ are obtained directly from the summation of A_L^S and A_L^D from $L = -\infty$ to $L = \infty$, respectively, [20,23,39]. Fig. 3 (Method II) shows that the integral breadths, β_{WA}^S and β_{WA}^E , which are related to size and strain broadening, respectively, are directly related to the inverses of the summations of the separated coefficients. Further, it is realised that the size and strain values derived from the summations of the Fourier coefficients have a slightly different meaning compared with those obtained from the WA-plots. Thus, the strain ϵ evaluated from the integral breadth should be approximately 25%

larger than the root mean square strain $\langle e_L^2 \rangle^{\frac{1}{2}}$ averaged over the dimension of the domain size $\langle N_3 \rangle$ [39], where the latter represents a lower limit for the particle size T [18].

The physical integral breadth, β_{St} , corrected for instrumental broadening by the Stokes method [13] is obtained by summing the Fourier coefficients A_L (Fig. 3, Method III). Because β_{St} depends upon the particle size, faulting probabilities and strain, some assumptions have to be made on the relation between β_{St} , β_{St}^S (size and faulting term) and β_{St}^E (strain term). Since the Fourier coefficients A_L^S , when plotted as a function of L , have a non-zero initial slope (cf. Fig. 5a), the profile related to size broadening should be close to a Lorentzian, whereas strain broadening can be represented by a Gaussian [23,51,52]. Other authors assume that both

sources of broadening will produce a Lorentzian peak shape [48,53]. Hence, assuming $\beta_{Sr}^S = \lambda/(\langle N \rangle \cos \theta)$ for size broadening and $\beta_{Sr}^E = 4\varepsilon_0 \tan \theta$ for strain broadening (which corresponds to the assumption of a rectangular strain distribution with FWHM = $2\varepsilon_0$) [7,54,55], the total integral breadth from both effects may be calculated using a quadratic (Gauss) or linear (Cauchy) relation. If β_{Sr} has been determined for several reflections hkl , one can decide whether a plot of $(\beta_{Sr} \cos \theta / \lambda)^2$ versus $(\sin \theta / \lambda)^2$ or that of $(\beta_{Sr} \cos \theta / \lambda)$ versus $(\sin \theta / \lambda)$ will give the best fit.

It should be emphasised that the methods treated so far are so-called multiple-line methods, because they require the Fourier coefficients of different orders of the same reflection, or, at least, of different reflections. Some other approaches based on a Fourier analysis have been developed as well, which need only the profile of a single line for separating size and strain effects. However, these methods are based on less realistic assumptions and therefore, they will not be treated here in more detail. A brief survey of these methods is given, for example, in [25]. The so-called 'Modified Voigt analysis' (Method IV in Fig. 3), can be considered as an improved single-line method [57,58] that may be placed between the Fourier and the Real-space methods (see next section).

(b) Real-space methods

Although the Fourier space methods based on the theory of *Warren* and *Averbach* allow a detailed and accurate analysis of the microstructure, it is often acceptable at the expense of some loss of accuracy to use more rapid techniques which are simple to apply. These methods are based on the analysis of the full width of the line profile at half the maximum intensity, $2w$, and the integral breadth, β , which can easily be determined. A limitation in the use of the dispersion parameters, $2w$ and β , is the need to ascribe an analytical function to the line profiles. The most widely employed functions in this field, are the Cauchy and the Gaussian functions. It has been demonstrated in various investigations that instrumental as well as particle size broadening are approximately Cauchy, whereas the profiles arising from lattice strains are more nearly Gaussian and, therefore, a good approximation is given by the convolution of these functions which is called Voigt function [57, 59-61].

Fig. 6 summarises the individual steps of the Voigt analysis for separating the integral breadths of size and strain broadening, β_C^f and β_G^f , respectively. To start with, it is assumed that all profiles involved, can be described by Voigtian functions. *Langford* [57] derived an explicit equation for the Voigt function and showed that the breadths of the constituent Gaussian and Cauchy components can easily be found from the so-called form factor, $2w/\beta$, which is a characteristic parameter of any line profile. Thus, the values of $2w/\beta$ related to the Voigt profiles which are to be determined experimentally, will lie between the limits of 0.63662 and 0.93949 for the Cauchy and Gaussian profile shapes, respectively. In order to avoid graphical methods or the interpolation from tables [57], the necessary calculations can be carried out readily using empirical formulae that have been given in [62].

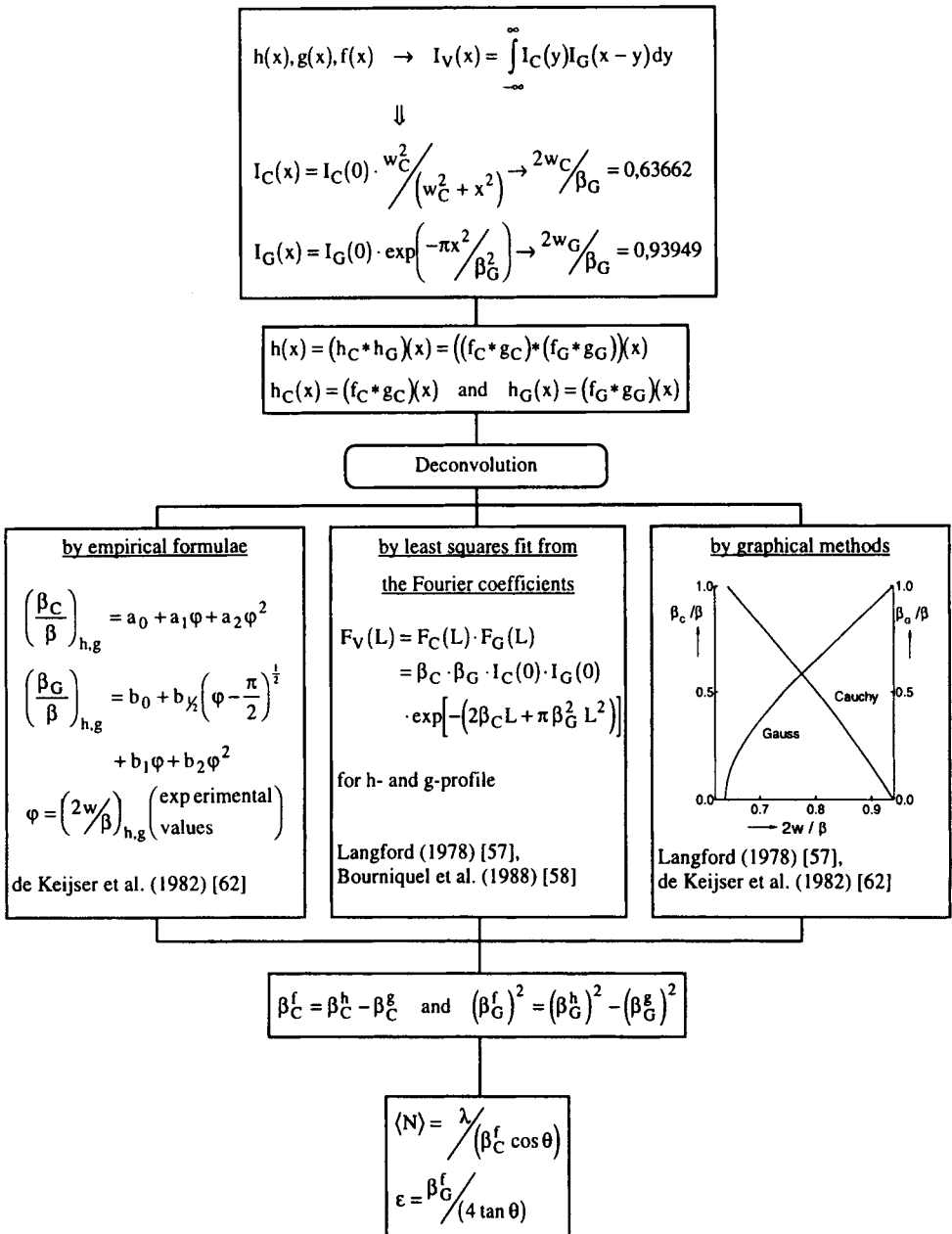


Figure 6. Separation of particle size and strain broadening by means of Voigt deconvolution;
 $a_0=2.0207$, $a_1=-0.4803$, $a_2=-1.7765$, $b_0=0.6420$, $b_1=1.4187$, $b_2=-2.2043$, $b_3=1.8706$

An improved method based on the analytical Fourier transform of the Voigt function, $F_V(L)$ (cf. Fig. 6), was suggested in [57,58]. According to the convolution theorem, $F_V(L)$ is given in a simple closed form by multiplying the transforms of its constituent Cauchy and Gaussian components, $F_C(L)$ and $F_G(L)$. The integral breadths β_C and β_G of both g and h profiles are then determined by a least squares fit of a parabola to the logarithm of the Fourier coefficients that are obtained by the *Stokes* method [13] (cf. Method IV in Fig. 3). Using the linear and quadratic relations between the integral breadths that hold for the convolution of two Cauchy and two Gaussian functions, respectively, one obtains the values β_C^f and β_G^f which are related to particle size and strain broadening, respectively.

It should be emphasised that the method of Voigt deconvolution is in principle a single-line procedure, i.e. it requires only one line profile of both the material to be investigated and the standard specimen. However, if two or more reflections are available, it is recommended to determine size and strain effects from the variation of β_C^f and β_G^f with hkl [61,63].

(c) Determination of dislocation densities

The methods employed in X-ray line profile analysis are mainly focused on the determination of the average particle size, $\langle N \rangle$, and the mean square strains, $\langle e_L^2 \rangle$ (WA- analysis), or, the microstrain ϵ (integral breadth methods). However, the results are difficult to be interpreted in terms of physical parameters related to the microstructure of the material. Thus, the values of $\langle N \rangle$ obtained by different methods generally differ considerably from each other and the physical meaning of the domain or particle boundaries is not yet understood in detail. A further difficulty arises from the definition of the non-uniform strains determined by the WA- analysis. Because the mean square strain $\langle e_L^2 \rangle$ depends on the correlation length, L , the evaluation of average residual microstresses according to

$$\sigma_{micro}^{RS} = E_{hkl} \langle \epsilon^2 \rangle^{\frac{1}{2}} \quad (16)$$

[64] must be related to a certain value of L , which is often settled by $L = 5\text{nm}$ [65] or $L = \langle N \rangle / 2$ [66].

Therefore, in order to understand the reasons of line broadening in a more physical manner, numerous efforts have been made to deduce from the X-ray diffraction line profiles both the density and the general arrangement of dislocations, which are known to cause line broadening by their accompanying strain fields [48, 67-69]. Table 2 briefly summarises the results of the most important investigations that have been done in this field. In the following, some general remarks will be made concerning the validity and the limitations of these methods.

Table 2

Methods for the determination of dislocation densities from the parameters of line profile broadening; b - Burgers vector, $\Gamma = \Gamma(hkl)$ - orientation factor, r_0 - inner cut-off radius of the dislocation line, R_0 , L_0 - outer cut-off radius, $B_0(hkl)$ - factor depending on the reflection used.

Assumptions	Procedures	References
Coherently diffracting domains bounded by dislocations	$\rho_D = \frac{3n}{\langle N_3 \rangle^2}$ Geometrical average	Williamson and Smallman (1956) [70]
Distorsions caused by strain components of the dislocations	$\rho_\epsilon = \frac{k}{b^2} \langle \epsilon^2 \rangle$ $\bar{\rho} = \sqrt{3nk} \frac{\langle \epsilon^2 \rangle^{\frac{1}{2}}}{b \langle N_3 \rangle}$	
Elastical strain energy of the aggregate ($-\langle \epsilon^2 \rangle$) composed of the elastical energy of the individual dislocations	$\langle \epsilon^2 \rangle = \frac{b^2}{\pi} \rho \frac{1}{24(1+\nu)} \ln \left(\frac{c_\epsilon}{\sqrt{\rho} b} \right)$ $C_\epsilon = R_0 \sqrt{\rho}$	Faulkner (1960) [71]
Random distribution of parallel dislocations on the $\{111\} \langle 110 \rangle$ fcc glide systems in elastically isotropic materials	$\langle \epsilon^2 \rangle = \left(\frac{b}{\pi} \right)^2 \rho f(\nu, \Gamma) \ln \left(\frac{1}{2\sqrt{\rho} r_0} \right)$	Ryaboshapka and Tikhonov (1961) [72]
Elastically isotropic cylindrical body of finite radius with randomly distributed dislocations parallel to the cylinder axis, fcc-lattice	$\beta^2 \cot^2 \theta = b^2 \rho f(\nu, \Gamma) \times$ $\times \ln \left[\frac{\pi}{\sqrt{12} \rho} R_0 \ln \left(\frac{\pi}{\sqrt{12} \rho} R_0 \right) \right]$	Krivoglaz and Ryaboshapka (1963) [73]
Restrictedly random distribution of parallel dislocations within isotropic cylindrical body	$\langle \epsilon^2 \rangle = \left(\frac{b}{\pi} \right)^2 \rho f(\nu, \Gamma) \ln \left(\frac{C_e}{\sqrt{\rho} b} \right)$ $C_e = R_e \sqrt{\rho}$	Wilkens (1967, 1969, 1970) [74, 75, 46]
Crystals containing small-angle grain boundaries of distance D with dislocation spacing d within the boundaries	$\langle \epsilon_L^2 \rangle = \frac{b^2}{4\pi^2} \frac{17\pi}{32} \rho \left[\ln \frac{d}{L} + c \right]$	Wilkens (1979) [47]
Restrictedly random distribution of dislocations, weak correlation between the defects	$-\frac{\ln(A_L)}{L^2} = \frac{1}{(\langle N_3 \rangle L)} + \left\{ \frac{2\pi^2 h_0^2 \langle \epsilon^2 \rangle}{a_0^2} + B_0(hkl) \rho \ln \left(\frac{L}{L_0} \right) \right\}$	Klimanek (1988, 1989) [76, 77]

I. The evaluation of dislocation densities from the average coherence length $\langle N_3 \rangle$ [70] only seems to be justified for a rough estimation of the order of magnitude, because the exact meaning of $\langle N_3 \rangle$ still remains unclear and the constants n and k involved in the calculations are assumed quite arbitrarily.

II. Most of the approaches [70-72, 74-76] are based on the concept of the mean square of the differential strain, which means the average of the 'true' local strain, e_0 , between neighbouring unit cells within the columns, i.e. assuming $\langle \epsilon^2 \rangle = \langle e_0^2 \rangle$ in Table 2. Due to experimental uncertainties, however, reliable values of $\langle e_L^2 \rangle$ are only obtained for a coherence length L of at least 5nm [65]. These values are, in general, considerably smaller than the differential mean square strain $\langle e_0^2 \rangle$ [44]. Therefore, a determination of dislocation densities from the $\langle e_L^2 \rangle$ values obtained from the WA- analysis yields only results of restricted reliability, even if the distribution of the dislocations is taken into account explicitly by the constants k , C_ϵ and C_e . Methods for calculating the differential mean square strain $\langle e_0^2 \rangle$ were suggested, for example, in [25,78].

III. A quite different formulation of the problem, which is directly based on the specific properties of the dislocation lines, was given by *Krivoglaz* and *Ryaboshapka* [73]. They calculated the integral breadth, β , of X-ray line profiles for cubic single crystals and polycrystals as a function of the dislocation density ρ , for the case of a random distribution of dislocations. Due to this assumption, however, the physical meaning of the outer cut-off radius R_0 involved in *Krivoglaz's* theory remains ill-defined. According to *Wilkens* [46,74,75], a statistically random distribution of dislocations should be rather unrealistic. He introduced the model of a 'restrictedly random' distribution by subdividing the cross section of the crystal into small areas of effective radius R_e . The dislocation distribution within the subareas is assumed to be completely random. In this case, the distribution of the dislocations can be characterised independently of their density itself, by a parameter $M = R_e \sqrt{\rho}$, which determines the shape of the line profile [79].

IV. *Klimanek* [76,77] combined the results of *Krivoglaz's* general theory of X-ray and neutron scattering [80] with *Wilken's* model of the restrictedly random distribution of dislocations and obtained the so-called 'Krivoglaz-Wilkens plot'. He demonstrated that for this case the dislocation density ρ of a real polycrystal can be determined independently of the peak shifts caused by lattice strains $\langle \epsilon^2(hkl) \rangle$ which are due to residual stresses of the 2nd kind. The applicability of this approach has been proved recently in various investigations using X-ray [81] as well as neutron diffraction methods [82-84].

2.174 Alternative approaches

Besides the Fourier space methods which are mainly based on the theory of *Warren* and *Averbach* and the line breadth methods based on the Voigtian analysis, numerous other approaches have been developed in the field of diffraction line profile analysis, but it is beyond the scope of this article to review these methods in detail. In the following, the most important of them will be summarised briefly.

Instead of the integral breadth, *Tournarie* [85] and *Wilson* [86,87] favoured the variance of the line profile as a measure of line broadening. Using the reciprocal space variable s , it is

defined as the second moment of the intensity profile:

$$v = \left(\int I(s) s^2 ds \right) / \int I(s) ds \quad (17)$$

From Eq. (17) it may be realised that the multiplication of the intensity $I(s)$ by the square of the variable s overemphasises the tails of the profile which are hardly to detect with sufficient accuracy [39]. Therefore, the variance method requires a very careful background correction of the profiles.

A quite different approach in line profile analysis is given by the method of 'line profile matching' which has been suggested in [88,89]. For two-phase materials consisting of a dispersion of fine particles in a matrix, the authors first calculated the residual stress/strain state due to the mismatch between particle and matrix by analytical models. In a second step, the accordingly simulated diffraction profiles were matched to the measured ones in order to prove the validity of the assumptions involved.

Starting from the finding that the WA- analysis breaks down at a certain value of L if the strain distributions are not Gaussian (cf. Eq.s (13,14)), *van Berkum et al.* [90,91] developed an alternative method for separating size and strain broadening. A basic assumption of this approach is the result of *Stokes and Wilson* [92] that for small L the strain gradients within the columns can be neglected. For this case, the fundamental equation for separating size and strain effects becomes

$$\ln \left[A \left(\frac{l}{n}, l \right) \right] = \frac{1}{l} \ln [A^S(n)] + \ln [A^D(n, l)] \quad (18)$$

[90] (n = harmonic number, l = order of reflection). Plotting $\ln[A(n/l, l)]$ versus $1/l$, the size Fourier coefficients are obtained from the slopes and the first order reflection strain coefficients are obtained from the intercepts of the curves. Fig. 7 shows the regions of applicability of the WA and the alternative analysis. They are bounded by iso-surfaces which are calculated for various combinations of three parameters characterising the microstructure in terms of (a) the relative amount of size and strain broadening, (b) the shape of the strain distribution $q(e_L)$ and (c) the degree of strain fluctuation within the columns [91].

In the so-called "double Voigt" approach, size as well as strain broadened profiles are described by Voigt [93-95] or Pseudo-Voigt [95-97] functions, taking into account that both contributions are superpositions of Gaussian and Cauchy components themselves. Other attempts are based on the use of the Pearson VII function [95,98]. In many of these cases, the determination of the crystallite-size and the lattice-strain parameters is performed by means of the profile refinement method of Rietveld [99,100]. A review of these methods is given, for example, in [101].

The probably most complex and comprehensive approach in line profile analysis that has been developed so far is due to *Klimanek* [76,77,102] who showed the line profiles to form so-called diffraction multiplets which can be treated on the base of a hierarchy of polycrystalline model structures taking into account the phase content, the structural inhomogeneity of the crystallites and the spatial distribution (position and orientation) of grains with different lattice disorder. The applicability of the multiplet approach has been proved recently for the substructure analysis in textured metallic materials using X-ray and neutron diffraction [103].

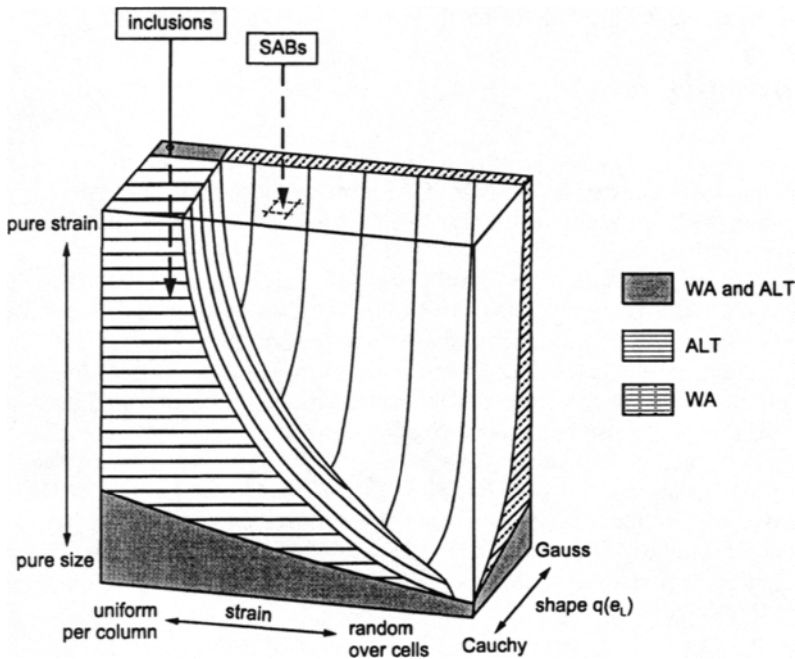


Figure 7. Regions of applicability of the WA and the alternative (ALT) analysis [91]. The arrows indicate the broadening caused by misfitting inclusions and small-angle boundaries (SABs) (for details see [90,91]).

2.175 Importance of line profile analysis for modern engineering and its relation to X-ray stress analysis

Today, it is well-known that the materials used in modern engineering have to meet increasing demands with regard to their mechanical, thermal and/or chemical properties. Consequently, new materials as well as combinations of materials like ceramics or thin film structures on certain substrates are developed and tested in addition to the traditional materials like steel. They often exhibit a complex microstructure within the near surface region with steep gradients of residual stresses, texture and/or chemical composition. A characterization of the microstructure requires a combination of investigations by several methods which complement each other in a suitable way.

When performing an X-ray analysis, one should exploit the full physical information contained in the diffraction profile. In the field of engineering the attention is often focused on macrostresses only. In the field of materials science, however, it is well recognised that changes of the material properties on the microscale can be equally important for the performance of the material [89]. Therefore, the evaluation of microstructural properties from the line profiles measured for macrostress evaluation, by one of the methods described above, should yield valuable additional information on the microstress state, domain sizes and the defect structure within the gauge volume.

Finally, it should be emphasised that all X-ray diffraction procedures are integral methods by nature and, therefore, only yield indirect results on the structure (in contrast to the methods of X-ray topography applied in the field of single crystal analysis). This holds true especially of the methods in line profile analysis, where many mathematical assumptions have to be made on the state of the microstructure to be investigated. For this reason, additional information on the defect structure (dislocations, stacking faults etc.) obtained by direct methods like transmission electron microscopy (TEM) should be used in order to prove the validity of the models on which the line profile analysis is based.

2.176 Recommendations

The experimental effort required for line profile analysis will largely depend on the accuracy desired for the microstructural parameters. The fundamental rule that should be followed for obtaining the most reliable results can be expressed as follows: 'The smaller the structural broadening, i.e. the smaller the difference in broadening between h and g (profile), the more careful, accurate measurement of profiles is required' [25]. Fig. 8 gives a survey of the main points that should be paid attention to in planning the experiment. A few remarks will be added in the following:

1. The preparation of well-defined standard specimens is non-trivial. As suggested in [26] they should be of the same chemical composition as the material to be investigated with a crystallite size above $1\mu\text{m}$. For metals, suitable standard specimens are obtained by normalising rather than from powders.

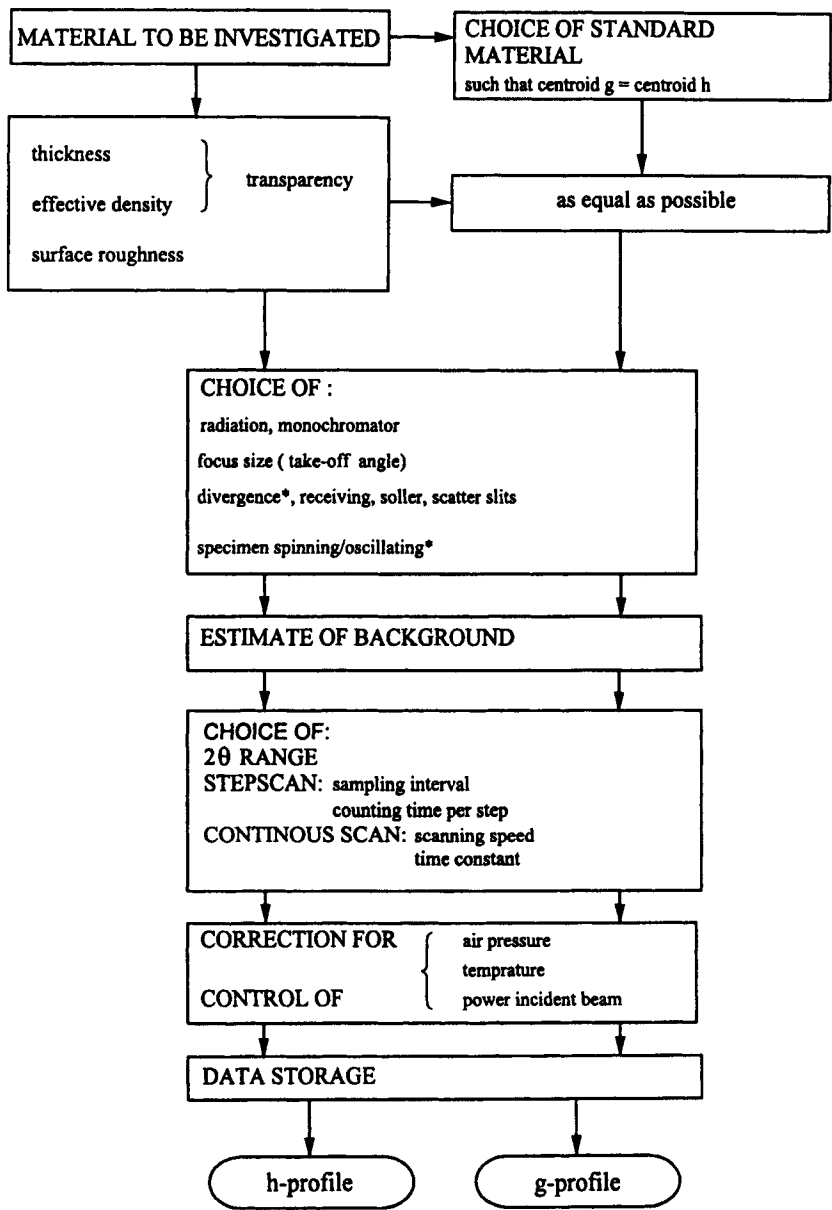
2. It is recommended to use the centroid of the h profile as a reference for the ideal standard profile which should be recorded such that the centroids of the h and g profiles coincide. The origins of both profiles should be chosen at the same position of the axis chosen (2θ -scale for $2\theta \leq 120^\circ$, reciprocal s -scale for $2\theta \geq 120^\circ$) in order to obtain the centroid of the resulting f profile at the origin.

3. It is emphasised to record both the h and the g profile under exactly the same experimental conditions. The use of a monochromator is strongly recommended to avoid complications concerning the α_1/α_2 -separation and to keep the background as low as possible. If available, a vacuum chamber on the diffractometer should be used to reduce air scattering. Fluorescence can be minimised by using suitable X-ray wavelength.

4. In order to improve the symmetry of the profiles, a small axial divergence (small aperture of the Soller slit system) is preferred. If necessary, the crystal statistics should be improved by oscillating the specimen around the θ -axis and/or rotating it around an axis perpendicular to its surface.

5. In order to minimise errors due to truncation, it is recommended to choose the measuring range as large as possible with regard to the constraints arising from overlapping neighbouring lines as well as the counting time available. Further, the sampling distance within the range chosen should be compatible with the required detailedness of information.

For more detailed and additional information the reader is referred to the comments and recipes given, for example, in [25,28], as well as to the corresponding Chapters in this book.



* These should be considered in relation to grain size (crystal statistics)

Figure 8. Outline of measuring procedure for X-ray diffraction line profiles [25].

List of principal symbols introduced in this chapter

A_L	Cosine Fourier coefficient;
a	period on the experimental 2θ - or the reciprocal $\sin\theta$ - scale;
a_0	lattice parameter of the cubic unit cell;
B	integral breadth (in degrees or radians 2θ) of the profile h from the specimen investigated;
B_L	sine Fourier coefficient;
b	integral breadth of the instrumental standard profile g ;
e_L	strain within the columns between two unit cells which are $n = L / D_{hkl}$ cells apart;
$\langle e_L \rangle^2$	mean square strain;
F_L	$A_L + i B_L$ complex Fourier coefficient of the profile f ;
f	structurally broadened profile;
G_L	complex Fourier coefficient of the profile g ;
g	instrumental standard profile;
H_L	complex Fourier coefficient of the profile h ;
h	$g * h$, profile from the specimen to be investigated;
h_0	$(h^2 + k^2 + l^2)^{\frac{1}{2}}$, order of the reflection;
L	$n \cdot D_{hkl}$, distance normal to the reflecting planes;
$\langle N \rangle$	$\lambda / (\beta \cos \theta)$, average crystallite size obtained from the integral breadth;
$\langle N_3 \rangle$	average column length (domain size) perpendicular to the reflecting planes;
p	crystallite size distribution function;
q	distribution function of the strains e_L ;
s	$2(\sin \theta - \sin \theta_{\max}) / \lambda$, variable on the reciprocal scale;
T	$\langle N_3^2 \rangle / \langle N_3 \rangle$, particle size obtained from the summarised Fourier size coefficients;
$2w$	full width at half maximum (FWHM) of the profiles;
$1.5\alpha + \beta$	term which contains the deformation and twin fault probabilities α and β , respectively;
β	integral breadth of the structural broadened profile f ;
ϵ	$\beta / (4 \tan \theta)$, microstrain obtained from the integral breadth.

Superscripts

S, D	Denotes that the parameter refers to size or distortion broadening;
f, g, h	denotes that the parameter refers to the f , g or h profile;

Subscripts

C, G	Denotes that the parameter refers to the Cauchy or Gaussian component of the profile;
V	denotes that the parameter refers to the Voigtian profile;
WA	denotes that the parameter is related to the Warren-Averbach analysis;
St	denotes that the parameter refers to a profile which was corrected by Stokes method.

2.177 References

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2.18 Residual stress analysis in single crystallites

W. Reimers

2.181 Introduction

From the view of diffraction methods polycrystalline materials have to be defined as coarse grained when the diffraction experiment gives evidence for a splitting of the Debye fringes into spatially localized Bragg reflections which can be attributed to individual crystallites. In this case the usually applied measuring and evaluation procedures for the analysis of residual stresses face two problems. Due to the inhomogeneous intensity distribution in the diffraction pattern, intensity measurements can be performed only at selected sample orientations. Furthermore the single crystal elastic anisotropy leads to a scatter of the experimentally obtained strain data [1] resulting in important error bars when calculating stress values under the usual assumption of elastic isotropy [2]. Since the number of crystallites contributing to the diffracted intensity is decisive for the observed intensity pattern, the transition from fine grained to coarse grained materials depends on the gauge volume under study, the experimental resolution and the grain size.

In applying X-ray diffraction in general only the near surface crystallites contribute to the diffraction pattern. Under usual experimental conditions the Debye fringes are splitted for crystallite diameters of $\varnothing \geq 100 \mu\text{m}$. By additional sample movements during the measurement, e.g. translation, rotation and inclination, the number of reflecting crystallites can be increased, so that the condition of quasi isotropic behaviour of the gauge volume may be fulfilled up to grain sizes of approximately $\varnothing \approx 200 \mu\text{m}$ [3, 4]. Using synchrotron radiation, however, separated Bragg reflections may already be observed at crystallite diameters of $\varnothing \approx 20 \mu\text{m}$ due to the high parallelity of the radiation. For neutron diffraction investigations a gauge volume of some mm^3 and a comparatively large divergence of the beam are usual due to intensity reasons. Grain size effects on the diffraction pattern are therefore to be expected at crystallite diameters of $\varnothing \geq 500 \mu\text{m}$.

In those cases where individual Bragg reflections are observed, the single crystal anisotropy has to be considered in calculating the stresses from experimental strain data. For this an evaluation of the orientation of the crystallite under study is necessary. Therefore in a first step the spatial orientation of two independent reflections (hkl) of the selected crystallite has to be determined. Using a four-circle diffractometer, this may be done in a straight forward and efficient way. From the knowledge about the spatial orientation of these two reflections the orientation matrix, which relates the crystallographic axes system to the fixed laboratory system, can be established. From there on the diffractometer settings for every reflection (hkl) to be studied are calculated and the precise reflection position is determined experimentally by a centering routine. This means that this method is well suited for an automatic measuring routine, which gives the strain tensor based on the experimentally determined diffraction angles 2Θ for several reflections of an individual crystallite. Since the

orientation of the crystallite has been determined, the stress tensor components are calculated from the experimental strain tensor components by applying Hooke's law for anisotropic materials.

The stress values obtained from the single grain measuring and evaluation technique represent the sum of the stresses of first and second kind [5]. By studying several neighbouring grains, the stress differences between the grains yield information about the second kind stresses whereas an averaging over the stress values gives the first kind stresses. Third kind stresses can be obtained either by analysis of the 2θ -profile or as a function of sample translation with respect to the measuring spot (X-ray or synchrotron radiation).

2.182 Historical review

The analysis of the deformation state of crystallites embedded in the polycrystalline matrix is of interest not only for the study of residual stress states but also for the investigation of crystallite-crystallite interactions as a typical physical feature of polycrystalline matter. By 1939 the inhomogeneity of elastic-plastic transition was studied by following the shift of Bragg reflections of different crystallites in a polycrystalline steel sample stepwise increasing the external uniaxial load [6]. Important progress in the interpretation of the results concerning the deformation behaviour of crystallites in polycrystals was achieved by applying methods for the systematic crystallographic indexing of the Bragg reflections [7]. For the measurements the Kipp-technique [8, 9] and the crystal-rotation technique [10] were applied in combination with film-techniques. The indexing was performed by graphical methods. The investigations on Fe-crystallites with grain sizes of 10mm^2 [8, 9] gave evidence for stress variations up to 100% between crystallites with different orientations. An increase of sensitivity for the registration of X-ray intensities and hence a reduction of the necessary measuring spot size under study could be realised by using Geiger-Müller counters. For this detector configuration, however, the precise spatial orientation of the Bragg reflection to be measured has to be known before hand. This needs the prior orientation of the crystallite, e.g. using the Laue-technique. Applying this two-step procedure, measurements were performed on single crystalline epitaxial layers [11, 12] and on an Al-bicrystal [13], where the stress profile over the diameter of the large crystals (grain size $\sim 3\text{ cm}^2$) was registered quasi point wise with a measuring spot diameter of $200\text{ }\mu\text{m}$. Important stress variations were observed near the grain boundaries. The in-situ orientation of the crystallites to be investigated is possible using a three circle diffractometer which allows the angular rotation of the sample by two axes perpendicular to each other. Based on this experimental set up, deformation measurements were performed on Fe-crystallites [14, 15] and the procedures for stress analyses on single crystalline Si-wafers are given in [16].

In comparison to three circle diffractometers the use of four circle diffractometer allows a more systematic and hence less time consuming analysis of the orientation of the crystallite to be studied [17-19]. Furthermore the additional sample rotation axis realised in this diffractometer concept enables the investigations of Bragg reflections at different incident angles of the radiation. So the penetration depth of the X-rays can be varied over a comparatively large range, hence allowing the analysis of near surface stress gradients, e.g. in Si-wafers [20, 21]. The use of four-circle diffractometers for the analysis of coarse grained materials is also supported by its already wide spread application in the field of texture Figures [23].

2.183 Basic principles of the single grain measuring technique

2.183a Evaluation of the orientation matrix

The geometry of a four-circle-diffractometer and the definition of the laboratory axes system are illustrated schematically in Figure 1. The crystallite selected for the study is located at the instrument center. Using X-rays the selection of the crystallite can be done either by collimating the beam or by shielding the sample surface with thin Pb-foils. Using neutron diffraction the 90 degree scattering technique is applied which guarantees that the gauge volume keeps constant for all sample orientations.

The incident and diffracted beams are in the horizontal plane. The counter (here: det) is moved in this plane about the vertical instrument axis ω and makes an angle 2Θ with the primary beam direction (here: col). The whole Eulerian cradle may be rotated around the vertical axis by ω . The χ -axis is in the horizontal plane and makes an angle ω with the primary beam direction. The sample is rigidly attached to the φ -shaft which is supported by the χ -ring. The rotation sense for the rotation movements is defined mathematically positive.

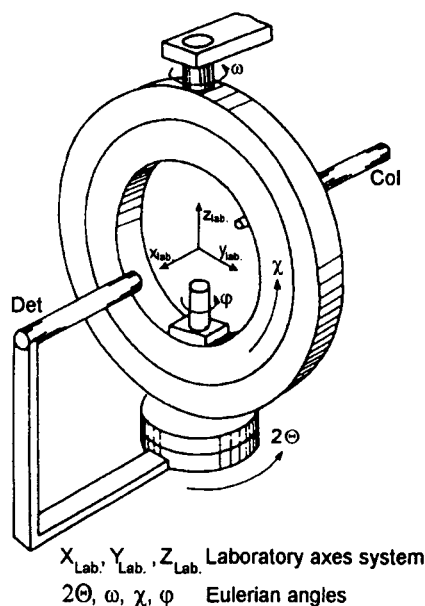


Figure 1. Four-circle-diffractometer.

The orientation of the crystallite under investigation is known when it is possible to relate its axes system to the fixed laboratory system. This coincides with the Φ -axes system when all instrument angles are zero.

x_{lab} parallel to the primary beam direction

y_{lab} parallel to the direction of the reflected beam for $2\Theta = 90^\circ$

z_{lab} parallel to the ω -axis

For transforming the systems it is convenient to define them in terms of Cartesian axes. A crystal direction $\underline{h}$ is therefore described by a Cartesian crystal direction $\underline{h}_c$:

$$\underline{h}_c = B\underline{h}$$

$$\text{for } \underline{h} = \begin{pmatrix} h \\ k \\ l \end{pmatrix}$$

$$\text{and } B = \begin{pmatrix} b_1 & b_2 \cos \beta_3 & b_3 \cos \beta_2 \\ 0 & b_2 \sin \beta_3 & -b_3 \sin \beta_2 \cos \alpha_1 \\ 0 & 0 & 1/a_3 \end{pmatrix} \quad (1)$$

a_i , α_i and b_i , β_i are the direct and reciprocal lattice parameters, respectively. Since $\underline{h}_c$ shall be described in terms of the Φ -axes system also the instrumental angles ω , χ , φ are represented in form of Cartesian axes systems Ω , X , Φ :

$$\Phi = \begin{pmatrix} \cos \varphi & -\sin \varphi & 0 \\ \sin \varphi & \cos \varphi & 0 \\ 0 & 0 & 1 \end{pmatrix}$$

$$X = \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos \chi & -\sin \chi \\ 0 & \sin \chi & \cos \chi \end{pmatrix}$$

$$\Omega = \begin{pmatrix} \cos(\omega - \omega_0) & -\sin(\omega - \omega_0) & 0 \\ \sin(\omega - \omega_0) & \cos(\omega - \omega_0) & 0 \\ 0 & 0 & 1 \end{pmatrix} \quad (2)$$

with $\omega_0 = 2\Theta/2$.

The angular set ω , χ , φ gives the diffractometer position where reflected intensity is observed. The corresponding crystal direction can then be described in the Φ -axes system in form of an unit vector $\underline{u}_\Phi$:

$$\underline{u}_\Phi = \tilde{\Phi} \tilde{\chi} \tilde{\Omega} \begin{pmatrix} 0 \\ 1 \\ 0 \end{pmatrix} \quad (3)$$

$\sim$ = transposed matrix

In the case of residual stress measurements, the material investigated and hence the cell parameters are usually known approximately. So the diffracting angle for a reflection $\underline{h}$ can be calculated and the detector is positioned. The spatial orientation of a reflection $\underline{h}_1$ is then searched by systematic variation of φ and χ with fixed ω . The angular position of this reflection is then denoted $\omega_1, \chi_1, \varphi_1$. The plane of a second reflection $\underline{h}_2$ is then defined by its angular relationship to the first reflection. In cases, where the angle between $\underline{h}_1$ and $\underline{h}_2$ has to be 90° for crystallographic reasons, $\underline{h}_2$ is found by a systematic search using $\Delta\omega$ -steps in the plane $(\chi_1 + 90^\circ, \varphi_1)$. The observation of two non-collinear reflections of known indices $\underline{h}_1, \underline{h}_2$ is sufficient to obtain the orientation matrix U [24] which transforms vectors in the crystal cartesian axes system into the Φ -axes system:

$$\underline{h}_{1\Phi} = U \underline{h}_{1c} \quad \underline{h}_{2\Phi} = U \underline{h}_{2c}$$

with $\underline{h}_{1\Phi} \parallel \underline{u}_{1\Phi}$

$$\underline{h}_{2\Phi} \parallel \underline{u}_{2\Phi} \quad (4)$$

Because of experimental errors and uncertainties in the knowledge of the cell parameters, the orthogonal matrix U does not, in general, satisfy both conditions. Therefore, an orthogonal unit-vector triple $\underline{t}_{1c}, \underline{t}_{2c}, \underline{t}_{3c}$ in the crystal Cartesian axes system is defined where $\underline{t}_{1c}$ is parallel to $\underline{h}_{1c}$, $\underline{t}_{2c}$ lies in the plane of $\underline{h}_{1c}$ and $\underline{h}_{2c}$ and $\underline{t}_{3c}$ is perpendicular to $\underline{t}_{1c}$ and $\underline{t}_{2c}$. In the Φ -axes system another triple $\underline{t}_1, \underline{t}_2, \underline{t}_3$ is defined based in the same way on $\underline{u}_{1\Phi}$ and $\underline{u}_{2\Phi}$. Since these two unit vector triples are orthogonal by definition they can be superimposed exactly onto each other:

$$\underline{t}_{i\Phi} = U \underline{t}_{ic} \quad i=1,2,3 \quad (5)$$

In matrix notation:

$$\underline{T}_\Phi = U \underline{T}_c \quad (6)$$

Then follows:

$$U = \underline{T}_\Phi \tilde{\underline{T}}_c \quad (7)$$

2.183b Angle calculations for any reflection (hkl)

After having evaluated the orientation matrix by two reflections, the coordinates in the Φ -axes system can be calculated using the orientation matrix UB for any reflection (hkl):

$$\underline{h}_{\Phi} = UB\underline{h}$$

$$\text{with } \underline{h}_{\Phi} = \begin{pmatrix} h_{\Phi 1} \\ h_{\Phi 2} \\ h_{\Phi 3} \end{pmatrix} \quad (8)$$

For the detector position the Bragg equation is used. The interplanar lattice spacing D is given by:

$$D = \frac{1}{\left(h_{c1}^2 + h_{c2}^2 + h_{c3}^2\right)^{1/2}} \quad (9)$$

The diffractometer setting for the symmetric position ($\omega_0 = 2\Theta/2$) is then obtained by:

$$2\Theta = 2 \arcsin\left(\lambda / 2 \cdot \left(h_{c1}^2 + h_{c2}^2 + h_{c3}^2\right)^{1/2}\right)$$

$$\omega_0 = \frac{2\Theta}{2}$$

$$\chi_0 = \arctan\left(\frac{h_{\Phi 3}}{\left(h_{\Phi 1}^2 + h_{\Phi 2}^2\right)^{1/2}}\right)$$

$$\varphi_0 = \arctan\left(\frac{h_{\Phi 1}}{h_{\Phi 2}}\right) \quad (10)$$

with λ = wavelength and index $_0$ for the symmetric position.

2.183c Rotation around the scattering vector

The rotation of the reflecting plane (hkl) around the scattering vector allows a systematic variation of the incidence angle of the beam for all cases where the reflecting plane is not parallel to the sample surface. So for most cases the penetration depth of the beam can be chosen in a defined way so enabling the analysis of strain-stress gradients. It is also sometimes useful to rotate the crystal around the scattering vector by the angle τ in order to

avoid the absorption of the incident or reflected beam by the Eulerian cradle or by the sample. With $\tau = 0^\circ$ the rotation matrix R_0 for the bisecting position is defined by:

$$R_0 = \Omega_0 \cdot X_0 \cdot \Phi_0 \quad (11)$$

The new rotation matrix is then calculated

$$R_\tau = \mathcal{T} \cdot R_0$$

$$\text{with } \mathcal{T} = \begin{pmatrix} \cos \tau & 0 & \sin \tau \\ 0 & 1 & 0 \\ -\sin \tau & 0 & \cos \tau \end{pmatrix} \quad (12)$$

The τ -rotation axis is defined to be parallel to the y_{lab} -axis for $\omega = \chi = \varphi = 0^\circ$.

R_τ is then generally represented by:

$$R_\tau = R = \Omega \cdot X \cdot \Phi \quad (13)$$

The solution of the equation system yields the new diffractometer setting for the reflection position after the rotation by the angle τ :

$$\begin{aligned} \omega &= \arctan\left(\frac{R_{\tau 13}}{-R_{\tau 23}}\right) \\ \chi &= \arctan\left(\frac{(R_{\tau 31}^2 + R_{\tau 32}^2)^{1/2}}{R_{\tau 33}}\right) \\ \varphi &= \arctan\left(\frac{R_{\tau 31}}{R_{\tau 32}}\right) \end{aligned} \quad (14)$$

2.183d Analysis of the mosaic spread

The mosaic spread describes the angular distribution of the lattice planes (hkl) of the crystallite under study. The mosaic spread is usually measured in form of rocking curves, where the reflecting plane is rotated in $\Delta\omega$ -steps through its reflection position keeping the detector at fixed 2Θ . Due to the atomic structure of crystals, the mosaic spread may be anisotropic, e.g. after plastic deformation. So it is necessary to measure the rocking curves around different crystal axes. Therefore, the diffractometer has to be positioned in such way that, at the same time, the reflecting conditions for the reflection $\underline{h}_1$ to be measured are

fulfilled and the crystal direction $\underline{h}_2$ is parallel to the ω -rotation axis [25, 26]. Hence the symmetric position for the reflection $\underline{h}_2$ is calculated. The calculated angle φ_{02} (symmetric position, $\underline{h}_2$) is kept fixed and χ is rotated corresponding to $\chi_1 = \chi_{02} + 90^\circ$. The reflection position for the reflection $\underline{h}_1$ to be investigated is then calculated by solving the following system with respect to Ω :

$$R_{01} = \Omega \cdot X_1 \cdot \Phi_{02} \quad (15)$$

R_{01} is the rotation matrix for the symmetric position of the reflection $\underline{h}_1$.

2.183e Strain and stress tensor

Since the strain tensor is defined as a symmetric tensor of second rank, at least 6 reflections have to be analysed concerning their precise reflection position. The procedure for the reflection position determination and the necessary correction factors to be applied are described in paragraph 2.184. According to Bragg's law the refined 2Θ -position gives the corresponding $D(\underline{h})$ -value so that the strain in $\underline{h}$ is obtained with:

$$\varepsilon(\underline{h}) = \frac{D(\underline{h}) - D_0(\underline{h})}{D_0(\underline{h})} \quad (16)$$

Some possibilities to determine the lattice spacings of the unstressed sample $D_0(\underline{h})$ are discussed case wise in paragraphs 2.186 and 2.187.

For expressing the measurements $\varepsilon(\underline{h})$ in terms of strain tensor components ε_{kl}^c , the cartesian crystal axes system is chosen as a fixed reference system:

$$\varepsilon(\underline{h}) = n_k \cdot n_l \cdot \varepsilon_{kl}^c \quad k, l = 1, 2, 3 \quad (17)$$

In this notation $\underline{n}$ is a unit vector parallel to the scattering vector whose components n_k are given in the cubic system by:

$$n_k = \frac{h_k}{\sqrt{h_1^2 + h_2^2 + h_3^2}} \quad (18)$$

For non orthogonal crystal axes systems, an orthogonalization has to be performed:

$$\varepsilon(\underline{h}) = p_k \cdot p_l \cdot \varepsilon_{kl}^p \quad k, l = 1, 2, 3 \quad (19)$$

Here the components p_k are defined by:

$$p_k = \alpha_{kl}^{-1} \cdot n_l \quad k, l = 1, 2, 3 \quad (20)$$

α_{kl} are the components of the orthogonalization matrix. In homogeneous media the symmetric strain tensor $\underline{\epsilon}^c$ can be determined by six measurements in non-coplanar directions. If more information is available a least-squares refinement can be applied. The stress tensor components σ_{ij}^c are calculated by applying Hooke's law for anisotropic, quasielastic materials. Here, the single crystal elastic constants are inserted:

$$\sigma_{ij}^c = c_{ijkl} \cdot \epsilon_{kl}^c \quad i, j, k, l = 1, 2, 3 \quad (21)$$

Whereas in the triclinic crystal system 21 independent elastic constants are present, their number is reduced to three ($c_{1111} \neq c_{2222} \neq c_{1212}$) in the case of the cubic system. The resulting stress tensor $\underline{\sigma}^c$ is referred to the cartesian crystal axes system whose orientation is dependent on the crystal orientation. In most cases, especially for comparing the values obtained in crystals which are in direct neighborhood to each other, it is preferable to transform the stress tensor into the macroscopic sample system. As the first step, the stress tensor $\underline{\sigma}^c$ is transferred into the fixed laboratory system (index L). As transformation matrix the UB-matrix can be used:

$$\underline{\sigma}^L = \tilde{T} \cdot \underline{\sigma}^c \cdot T \quad (22)$$

In the second step the stress tensor $\underline{\sigma}^L$ has to be transformed into the common reference system. Therefore, the angles describing the orientation of the reference system relative to the laboratory system have to be determined. The transformation matrix T is then represented by a rotation matrix R, which components are given by ω, χ, ϕ .

2.184 Experimental details and data correction

2.184a Adjustment of the diffractometer and reflection centering

After an optical preadjustment of the diffractometer set up, the final adjustment is performed analysing the reflection positions of crystallographically equivalent reflections of a single crystal. According to the procedure described in [27] a reflection (hkl) of the test crystal is measured in 8 equivalent positions (Table 1):

Table 1. 8 symmetrically equivalent reflection positions

	T	D	A	
1	2 Θ	ω	χ	ϕ
2	2 Θ	ω	$-\chi$	$\phi+180^\circ$
3	-2 Θ	$-\omega$	$-\chi+180^\circ$	ϕ
4	-2 Θ	$-\omega$	$180^\circ-\chi$	$\phi+180^\circ$
5	-2 Θ	$-\omega$	$-\chi$	ϕ
6	-2 Θ	$-\omega$	$-\chi$	$\phi+180^\circ$
7	2 Θ	ω	$\chi-180^\circ$	ϕ
8	2 Θ	ω	$180^\circ-\chi$	$\phi+180^\circ$

Each reflection position is determined precisely applying a centering routine. With φ fixed, some cycles of independent ω , χ and ω - 2Θ -step scans are performed until the shift from one cycle to the next is smaller than 0.01° . For each cycle the intensities are corrected for background and the net intensities are then used to calculate the peak position by the center of gravity. The systematic comparison of the 8 reflection positions measured gives then the data for the readjustment of the diffractometer:

Δx -translation of the diffractometer:

$$\Delta x = -0.5 \frac{C_y \cdot R_c + S_y \cdot R_s}{\sin \Theta} \quad (23)$$

$$\text{with } C_y = 0.125 \cdot (T[1] - T[2] - T[3] + T[4] - T[5] + T[6] + T[7] - T[8]) + S_y$$

$$S_y = 0.125 \cdot (-D[1] + D[2] + D[3] - D[4] + D[5] - D[6] - D[7] + D[8])$$

R_c - distance sample to counter

R_s - distance source to sample

Δy -translation of the diffractometer:

$$\Delta y = -0.5 \cdot \frac{C_x R_c + S_x R_s}{\cos \Theta} \quad (24)$$

$$\text{with } C_x = 0.125 \cdot (T[1] - T[2] - T[3] + T[4] + T[5] - T[6] - T[7] + T[8]) - S_x$$

$$S_x = 0.125 \cdot (D[1] - D[2] - D[3] + D[4] + D[5] - D[6] - D[7] + D[8])$$

Δz -translation of the diffractometer:

$$\Delta z = 2 \cdot \frac{C_z \cdot \sin \Theta (R_c + R_s)}{\cos \omega \cdot R_c \cdot R_s} \quad (25)$$

$$\text{with } C_z = 0.125 \cdot (-A[1] - A[2] - A[3] - A[4] + A[5] + A[6] + A[7] + A[8])$$

$$\text{counter height } \Delta z_c: \quad \Delta z_c = -2 \cdot \frac{\sin \Theta}{\cos \omega} \cdot R_c \cdot \Delta \chi \quad (26)$$

$$\text{with } \Delta \chi = 0.125 \cdot (-A[1] - A[2] + A[3] + A[4] + A[5] + A[6] - A[7] - A[8])$$

2.184b Selection of a crystallite in a polycrystalline environment

Using X-ray or synchrotron radiation only near surface crystallites are accessible due to the limited penetration depth of these radiations. So the measuring spot can be defined applying thin Pb-diaphragms (thickness $8\text{ }\mu\text{m}$) on the sample surface. The measuring spot is then optically adjusted to the center of the Eulerian cradle by means of X, Y, Z translations. After having established the orientation matrix, several reflections are measured at $\tau = 0$ (symmetrical position) and at $\tau = 180^\circ$ (rotation around the reflecting vector) for checking and optimizing the adjustment of the crystallite under study.

Due to the penetration depth of neutrons in the magnitude of some 10 mm in principle all crystallites along the path of the primary beam may give an intensity signal. Therefore small diaphragms (approximately $1 \times 1\text{ mm}^2$) before and after the sample are set up for protecting the gauge volume. Moreover it is useful to perform the reflection search at approximately $2\theta \sim 90^\circ$ by adopting the wavelength of the neutron beam to the D-spacing of the selected reflection. This way it is assured that the sample volume under study is kept unchanged for all sample orientations (Figure 2).

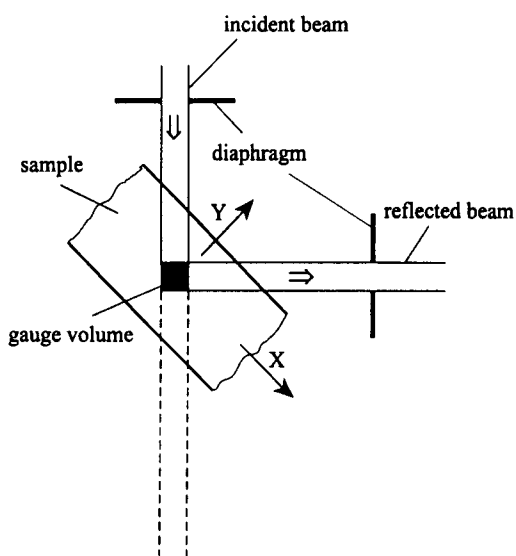


Figure 2. 90° scattering technique.

The refining of the adjustment of the crystallite selected in the center of the diffractometer is done by comparing the peak positions of Friedel pairs (hkl) , $(\bar{h}\bar{k}\bar{l})$, which are usually both accessible using neutron diffraction by combining reflection and transmission mode measurements.

2.184c Special experimental set ups and measuring routines

Due to the monocrystalline properties of the crystallites, the observed intensity to background ratio is in most cases excellent in comparison to the effort necessary for measuring methods based on the analysis of Debye fringes. It is therefore possible to reduce the measuring spot down to crystallite sizes of approximately $40\text{ }\mu\text{m}$ [28].

Investigating large crystallites or monocrystals the intensity situation then allows to perform high resolution X-ray diffraction experiments when. Figure 3 demonstrates the experimental set up of a high resolution double crystal diffractometer yielding a primary beam divergence to $2'$.

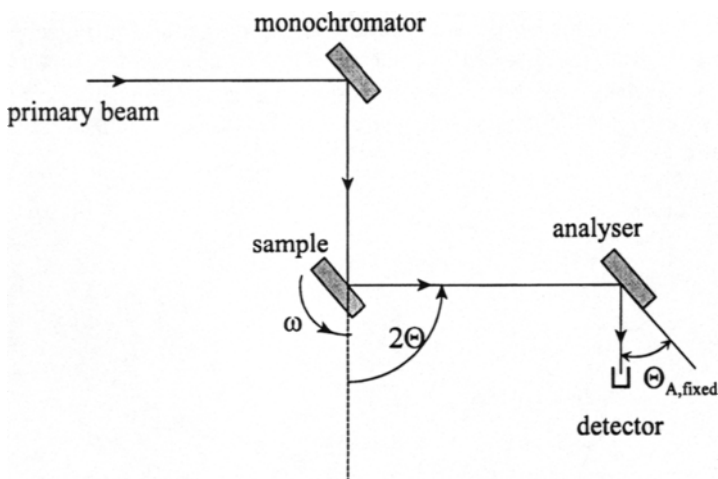


Figure 3. High resolution double crystal diffractometer.

A high resolution arrangement was successfully used for the analysis of the stress distributions in γ -hardened monocrystalline Ni-base superalloys. Technical monocrystals may exhibit important orientation distributions, which can make difficult the definition of the reflection center. For the analysis of the intensity distribution therefore a mapping technique was applied including the measurement of rocking curves as a function of the diffraction angles 2Θ . The high resolution of the experiment then allows the integration over the rocking angles ω . This projection on the 2Θ -axis results in a function independent from the mosaicity [29]. Furthermore a high accuracy for the determination of the absolute Bragg angles is achieved by applying the Bond-method [30] which means measurements at positive 2Θ as well as at negative 2Θ .

2.184d Correction factors and data processing

The data processing and the data correction of the experimental reflection profiles is analogous to the procedure described in chapter 2.04.

2.185 Deformation behaviour of crystallites under applied load

Neutron diffraction in combination with the single-grain measuring and evaluation technique is suited for the analysis of residual volume stresses in large grained materials or also for the investigation of crystallite-crystallite interactions. Since the deformation of the whole grain is measured, the effect of the free surface can be neglected.

As sample material for the experiments under external load the Ni-base alloy IN 939 was chosen. The grain sizes range from $\varnothing \approx 1$ mm up to $\varnothing \approx 5$ mm. Flat tensile specimen were prepared and installed in a tensile apparatus which allows the application of defined uniaxial stresses. The applied stress is examined by calibrated strain gauges on the sample holder. The grain selection, the measurements and the data processing were performed as described in paragraph 2.184. In each flat tensile specimen several grains were investigated. The grains were investigated at three stress levels within the elastic deformation region ($\sigma_{\text{ext}} = 0; 340$ MPa; 510 MPa) [31]. The comparison of the interplanar lattice spacings of Friedel-equivalents (D_{hkl} and $D_{\bar{h}\bar{k}\bar{l}}$) indicates that $\Delta D_{hkl} / D_{hkl}$ could be determined for most of the reflections with an accuracy of $\pm 0.1 \%$.

The strain values $\epsilon(hkl)$ are calculated from the D_{hkl} values measured at the different stress levels σ_{ext} by means of a least-squares fit of the regression coefficient from D_{hkl} vs. σ_{ext} . The strain tensor ϵ_{exp} is refined including the whole set of reflections for the applied stress of $\sigma_{\text{ext}} = 510$ MPa. Using the least-squares algorithm, a weighting factor of $1/\sigma^2$ was applied, where σ is the standard deviation of each measurement as obtained from the regression coefficient. The typical agreement between the experimental and calculated values of $\epsilon(hkl)$ is demonstrated for one grain in Table 2.

Table 2. Comparison between the experimental and calculated strains $\epsilon(hkl)$ (in 10^{-6})

h	k	l	$\epsilon(hkl)_{\text{cal}}$	$\epsilon(hkl)_{\text{exp}}$	$\sigma(\epsilon(hkl)_{\text{exp}})$
2	0	0	3149	3270	140
0	2	0	-1338	-1300	30
0	0	2	-1029	-1070	30
2	2	0	952	910	30
0	2	2	-1136	-1130	40
2	0	2	1003	1040	30
2	-2	0	850	840	20
-2	0	2	1115	1120	10
0	2	-2	-1230	-1220	50

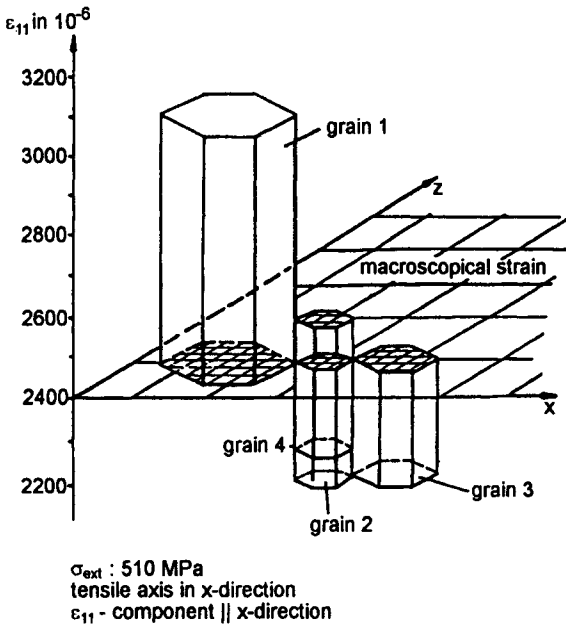


Figure 4. Comparison of the macroscopical and microscopical strain tensor component ϵ_{11} .

The relative position of the grains in specimen 1 and the tensor component ϵ_{11} in the direction of the tensile axis are shown in Figure 4. For comparison, the mean macroscopic strain tensor component ϵ_{11} ($\epsilon_{11} = 2400 \times 10^{-6}$) is shown as a reference plane.

The observed strain variations in the sample amount up to 30 %. The strong deformation of grain 1 is due to its special orientation where the crystal direction [100] is almost parallel to the applied stress direction. Thus grain 1 exhibits a larger longitudinal deformation than the mean value ϵ_{11} . The smaller grains are less deformed so that the deformations of the individual grains are almost averaged out over the four grains.

Considering the macroscopical strain value in tensile direction and the corresponding value for the free single crystal for grain 1 it is evident that in the matrix an intermediate value is realized (Figure 5).

Due to the volume relationship and to the favourite orientation of grain 1, the neighbouring grains are relatively compressed so that their elongations along the tensile axis are smaller than the free single crystal elongation and the mean value, respectively.

Whereas neutron diffraction gives integral deformation and stress values for the crystallites under investigation, the strain-stress distribution over the grain diameter can be studied by synchrotron radiation, whose high parallelity allows a high resolution registration of the deformation state. Experiments performed at HASYLAB, DESY, also on the Ni-base alloy IN 939, gave evidence for inhomogeneities over the grain diameter (Figure 6), which are in the same order of magnitude as the variations found from grain to grain.

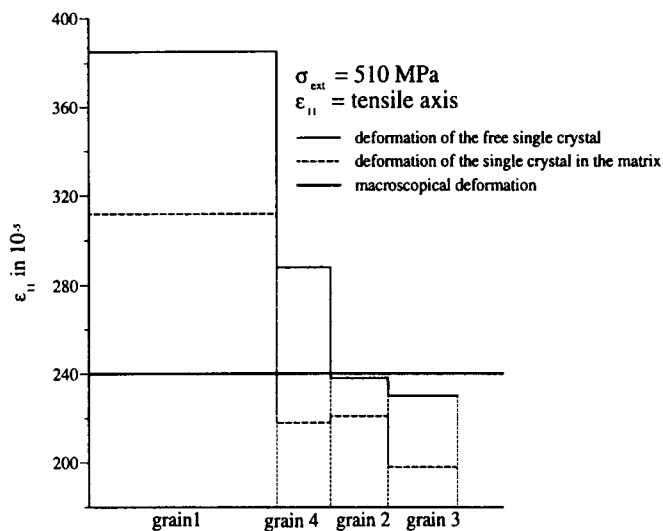
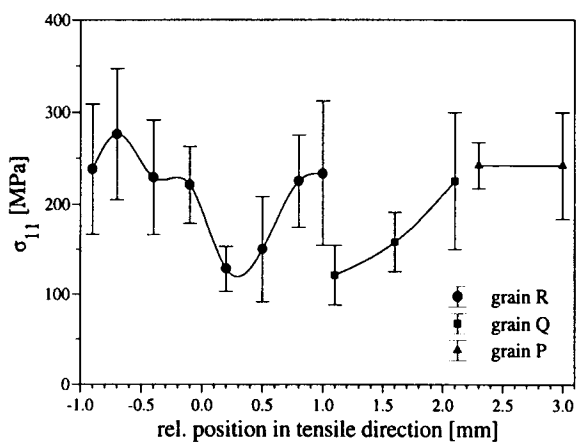


Figure 5. Strain tensor component ϵ_{11} .



$\sigma_{\text{ext}} = 248 \text{ MPa}$

Measuring spot: $250 \times 250 \mu\text{m}^2$

σ_{11} -component || tensile direction

Figure 6. Variation of load stresses, measured by synchrotron radiation [35].

2.186 Residual stress analysis

2.186a Residual stress in a welding zone of a ferritic steel

X-ray diffraction experiments are especially suited for near surface investigations but also for investigations with high local resolution. Since strong stress gradients can be expected in the vicinity of weldings, often X-ray diffraction is used for their analysis. The heat impact of the welding process, however, may lead to a coarsening of the grains, so that the conventional measuring technique can no longer fulfill the demand for small measuring spot sizes. For the investigation of the stress distribution in a plasma welded steel (German grade: X8 Cr17) [28] the single grain measuring technique was applied. The rough positions of the crystallites selected in the sample surface were defined by applying a lead diaphragm. The correct positioning of the diaphragm in the X, Y directions on the sample surface was checked by intensity measurements. The grain boundaries and the grain numbering are shown in Figure 7.

The orientations of the crystals were determined by means of analysing the angular orientations of the {200} and {110} reflections. The measurements of the interplanar lattice spacings then were performed on the {211} reflections. For the calculation of the stress tensor components the single crystal elastic constants for Fe were used. For the evaluation it was assumed $\sigma_{33} = \sigma_{13} = \sigma_{23} = 0$. From these conditions the value D_0 was refined individually for each grain. A good agreement was found for the grains investigated with $D_0 = 2,87264$ (25). This result gives evidence that the assumption $\sigma_{i3} = 0$ is fulfilled in this sample due to the small penetration depth of the Cr-K α -radiation. Furthermore, there is no evidence for significant variations in the chemical composition.

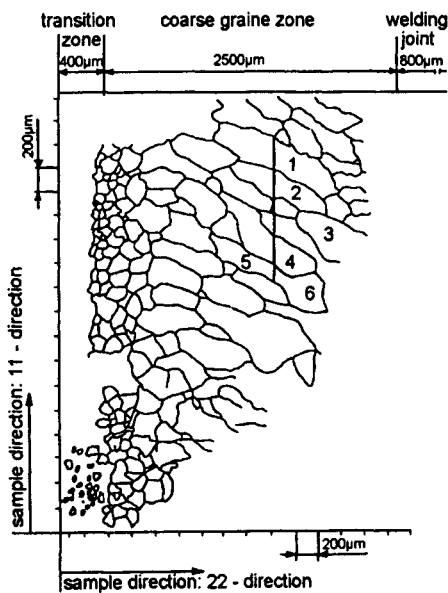


Figure 7. Grain boundaries corresponding to the micrograph and numbering of the grains investigated.

The results of the measurements in the coarse grain zone are summarized in Table 3.

Table 3. Stress tensor components in individual grains situated in the coarse grain zone of a plasma welding joint (value in MPa, standard deviations in parenthesis)

Stress tensor components (σ_{11} parallel and σ_{22} perpendicular to the welding joint direction, σ^m : main stresses)

grain no.	σ_{11}	σ_{22}	σ_{12}	σ_{11}^m	σ_{22}^m
1.	-141(31)	-198(26)	-76(27)	-88(25)	-250(32)
2.	-29(41)	-156(28)	-156(32)	75(38)	-261(32)
3.	-82(17)	-73(17)	-112(16)	34(10)	-190(29)
4.	-38(35)	-22(19)	-52(31)	22(28)	-83(35)
5.	-163(28)	-145(24)	-143(26)	-10(5)	-297(29)
6.	-108(36)	-80(28)	-169(31)	75(19)	-263(28)
Ø value	-94(13)	-112(10)	-118(11)	18(10)	-224(13)

Table 3 gives evidence for the extent of stress variations from grain to grain. The evaluation of the deformation data obtained from the individual grains gives evidence that the stress tensor component σ_{12} is significantly different from zero. This finding is interpreted as a result of the heat distribution during the plasma welding process. At any time the heat is very localized, so that a radial stress component arises due to the temporary displacement of the heat impact during the welding process. The diagonalization of the stress tensor for the individual grains shows that in fact a nearly uniaxial stress state is present.

2.186b Residual stresses in a polycrystalline γ' -hardened nickelbase-superalloy

γ' -hardened nickelbase-superalloys are of practical interest since the microscopic stresses induced by a lattice parameter mismatch between the γ -matrix and the γ' -particles are made responsible for the excellent mechanical-technological properties, e.g. the high temperature creep resistance.

Choosing the polycrystalline nickelbase-superalloy IN 939 as an example the macroscopic and microscopic residual stresses were investigated at defined intervals especially in the early stage of high temperature cyclic loading ($T = 850\text{ C}$) [33].

From the sample material flat tensile specimens were prepared by electroerosion and subsequent electrolytical polishing of a surface zone of approximately $50\text{ }\mu\text{m}$. Since the heat impact of the electroerosion extends over a depth of $\sim 200\text{ }\mu\text{m}$, tensile residual stresses could be produced in the near surface zone, which were then quantitatively analysed by X-ray diffraction. The average grain diameter in the specimen is $\bar{\phi} \approx 1\text{-}2\text{ mm}$. In these grains the γ' -particles (with a diameter of $\bar{\phi} \approx 200\text{ nm}$) are orientationally coherent with the matrix. Since furthermore the lattice parameters of γ and γ' differ only in the order of magnitude of 1‰, the reflections of the two phases are superimposed, Figure 8.

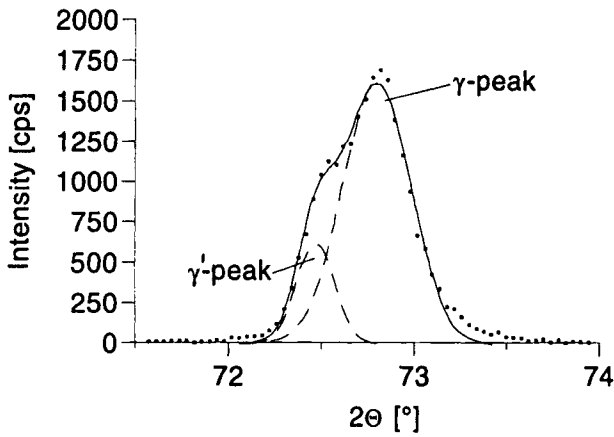


Figure 8. X-ray reflection profile from the γ' -hardened Ni-base-superalloy IN 939.

The analysis of the reflection profiles in different crystalline directions revealed the same 2θ -splitting and the same intensity ratios for the γ - and γ' -phase. So in a first step the reflection profiles were evaluated using the center of gravity method. These data then result in integral residual stress values for the crystallite under study, which represent the sum of the stresses of first and second kind. The in plane stresses for the investigated crystallites in three different samples A, B, C are displayed in Figure 9.

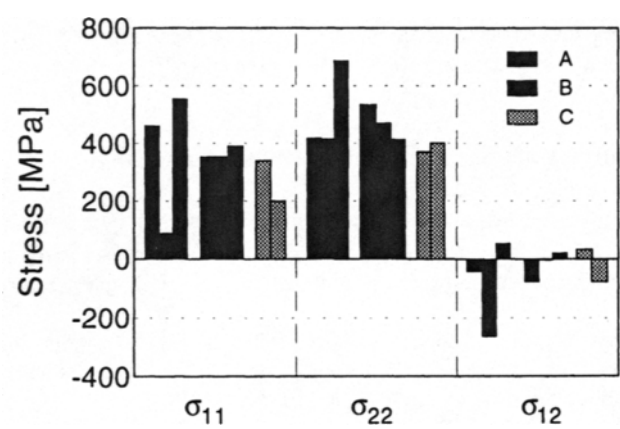


Figure 9. Stress values for the investigated crystallites.

As discussed in paragraph 2.185, the stress variations from grain to grain are due to the elastic anisotropy of the crystallites and to the crystallite-crystallite interactions in the

polycrystal. The averaging over the stress tensor components for the investigated crystallites gives values of $\sigma_{11} = (350 \pm 140)$ MPa and $\sigma_{22} = (450 \pm 100)$ MPa. These stress values are the macroscopic surface stresses (stresses of first kind), which are due to the above mentioned sample treatment. Consequently it could be shown that after further electrolytical polishing, the macroscopic stresses vanish.

The development of the macroscopic residual stresses in the early stage of cyclic loading is demonstrated in Figure 10. This figure shows the stresses for three grains in sample A which was subjected to an upper stress level of $\sigma_u = 300$ MPa and for further three grains in sample B with an upper stress level of $\sigma_u = 350$ MPa. The stresses were found to be lowered significantly after 16 cycles reaching nearly zero for both samples. This stress reduction can be explained by a prior plastic deformation of surface material caused by the tensile residual stresses in the initial state. As known from stretching, a technically applied method e.g. for residual stress reduction, the plastic deformation has caused an equalization of the strong inhomogeneous stress distribution between surface and bulk material. The macroscopic stresses then stayed near the zero level with increasing load cycles.

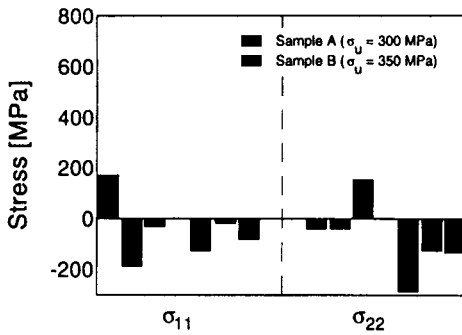


Figure 10. Residual stresses of individual grains after 16 cycles at 850°C (σ_{11} : perpendicular to the load axis, σ_{22} : parallel to the load axis).

A more detailed insight in the actual stress situation in the NI-base alloy IN 939 can be obtained also by analysing the stresses in the γ - and the γ' -phases. For this, the reflection profiles (see Figure 8) are evaluated separately for the γ and γ' -phases. The intensity analysis yields the volume ratio of the phases. In agreement with the data published in [34], the γ' -volume was determined to (30 ± 5) %. The splitting in 2θ of the γ - γ' -profiles gives the corresponding ΔD -values. These experimental data are composed from two components:

1. The difference in the crystallographic lattice parameters of the γ - and the γ' -phase due to the different chemical composition. This difference is used for the calculation of the γ - γ' -misfit:

$$\delta = \frac{a_{\gamma'} - a_{\gamma}}{1/2(a_{\gamma'} + a_{\gamma})} \quad (27)$$

2. The individual strain states of the γ - and the γ' -phase due to the coherency of the phases. In this context it has to be mentioned that the heat treatment of the material strongly influences the degree of coherency present in the heterogeneous material. It could be shown for some alloys by [35] that an extensive heat treatment reduces the coherency until the unconstrained misfit corresponding to equation (27) is achieved.

So from the point of view of diffraction investigations at the given material the analysis of the ΔD -values gives the constrained misfit. For the sample material investigated in this study the misfit was determined to be $\delta_{\text{constrained}} = 3.8\%$ and no anisotropy could be found. A further quantitative separation of this value into the parts unconstrained misfit and coherency strains requires the knowledge of the D_0 -values for the γ - and the γ' -phases.

Using X-ray diffraction with its near surface information depth, the D_0 -value can mostly be calculated by benefiting from the boundary condition $\sigma_{33} = 0$. This, however, is not admitted in the case of heterogeneous materials. Especially the diameter of the γ' -particles with less than $0.2 \mu\text{m}$ in comparison to the penetration depth of the X-radiation with $10 \mu\text{m}$ indicates that already three dimensional stress states are registered. Therefore, the coherency stresses between the γ - and the γ' -phases had to be estimated by using the approximation proposed [39], where about 40% of the measured in-situ misfits are attributed to coherency strains. Using this assumption tensile coherency stresses of up to 300 MPa in the matrix are calculated. Considering this data and adding the stresses of first and second kind in Fig. 9, maximum local tensile stresses up to 700 MPa are present in the sample material in the initial state. Due to the high tensile prestresses in the γ -matrix, the cyclic loading leads to strong changes of the stress values of the matrix phase. The γ' -phase, however, shows compressive stresses in the initial sample state. Consequently, this phase is much less affected by the external load and its stress values are less changed.

2.187 Residual stress analysis in technical single crystals

2.187a Monocrystalline γ' -hardened nickelbase superalloys

The nickelbase superalloy SC 16 is the single crystalline successor of polycrystalline IN 738 LC used as stationary gas turbine blade material. Profiting from the anisotropy of the single crystal structure, an improvement of the creep resistance in $[001]$ -direction at higher temperatures is achieved. During creep the γ' -precipitates undergo directional coarsening from spherical or cuboidal into a more platelike morphology [37-39]. This shape change is controlled by diffusion and internal stresses so that there is an important interest in characterizing the stress states in the initial material state as well as after creep deformation.

The orientational distribution of the dendrites in monocrystalline nickelbase superalloys and also the small lattice parameter mismatch between the γ -matrix and γ' -particles ($\delta \sim -1\%$) leads to an overlap of the reflection contributions due to the dendrites and to the two phases. Therefore high-resolution X-ray measurements were performed using the mapping technique described in section 2.184c. After integrating the intensity data over the orientation angles ω , asymmetric reflection profiles are obtained, whose interpretation needs the introduction of a model about strain distributions on the microstructural scale (Figure 11).

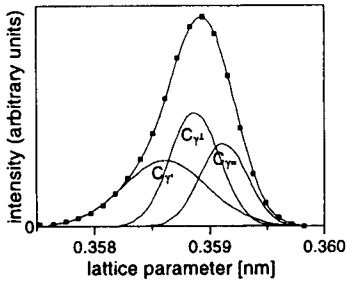


Figure 11. Example for an integrated reflection profile of a SC 16 specimen, (400) - the initial material state.

REM-micrographs of the initial material state give evidence for a cuboidal morphology of the γ' -particles, whereby their diameter is about 400 nm and their volume fraction amounts to approximately 40% [40]. Due to the coherency between the matrix phase and the γ' -particles, microstresses are present which require the distinction between D-spacings of the lattice planes parallel and perpendicular to the γ/γ' -interfaces. So for a measuring direction [100], three reflection contributions have to be expected:

γ' -particles with an intensity contribution of $\sim 40\%$,

γ -matrix with lattice planes parallel to the interface with an intensity contribution of $\sim 20\%$ and γ -matrix with lattice planes perpendicular to the interface with an intensity contribution of $\sim 40\%$ (Figure 12).

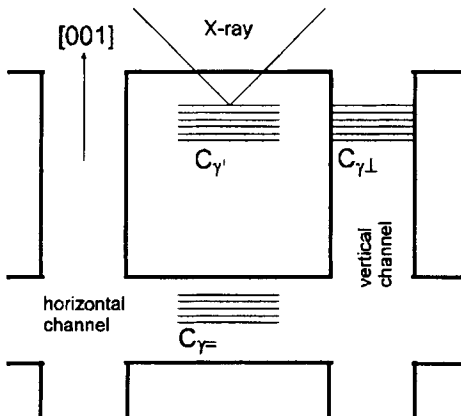


Figure 12. Schematic distinction of lattice parameters in the initial state [41].

Due to the reduced crystallographic symmetry of the γ' -phase, superstructure reflections e.g. of type (h00) with $h = 2n + 1$ are present. The measurement of these reflections is used for fixing the γ' -reflection contribution in the fundamental reflections of type (h00) with $h = 2n$.

The deconvolution of the intensity profile yields the following constrained lattice parameters for the undeformed material state [42]:

$a_{\gamma'}$		$c_{\gamma=}$
0.35863(3) nm	0.35885(3) nm	0.35913(3) nm

For the calculation of stress values additionally the stress free lattice parameters are necessary.

The idealized microstructure can be used to calculate this data by applying equilibrium conditions. The mechanical equilibrium condition (macroscopical) is given by:

$$\iint \sigma_{ii} dx_j dx_k = 0 \quad (28)$$

This condition must be fulfilled in a unit cell if the precipitates are arranged periodically. Particularly, the stresses perpendicular to the $\gamma / \gamma' -$ interface must become minimized:

$$\sigma_{33} = 0 \quad (29)$$

Perpendicular to the interface, the lattice can be strained free resulting in minimal stress values (here assuming to be 0). In the interface the coherency condition causes a plain stress state. Using Hooke's law for anisotropic media with the stiffness c_{ij} , the stress tensor component for horizontal orientated matrix channels $\sigma_{33\gamma}$ is given by:

$$\sigma_{33\gamma} = c_{11}\epsilon_{33} + c_{12}(\epsilon_{11} + \epsilon_{22}) \quad (30)$$

In equation (4) the strain components are now replaced by the expressions of the lattice parameters:

$$\sigma_{33\gamma} = c_{11} \frac{c_{\gamma=} - a_0\gamma=}{a_0\gamma=} + 2c_{12} \frac{a_{\gamma=} - a_0\gamma=}{a_0\gamma=} \quad (31)$$

Solving this equation, the stress free lattice parameter for the matrix can be calculated:

$$a_0\gamma= = \frac{\frac{c_{11}}{2c_{12}} c_{\gamma=} + a_{\gamma=}}{1 + \frac{c_{11}}{2c_{12}}} \quad (32)$$

The same procedure can be applied to the vertical matrix channels. So, stress free lattice parameters can be calculated directly from experimental data and absolute strain and stress values can be given. By means of the phase equilibrium condition, the stress state of the $\gamma' -$ phase can also be determined:

$$\Delta f_{\gamma}, \sigma_{ii\gamma'} = -\Delta f_{\gamma} \sigma_{ii\gamma} \quad (33)$$

$\Delta f_{\gamma'}$, Δf_{γ} -volume fractions of the γ' -phase and of the γ -phase

Then, the stress free lattice parameter of the γ' -phase can be calculated as follows [43]:

$$a_{0\gamma'} = \frac{c_{11}c_{\gamma'} + 2c_{12}a_{\gamma'}}{\sigma_{33\gamma'} + c_{11} + 2c_{12}} \quad (34)$$

This stress free lattice parameter can be used to calculate the full strain and stress tensor in the precipitates and in each kind of matrix channel. A reference such as the undeformed material state is not necessary. Applying the mechanical equilibrium conditions corresponding to equation (32) and (34) and assuming a γ' -volume fraction of 40%, the stress free lattice parameters can be determined:

$a_{0\gamma'} = 0.35859(3)\text{nm}$ $a_{0\gamma} = 0.35898(3)\text{nm}$

Using the elastic constants c_{ij} of this material [44], the coherency stress tensor can be calculated:

$$\sigma_{\gamma'} = \begin{pmatrix} 90 & 0 & 0 \\ 0 & 90 & 0 \\ 0 & 0 & 90 \end{pmatrix} [\text{MPa}] \quad \sigma_{\gamma} = \begin{pmatrix} -90 & 0 & 0 \\ 0 & -90 & 0 \\ 0 & 0 & 0 \end{pmatrix} [\text{MPa}]$$

The matrix shows compressive stresses in plane. Due to the symmetry of the γ/γ' unit cell a hydrostatic stress state in the γ' -precipitates is found. These coherency stresses can be used as a reference to characterize the deformation-induced changes.

Further investigations have been prepared on creep deformed sample material. The sample material was creep deformed in [001]-direction at 950°C with creep stresses of 120 MPa, 150 MPa and 200 MPa, respectively [43]. TEM observations and the X-ray reflection profile analysis demonstrate that the γ' -volume ratio is not changed significantly under the given creep conditions.

In order to analyse the strain/stress state in the creep deformed samples the model for the interpretation of the reflection contributions has to be extended. Due to the creep axis [001] as preferred direction, the deformation behaviour in horizontal and vertical channels has to be distinguished (Figure 13).

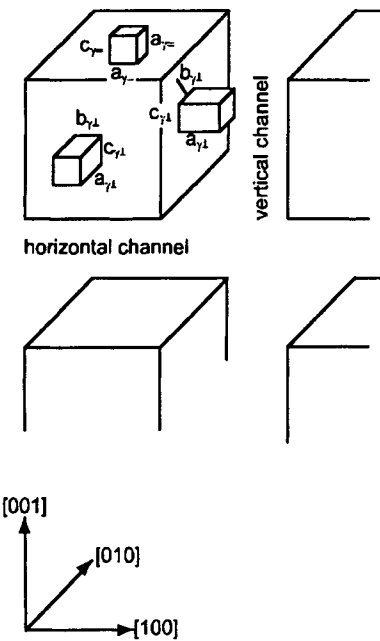


Figure 13. Schematic distinction of lattice parameters after creep deformation.

According to Figure 13, three reflection contributions have to be expected for the deformed state for a (00l)-reflection with $l=2n$: the γ -phase contributes with c_{γ} , the horizontal channels with $c_{\gamma=}$ and the vertical channels with $c_{\gamma\perp}$ lattice spacings having an intensity weight of 1/3 and 2/3 of the matrix, respectively (Figure 14).

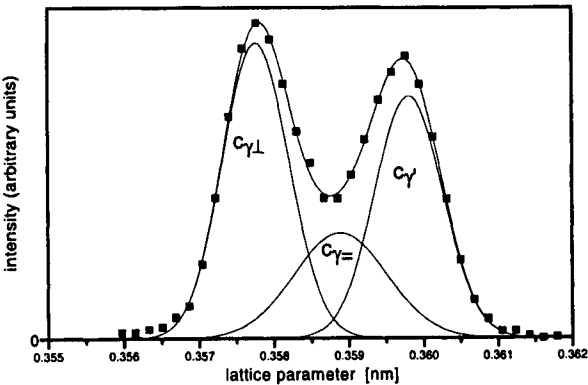


Figure 14. Integrated reflection profile (004) of a SC 16 specimen creep deformed at 200 MPa.

For analysing (0k0)- or (h00)-reflections measured in the deformed state a further distinction between the D-spacings in both channel types has to be introduced due to due to their different orientations relative to the external load axis. Figure 13 indicates that an orthorhombic lattice distortion arises for the vertical channels whereas a tetragonal distortion is expected in the horizontal channels. So for (0k0)- or (h00)-reflections the following contributions have to be expected: a_γ for the γ -phase and the matrix lattice spacings $a_{\gamma\parallel}$ for horizontal channels and $a_{\gamma\perp}$ and $b_{\gamma\perp}$ for vertical channels where each matrix contribution has a weight of 1/3. The assignment of the matrix contributions to the different channel types can be done by comparison with FEM-calculations [41] which predict tensile stresses in the horizontal and compressive stresses in the vertical channels. Furthermore the mechanical equilibrium must be satisfied leading to the condition that stresses normal to the interfaces have to be close to zero. It is a prerequisite condition of the data analysis method described here that the shear components of strains and stresses vanish.

The lattice parameters obtained from the evaluation are presented in Figure 15 and Figure 16 for the different material states.

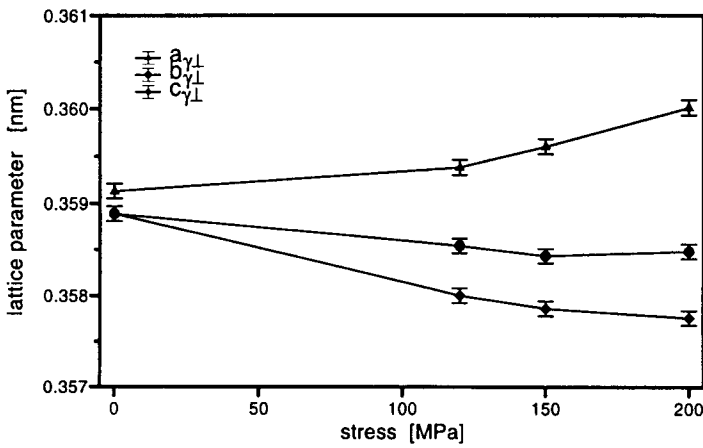


Figure 15. Lattice parameters as defined in Figure 13 for the vertical channels vs. applied creep stress.

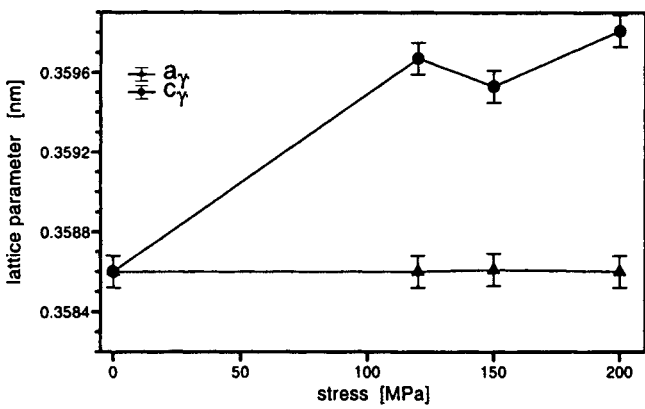


Figure 16. Lattice parameters as defined in Figure 13 for the γ -phase vs. applied creep stress.

Again using equation (32) and (34) for the D_0 -calculation, and equation (21) for the calculation of the stresses, the results summarized in Figure 17 were obtained.

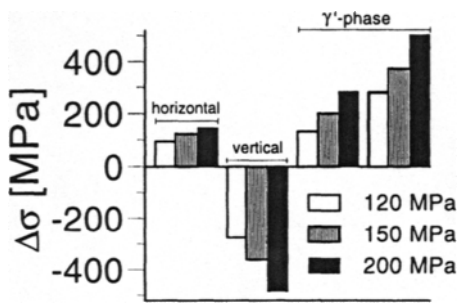


Figure 17. Creep induced stresses for different creep stresses.

After creep deformation tensile stresses can be found inside the horizontal matrix channels. Very high compressive stresses occur inside the vertical channels. Due to the mechanical equilibrium, the stress state inside the precipitates shows a strong hydrostatical component. The absolute stress values seem to be proportional of the external applied load in the investigated range of stress rates. Further results on stress analyses in single crystalline nickelbase superalloys are reported in [42, 43, 45-51].

2.187b Grinding stresses in silicon wafers

In microelectronics industry abrasive machining (e.g. ID cut-off grinding, surface grinding, lapping) is used to manufacture thin silicon wafers as substrate materials as well as for the thinning of completely processed wafers. By these treatments crystal damage is caused and residual stresses are induced. These stresses lead to geometrical deformations of the wafers, which are not compatible with the achieved high degree of automatic control in the microchip technology. They may also induce the formation of dislocations, which can influence the diffusion behaviour of impurity atoms. So for optimizing the manufacturing of Silicon wafers information about near surface crystal damage and residual stresses is required.

Due to the brittleness und hardness of the material steep stress gradients are present in the near surface zone. So for the analysis of these inhomogeneous stress fields in axial direction several series of measurements each with a defined information depth have to be performed. Different radiations (Cr-K α and Cu-K α) are used for this measurements. Furthermore, the beam path of the X-rays in the sample and hence the penetration depth of the radiation depends on the incidence angle of the X-rays relative to the sample surface. So the selection of the reflection type (hkl) to be measured and the individual orientation of the wafer during measurement are further parameters for defining the thickness of the surface zone investigated. The penetration depth p of each reflection can be calculated from the Eulerian angles (the sample surface is parallel to the φ -axis):

$$p = \frac{1}{\mu} \frac{(\cos \chi - 1) \cdot \sin(2\Theta - \omega) \cdot \cos \varphi + \sin(\omega + \varphi)}{(\cos(\chi - 1) \cdot \cos \varphi \cdot (\sin \omega + \sin(2\Theta - \omega)) + \sin(\omega + \varphi) + \sin(2\Theta - \omega - \varphi)) \cdot (\cos \varphi (\cos \chi - 1) \sin(2\Theta - \omega - \varphi))} \quad (35)$$

For a given reflection (hkl), the maximum penetration depth is obtained in the symmetrical diffractometer position ($\omega_0 = 2\Theta/2$). The penetration depth can be raised over a wide range by rotating the sample around the scattering vector (see section 2.183b). The penetration depths are given for a list of reflections in Table 4.

Table 4. Penetration depths of Silicon reflections: wafer orientation [100], symmetrical position.

Cu-Kα (λ = 0.15418 nm)			Cr-Kα (λ = 0.22909 nm)		
penetration depth p	hkl	2Θ [°]	penetration depth p	hkl	2Θ [°]
5.0 μm	111	28.4	2.4 μm	111	42.8
	113	56.1		113	88.7
	133	76.4		133	133.7
	115	95.0	4.8 μm	220	73.2
	135	114.3			
10.1 μm	220	47.3			
	242	88.1			
	260	127.7	7.2 μm	311	88.7
15.1 μm	311	56.1		311	133.7
	331	76.4	9.6 μm	400	115.0

For each penetration depth a sufficient number of measurements have to be performed. In a first step the stress states in four different, defined depths are considered whereby the stress values $\sigma(p)$ represent the integral of the stress distribution over the penetration depth p :

$$\sigma(p) = \frac{\int \sigma(z) \cdot e^{-z/p} dz}{\int e^{-z/p} dz} \tag{36}$$

For the stress analysis only plane stress states are considered . The D_0 values were measured at non-machined Silicon wafers of the same series the ground wafers are taken from. The stress tensor was calculated in the crystal axes system and afterwards transformed into the reference system of the grinding process. In this way the σ_{11} axis is defined parallel to the grinding direction and the σ_{22} axis perpendicular to it.

Table 5 presents the residual stresses for single-side ground wafers obtained for two penetration depths. In both cases compressive stresses are found. As expected the stresses perpendicular to the grinding direction are larger than those parallel to it. The degree of anisotropy of the stress distribution is lower in the case of tangential grinding. This can be explained with the different kinematics of the two grinding processes, because in contrast to plunge grinding in the case of tangential grinding the machined wafer is additionally moved perpendicular to the main grinding direction [52]. The absolute values of the stresses decrease rapidly with increasing penetration depth, indicating that the surface compressive stresses are compensated by tensile residual stresses in the bulk material at a material depth of a few microns. Therefore, it can be concluded that the absolute values of the actual stresses at the surface are even higher than the results for $p = 2.4 \mu\text{m}$ and that steep stress gradients are present.

Table 5: Residual stresses of ground Silicon wafers

penetration depth [μm]	plunge ground	tangential ground	error
2.4	$\sigma_{11} = -6 \text{ MPa}$ $\sigma_{22} = -152 \text{ MPa}$	$\sigma_{11} = -116 \text{ MPa}$ $\sigma_{22} = -208 \text{ MPa}$	$\pm 40 \text{ MPa}$
7.2	$\sigma_{11} = -18 \text{ MPa}$ $\sigma_{22} = -58 \text{ MPa}$	$\sigma_{11} = -49 \text{ MPa}$ $\sigma_{22} = -80 \text{ MPa}$	$\pm 40 \text{ MPa}$

In order to obtain the stresses at the surface and the stress gradients, results for additional penetration depths were added to the data of table 5. Figure 18 shows a sufficient data set $\sigma_{22}(p)$ of a tangential ground wafer.

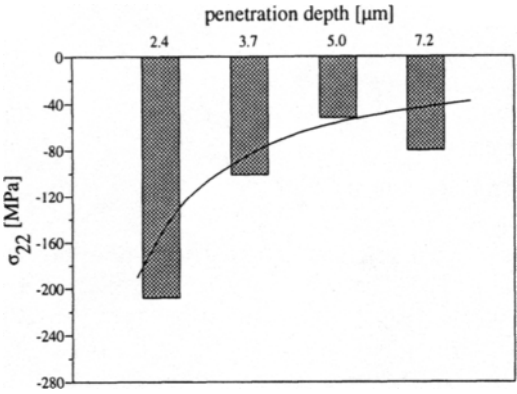


Figure 18. Residual stresses $\sigma_{22}(p)$ for a tangential ground wafer.

The continuous line in the diagram represents the following fit function:

$$\sigma(\tau) = a \left[\left(\frac{1}{\tau + \alpha} \right)^{-2} - z_0 \left(\frac{1}{\tau + \alpha} \right)^{-1} \right] \quad (37)$$

Equation (37) was chosen as fit function because it represents the inverse Laplace transformation from the general and usual form to describe stress depth profiles:

$$\sigma(z) = a(z - z_0) \exp(-\alpha z) \quad (38)$$

For the backtransformation from $\sigma(p)$ to $\sigma(z)$ mathematical tables were used.

Figure 19 shows the actual stress depth profile evaluated from the data given in Figure 18. The diagram shows surface stresses of about -550 MPa and a transition depth to tensile stresses of 1.8 μm .

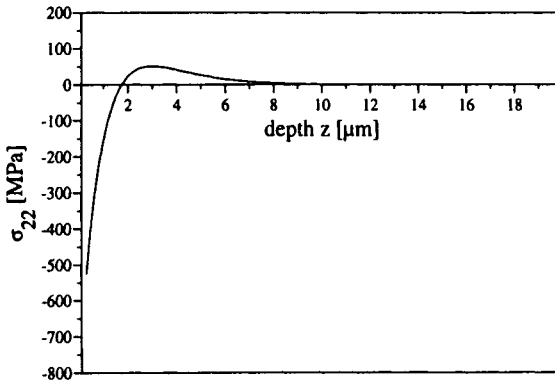


Figure 19. Stress depth profile evaluated from the data of Figure 18.

The formalism applied to the σ_{22} data of a plunge ground wafer yields surface stresses of -300 MPa and a transition depth of 1.7 μm . Although the surface stresses are extrapolated and hence affected with important error bars undoubtedly the maximum stresses are present in a near surface zone of an axial extension less than 2 μm .

2.188 Summary and outlook

Residual stresses in large grained polycrystalline and in single crystalline materials can be analysed using the single crystal measuring and evaluation technique. The physical precondition for applying single crystal diffraction methods is the splitting of Debye fringes into localized Bragg peaks with sufficient intensity. These Bragg peaks are observed the smaller the experimental divergence and the smaller the number of crystallites illuminated. So applying neutron diffraction methods with usual gauge volumes in the order of some mm^3 and comparatively low angular resolution crystallite diameters of $\sim 1 \text{ mm}$ are necessary for analysing Bragg reflection positions. Using X-ray and synchrotron radiation, crystallite diameters down to $\sim 40 \text{ }\mu\text{m}$ can be investigated up to now. Here a new future program can be expected from the use of recently developed glass capillary focusing systems which allow the reduction of the diameters of synchrotron and X-ray beams from $\varnothing = 300 \text{ }\mu\text{m}$ to $\varnothing \sim 10 \text{ }\mu\text{m}$ with only small intensity losses. This way it seems possible to analyse the strain and stress state of crystallites with diameters of $\varnothing \sim 10 \text{ }\mu\text{m}$ hence achieving a local resolution of the stress analysis which may be of interest in the field of experimental fracture mechanics as well as investigating microelectronic devices.

The stress values obtained from the single crystal measuring and evaluation technique represent the sum of stresses of first and second kind. If only the stresses of first kind are of interest, measurements on several differently oriented crystallites with a subsequent averaging over the individual stress states is necessary. Here the determination of the different orientation matrices is the time determining step. An important speed up of this procedure can be expected from the use of currently available two-dimensional position sensitive detectors. At appropriate sample to detector distances angular ranges are covered which allow the simultaneous registration of two reflections, and so enabling a direct orientation matrix determination.

This technical progress will also enhance the use of the single crystal measuring and evaluation method for microstructural investigations on the crystallite-crystallite interactions in the elastic as well in the plastic deformation region. Due to the complexity of the polycrystalline state statistical functions e.g. for the orientation distribution function or the orientation correlation function have to be introduced which need a data base built up from the investigation minimum of 150 crystallites [53].

2.189 Recommendations

It is recommended

to check the adjustment of the crystallite selected for the investigation in the precise center of the diffractometer by performing intensity versus X- and Y-translational scans and subsequent τ -scans

to use a reflection centering routine for the reflection position measurement

to measure a number of independent reflections which is at least twice the number of unknown parameters, e.g. σ_{ij} , D_0

to establish the list of reflections to be measured under the aspect of covering the widest possible range in φ and χ

to include the Friedel pairs (hkl) , $(\overline{h}\overline{k}\overline{l})$ in the list of reflections to be measured when using neutron diffraction.

2.1810 References

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3 Neutron diffraction methods

L. Pintschovius

3.1 Historical review

X-ray stress analysis is a well-established method for the non-destructive measurement of residual stresses as well as load stresses. Using this methodology and replacing X-rays by neutrons, it is possible to obtain information on stresses not only from near surface regions, but also from the bulk of the material, as neutrons can penetrate deeply (some cm) into most materials. The feasibility of the method has been demonstrated more than ten years ago by three independent groups [1-5]. The early experiments aimed at validating the method by investigating samples of known strain. The results of these experiments was very encouraging and therefore the method was soon applied to problems of practical importance. As a consequence, further groups joined the field. To get started, they usually performed a validation experiment of their own, so that the neutron diffraction method is now amply tested and can be considered as fully trustworthy. In particular, it has been well established that neutron and X-ray results are perfectly compatible with each other.

Over the years, the number of groups working in this field has grown continuously and neutron stress analysis was applied to a variety of problems. In the beginning, most investigations were of a fundamental character, and investigations of this kind still keep an important role in neutron stress analysis. However, in the meantime more and more groups have established close contacts with industry [6] and sometimes neutron stress analysis is conducted as a commercial service.

For a long time, the studies were carried out on available instruments which were not designed for stress analysis and hence not optimized for this application. Often, the special set-up for neutron stress analysis had to be improvised and cleared away after the experiment to make room for experiments of a totally different nature. The obvious inconvenience of such working conditions led to the conversion of existing diffractometers into dedicated instruments for stress analysis. However, it is only very recently that dedicated instruments for stress analysis are built from scratch, so that the special requirements of stress analysis can be fully given consideration to. The dedicated instruments will allow one to fully utilize the potential of the technique. This will widen the application field of neutron stress analysis and will help to establish it as a routine method.

3.2 Principles

Neutron stress analysis is completely analogous to X-ray stress analysis, i.e. it is based on precise measurements of interplanar lattice spacings in different directions of the sample. The notations and basic formula of X-ray diffraction apply to neutron diffraction as well, in particular Bragg's law:

$$D_{\{hkl\}} = \frac{\lambda}{2\sin\Theta_{\{hkl\}}} \quad (1)$$

which connects the neutron wavelength λ and the scattering angle $\Theta_{\{hkl\}}$ of a diffraction peak with the interplanar spacing $D_{\{hkl\}}$, $\{hkl\}$ being the Miller indices. For further details see the Chapter 2 in this book. In the following we will restrict ourselves to the neutron specific points.

The wavelength λ of thermal neutrons is of the order of 1 Å, i.e. about the same as that of the most widely used X-ray tubes. Hence the same reflection lines are used in X-ray and in neutron stress analysis or at least lines with similar D spacing. In case of complex structures with more than one chemical species in the unit cell the structure factors $S_{\{hkl\}}$ of the different reflection lines may be very different in the X-ray and the neutron case: for X-rays the atomic scattering power is proportional to the square of the atomic charge, Z^2 , whereas the neutron cross section varies in an irregular manner with Z . As a consequence, different reflection lines may give optimum intensities for X-rays or neutrons.

In general, materials suited for X-ray stress analysis are suited for neutron stress analysis as well. There are a few exceptions where the atoms are either too strongly absorbing (e.g. Cd and B) or where a combination of positive and negative neutron scattering lengths leads to a near cancellation of the coherent cross-section (e.g. Ti-Al-alloys with high Al content). The reader interested in verifying that a particular material is suited for neutron stress determination is referred to Ref. 7 where the neutron absorption and neutron scattering cross-sections are tabulated.

As neutron and X-ray stress analysis share the same basic principles, they both

- (1) can be applied to crystalline solids only.
- (2) register only elastic strains, i.e. they inherently discriminate between elastic and plastic deformations. Strong plastic flow may show up by side effects such as reflection line broadening and / or texture.
- (3) apply to residual as well as load stresses. As neutron spectrometers can accommodate bulky items, it is not difficult to load a specimen under investigation.
- (4) are phase specific. Stresses can be determined for each crystalline phase of a multiphase material provided this phase is sufficiently abundant ($\geq 10\%$).
- (5) are sensitive to macro- and microstresses. In the presence of large microstresses neutrons do not better, but also not worse than X-rays to isolate macrostresses.

- (6) are really non-destructive (see also Section 3.6).

The most important difference between neutrons and X-rays is their penetrating power, which is typically 3 orders of magnitude larger for neutrons. This allows one to investigate stresses in the interior of technical components, which is the major reason to use neutrons instead of X-rays.

3.3 Instruments for stress measurements

There are two types of neutron sources for the production of intense neutron beams, i.e. research reactors and accelerator based installations, so-called spallation sources. In reactors the neutrons are released after fission of U nuclei, whereas in spallation sources neutrons are generated via the disintegration of heavy nuclei (Bi, W, U etc) by means of bombardment with high energy (600...1000 MeV) proton beams. In both cases the as-created neutrons are much too fast to be useful for diffraction purposes, and so they have to be slowed down ("thermalized") in a moderator made of light elements (light water, heavy water or beryllium). In the end the neutron spectra provided by reactors and spallation sources are similar to each other. There is, however, an important difference between the two types of sources concerning the time-structure: whereas reactors (except the Dubna reactor) are steady-state sources, spallation sources (except the Würenlingen installation) are pulsed sources. Depending on the time-structure of the source, different types of instruments are best suited for stress measurements.

3.31 Instruments for steady-state sources

Neutrons can be monochromatized either by a Bragg reflection on a suitable single crystal or by selecting a narrow velocity band with the aid of mechanical choppers. Diffractometers operated at reactors are nearly exclusively based on single crystal monochromators, and hence this section will be devoted to this type of instrument. There has been a recent attempt to develop a chopper-based instrument for stress measurements at a reactor [8,9]. It is very similar to a typical instrument used at a pulsed source and therefore will be dealt with in the next section.

A schematic view of a crystal diffractometer is depicted in Fig. 1. The polychromatic ("white") beam coming from the neutron source (i.e. the moderator) is narrowed down by Soller slits with divergence α_1 and then impinges on the crystal monochromator. The wavelength λ of the Bragg reflected neutrons depends on the interplanar lattice spacing $D_{\{hkl\}}$ and the take-off angle $2\Theta_m$ according to Bragg's law (Equ.1). The monochromatic beam passes through another Soller collimator with divergence α_2 , is then diffracted by the sample and finally reaches the detector through a third collimator with divergence α_3 . Slits in the incoming and the diffracted beam define the internal probe region in the sample (see Sect. 3.5.1a). The wavelength is usually chosen such as to make the scattering angle $2\Theta_s$ at the sample close to $2\Theta_s = 90^\circ$ as this choice will lead to the best spatial resolution for given slit widths.

As neutron measurements are very time-consuming great care has to be taken to properly choose the angular divergences α_i of the collimators, the monochro-

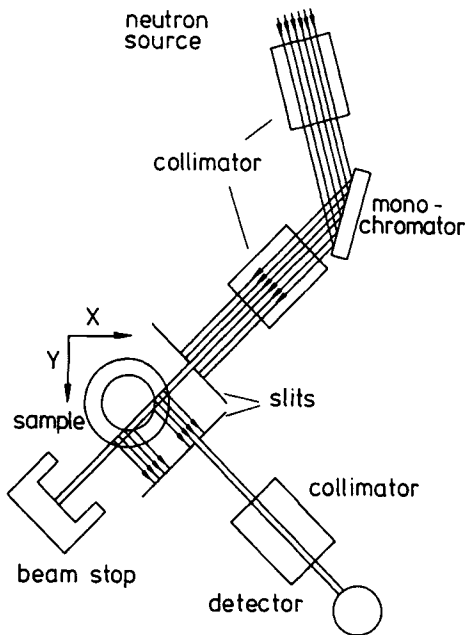


Fig. 1. Schematic drawing of a neutron diffractometer for stress measurements

mator take-off angle $2\Theta_m$ and the monochromator D spacing. Optimization has often been based on the experience with general-purpose neutron diffractometers. Margaca [10] has shown that the special requirements of stress measurements may lead to a different optimal configuration. The following guide-lines can be given:

- (i) The monochromator take-off angle $2\Theta_m$ should be large, i.e. $90^\circ \leq 2\Theta_m \leq 140^\circ$, and the monochromator D-spacing has to be chosen accordingly to obtain the required neutron wavelength. Unfortunately, not many neutron diffractometers allow one to meet this requirement, either because of geometrical constraints or because of the non-availability of suitable monochromator crystals. In such a case it may be advantageous to use a so-called triple-axis spectrometer instead of an ordinary diffractometer. These instruments are not designed for diffraction measurements, but are usually very flexible and therefore can be operated in a high-resolution diffractometer mode. The difference with respect to a diffractometer sketched in Fig. 1 lies in the fact that the beam diffracted by the sample is Bragg reflected by an analyzer crystal before it reaches the detector. The second reflection reduces the background considerably and improves the resolution (it is equivalent to a normal diffractometer with a large $2\Theta_m$) but leads to a loss in intensity of about a factor 2 due to the finite reflectivity of the analyzer.
- (ii) Although the precision to which a peak position can be determined is in principle not limited by the peak width, provided the counting statistics can

be made good enough, in practice the peak width *does* limit the accuracy to about 1% of the width at best. Typically lattice strains have to be registered with an accuracy of 10^{-4} (for ceramic materials this may be still insufficient) and therefore the resolving power of the instrument should be $\Delta D/D \approx 0.5\%$ or better. The required resolution can be achieved by an appropriate choice of the collimation for any monochromator take-off angle $2\Theta_m$. However, large $2\Theta_m$ as recommended above will give a higher luminosity for the same resolution as small ones. When $2\Theta_m$ is close to $2\Theta_s$ (i.e. in general close to 90°) α_2 will have little influence on the resolution and hence may be relaxed to increase the intensity.

We note that the recommendation to aim at a high resolution is valid only for the case that the reflection lines are not intrinsically broadened due to, e.g., high dislocation densities, very small grain size, etc. In such a case the instrumental resolution should be matched to the intrinsic linewidth to achieve the best compromise between peak width and peak intensity.

Among the conventional powder diffractometers best suited for stress measurements is the D1A instrument at the high flux reactor at Grenoble. The high performance is due to the following reasons (apart from the fact that it is located at a high flux reactor): (i) a large monochromator take-off angle, i.e. $2\Theta_m = 130^\circ$, (ii) multiple wavelengths available by choosing a proper set of reflecting planes of a Ge monochromator (Ge111, Ge311, etc). (iii) a vertically focussing monochromator giving a considerable gain in intensity compared to a standard flat monochromator. (iv) a proper choice of collimators leading to a high resolution $\Delta D/D \approx 0.2\%$. The high resolution is particularly valuable for measurements on ceramic samples. We note, however, that even the D1A instrument is not fully optimized for stress analysis as it was not designed for such a purpose.

In recent years dedicated instruments have been developed or are under construction at several places. The following ideas have been exploited to optimize the instruments for stress measurements:

- (i) The Soller collimators used in general-purpose diffractometers are appropriate only when a wide beam is needed. When narrow slits are put before and after the sample two further slits just after the monochromator and before the sample advantageously replace the corresponding Soller collimators: the slit system has a nearly rectangular transmission curve, whereas a Soller collimator has a triangular one (Fig. 2). For the same mean square divergence a rectangular transmission curve offers a gain in intensity of $\sqrt{2}$ compared to a triangular transmission curve. Moreover, a slit system does not suffer from the losses associated with the finite thickness of the blades in a Soller collimator.
- (ii) For most measurements a large vertical acceptance of the detector (up to about $\pm 10^\circ$) is not detrimental and hence can be used to maximize the intensity. We emphasize that a large vertical divergence after the sample *does not* affect the spatial resolution (whereas, as will be discussed in sect. 3.5.1a, a large vertical divergence before the sample *does* so). We further emphasize that a large vertical divergence *does not* affect the resolution in $\Delta D/D$ as long as $2\Theta_s \approx 90^\circ$.
- (iii) Multidetectors usually give a considerable intensity gain when compared to a single detector. Different set-ups have been reported in the literature [11-13], each having its pros and cons. Most promising seems to be a system using a

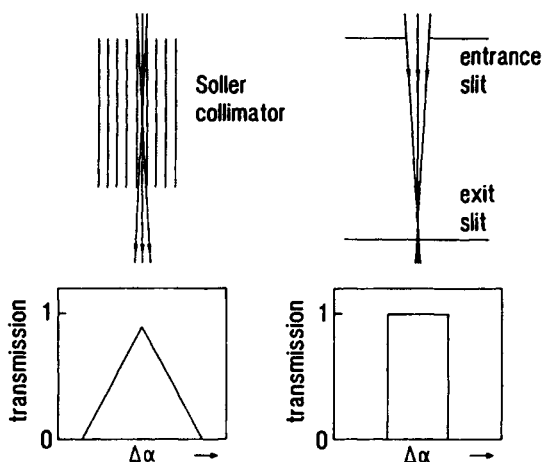


Fig. 2. Comparison of the transmission curve of a Soller collimator to that of a slit system

position-sensitive detector (PSD) covering a few degrees only but with a high angular resolution ($\sim 0.1^\circ$). The calculated gain factor of such a PSD compared to a single detector with *optimized* horizontal acceptance scanned through an *optimized* angular range is about 4. As it is difficult to operate single detectors always under optimal conditions gain factors will in practice be considerably larger. On the other hand, the gain in intensity may be partly offset by an increase in the background counting rate.

Using a PSD care has to be taken to place the beam mask after the sample very close to the internal probe region to ensure that all the neutrons reaching the PSD come from essentially the same spot. In case that this requirement cannot be fulfilled a radial Soller collimator may be used, but at the expense of a drastic loss in intensity.

- (iv) Attempts have been made to produce a well-focussed beam by using perfect or nearly perfect bent crystals as monochromator (called "microfocussing"). A size of the focus down to about $2.5 \times 4 \text{ mm}^2$ has been reported [14]. The beam intensity at the focus was very high, but the resolution $\Delta D/D$ achieved with this set-up was only moderate, as the requirements for focussing in real space are conflicting with those for focussing in reciprocal space. Optimized monochromators may give higher gains as achieved so far, so that the potential of "microfocussing" techniques cannot yet be assessed realistically.

3.32 Instruments for pulsed sources

Instruments for pulsed sources are so called time-of-flight (TOF) instruments, as the neutron wavelength is determined from the neutron velocity via a measurement of the TOF for a well-known distance. TOF-diffractometers operated at pulsed sources are very simple machines (see Fig. 3): the white beam impinges

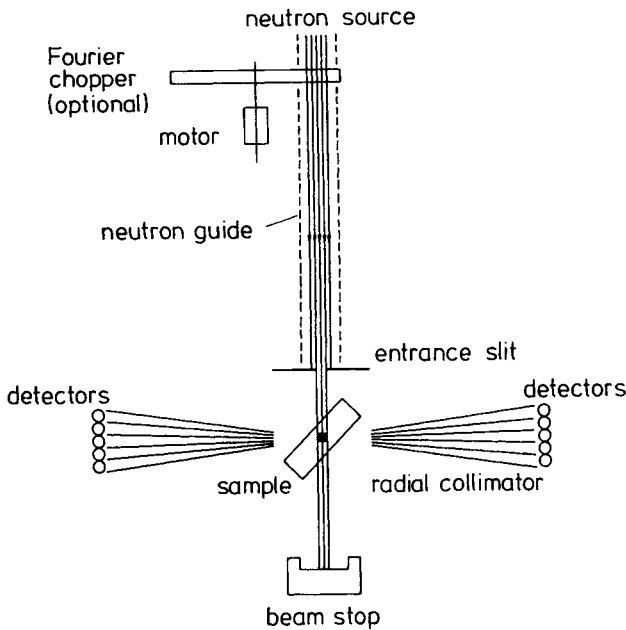


Fig. 3. Schematic drawing of a TOF-spectrometer for stress measurements

on the sample and the diffracted neutrons are detected in stationary neutron counters in a certain angular range $2\theta_{\min} < 2\theta_s < 2\theta_{\max}$. The TOF is given by the time lag between the neutron burst generated in the source and the arrival time at the detector. The resolution $\Delta D/D = \Delta t/t$ is given by the ratio of the pulse width and the total TOF from the moderator to the detector. The total flight path may be made rather long (50 m or even more) without a serious loss in intensity if the neutrons are travelling through so-called neutron guides. Hence diffractometers at pulsed sources easily achieve a high resolution (0.1...0.2%). The Dubna pulsed neutron source is an exception, as this a pulsed reactor and not a spallation source. The pulse width of a pulsed reactor is inherently much longer ($\sim 200 \mu\text{s}$) than that of spallation sources ($\sim 30 \mu\text{s}$). To obtain a high resolution despite a rather long pulse requires either an extremely long flight path or a pulse shaper. An instrument recently developed at Dubna [15] uses the second possibility: before hitting the sample the beam is modulated by a so-called "Fourier-chopper". Such a Fourier-chopper modulates the neutron intensity in a sinusoidal way from zero to more than 150 kHz. The period of the highest modulation frequency will determine the resolution, apart from uncertainties in the flight-path length and the scattering angle. Therefore this method can be used with any pulse length and even at a steady-state source [8,9]. However, there is a definite advantage for the Fourier method if the source is pulsed: whereas for a steady-state source all diffracted neutrons contribute to the statistical error at any point in the spectrum irrespective of their wavelength, only neutrons in a narrow wavelength band (related to the pulse width and the distance from the source to the sample) contribute to the statistical error of a particular point if the source is pulsed. Therefore,

whereas the Fourier method used at a reactor has not yet proven its merits, it looks very promising for the Dubna reactor.

Detectors are often placed at $2\Theta_s \approx 90^\circ$ on both sides of the beam (as in Fig. 3). When the sample is aligned as to have a principal stress direction at an angle of 45° to the incident beam, two principal strains can be measured at a time.

Measurements at spallation sources were aiming so far mostly at phase-specific stresses and hence could use a wide beam. Measurements with high spatial resolution could not be improvised (like at a reactor) as they require a fully automated control of the sample position, slit width etc., because there is no access to the instrument when the beam is on. The necessary technical developments were recently brought to a close at the ISIS spallation source [16].

There are no systematic studies which would allow one to compare the performance of TOF-stress scanners to that of diffractometers. In one respect, however, the TOF-instruments have a definite advantage: they always yield peak shifts for more than a single reflection line which is a very valuable information to check the presence of microstresses.

3.4 Data evaluation procedures

As neutron and X-ray stress analysis share the same basic principles, data evaluation procedures have much in common. The evaluation of X-ray diffraction data has been explained in detail in Chapter 2. Therefore only the neutron specific aspects will be dealt with here.

3.41 The $\sin^2 \psi$ -method

The $\sin^2 \psi$ -method, which is the standard technique in X-ray stress analysis, is rarely used with neutrons for two reasons: (i) it is too time-consuming to measure strains for many ψ -values and (ii) application of the $\sin^2 \psi$ -method is restricted to rather thin samples. The beam path through the sample and hence the beam attenuation strongly depends on ψ . As a consequence only a very limited ψ -range is accessible with thick samples (e.g. $0 \leq \sin^2 \psi \leq 0.25$ and $0.75 \leq \sin^2 \psi \leq 1$) giving not much more information than the end points of the D vs $\sin^2 \psi$ -relation, i.e. $\sin^2 \psi = 0, 1$.

The $\sin^2 \psi$ -method was primarily used for the study of microstresses, as these are often associated with pronounced non-linearities in the D vs $\sin^2 \psi$ -relation. An example will be given in Section 3.5.4. A further case for the $\sin^2 \psi$ -method is the determination of stresses in near-surface regions of very thick samples by measurements for $0 \leq \sin^2 \psi \leq 0.5$ in complete analogy to the X-ray technique.

3.42 Determination of principle stresses

In the bulk of the neutron studies performed so far strains were measured only in three directions assumed to be the principle stress directions as inferred from the sample geometry. It is in general implicitly assumed that the D vs $\sin^2 \psi$ -

relation is linear. Since it is difficult, as explained in the previous paragraph, to check the linearity of the D vs $\sin^2 \psi$ -relation with neutrons, complementary X-ray measurements performed on the same or a similar sample may help to clarify the situation. We note that stress values calculated from strains observed at $\sin^2 \psi = 0, 1$ are not as sensitive to non-linearities in D vs $\sin^2 \psi$ as values calculated from the slope of D vs $\sin^2 \psi$ in a limited ψ -range.

In case that the macroscopic Young's modulus E and Poisson's ratio ν can be used there is the following relationship between the principal stresses σ_i and the principal strains ϵ_i .

$$\sigma_i = \frac{E(1-\nu)}{(1+\nu)(1-2\nu)} \epsilon_i + \frac{\nu E}{(1-2\nu)(1+\nu)} \sum_j \epsilon_j \quad j=1,2,3 \quad j \neq i \quad (2)$$

or, using the X-ray elastic constants S_1 and $\frac{1}{2} S_2$,

$$\sigma_i = \frac{2(4S_1 + S_2)}{S_2(6S_1 + S_2)} \epsilon_i - \frac{4S_1}{S_2(6S_1 + S_2)} \sum_j \epsilon_j \quad j=1,2,3 \quad j \neq i \quad (3)$$

3.43 Determination of stress tensors

In case that the directions of the principal stresses cannot be inferred from the sample geometry measurements are required in more than just 3 directions. In principle, measurements in 6 independent directions are enough for a complete determination of the strain tensor. However, as has been pointed out in [17], it is highly desirable to overdetermine the system by performing measurements in about 12 directions. If this is too time-consuming, the directions in which the measurements are made have to be chosen with great care (see Ref. 15).

3.44 The D_0 -problem

The quantities directly determined by diffraction methods are lattice spacings, not strains. The evaluation of strains from the observed lattice spacings requires an accurate knowledge of the lattice spacing D_0 of the stress free state. This applies both to X-ray and neutron stress analysis, but for neutrons the D_0 -problem is much more severe: as there can be no macrostress perpendicular to the surface evaluation of X-ray data can usually be based on the assumption that the stress component σ_{33} is zero in the thin surface layer probed by X-rays. This boundary condition allows one to determine D_0 together with the surface stresses. Only in those cases where large microstresses give rise to non-zero σ_{33} components D_0 has to be determined independently (see the Chapter 2.11 in this volume). In the interior of a bulk solid, however, there is no boundary condition which might be utilized to determine D_0 .

The most widely used solution to the D_0 -problem consists in annealing a reference sample to make it essentially stress-free. Unfortunately, this simple solution does not work in all cases. In particular, multiphase materials usually retain considerable phase specific stresses after the annealing procedure due to differences in thermal expansion. Further the sample may contain chemical

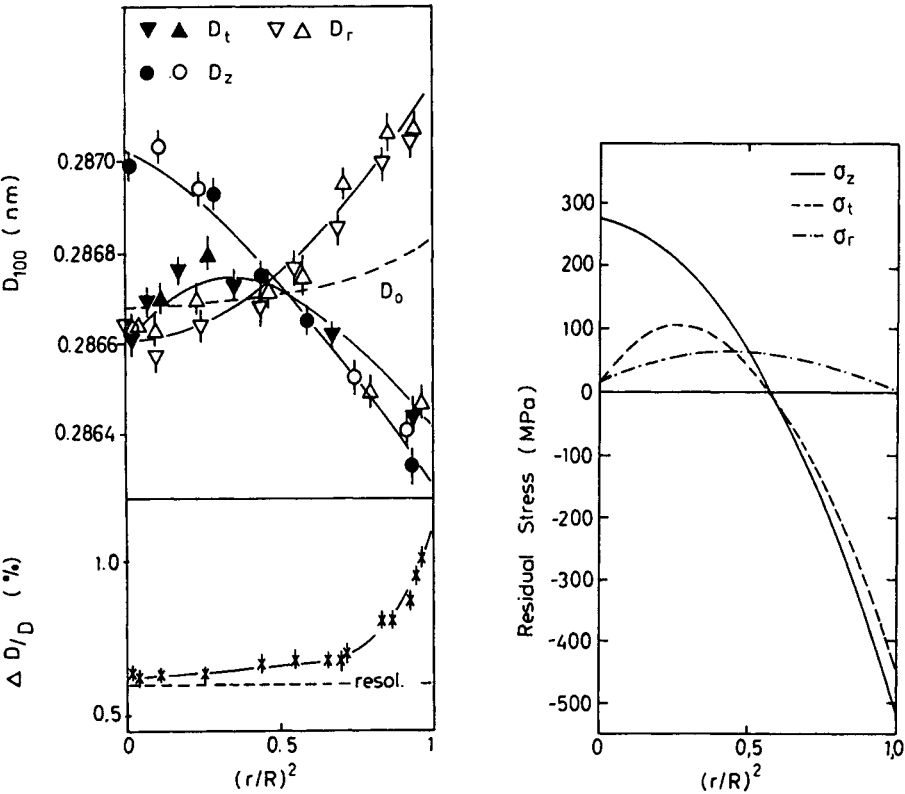


Fig. 4. left, top: lattice spacings measured in a cylinder made from a plain carbon steel (German grade Ck45) quenched from $t=800^{\circ}\text{C}$ in oil.
left, bottom: observed widths of the {211} reflection line
right: residual stress distributions corresponding to the lines in the left hand part of the figure

gradients so that D_0 varies accordingly. In this case it may be necessary to section the reference sample before the annealing process and then determine D_0 at various parts of the sample, as has been done in Ref. [18] for a weldment. In those cases where the chemical gradient is produced by rapid quenching even the section - and - anneal procedure fails as annealing will alter the chemical composition. For instance, it will convert a martensite into a mixture of ferrite and cementite. In such a case D_0 has to be estimated from an analysis of the microstructure or quantities like the microhardness etc. For instance, in Ref. 19 the variation of D_0 was estimated from the variation of the width of the {211} reflection line as martensite gives rise to a pronounced line broadening (see Fig. 4).

Provided a residual strain distribution is determined across the whole cross-section of the specimen, D_0 may be properly adjusted to make the calculated residual stresses fulfill the balance of forces. (We note that the residual stress distribution shown in Fig. 4 *does* fulfill the balance of forces). The assumption that the observed stresses have to be balanced seems obvious, but may fail nevertheless. Firstly, strains evaluated from a particular reflection line may not be representative for the macrostress state, in particular in strongly textured samples. This can be checked by using several different reflection lines. Secondly, after heavy cold-working even the use of several different reflection lines does guarantee that the calculated stresses can be interpreted as macrostresses, a phenomenon which is not understood so far. Examples are given in Ref. 20.

An improper assumption of the D_0 -value leads to an error in the calculated stresses which corresponds a hydrostatic component, whereas the so-called stress deviator remains unchanged. Fortunately, the hydrostatic stress component is in general of lower importance for the assessment of the stress state than the stress deviator.

3.45 Separation of macro- and microstresses

As neutrons and X-rays are based on the same principles, they both have the potential to get information not only on macro- but also on microstresses. On the other hand, both techniques suffer from the same problems in separating the two types of stresses. For details see the corresponding paragraphs in Chapter 2. There is, however, one particular advantage of neutrons which can be exploited for the separation: using a poor spatial resolution on purpose, i.e. a wide neutron beam covering the whole cross-section of the specimen, the observed strains are associated with microstresses only as the macrostresses cancel each other when averaged over the cross-section. In reflection geometry the finite penetration depth leads to a non-uniform sampling of the cross-section, and therefore this method is restricted to relatively thin samples (e.g. ≈ 5 mm for steel). An application of this method will be presented in Section 3.5.4.

3.5 Fields of application

3.51 Stress measurements in the interior of bulk solids

The major application field of neutron stress analysis is the non-destructive determination of stresses in the interior of technical components exploiting the high penetrating power of neutrons. This method has a great potential, but also its inherent limitations, which will be discussed in the following.

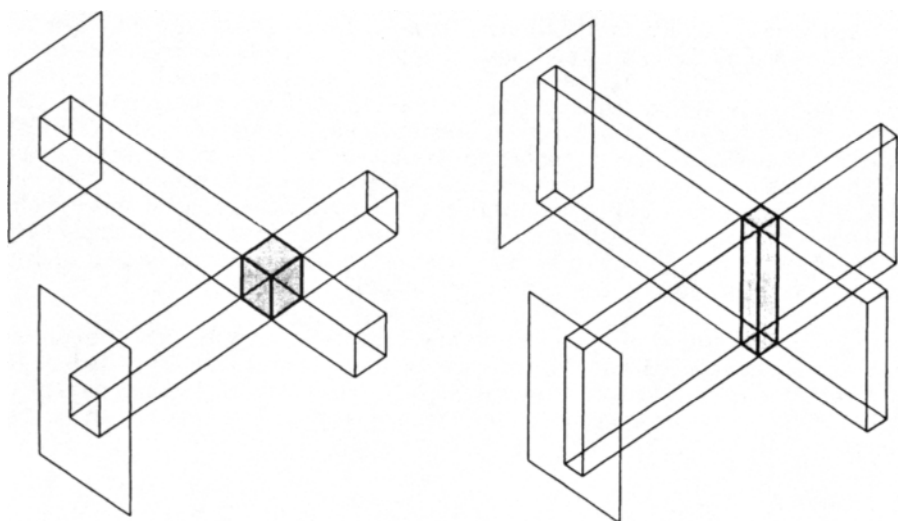


Fig. 5. Shape of the internal probe region created by different sets of slits in the incident and the diffracted beam

3.51a Spatial resolution

The shape and the size of the internal probe region is determined by slits in the incoming and the diffracted beam. This is illustrated in Fig. 5 for a scattering angle $2\Theta_s = 90^\circ$, which gives the best spatial resolution for given slits widths and therefore is the standard choice.

The volume V of the internal probe region is usually chosen in the range 5 to 30 mm^3 to ensure reasonable counting rates. With high performance instruments and relatively thin samples (i.e. low absorption) V may be decreased to about 1 mm^3 .

As is illustrated in Fig. 5 the internal probe region need not to be shaped as a cube. When stress gradients are low in one or two directions this can be exploited to gain intensity while maintaining a high spatial resolution in the direction of the large stress gradients. This also means that the above mentioned minimum $V \approx 1 \text{ mm}^3$ is compatible with a spatial resolution better than 1 mm in a certain direction. For instance, 0.5 mm have been achieved in Ref. 21 (see Fig. 9). However, it is difficult to improve the spatial resolution even further: as is illustrated in Fig. 6 the finite divergence of the neutron beam gives rise to penumbra regions and the importance of the penumbra relative to the main beam increases inversely as the slit dimensions. Penumbra effects are particularly severe in the vertical direction as practically all neutron diffractometers use a much larger divergence in the vertical direction than in the scattering plane. (The case shown in Fig. 6 is, however, an extreme). In order to minimize penumbra effects the slits should always be brought as close as possible to the sample.

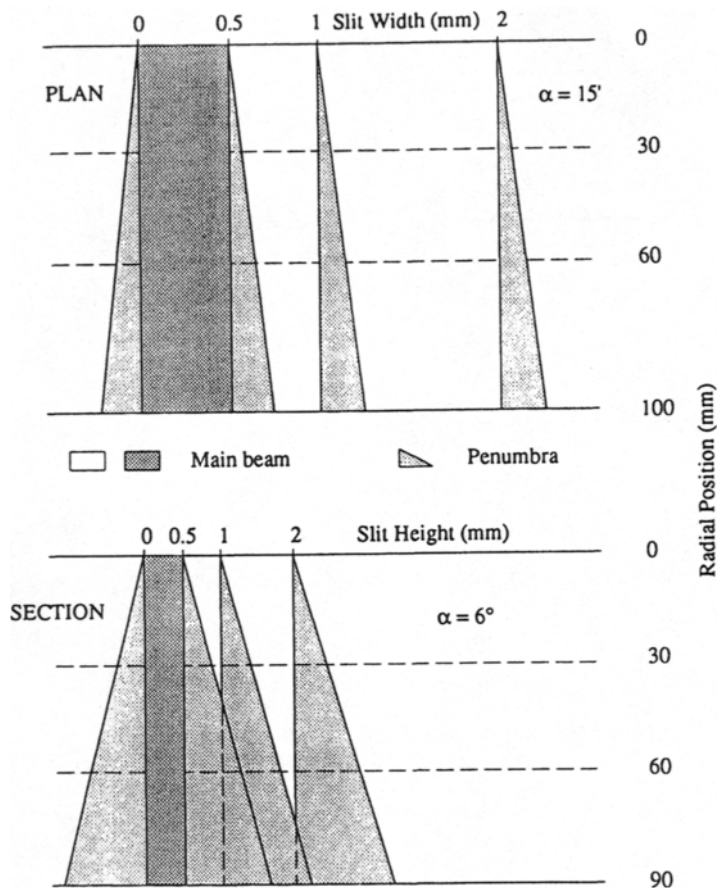


Fig. 6. The effects of horizontal and vertical beam divergence, and radial position of the input slit, on the gauge volume definition for the D1A instrument at the high flux reactor at Grenoble (after [22]).

3.51b Sample dimensions

Although the penetration depth of neutrons is large compared to that of X-rays, it is not large compared to the dimensions of many technical components. Fig. 7 shows the variation of the diffracted intensity with the sample thickness for a Bragg angle $2\theta = 90^\circ$, i.e. the angle which leads to the best spatial resolution for a given volume of the internal probe region. For most materials the attenuation of the neutron beam is mostly due to scattering processes and not due to absorption. Therefore, materials which give a high diffraction intensity for thin samples have low penetration depths and vice versa. The maximum thickness which can be tolerated depends on the spatial resolution required and, of course, on the the time which one is willing to spend on the measurements. The lines shown in Fig. 7 refer

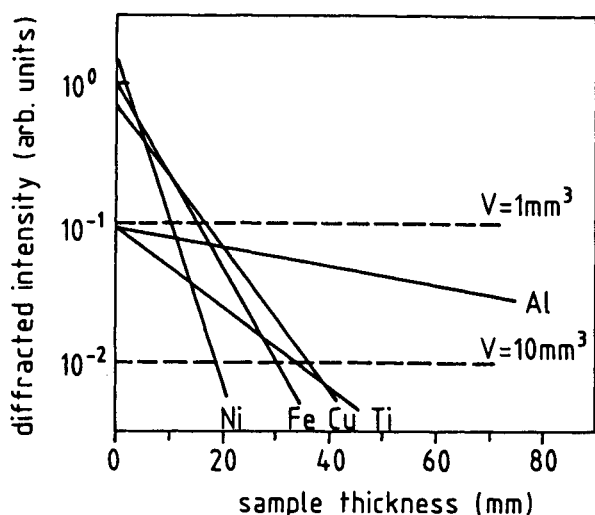


Fig. 7. Diffraction intensity versus thickness of the sample for $2\theta_s \approx 90^\circ$ for various metals.

to an instrument at a high flux reactor for reasonable counting rates, i.e. ≈ 2 hours/peak. So as a rule of thumb steel samples should not exceed 30 mm thickness, whereas aluminium samples may be about three times as thick. Other sample dimensions may be much larger, as neutron diffractometers can accommodate rather large specimens.

3.51c Accuracy

It seems to be common practice in neutron studies to aim at a precision $\Delta D/D \leq 10^{-4}$. As the mechanical precision of neutron diffractometers is usually quite high, lower statistical errors can be achieved if long counting times can be tolerated. Using the high resolution diffractometer D1A at the high flux reactor Grenoble a precision as high as $\Delta D/D \approx 2 \cdot 10^{-5}$ has been achieved in a study of the residual stress state of a ceramic sample [23] (see Fig. 8). However, the above mentioned precision $\Delta D/D \approx 10^{-4}$ (corresponding to an error $\Delta \sigma \approx 20$ MPa for specimens made of steel) seems to be sufficient for most applications. Very often the accuracy of the experimental stress values is limited not so much by the statistical errors than by effects associated with elastic and plastic anisotropy.

3.51d Applications

Neutron stress analysis is applied to the same type of problems as X-ray stress analysis ranging from quality control to questions of basic character. However,

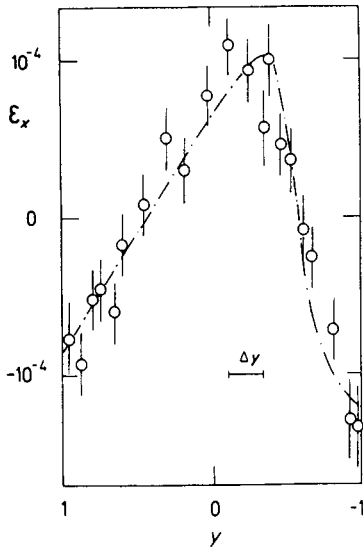


Fig. 8. Residual strains ϵ_x as determined by neutron diffraction in an Al_2O_3 ceramic sample after a creep test [24]. Note the small range of ϵ_x . The bar ΔY denotes the spatial resolution (0.4 mm). The dash-dotted line was calculated from a non-symmetric creep law.

there is rarely enough beam time for routine measurements so that most neutron studies are dealing with rather fundamental problems. In particular, in-depth measurements of residual stresses by neutron diffraction are widely used to validate theoretical predictions based on the finite-element method. An example for results of this kind is shown in Fig. 8.

3.52 Stresses at surfaces and interfaces

The investigation of surface stresses is certainly a domain of X-ray diffraction and in general it makes no sense to try to compete in this field with neutrons. However, the depth range in which stresses can be explored non-destructively by X-rays is very limited and it may be of interest to follow the residual stresses deeper into the material. When the surface is covered by some other material and thereby is forming an interface, neutron diffraction is usually the only means to explore the stress state in this region (an example of stress measurements at an interface is depicted in Fig. 9).

What is the minimum depth to be probed by neutrons? Using very narrow input and exit slits there is no principal limit but in practice the slit width cannot be made much smaller than 0.5 mm for intensity reasons even when the slits are rather long. With slits 0.5 mm wide and $2\Theta_s = 90^\circ$ the minimum distance of the center of the gauge volume from the surface is $l = 0.35$ mm as long as the gauge area is kept completely within the sample. The distance is further reduced if the gauge area is only partially immersed in the sample. This partial immersion

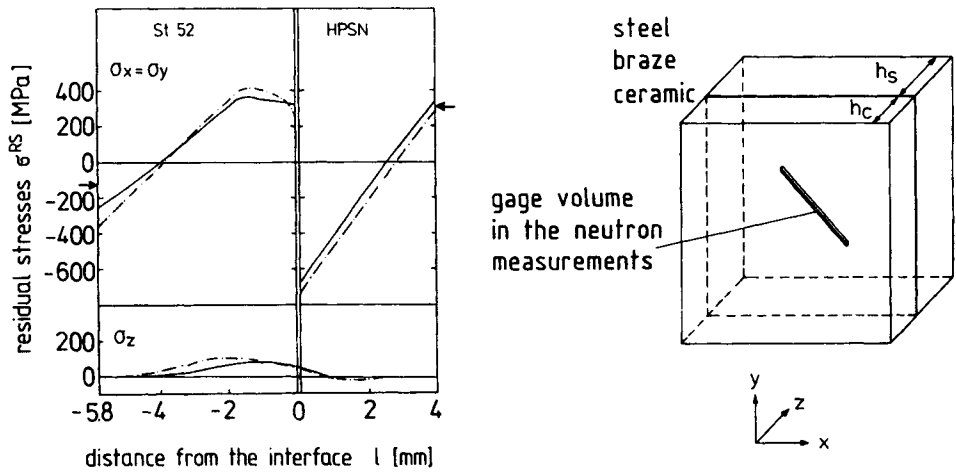


Fig. 9. Experimental (solid lines) and calculated (broken lines) residual stress distributions along the central line of brazed ceramic-steel component (ceramic: hot pressed silicon nitride, steel: German grade St52). The arrows denote X-ray results. The specimen is depicted at the right hand part of the figure. The calculations were based on the temperature dependent elastic-plastic behavior of the materials [23].

causes (i) a reduction of the diffraction intensity and (ii) in general systematic changes of the diffraction peak profile not associated with strains but with the finite divergences of the incoming and the diffracted beam (see Fig. 10). The latter effect is discussed in some detail in Ref. 20. It can be largely corrected for by measurements on a stress-free sample as a reference using the same geometry (i.e. transmission or reflection). Nevertheless, a practical limit is usually reached when the gauge area is immersed less than 50%. With 0.5 mm slits this leads to a minimum distance of the center of mass of the gauge volume from the surface of about 0.1 mm. Such a high spatial resolution has been achieved, e.g., in Refs. 19 and 23. Fig. 11 was taken from Ref. 23 showing steep stress gradients in a 0.5 mm thick surface layer of a shot peened sample made of a nickel superalloy.

3.53 Phase specific stresses

The large penetration depth of neutrons gives them an edge on X-rays for the investigation of composite materials. In order to get strain values which are representative of the bulk, the domains of the different constituents have to be small compared to the penetration depth. If the material is composed of fibres or grains which are several μm thick or even larger, X-ray results will be strongly affected by surface effects, whereas neutron results will be not.

Most of the neutron studies of composites were performed at spallation sources. The TOF-technique employed at spallation sources has the advantage that it yields information simultaneously for the matrix and the reinforcement material. With detectors placed at different scattering angles a single run is often sufficient to obtain enough data to fully characterize the stress state of the composite. When

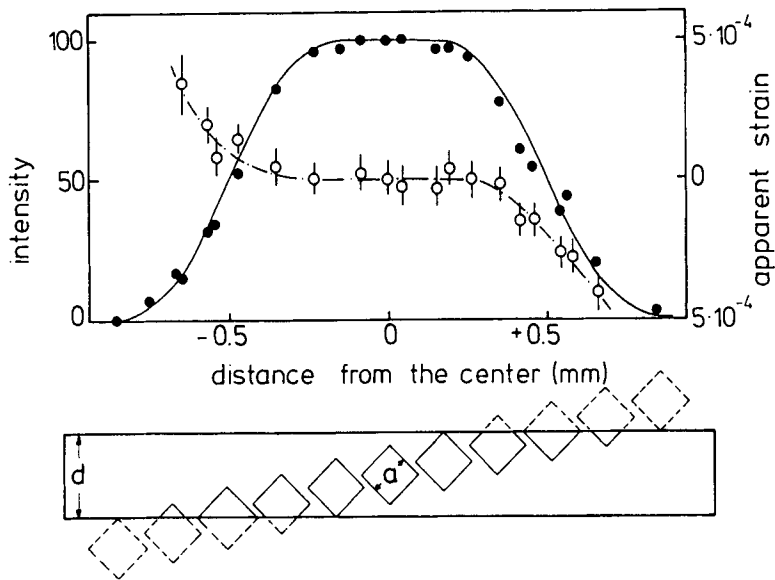


Fig. 10. Full dots: Intensity versus distance of the gauge area ($0.5 \times 0.5 \text{ mm}^2$) from the center of a 1 mm thick steel strip [19].
open circles: Apparent strain versus distance of the gauge area from the center. As the sample was essentially stress-free the apparent strains are associated with systematic errors resulting from incomplete immersion of the gauge area.

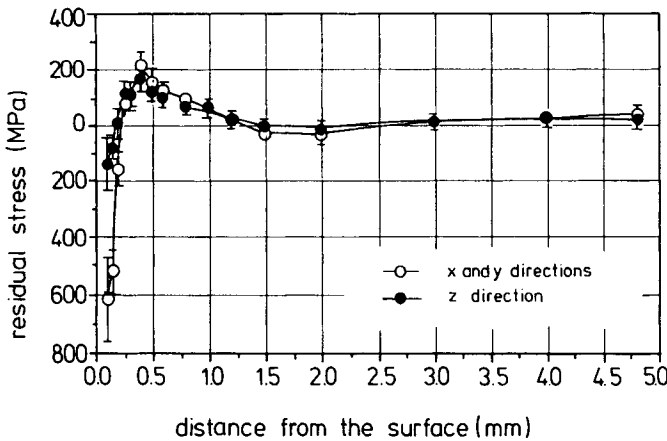


Fig. 11. Residual stress field through half-thickness of a shot-peened plate [25].

the spatial variation of the stresses is not of primary interest a wide beam can be used which makes the measurements very quick. An example of data obtained in this way is shown in Fig. 12.

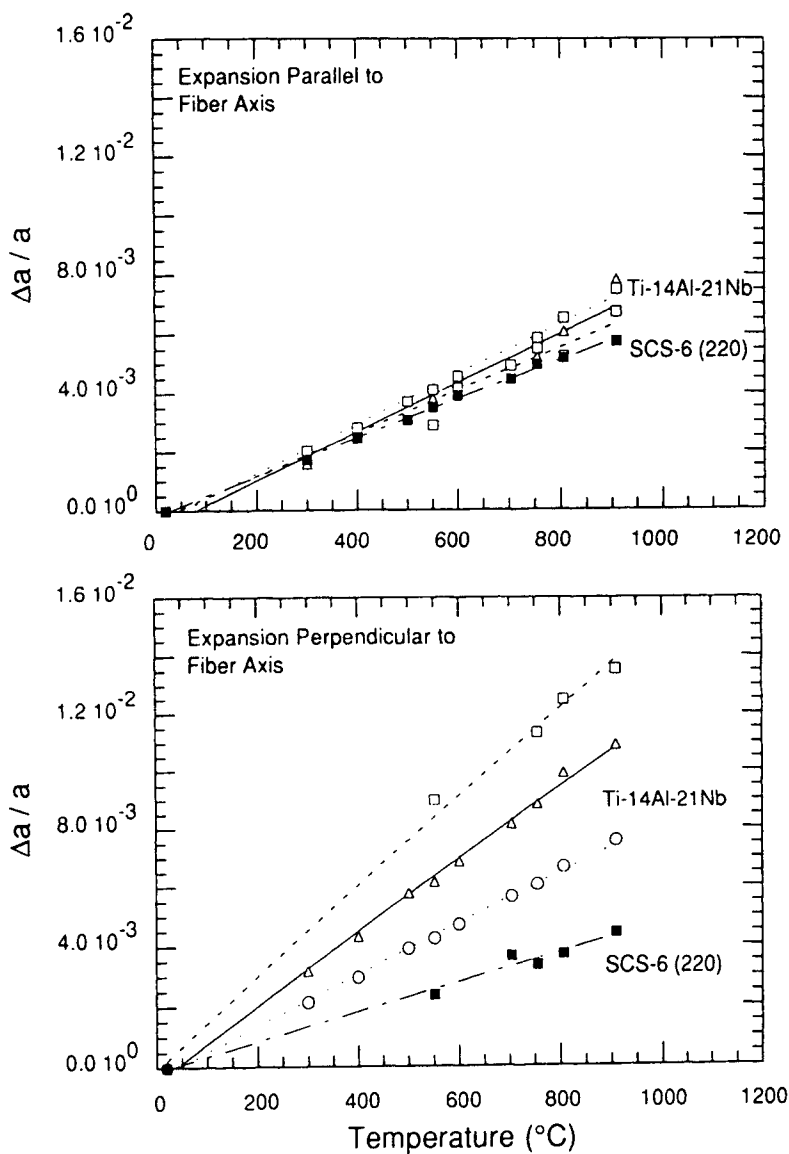


Fig. 12. Lattice parameter relative to room-temperature value, versus temperature in a SiC-fibre reinforced Ti alloy as determined from the SiC {220} plane and various Ti alloy lattice planes [26].

3.54 Microstresses

As mentioned in Sect. 3.4.5 the high penetrating power of neutrons can be exploited for the separation of macro- and microstresses by using a poor spatial resolution on purpose so that the observed strains are averages over the whole cross-section and hence macrostresses are averaged out. This was done in, e.g., Ref. 11 for the separation of macro- and microstresses in a cold rolled steel strip. Representative data are shown in Fig. 13.

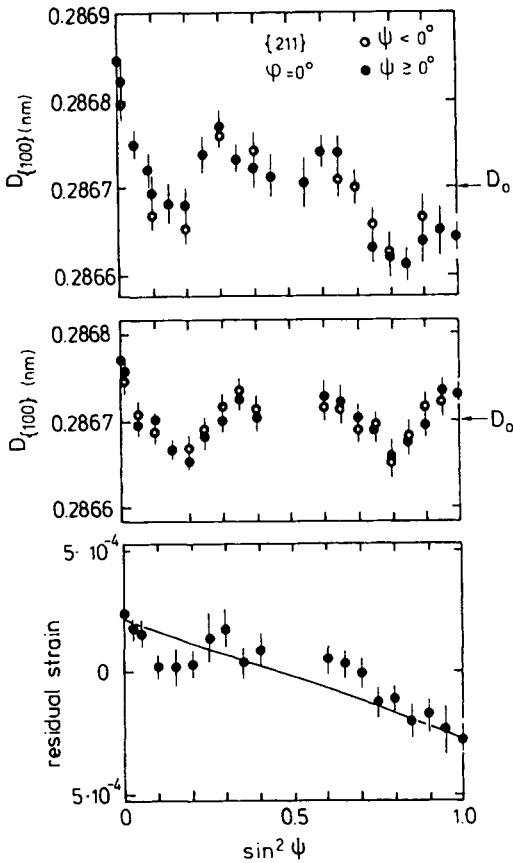


Fig. 13. above: D vs $\sin^2 \Psi$ -distribution in the rolling direction of a cold rolled steel strip as observed in the center of the specimen
middle: as above, but with wide slits in the incoming and diffracted beam to obtain an average over the cross-section
below: difference between the above data representing the strains associated with macrostresses. The deviations from a linear distribution are due to elastic anisotropy (after [21]).

Another advantage of neutrons is the fact that D vs $\sin^2\psi$ -distributions can be determined up to $\sin^2\psi=1$. Progress in X-ray diffraction now allows to cover a wide range in $\sin^2\psi$ as well, i.e. up to $\sin^2\psi=0.9$, but in strongly textured

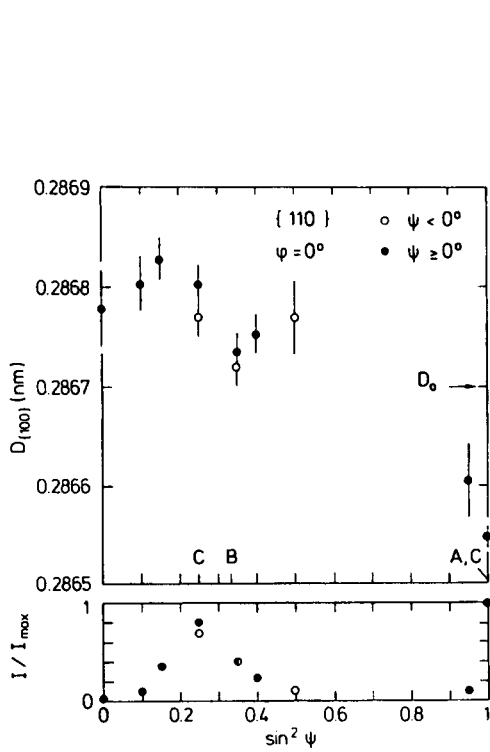


Fig. 14.
Lattice parameter and relative intensity vs $\sin^2\psi$ as measured in the rolling direction of a cold rolled steel strip using the {110} reflection line [21]

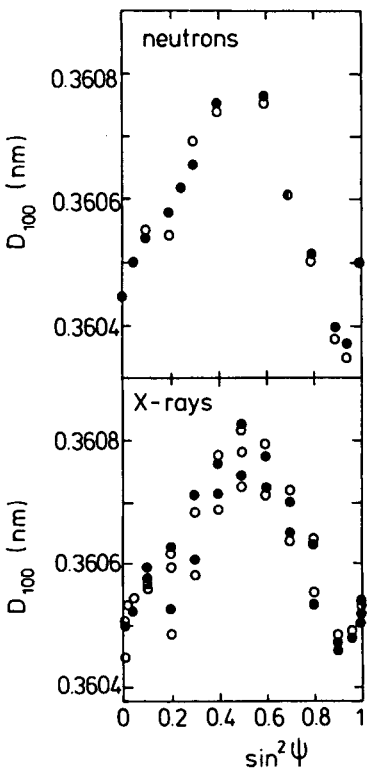


Fig. 15.
Lattice parameter vs $\sin^2\psi$ as measured in the direction of plastic strain of a tensile test specimen made of steel X2CrNiMoN225. The {220} reflection line was used both in the neutron and the X-ray experiment. The neutron values represent averages over the whole cross section. The X-ray values were obtained by measurements on three surfaces making different angles with the strain direction and plotted in the system of co-ordinates of the neutron experiment (after [20].)

specimen the missing region $0.9 < \sin^2\psi \leq 1$ may be still of importance. For instance, in cold rolled steel the majority of the grains have an 110-axis in the rolling direction and therefore very little diffraction intensity is found for the {110} reflection line for $0.5 < \sin^2\psi < 0.95$. As a consequence only neutrons can probe the D_{110} -values in the rolling direction (see Fig. 14). In other cases there may be strong changes in D close to $\sin^2\psi = 1$ which cannot be anticipated from measurements for $\sin^2\psi \leq 0.9$ (see Fig. 15).

3.6 Possible hazards

Neutron stress analysis is sometimes met with reservation because it is feared that irradiation by neutrons may adversely affect the sample properties. Suspicions that the neutrons may induce radiation damage are certainly unfounded: the slow neutrons used with neutron diffraction have too little energy to do so. On the other hand, activation associated with neutron absorption cannot always be excluded. However, activation levels can be usually kept to negligible levels when the neutron beam is properly masked. There are a few exceptions like U and its compounds, where activation might pose a problem, but these materials are a special case anyway for other reasons. In summary, concerns about possible hazards associated with neutron stress analysis can be dismissed in almost all cases of practical interest.

3.7 Neutron diffraction versus x-ray diffraction and other techniques

Neutron stress analysis is now a well established method and therefore it should not be used to duplicate results obtained by X-ray diffraction. As neutron diffraction is relatively expensive (or neutron beam time is scarce for this very reason) its use should be restricted to those fields of application where neutrons have a clear edge on X-rays: firstly, it is the non-destructive in-dept determination of residual or load stresses. Secondly, it is the investigation of bulk stresses in composite materials. Thirdly, neutrons might also have an advantage for the investigation of near-surface stresses and microstresses in special cases (examples have been given in Sec. 3.5.4).

Sometimes, neutron diffraction is used to establish that residual stresses measured at the surface by X-rays are representative for the bulk, so that further investigations can be based primarily on X-ray diffraction. Results of this kind are shown in Fig. 16.

When compared to other techniques different aspects might give neutron stress analysis the preference:

- (i) neutron stress analysis does normally not require a calibration (and even if it does calibration is relatively easy) and therefore it may be used to calibrate other methods based on ultrasonic or magnetic measurements.
- (ii) it is non-destructive whereas sectioning, layer-removal or hole drilling techniques are not (a comparison between results obtained by neutron diffraction and strain-gauged layering measurements is shown in Fig. 17, a comparison of residual hoop stresses determined by the neutron diffraction technique and the Sachs boring method for an autofrettaged ring sample is shown in Fig. 18).

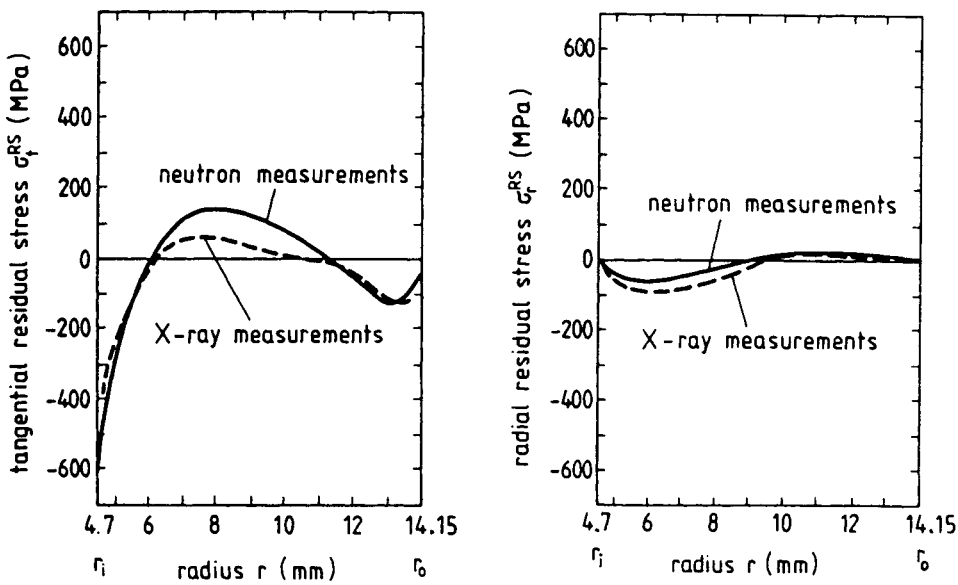


Fig. 16. Comparison between tangential (left) and radial (right) residual stress distributions measured by neutron diffraction in the central part of an autofrettaged steel tube and by X-ray diffraction on the face of a ring cut out the tube (after [27]).

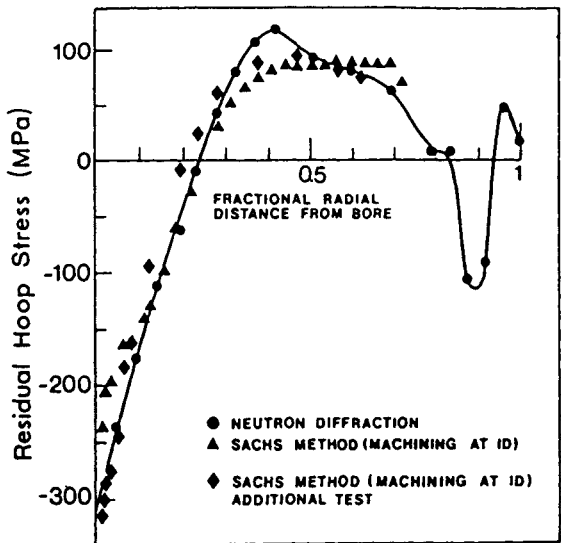


Fig. 17. Comparison of residual hoop stresses, as a function of radial distance from the bore, determined by the neutron diffraction technique and by the Sachs boring method for an autofrettaged ring sample [28].

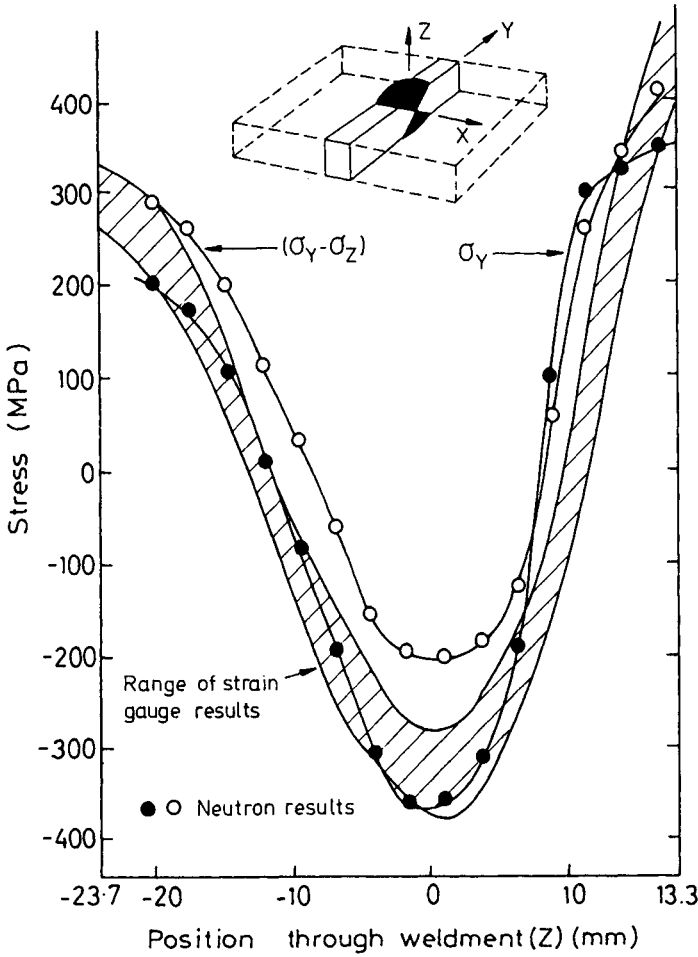


Fig. 18. Comparison of neutron diffraction and strain-gauged layering measurement of the residual stresses across a mild steel double-vee weld section [29].

- (iii) the spatial resolution achievable with neutrons is usually better than with most sectioning or hole drilling techniques, let alone ultrasonic or magnetic measurements. Microhole drilling may compete in spatial resolution, but does not allow in-depth measurements.

In summary, there are many cases of practical interest where neutron diffraction is the most appropriate choice for stress analysis. It would be much more widely used than today if more suitable instruments could be made available to its potential users.

3.8 Recommendations

The first step in planning a neutron stress measurement is to check if the proposed experiment is actually feasible. Proposers are often not very familiar with the neutron diffraction technique and may have unrealistic ideas about the penetration depth of neutrons, the spatial resolution achievable, the number of samples to be studied during the available beam time, etc. Often, a compromise has to be found between what is desirable and what is realistically achievable, which requires the expertise of the neutron scattering specialist already in the initial stage of the experiment.

In view of the general shortage of neutron beam time every effort should be undertaken to define an efficient strategy for the neutron measurements. In most cases X-ray measurements on the samples in question will yield very valuable information on a number of issues:

- (i) How large are the stress gradients? This will determine the dimensions of the gauge volume needed to obtain meaningful results.
- (ii) Are the D vs $\sin^2\psi$ -distributions linear or at least approximately so? If yes, it will suffice to restrict the measurements to the determination of principal strains.
- (iii) What is the ratio of macro- to microstresses? In case that microstresses are relatively small - but only in this case - it will be enough to use a single reflection line. If the corresponding XEC's are not known with sufficient precision they should be determined by X-ray loading strain measurements. In the event that in spite of the presence of considerable microstresses only a single reflection line can be used for lack of beam time, X-ray measurements may help to choose an appropriate $\{hkl\}$.
- (iv) Are the reflection lines inherently broadened, and if yes, to what extent? High resolution set-ups are good policy only if inherent line-broadening is small. Otherwise, the resolution may be relaxed to gain intensity.
- (v) There are many cases where neutron measurements are aimed at the spatial distribution of stresses. On the other hand, there is rarely time enough to systematically scan the strains throughout the specimen. X-ray measurements will help to define the regions of highest interest.
- (vi) Last not least, X-ray measurements performed at the surface may turn out to be sufficient for series measurements, as soon as it has been established by neutron measurements that the stresses found at the surface are representative for the stresses observed in the bulk.

A problem for which X-rays are generally not very helpful is the D_0 -problem. Even when it is possible to determine D_0 with X-rays with high precision this information is often of limited use only, as the calibration of neutron diffractometers depends on a number of parameters which are frequently changed from experiment to experiment. Hence, D_0 should be determined with the set-up used in the actual measurements. If ever possible, a stress-free reference sample should be prepared for the determination of D_0 (see Section 3.4.4).

The choice of the instrument will depend on criteria discussed above: the width of the wave-length band necessary to use all the reflection lines of interest, the required resolution in $\Delta D/D$, the required spatial resolution (which translates into a certain minimum luminosity), etc. In practice, however, the ultimate criterion for the choice of the instrument is very often accessibility. Given the overload of the few excellent neutron strain scanners around the world this has to be accepted, but nevertheless it should be carefully considered in each case if the available instrument meets minimum requirements for the planned experiment.

In view of the diversity of instruments used for neutron stress analysis it is beyond the scope of this article to describe a detailed procedure for the alignment. Some methods for the alignment of instrumentation for residual stress measurements can be found in [22,30]. However, the necessary auxiliary devices are often not available at a particular instrument. Therefore, the occasional user of a neutron strain measurement instrument is advised to apply to the instrument responsible and/or to a neutron scattering expert familiar with this instrument for detailed instructions. A discussion with these persons will further be helpful for the search of an optimum configuration of the instrument. Some guide-lines for the optimization of a crystal diffractometer for residual stress measurements have been presented in this chapter. However, existing instruments frequently suffer from constraints which do not allow one to optimize the instrument as desired (e.g., the monochromator take-off angle may be restricted to rather low values or may even be fixed). Only a discussion with the instrument responsible will clarify how flexible the instrument is to allow one to optimize it for the planned experiment. Finally, based on the frequent observation of a certain carelessness the following point of practical importance is emphasized: as has been discussed in Sect. 3.5.1a the finite divergence of the incident and the diffracted beam gives rise to penumbra effects which results in a poorer spatial resolution than expected from the slit dimensions. Therefore, choosing the slit positions rather far away from the sample in order to avoid any risk of collision when the sample is translated or rotated may seriously impair the spatial resolution.

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4. Ultrasonic techniques

E. Schneider

4.01 Historical review

Since 1929, when Sokolov first proposed the use of ultrasound to find hidden flaws in metals using a through transmission technique, the nondestructive testing of materials using ultrasound has been experiencing an impressive development. The possibility of visualizing flaws in their three dimensions and the availability of acoustic microscopes with resolutions of the order of a few micrometers in both the lateral and the depth directions are picked in order to illustrate the state of the art.

The development of ultrasonic techniques for the evaluation of material properties and for the characterization of the state of materials began in the 1950s. Using measurements of ultrasonic wave velocities, the elastic constants of materials and their changes with temperature and external load were evaluated; effects of heat treatments on the microstructural state of materials were investigated by measurements of the ultrasonic attenuation and its change with the ultrasonic frequency.

Developments in acoustoelasticity have been stimulated by interest in the evaluations of third order elastic constants. Based on Murnaghan's /1/ theory of nonlinear elasticity, Hughes and Kelly /2/ developed a theory of acoustoelasticity in 1953. They described the change of ultrasonic velocities as function of the elastic strains of an isotropic solid. It was in 1958/59 when the birefringent phenomenon of acoustic waves was discovered by Bergman and Shahbender /3/ and by Benson and Raelson /4/. In a metallic tensile test sample, the velocity of a shear wave polarized along the axis of applied stress was found to change differently with stress than the velocity of the wave vibrating perpendicular to the stress axis. This phenomenon is analogous to the changing of light speed in a stressed transparent body. Since the photoelastic effect has already been used to determine the state of stress, the applicability of the ultrasonic birefringence effect for stress analysis has been investigated and developed.

Crecraft /5/ showed in 1967 that the acoustoelastic effect can be used for the evaluation of stress states in engineering components. Measurements of the stress induced changes of ultrasonic velocities in steel samples were done by Egle and Bray /6/ in 1976. They published all of the five possible relative changes in longitudinal and shear wave velocities for an uniaxial state of stress and confirmed the theoretical predictions.

Until about 1990, a large number of individual researchers and numerous groups worldwide improved the measuring techniques and developed different approaches to evaluate stress states in samples and components. Reviews are given by Pao, Sachse, Fukuoka /7/ as well as by Thompson, Lu and Clark /8/. Fukuoka /9/ reviews the Japanese activities and Boborenko, Vangeli and Kuzenko /10/ summarize research and developments in the former Soviet Union.

Due to their contribution to the applicability of ultrasonic techniques on components or to the acceptance of this new technique by industrial partners, the following authors are selected and their work is highlighted.

Kino and co-workers /11/ developed a two pulse echo measuring technique and evaluated the inhomogeneous stress states around a crack and a circular hole in tensile test samples. They also evaluated the stress intensity factor of cracks and the J integral for an aluminium center cracked panel.

A pulse phase locked loop technique was developed by J. Heyman and co-workers /12/, which they used to evaluate the axial stress in fasteners. That stimulated various manufacturers to introduce ultrasonic devices for the strain and stress analyses on fasteners and bolts.

A.V. Clark and colleagues /13/ used a modified zero crossing technique to evaluate welding stresses in thin aluminium and thick steel plates.

Approaches to evaluate stresses in textured materials were investigated and reviewed by R.B. Thompson and co-authors. They also developed a Fourier transform phase slope technique for the ultrasonic measurement of texture and stress /8,14/.

Another commercially available ultrasonic setup for the evaluation of stress states was built by the group of J. Deputat. The semi automated system was optimized for the analysis of stresses in rails and rail road wheels /15,16/.

A setup for the automated time and locus continuous stress evaluation was introduced to the market by E. Schneider and co-workers. The system is in industrial use for the evaluation of surface stresses in hardened rolls /17,18/.

Fukuoka and co-workers developed an ultrasonic resonance method and measuring system for the analysis of stress in thin plates /19/.

In recent years, the widest use of ultrasonic techniques has been made for the evaluation of stress states in railroad wheels. Based on the DEBRO 30 setup, J. Deputat and co-workers developed the DEBBIE system which is fairly new on the market /20,21/. This portable system is optimized for the use in field. The UER systems, developed by E. Schneider, R. Herzer and co-workers, are used in the wheel inspection lines in railroad workshops for the fully automated evaluation of the stress profile along the height of the wheel rims /22,23/. Investigations of ultrasonic techniques for the evaluation of stress states in rails are still going on /24,25,26/.

4.02 Symbols and abbreviations

ρ	Density of the material
v_L	Velocity of a longitudinal wave in an isotropic solid
v_T	Velocity of a shear wave in an isotropic solid
v_R	Velocity of a Rayleigh wave in an isotropic solid
v_{SL}	Velocity of a skimming longitudinal wave in an isotropic solid
v_{SH}	Velocity of a dispersion-free shear horizontal wave in an isotropic solid
λ, μ	Lamé moduli of an isotropic solid
K	Bulk modulus
G, E	Shear, Young's moduli
ν	Poisson ratio
$\phi(\epsilon)$	Strain energy of deformation per unit volume
ϵ	Elastic strain
C_{ijkl}	Tensor of the second order elastic constants of a solid
C_{ijklmn}	Tensor of the second and third order elastic constants of a solid
l, m, n	Third order elastic constants of an isotropic solid
I_1, I_2, I_3	Invariants of the Lagrangian strain tensor
$\epsilon_i, \epsilon_j, \epsilon_k$	Principal strain components
v_{ii}	Velocity of a longitudinal wave, propagating in i-direction
v_{ij}, v_{ik}	Velocities of shear waves, propagating in i-direction vibrating in j- or k-direction, respectively
i, j, k	axes of a Cartesian coordinate system
$\sigma_i, \sigma_j, \sigma_k$	Principal stress components
AEC_{ij}	Acoustoelastic constant of an ultrasonic wave propagating in i-direction, vibrating in j-direction
v_{RR}	Velocity of a longitudinal wave propagating in the rolling direction of a textured sample
v_{RW}	Velocity of a shear wave propagating in the rolling direction, vibrating in the transverse direction of a textured sample
R	Rolling direction of a textured sample
W	Transverse direction of a textured sample
N	Normal direction of a textured sample
C	Anisotropy factor $C=C_{11}-C_{12}-2C_{44}$
C_{11}, C_{12}, C_{44}	Single crystal elastic constants
$C_4^{11}, C_4^{12}, C_4^{13}$	Expansion coefficients of the orientation distribution function
T	Transmitter of ultrasonic wave
R	Receiver of ultrasonic wave

4.03 Physical fundamentals

4.031 Influence of stress states on ultrasonic velocities and the acoustoelastic effect

Elastic waves propagate isotropic solids with a velocity which is characteristic for the material under test. The velocities of a longitudinal wave (v_L) and a shear wave (v_T) are given by:

$$\rho v_L^2 = \lambda + 2\mu = K + \frac{3}{4}\mu \quad (1)$$

$$\rho v_T^2 = \mu \quad (2)$$

respectively, where ρ is the density, λ and μ are the Lamé moduli and K is the bulk modulus. λ and μ describe the elastic behavior of the solid in first approximation (Hooke's law). In engineering, the elastic properties are more often characterized by the Young's (E) and Shear (G) moduli:

$$G = \mu \quad (3)$$

$$E = \mu (3\lambda + 2\mu) / (\lambda + \mu) \quad (4)$$

$$K = (3\lambda + 2\mu) / 3 \quad (5)$$

Longitudinal and shear waves propagate the bulk of components. By modifying the angle of incidence or the principle of wave generation, the sound beam can be turned into a surface near layer. The velocity of the skimming longitudinal also called creeping wave (v_{SL}), propagating in the surface layer, is also given by equation (1) as the velocity of the dispersion free SH_0 wave (v_{SH}) is determined by equation (2). The Rayleigh- or surface wave is a guided wave, it follows the surface shape of the component. Its velocity (v_R) is usually described by the Bergman approximation /27/:

$$v_R = v_T (0.87 + 1.12\nu) / (1 + \nu) \quad (6)$$

ν is the Poisson ration for the material.

The most widely used model for the description of the acoustoelastic effect, that is, the influence of strain states on the propagation velocities of ultrasonic waves, is given by Hughes and Kelly /2/ using Murnaghan's /1/ theory of finite deformations and third order terms in the elastic energy of a solid.

The strain energy of deformation per unit volume $\Phi(\epsilon)$ may be written as a power series in the elastic strain ϵ :

$$\Phi(\epsilon) = \Phi_0 + g \cdot C_{ij} \cdot \epsilon_{ij} + \frac{1}{2} C_{ijkl} \cdot \epsilon_{ij} \cdot \epsilon_{kl} + \frac{1}{6} C_{ijklmn} \epsilon_{ij} \cdot \epsilon_{kl} \epsilon_{mn} + \dots \quad (7)$$

If the strain energy is zero before the deformation, Φ_0 is zero. The second term is a potential energy which can be set equal to zero since the reference level is not important. C_{ijkl} and C_{ijklmn} are the tensors of the elastic second and third order constants of the solid. The number of elastic constants reduce in case of an isotropic solid to two independent second order (λ , μ) and three independent third order constants (l , m , n). The elastic constants are named from their positions in terms of the strains in equation (7). The coefficients of the term with the quadratic strains are second order, the ones of the term with the cubic strains are third order constants. In an isotropic solid, the strain energy density depends only on the invariants I_1 , I_2 and I_3 of the Lagrangian strain tensor since the elastic constants are invariant under arbitrary rotations:

$$\Phi(\epsilon) = (\lambda + 2\mu) I_1^2 / 2 - 2\mu I_2 + (l + 2m) I_1^3 / 3 - 2m I_1 I_2 + n I_3 \quad (8)$$

Also the density of the deformed body can be expressed in terms of the invariants:

$$\rho(\epsilon) = \rho / (1 + 2 I_1 + 4 I_2 + 8 I_3) \quad (9)$$

where ρ is the density of the solid at zero strain.

In case of the propagation of a plane wave along one principal axis of strain, there are only three non-vanishing strain components ϵ_{ii} , $\epsilon_{ij} = \epsilon_{ji}$, $\epsilon_{ik} = \epsilon_{ki}$ to be considered. Hence, the strain invariants reduce to

$$\begin{aligned} I_1 &= \epsilon_{ii} \\ I_2 &= -(\epsilon_{ij} \epsilon_{ji} + \epsilon_{ik} \epsilon_{ki}) \\ I_3 &= 0 \end{aligned}$$

Differentiating the equation (8) with respect to the strains yield the stress components. As described in [2] and in more detail in [28], the solution of the wave equation results in three expressions for the propagation of a pure longitudinal and two pure shear waves polarized to one principal direction of strain each.

These principal solutions can be generalized for the case of sound propagations in each of the three principal directions of a strained solid with cubic structure:

$$\rho v_{ii}^2 = \lambda + 2\mu + (2l + \lambda) (\epsilon_i + \epsilon_j + \epsilon_k) + (4m + 4\lambda + 10\mu) \epsilon_i \quad (10)$$

$$\rho v_{ij}^2 = \mu + (\lambda + m) (\epsilon_i + \epsilon_j + \epsilon_k) + 4\mu\epsilon_i + 2\mu\epsilon_j - 0.5n\epsilon_k \quad (11)$$

$$\rho v_{ik}^2 = \mu + (\lambda + m) (\epsilon_i + \epsilon_j + \epsilon_k) + 4\mu\epsilon_i - 0.5n\epsilon_j + 2\mu\epsilon_k \quad (12)$$

The first index of v represents the direction of sound propagation, the second the direction of vibration. i , j and k are the axes of a Cartesian coordinate system. l , m and n are the third order elastic constants of the material under consideration. v_{ii} is the velocity of a longitudinal wave propagating in the i -direction; v_{ij} and v_{ik} are the velocities of two shear waves polarized perpendicular to each other.

The equations (10), (11) and (12) are the fundamental equations for the ultrasonic evaluation of load or residual stress states. They describe the acoustoelastic effect. It should be

noted that these equations can only be used if the sound wave propagates and vibrates along principal axes. A former isotropic solid, subjected to a mechanical stress state becomes anisotropic. The velocities of ultrasonic waves become dependent on the magnitude and direction of the stresses causing the strain states in the solid.

In order to develop equations describing the influence of stress states on the ultrasonic velocities it is also assumed that the principal axes of strain coincide with the principal axes of stress. Using the generalized Hooke's law, the strains are replaced by the stresses and the elastic moduli. Replacing $\lambda + 2\mu$ in equation (10) by ρv_L^2 and using the approximation $v_{ii} + v_L = 2v_L$, simple treatments result in [17]:

$$\frac{V_{ii} - V_L}{V_L} = \frac{A}{C} \cdot \sigma_i + \frac{B}{C} \cdot (\sigma_j + \sigma_k) \quad (13)$$

A similar procedure yields:

$$\frac{V_{ij} - V_T}{V_T} = \frac{D}{K} \cdot \sigma_i + \frac{E}{K} \cdot \sigma_j + \frac{F}{K} \cdot \sigma_k \quad \text{and} \quad (14)$$

$$\frac{V_{ik} - V_T}{V_T} = \frac{D}{K} \cdot \sigma_i + \frac{F}{K} \cdot \sigma_j + \frac{E}{K} \cdot \sigma_k \quad (15)$$

A, B, C, D, E and F are combinations of the elastic constants of the material:

$$\begin{aligned} A &= 2(\lambda + \mu)(4m + 5\lambda + 10\mu + 21) - 2\lambda(21 + \lambda) \\ B &= 2(21 + \lambda)(\lambda + \mu) - \lambda(21 + \lambda) - \lambda(4m + 5\lambda + 10\mu + 21) \\ C &= 4\mu(\lambda + 2\mu)(3\lambda + 2\mu) \\ D &= 2(\lambda + \mu)(\lambda + m + 4\mu) - \lambda(2\lambda + 2m + 2\mu - 0.5n) \\ E &= 2(\lambda + \mu)(\lambda + m + 2\mu) - \lambda(2\lambda + 2m + 4\mu - 0.5n) \\ F &= 2(\lambda + \mu)(\lambda + m - 0.5n) - \lambda(2\lambda + 2m + 6\mu) \\ K &= 4\mu^2(3\lambda + 2\mu) \end{aligned} \quad (16)$$

The equations (11) and (12) only differ on the terms which consider the principal strains in the directions of vibration. Normalizing the difference $v_{ij} - v_{ik}$ to one of the velocities, e.g., to v_{ik} yield the relationship $(v_{ij} - v_{ik}) / v_{ik} = ((4\mu + n) / 4\mu) / (\epsilon_j - \epsilon_k)$ if the approximation $(v_{ij} + v_{ik})v_{ik} = 2v_T^2$ is used. The strains can be replaced by stress values using Hooke's law: $\epsilon_j - \epsilon_k = (1 / 2\mu)(\sigma_j - \sigma_k)$. Expressing velocity by time-of flight and path length, which is identical for both shear waves, yield expressions which are independent of the quantity path length [29]:

$$(t_{ik} - t_{ij}) / t_{ij} = (4\mu + n)(\sigma_j - \sigma_k) / 8\mu^2 \quad (17)$$

$$(t_{ji} - t_{jk}) / t_{jk} = (4\mu + n)(\sigma_k - \sigma_i) / 8\mu^2 \quad (18)$$

$$(t_{kj} - t_{ki}) / t_{ki} = (4\mu + n)(\sigma_i - \sigma_j) / 8\mu^2 \quad (19)$$

The equations (18) and (19) are derived from the equation (17) by cyclic permutation of the indices. The relationships (17), (18) and (19) describe the ultrasonic birefringence effect; they allow the characterization of stress states in terms of the difference of two principal stresses which is of particular benefit if yield criteria (Tresca, von Mises) are of interest. In addition to the independence from the ultrasonic path length, the birefringence equations contain only one third order elastic constant. As will be shown later, this constant n has been found to be less effected by changes of the microstructure of the material under test than l and m .

The stress or strain influence on the velocities of the skimming longitudinal wave and on the SH_0 wave propagating the near surface layer is the same as their influence on the waves propagating the bulk. Adjusting the direction indexes according to the directions of the principal axes and of the propagation and vibration directions, the equations (13) and (14) can be used.

The generalization of equations (10) and (11) to propagation directions not along the principal axes describe the angular dependence of the relative velocity changes of the skimming longitudinal (v_{SL}) and SH_0 (v_{SH}) waves:

$$\frac{V_{SL}(\Theta) - V_L}{V_L} = \frac{A+B}{2C}(\sigma_i + \sigma_j) + \frac{A-B}{2C}(\sigma_i - \sigma_j) \cos 2\Theta \quad (20)$$

$$\frac{V_{SH}(\Theta) - V_T}{V_T} = \frac{D+F}{2K}(\sigma_i + \sigma_j) + \frac{D-F}{2K}(\sigma_i - \sigma_j) \cos 2\Theta \quad (21)$$

Here, σ_i and σ_j are the principal in plane stresses and Θ is the angle between propagation direction and σ_i .

Rayleigh waves are guided waves; they follow the free surface. The mass particles vibrate along ellipses which have the large axis perpendicular to the surface and the small axis in the surface and both axes are perpendicular to the propagation direction. The major part of the energy of these waves propagates in the surface layer of one wavelength of depth. Hence, Rayleigh waves can be used to evaluate stress gradients by adjusting the center frequency f of the wave according to the relationship $d = V_R / f$ with d as the penetration depth. But special care must be taken: A stress gradient within the penetrated depth causes a frequency dependence of the wave velocity and a stress gradient goes along with a gradient of microstructural state in most cases and microstructural changes vary the acoustoelastic constants. Hence, calibration experiments are strongly recommended. It is also important to take into account that the Rayleigh wave velocity becomes frequency dependent if the radius of the surface curvature in the wave propagation direction is smaller than about ten times the wavelength. The influence of stress or strain states on the velocity of Rayleigh waves is given in a first order approximation in terms of the Poisson ratio and the third order elastic constants of the material /30/. The influence of stress and orthorhombic anisotropic (texture) is treated in perturbation /31/ and exact analysis /32/. In case of an isotropic solid the following equation is yielded:

$$\frac{V_{RS}(\Theta) - V_R}{V_R} = \frac{R_1 + R_2}{2}(\sigma_1 + \sigma_2) + \frac{R_1 - R_2}{2}(\sigma_1 - \sigma_2) \cos 2\Theta \quad (22)$$

Here V_{RS} is the Rayleigh wave velocity in the stressed solid; Θ the angle between wave propagation and the direction of the principal stress σ_1 . The constants R_1 and R_2 can be given in terms of the second and third order elastic constants of the material but, since the constants become small in value, it is strongly recommended to evaluate these constants in a tensile test experiment using a representative sample. R_1 and R_2 are the acoustoelastic constants for the Rayleigh wave propagation along and perpendicular to the direction of applied uniaxial stress, respectively.

It is important to note that all equations given in this section can only be used for the stress analysis of materials in which the stress or strain state is the only reason for the direction dependency of the ultrasonic velocities. It should also be mentioned that the application is limited to materials with cubic single crystal symmetry.

4.032 Evaluation of third order elastic constants and acoustoelastic constants

In order to achieve a quantitative evaluation of stress states using ultrasonic techniques, the material-dependent elastic constants have to be known.

The second order constant λ and μ or Young's and shear moduli can be evaluated according to equations (1) to (4) using the measured ultrasonic longitudinal and shear wave velocities and the density of the material.

The five possible different changes of ultrasonic longitudinal and shear waves as function of elastic strain in a tensile test experiment were measured by Egle and Bray for the first time [6]. The waves propagated the sample in directions parallel and perpendicular to the load direction. Figure 1 sketches the relative changes of sound velocities as function of the elastic strain.

The velocity of a longitudinal wave, propagating in the direction of the load (v_{11}) shows the strongest influence of the strain or stress. The velocity of a shear wave, propagating along the thickness direction decreases significantly if the vibration is parallel to the load axis (v_{31}) and increases slightly if the polarization is perpendicular (v_{32}) to the load direction. Comparing the velocities v_{31} and v_{13} it shows that the influence of stress or strain on the shear wave velocities is stronger if the polarization direction is along the load axis.

The displayed results are generally found for all steels and Al-alloys. The slopes of the lines are dependent on the material and on the microstructural state of the material under test.

The result of an experiment like this yields the evaluation of the 5 elastic constants of the material, namely the two second order and the three third order constants. Since the second order constants can simply be evaluated as described, it is sufficient to measure the influence of strain on the velocities of only 3 ultrasonic waves.

Usually, the propagation directions of a longitudinal and a shear wave are oriented in the through thickness direction perpendicular to the direction of load. The vibration direction of the linearly polarized shear wave is parallel (v_{31}) and perpendicular to the load (v_{32}).

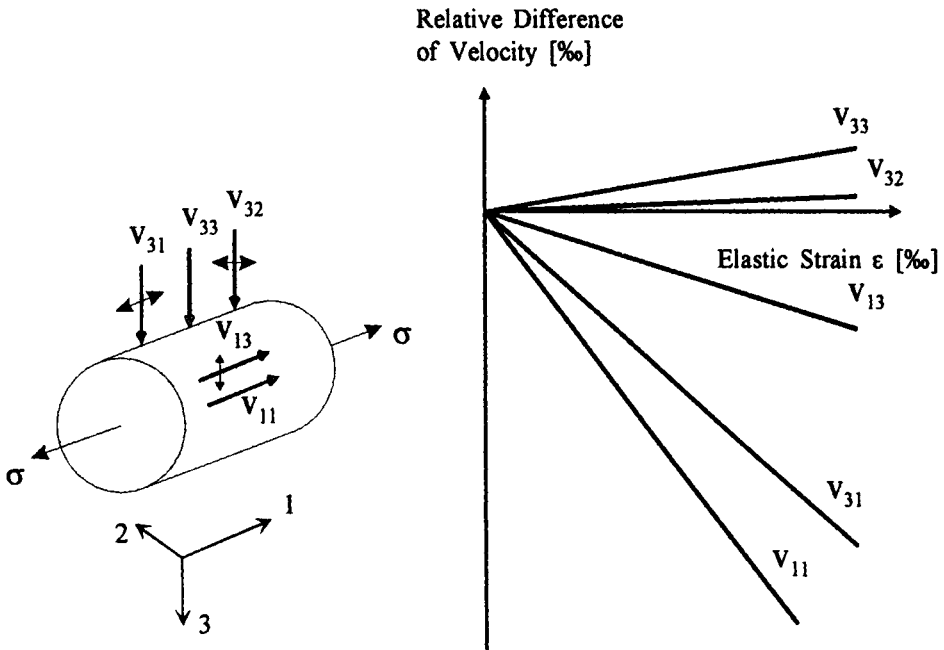


Figure 1. Relative change of wave velocities as function of elastic strain of a metallic sample.

Having the load in the 1-direction and the propagation directions of the ultrasonic waves in the 3-direction, the evaluation equations for the third order constants are:

$$l = \frac{\lambda (\lambda + \mu)}{\mu} \left[\frac{\lambda + 2\mu}{\lambda} \text{AEC}_{33} + \frac{4(\lambda + \mu)}{3\lambda + 2\mu} \left(\text{AEC}_{31} + \frac{\lambda}{2(\lambda + \mu)} \text{AEC}_{32} \right) + \frac{\lambda}{\lambda + \mu} \right] \quad (23)$$

$$m = \frac{2(\lambda + \mu)}{3\lambda + 2\mu} [\lambda \text{AEC}_{32} + 2(\lambda + \mu) \text{AEC}_{31}] - 2\mu \quad (24)$$

$$n = \frac{8\mu(\lambda + \mu)}{3\lambda + 2\mu} [\text{AEC}_{31} - \text{AEC}_{32}] - 4\mu \quad (25)$$

The AECs are the acoustoelastic constants. They describe the slope of the linear change of the relative difference of the velocity as function of the elastic strain ϵ . As before, the first index of the AEC describes the direction of propagation, the second one the direction of vibration of the corresponding wave. Figure 2 shows the relative differences of ultrasonic velocities versus the elastic strain ϵ of a fine grained steel sample and of an Al-alloy sample.

In some cases, especially if the thickness of the sample under test (3-direction) is small it is of advantage to use a skimming longitudinal wave, propagating in the 1-direction between transmitter and receiver probes. With the AEC_{11} the constant l is given as:

$$l_{11} = \frac{\lambda + \mu}{\mu} \left[(\lambda + 2\mu) AEC_{11} - \frac{4(\lambda + \mu)}{3\lambda + 2\mu} (\lambda AEC_{32} + 2(\lambda + \mu) AEC_{31}) - (2\lambda + \mu) \right] - \frac{\lambda}{2} \quad (26)$$

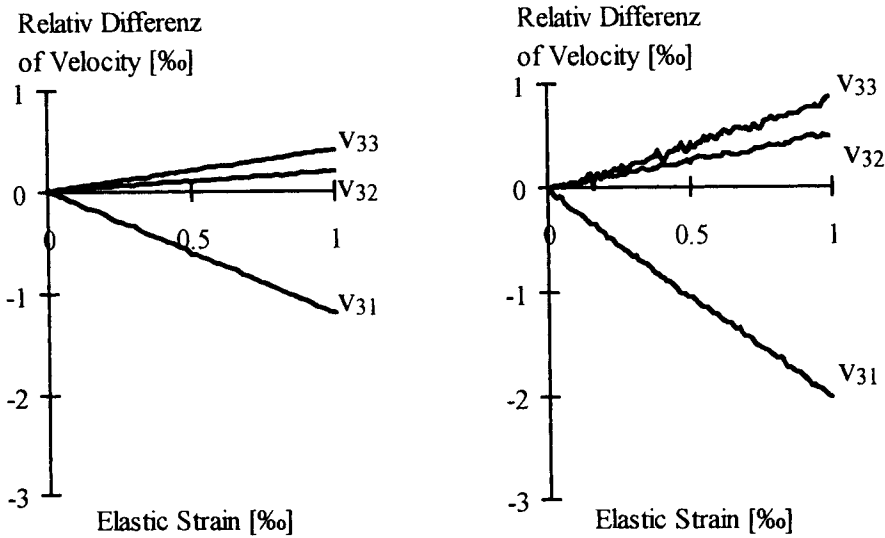


Figure 2. Relative change of longitudinal (v_{33}) and shear wave velocities (v_{31}, v_{32}) versus elastic strain of a fine grained steel sample (left) and of an Al-alloy sample (right).

Table 1 summarizes elastic constants of different materials.

Table 1
Second and third order constants [GPa] of materials.

Material	λ	μ	l	m	n
Armco-Iron /2/	110 ± 0.4	82 ± 1	-348 ± 65	-1030 ± 70	$+1100 \pm 1100$
Steel ferrit. / pearlitic	110	81	-270	-580	-710
Steel 0.12% C /36/	115	82	-301 ± 37	-666 ± 6.5	-716 ± 4.5
St 42	110	81	-48	-503	-652
St E 355 /33/	109	82	-192	-565	-724
Rail Steel /6/	$116 \pm 2\%$	$80 \pm 2\%$	$-248 \pm 3\%$	$-623 \pm 4\%$	$-714 \pm 3\%$
Rail Steel /6/	$110 \pm 2\%$	$82 \pm 2\%$	$-302 \pm 3\%$	$-616 \pm 4\%$	$-724 \pm 3\%$
Rail Steel	$112 \pm 1\%$	$81 \pm 1\%$	$-358 \pm 5\%$	$-650 \pm 3\%$	$-721 \pm 3\%$

Table 1 continued.

Material	λ	μ	l	m	n
Steel Hecla 37 /34/	111 ± 1	82	-461 ± 65	-636 ± 46	-708 ± 32
Steel Hecla 17 /34/	111	82	-328 ± 30	-595 ± 32	-668 ± 24
Steel Hecla 138A /34/	109	82	-426 ± 55	-619 ± 50	-708 ± 40
Steel A 533 B /35/	119	79	-218	-486	-564
Steel A 471 /35/	120	79	-179	-496	-628
Steel Hecla /34/ austenite	87	72	-535 ± 90	-752 ± 100	-400 ± 40
17 Cr Ni Mo 6	109	81	-58 ± 38	-517 ± 14	-718 ± 18
24 Cr Mo 5V	112	83	-350	-624	-702
24 Cr Mo 5V	112	82	$-440 \pm 10\%$	$-600 \pm 2\%$	$-670 \pm 2\%$
30 Cr Mo Ni V 5 11	109	83	-357	-574	-670
22 Ni Mo Cr 3 7	109	82	-185	-503	-652
22 Ni Mo Cr 3 7	$109 \pm 2\%$	$82 \pm 1\%$	$-196 \pm 10\%$	$-520 \pm 2\%$	$-657 \pm 2\%$
22 Ni Mo Cr 3 7	$111 \pm 2\%$	$82 \pm 1\%$	$-190 \pm 10\%$	$-555 \pm 2\%$	$-659 \pm 2\%$
24 Ni Cr Mo V14 5	110	80	-90	-439	-546
Ni-Steel	109	82	-328	-578	-676
Ni-Steel S/NTV /5/	109 ± 1	82	-56 ± 20	-671 ± 6	-785 ± 7
Ni-Steel Rex 535 /5/	91	78	-46	-590	-730
Ni-Steel Rex 535 /34/	109	82	-327 ± 75	-578 ± 80	-676 ± 60
15 Mn Ni 6 3	$110 \pm 2\%$	$81 \pm 2\%$	$-270 \pm 10\%$	$-580 \pm 2\%$	$-710 \pm 2\%$
X6 Cr Ni 1811	101	75	-370	-532	-236
Roll H-Cr R 78/80	$129 \pm 2\%$	$84 \pm 2\%$	$-573 \pm 8\%$	$-775 \pm 3\%$	$-996 \pm 3\%$
WC-Co Sintered Metal	178	256	-464	-1390	-2108
WC-Co Sintered Metal	178	257	-552	-1453	-2153
Al 99% /5/	61 ± 1	25	-47 ± 25	-342 ± 10	-248 ± 10
Al 99.05% /36/	64	27	-319 ± 10	-373 ± 5	-354 ± 3
Al 99.3% /34/	57-	28	-311 ± 12	-401 ± 138	-408 ± 136
Al B 53 S /34/	62	26	-201 ± 23	-305 ± 27	-300 ± 24
Al B 53 S /34/	58	26	-223 ± 40	-237 ± 20	-276 ± 20
Al D 54 S /34/	49	26	-387 ± 125	-358 ± 15	-320 ± 12
Al JH 77 S /34/	58	27	-337 ± 25	-395 ± 24	-436 ± 28
Al Mg 3	56	28	-212	-309	-369
Al Mg 3	58	27	-245	-313	-350
Al 8091	45	31	-218	-378	-435
Al 7064	60	27	-324	-397	-403
Cu 99.85% /36/	107	47	-8378 ± 131	-2429 ± 16	-527 ± 4
Cu 99.9% /5/	104	46	-542 ± 30	-372 ± 5	-401 ± 5

Knowing the second and third order constants has the advantage that the influence of strain or stress states on the ultrasonic velocities can be evaluated. Hence, it is possible to find a wave which is strongly influenced on the stress or strain to be evaluated and another wave with different direction of propagation and / or vibration which is negligibly effected. This wave

velocity might be useful for the characterization and discrimination of microstructural influences.

In many cases only the AEC values are evaluated. This is particularly sufficient if the tensile test experiment is carried out under the same experimental conditions as the later analysis of the stress or strain state. The definition of the AEC is not uniform in literature. It is used for the relative change of ultrasonic velocity per applied stress or per applied strain. Sometimes the reverse value is also abbreviated as AEC.

4.033 Influence of texture on ultrasonic velocities and on the acoustoelastic effect

Most structural materials are polycrystalline aggregates and their exposure to plastic deformation and / or heat treatment during manufacturing leads to the alignment of the single crystals relative to the forming geometry or the temperature gradient. This development of preferred orientation or texture is the main reason for the anisotropic behavior of these materials. Electric, magnetic, elastic and plastic properties become direction dependent. This is desirable, for example, for transformer sheets in order to minimize the loss of energy during the magnetic reversals. And a certain texture is also needed in order to optimize the plastic deformation of rolled sheets during the deep drawing of e.g. automotive body parts or cans. In other cases, especially designed thermal and / or mechanical treatments are applied to yield components with isotropic properties. Industrial practice for characterizing texture is the measurement of X-ray pole figures from which the expansion coefficients of the orientation distribution function (ODF) are evaluated /37,38/.

The elastic anisotropy caused by texture influences the ultrasonic velocities and is, in addition to the strain or stress state, another reason for the direction dependences of the wave velocities. Both influences, the texture and the stress influence superimpose each other. In order to evaluate stress states, the texture influence on the velocities has to be separated or discriminated.

In cubic materials (steels, aluminium-alloys) an orthotropic texture develops e.g. during rolling. The texture is characterized by three orthogonal mirror planes. Because of the cubic crystal symmetry on the one hand, and the orthotropic texture symmetry on the other hand, only the three fourth order expansion coefficients C_4^{11} , C_4^{12} and C_4^{13} need to be considered for texture description in a first approximation. The velocities of ultrasonic shear and longitudinal waves, propagating a stress free cubic material with orthotropic texture are /39/:

$$\rho v_{RR}^2 = C_{11} - C \left(\frac{2}{5} - \frac{1}{70} \sqrt{\frac{7}{3}} \left(C_4^{11} - \frac{2}{3} \sqrt{5} C_4^{12} + \frac{1}{3} \sqrt{35} C_4^{13} \right) \right) \quad (27)$$

$$\rho v_{WW}^2 = C_{11} - C \left(\frac{2}{5} - \frac{1}{70} \sqrt{\frac{7}{3}} \left(C_4^{11} + \frac{2}{3} \sqrt{5} C_4^{12} + \frac{1}{3} \sqrt{35} C_4^{13} \right) \right) \quad (28)$$

$$\rho v_{NN}^2 = C_{11} - C \left(\frac{2}{5} - \frac{1}{70} \sqrt{\frac{7}{3}} \frac{8}{3} C_4^{11} \right) \quad (29)$$

$$\rho v_{RW}^2 = C_{44} + C \left(\frac{1}{5} + \frac{1}{70} \sqrt{\frac{7}{3}} \left(\frac{1}{3} C_4^{11} - \frac{1}{3} \sqrt{35} C_4^{13} \right) \right) = \rho v_{WR}^2 \quad (30)$$

$$\rho v_{WN}^2 = C_{44} + C \left(\frac{1}{5} - \frac{1}{70} \sqrt{\frac{7}{3}} \left(\frac{4}{3} C_4^{11} + \frac{2}{3} \sqrt{5} C_4^{12} \right) \right) = \rho v_{NW}^2 \quad (31)$$

$$\rho v_{NR}^2 = C_{44} + C \left(\frac{1}{5} - \frac{1}{70} \sqrt{\frac{7}{3}} \left(\frac{4}{3} C_4^{11} - \frac{2}{3} \sqrt{5} C_4^{12} \right) \right) = \rho v_{RN}^2 \quad (32)$$

The first subscript of the velocity v indicates again the propagation direction, the second one indicates the polarization direction of the wave. Since the rolling texture is of orthotropic symmetry and frequently found in half products, this type of texture is chosen to describe the texture influence on ultrasonic velocities. R, W and N represent the rolling, transverse and normal directions, respectively. $C = C_{11} - C_{12} - 2C_{44}$ is the anisotropy factor, a measure of the elastic anisotropy of a single crystal. C_{11} , C_{12} , C_{44} are the single crystal elastic constants. C_4^{11} , C_4^{12} and C_4^{13} are the expansion coefficients according to the Bunge notation. These expansion coefficients carry the texture information. The Roe notation is also often found, so that the following relationships can be helpful:

$$W_{400} = C_4^{11} \cdot \sqrt{2} \cdot \sqrt{\frac{7}{3}} / 48 \pi^2 \quad (33)$$

$$W_{420} = C_4^{12} \cdot \sqrt{\frac{7}{3}} / 48 \pi^2 \quad (34)$$

$$W_{440} = C_4^{13} \cdot \sqrt{\frac{7}{3}} / 48 \pi^2 \quad (35)$$

As can be seen from the equations (30), (31), (32), the influence of texture on the shear wave velocities remains unchanged if the directions of propagation and vibration are interchanged. The velocity of the longitudinal wave propagating in the normal direction depends only on one expansion coefficient (equation 29). It follows from the equations (31) and (32) that the combined use of shear wave velocities yield also relationships with one expansion coefficient only

$$\rho (v_{NR}^2 + v_{NW}^2) = 2 C_{44} + 2 C \left(\frac{1}{5} - \frac{1}{70} \sqrt{\frac{7}{3}} \cdot \frac{4}{3} \cdot C_4^{11} \right) \quad (36)$$

$$\rho (v_{NR}^2 - v_{NW}^2) = 2 \cdot C \cdot \frac{1}{70} \sqrt{\frac{7}{3}} \cdot \frac{2}{3} \sqrt{5} \cdot C_4^{12} \quad (37)$$

and from the equations (29) and (36) the following relationship, which is independent of texture:

$$\rho (v_{NN}^2 + v_{NR}^2 + v_{NW}^2) = C_{11} + 2 C_{44} \quad (38)$$

Out of the numerous possibilities of determining the expansion coefficients from equations (27) - (32) those which fit best the requirement of low error propagation are /39/:

$$C_4^{11} = \frac{210}{8C} \sqrt{\frac{3}{7}} \cdot \left(2\mu - (C_{11} + 2 C_{44}) \cdot \frac{v_{NR}^2 + v_{NW}^2}{v_{NR}^2 + v_{NW}^2 + v_{NN}^2} \right) \quad (39)$$

$$C_4^{12} = \frac{210}{4\sqrt{5}C} \sqrt{\frac{3}{7}} \cdot \left((C_{11} + 2 C_{44}) \cdot \frac{v_{NR}^2 - v_{NW}^2}{v_{NR}^2 + v_{NW}^2 + v_{NN}^2} \right) \quad (40)$$

$$C_4^{13} = \frac{210}{8\sqrt{35}C} \sqrt{\frac{3}{7}} \cdot \left(6\mu - \lambda + (C_{11} + 2 C_{44}) \cdot \frac{v_{NN}^2 + 8v_{RW}^2}{v_{NR}^2 + v_{NW}^2 + v_{NN}^2} \right) \quad (41)$$

$$C_4^{11} = \frac{210}{2C} \sqrt{\frac{3}{7}} \cdot (\rho(v_{SH0}^2(0^\circ) + v_{SH0}^2(45^\circ)) - 2\mu) \quad (42)$$

$$C_4^{13} = \frac{210\mu}{32\sqrt{35}C} \sqrt{\frac{3}{7}} \cdot (1 - 2 \cos^2 \theta)^{-2} \cdot \left(1 - \sqrt{1 - 16 \frac{(1 - 2 \cos^2 \theta)^2}{\cos^2 \theta \sin^2 \theta} \cdot \frac{v_{SH0(\theta)} - v_{SH0(0^\circ)}}{v_{SH0(0^\circ)}}} \right) \quad (43)$$

Not only the velocities of ultrasonic waves but also their stress or strain dependence (acoustoelastic effect) is influenced by the texture.

The most comprehensive theoretical treatment of the texture influence on the second and third order constants is done by Johnson /40/. In order to describe the elastic behavior of a material with cubic crystal symmetry and rolling texture, 9 second order and 20 third order constants are needed. The technique employed in order to obtain the estimates is a simple Voigt-type procedure, in which the second and third order stiffness tensors are averaged over the orientation distribution function for the crystallites. The results for the second order constants are equivalent to previous estimates of such quantities by Bunge /41/, Morris /42/ and Sayers /43/. The results for the third order constants contain as a special case the earlier work by Johnson /44/, who gives estimates for an aggregate that exhibits transverse isotropy.

In case of transverse isotropy, only the two expansion coefficients W_{400} and W_{600} are needed. In case of orthotropic texture (e.g. rolling texture) the three fourth order expansion coefficients W_{400} , W_{420} , W_{440} and the sixth order expansion coefficients W_{600} , W_{620} , W_{640} and W_{660} contribute to the second and third order elastic constants.

Since in most cases of application the texture is not known in terms of the expansion coefficients of the orientation distribution function, the experimental evaluation of the second and third order elastic constants using representative samples is a straight forward approach. Table 2 summarizes experimental results on samples cut parallel to each of the two principal texture directions. The propagation of the ultrasonic waves was along the third texture axis. Chatellier and Touratier describe a model and a more comprehensive experimental procedure to determine the acoustoelastic constants of cubic materials with an orthotropic texture /45/.

Table 2
Second and third order constants [GPa] evaluated using samples cut along the two principal directions of texture.

Material	λ	μ	l	m	n
Steel /46/	80	84	-283 ± 16	-666 ± 30	-837 ± 29
	80	84	-253 ± 21	-635 ± 30	-793 ± 28
Roll H-Cr-R 78/80	128	87	-597	-786	-927
	128	87	-712	-812	-926
Roll H-Cr-R 78/80	130	91	-365	-614	-837
	129	91	-471	-676	-861
Al 99.5% /46/	49	26	-73 ± 25	-264 ± 4	-352 ± 2
	49	26	-55 ± 31	-249 ± 9	-316 ± 4
Al 7075	57	27	-185 ± 75	-316 ± 19	-350 ± 17
			-117 ± 81	-272 ± 15	-326 ± 14
Al 7475	57	27	-192 ± 70	-335 ± 17	-358 ± 16
			-135 ± 69	-276 ± 15	-331 ± 14

4.034 Influence of temperature on ultrasonic velocities and on the acoustoelastic effect

With the temperature of a material, also its density and its elastic behavior change. Hence, the velocities of ultrasonic waves propagating the material become temperature dependent.

Using ultrasonic frequencies between 1 and 20 MHz, experimental investigations in the temperature range between -10°C and +40°C result in linear decreases of longitudinal and shear velocities with increasing temperature. There is no difference found, using samples of different ferritic pearlitic steels. The longitudinal wave and shear wave velocities decrease about $0.1\% \pm 0.01\%$ and $0.12\% \pm 0.02\%$ respectively as the temperature increases by $10^\circ\text{C} \pm 0.1^\circ\text{C}$.

Investigations of the temperature influence up to 1200°C using electromagnetic transducers /47/ and laser generated ultrasonic waves /48,49,50/ show different results for ferritic and austenitic steels.

Using the sound velocities and the density, the Youngs and shear moduli are calculated for the temperature range between -20°C and +200°C. For both ferritic and austenitic steel, the Youngs and shear moduli decrease with $0.3\% \pm 0.01\%$ and $0.33\% \pm 0.02\%$ if the sample's temperature increases by $10^\circ\text{C} \pm 0.1^\circ\text{C}$.

Like the second order, also the third order constants change with the material's temperature. Experimental investigations in the range between room temperature and 60°C using different Al-alloys, a ferritic and an austenitic steel sample are described in /51,52/. It is found that the third order constants l and m decrease linearly with temperature while the constant n does not change significantly.

Theoretical models describing the temperature induced change of the elastic properties of a solid are often described in literature. A most recent and comprehensive one is given by Cantrell /53/. The same author developed a theoretical model describing the stress or strain induced change of the temperature dependence of the sound velocities /54/.

Table 3
Relative change of longitudinal and shear velocities in steels and Al-alloys with temperature. The change of velocity is related to the velocity measured at 20°C sample temperature.

Relative change of sound velocities per 10°C raise of temperature.			
Material	Temperature Range	Longitudinal Wave	Shear Wave
Ferritic-Pearlitic Steel	-10°C to + 40°C	-0.11% ± 0,01%	-0.12% ± 0,02% -0.16
Austenitic Steel X6 CrNi 1811	+25°C to + 85°C	-0.15% ± 0,01%	-0.25% ± 0,02%
Austenitic Steel 18/8 /47/	+20°C to + 1200°C	-0.13%	
Austenitic-Ferritic Steel AF 22 /48/	+20°C to + 500°C	-0.13%	
	+500°C to + 900°C	-0.16%	
	+1000°C to + 1200°C	-0.2%	
Low Alloyed Carbon Steel St E 450.7 TM /49/	+20°C to + 500°C	-0.06%	
	+500°C to + 800°C	-0.34%	
	+800°C to + 1200°C	-0.08%	
Mild Steel BS 4360 grade 43 A /48/	+17°C to + 700°C	-0.15%	-0.22%
	+800°C to + 1200°C	-0.1%	-0.21%
Stainless Steel BS 970 /48/	+17°C to + 1200°C	-0.11%	-0.21%
Al, DURAL /50/	+5°C to + 300°C	-0.15%	
Al Alloy 2024 - T 351	-15°C to + 10°C	-0.16% ± 0,01%	-0.27% ± 0,02%
Al Mg 3	+20°C to + 60°C	-0.18% ± 0,01%	-0.33% ± 0,02%

Table 4
Relative change of third order constants of steels on Al-alloys if material's temperature increases by 10°C. The change is related to the values measured at 20°C sample temperature.

Material	l	m	n
24 Cr Mo 5V	-6.4%	-1.2%	0%
X6 CrNi 1811	-7.8%	-3.0%	0%
Al Mg 3	-24%	-5.1%	0.9%
Al 8091	-5.5%	-0.5%	-1.3%
Al 7064	-16%	-2.5%	-0.8%

Based on considerations of the Helmholtz free energy of the solids, the effect is expressed in terms of second, third, fourth and fifth order elastic constants of the crystal. Calculations of the fourth and fifth order constants are obtained using the central force Born-Mayer potential between ion pairs. The effect was experimentally investigated by Salama and co-authors /55,56,57/.

It was found that the temperature dependence of ultrasonic wave velocity is a linear function of the applied stress to metallic tensile test samples:

$$\left[\left(\frac{\Delta V}{\Delta T} \right)_{\text{stressed}} - \left(\frac{\Delta V}{\Delta T} \right)_{\text{not stressed}} \right] / \left(\frac{\Delta V}{\Delta T} \right)_{\text{not stressed}} = P \cdot \sigma \quad (44)$$

Applying a longitudinal wave propagating an Al-alloy sample in the direction perpendicular to the load, the factor of proportionality P was evaluated to $25 \times 10^{-4} \text{ MPa}^{-1}$ /56/. Similar investigations, using a different Al-alloy, resulted in $P = 22.5 \times 10^{-4} \text{ MPa}^{-1}$ /52/. Calculations using the theoretical model /54/ yielded $P = 18 \times 10^{-4} \text{ MPa}^{-1}$.

Taking into account that both the temperature and the stress or strain dependence of ultrasonic wave velocities are dependent on the material and on the microstructural state of a sample, the above mentioned agreement between experimental and calculated values of P is regarded as very good.

The effect of stress or strain induced changes of the temperature dependence of ultrasonic waves is not widely used for the evaluation of stress states in samples and components. This is mainly because of the extensive measuring effort.

4.035 Influence of microstructural changes on ultrasonic velocities and on the acoustoelastic effect

The microstructural state determines the elastic behavior of a material and hence the ultrasonic velocities. Ultrasonic techniques for the evaluation of mean grain sizes, porosities, creep and fatigue damages are developed for individual cases of application. An overview is given in /58,59/.

Because of the complex interaction between microstructural elements like grains, grain boundaries, second phases, precipitates, pores, dislocations, interstitials, vacancies and the ultrasonic wave propagation and the fact that their influences on the sound velocities is in the same order of magnitude as the influence of strain or stress, there is no generalized approach which can be used to evaluate stress states in materials in which the microstructural state varies in the inspected area.

The only meaningful procedure is the experimental investigation of ultrasonic velocities and their stress or strain dependence using representative samples with different microstructural states.

Microstructure

In order to allow the ultrasonic evaluation of stress states in and around a weld seam in ferritic steel structures, samples were cut from a plate of the same material, welded with the same welding parameters as done in the structure. Using the results of the ultrasonic velocity measurements and of their changes as function of a controlled elastic tensile strain, the material dependent elastic constants are evaluated.

It is to be seen from Table 5 that the change of microstructure does not effect the second order constants Young's and shear moduli. The third order elastic constants are dependent on the microstructural state. The largest relative differences, related to the values, evaluated for the ferritic / pearlitic states are 33% for the constant l , 15% and 13% for the constants m and n , respectively.

Similar experimental investigations are reported in /60/. Samples of the same steel grade 15 MnNi 6 3 were heat treated in order to simulate the microstructure in and around the weld seam. It was also found that the second order constants remained unchanged. The largest relative difference of the third order constants was found to be $30\% \pm 5\%$ in case of l and $5\% \pm 1\%$ and $7\% \pm 1\%$ for the constants m and n.

Table 5

Elastic constants [GPa] of steel 15 MnNi 63 with different microstructural states.

Microstructure	Young's Modulus	Shear Modulus	l	m	n
Ferrite / Pearlite	$210 \pm 2\%$	$81 \pm 2\%$	$-397 \pm 8\%$	$-617 \pm 5\%$	$-710 \pm 3\%$
Ferrite > 50%					
Ferrite 30% - 40% + Intermediate	210	81	-416	-636	-689
Intermediate	210	81	-348	-601	-694
Intermediate + Wittmannstätten	209	80	-285	-543	-619
Intermediate + Retained Austenite	210	81	-342	-588	-673

Measurements of the velocity of a longitudinal wave propagating perpendicular to the load axis as function of the applied tensile load were performed in order to investigate the influence of C-content on the acoustoelastic effect of different steel samples /61,62/:

Within the measuring error, a linear correlation is found between the acoustoelastic constant and the contents. From the fact that the AEC of the higher alloyed steel ASTM A533B fits into the linear relationship it is concluded that the carbon content of a steel is of stronger influence than other alloying elements.

Table 6

The acoustoelastic constant (AEC_{33}) of a longitudinal wave propagating perpendicular to the load for steel samples with different content on carbon or on ferrite phase.

Steel	C-content [%]	Ferrite phase [%]	Acoustoelastic constant [10^{-6} MPa^{-1}]	Change, related to AISI 1020 values
AISI 1020	0.18-0.23	97	$2.39 \pm 2\%$	0
ASTM A533 B	0.25	96.3	$2.44 \pm 2\%$	+2%
AISI 1045	0.43-0.50	93.3	$2.67 \pm 2\%$	+11%
AISI 1095	0.90-1.03	85.8	$3.09 \pm 2\%$	+29%

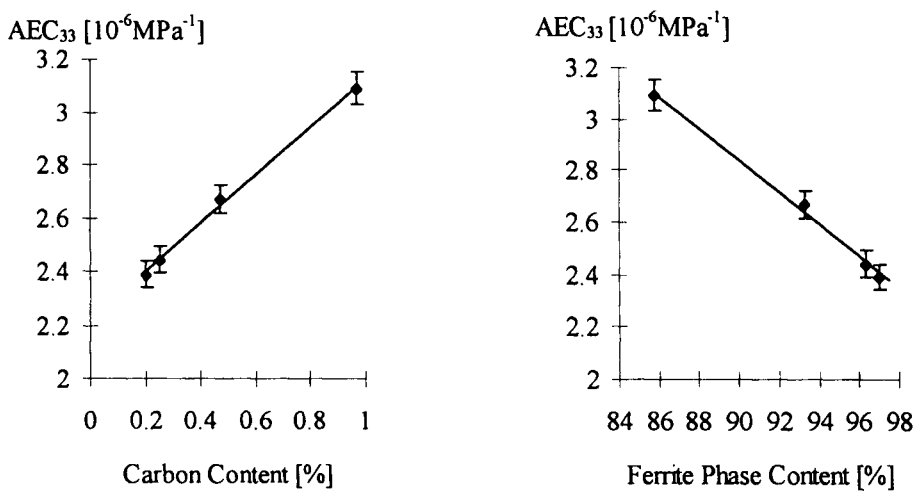


Figure 3. The acoustoelastic constant AEC_{33} of a longitudinal wave propagating perpendicular to the load for steel samples with different content on carbon (left) or on ferrite phase (right).

Similar experiments using samples of the work hardening Al-alloys 5052, 3003, 1100-0, and of the precipitation hardening Al-alloys 6061-T6, 2024-T351 also yielded a linear correlation with the percentage of solid solution phase. Furthermore, correlations between the acoustoelastic constant and the yield strength of the alloys are found /63/. The influence of the constant of SiC particles in SiC reinforced Al matrix composites on the second and third order elastic constants is described in /64/ and /52/. Calculations based on a model in which the precipitates are represented by a dilute elastic suspension of spherical particle inclusions in an infinite matrix confirmed the experimental results /62,65/.

Table 7

The acoustoelastic constant (AEC_{33}) of a longitudinal wave propagating perpendicular to the load for Al-alloys with different content on solid solution phase. The values for yield strength and Brinell number are also given.

Al-Alloy	Solid-Solution Phase (%)	Acoustoelastic Constant ($\times 10^{-6} MPa^{-1}$)	Yield Strength (MPa)	Brinell Number (500kg load, 10mm ball)
5052	99	$14.1 \pm 2\%$	155	63
3003	96	$12.9 \pm 2\%$	100	40
1100-0	92	$11.1 \pm 2\%$	20	23
6061-T6	98	$12.6 \pm 2\%$	240	94
2024-T351	96	$13.4 \pm 2\%$	270	120

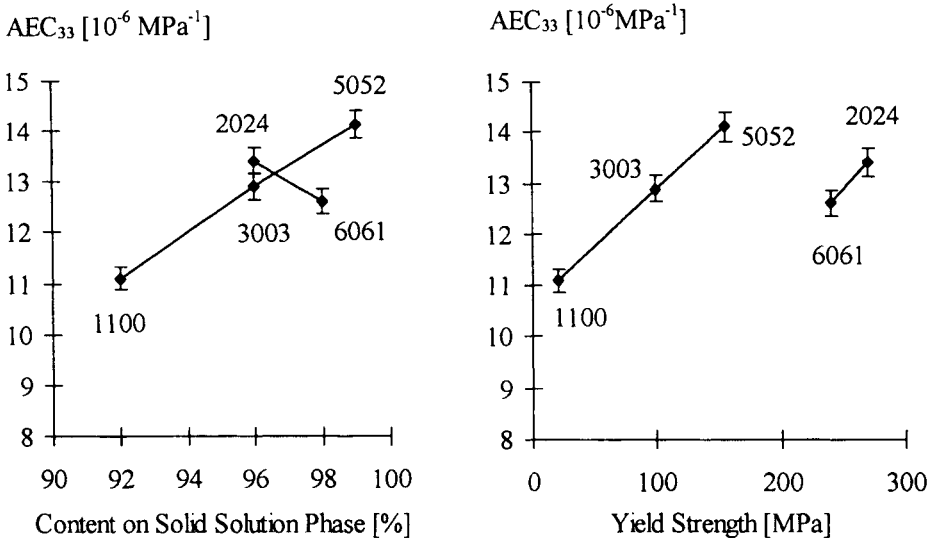


Figure 4. The acoustoelastic constant AEC_{33} of a longitudinal wave propagating perpendicular to the load for work hardening and precipitation hardening Al-alloys with different content on solid solution phase (left) and with different yield strengths (right).

Plastic deformation

Since the plastic deformation of a metal goes along with movements of dislocations and the increase of dislocation density, the dependency of sound velocities and higher order elastic constants on elastic deformation is expected.

Measurements of ultrasonic velocities as function of elastic-plastic strain of steels and Al-alloys are described in literature /7,66/. A model given by Johnson /66/ allows the calculation of ultrasonic velocities propagating a plastically deformed material with considerable algebraic effort. Calculations using a simplification of the model are to some extent in agreement with experimental results /67/. The measuring effect depends on the material and on the ultrasonic propagation and vibration direction with respect to the load and is different in the tensile and compressive regime.

Measurements of ultrasonic longitudinal and shear waves as function of the elastic strain were performed in order to evaluate the second and third order constants of steel samples which were plastically deformed by tensile stress. It has been found that Young's and shear moduli as well as the third order constant n do not change within the experimental error ($\pm 1\%$ and $\pm 7\%$, respectively) if the plastic deformation is less than about 8%. The third order constants l and m show a strong dependence of plastic deformation. These results are found for the ferritic 22 NiMoCr 3 7 steel as well as for the austenitic X6 CrNi 18 11 steel /60/. Results of recent experimental investigations, shown in Figure 5, illustrate the influence of plastic strain on the acoustoelastic effect of steel samples.

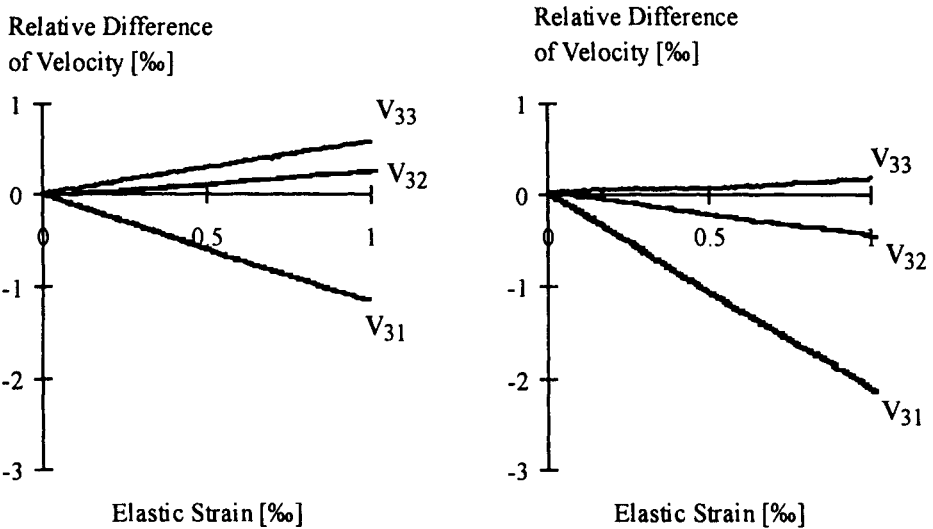


Figure 5. Relative change of ultrasonic longitudinal (v_{33}) and shear waves (v_{31}, v_{32}) versus elastic strain of a steel sample without (left) and with 12% plastic prestrain (right).

A model describing the interactions of ultrasonic waves with dislocations is given by Granato and Lücke /68/. The dislocations are considered as short strings which vibrate in the elastic field of the ultrasonic wave. The model mainly treats the ultrasonic attenuation due to dislocation damping in terms of the dislocation density, the loop length of the dislocation between pinning points, the dislocation tension and the ultrasonic frequency. The model predicts a very small (less than 0.1 %) influence on the wave velocities. The influence of dislocation density on the velocity and on the acoustoelastic effect is part of a present study. Using steel samples of the same grade and microstructural state, with the same mean grain sizes, and with dislocation densities of $1.4 \times 10^{10} \text{ cm}^{-2}$, $2.4 \times 10^{10} \text{ cm}^{-2}$ and $3.1 \times 10^{10} \text{ cm}^{-2}$ the absolute sound velocities of 5 MHz ultrasonic waves at room temperature was measured. The sound velocities changed only some tenth of a percent. But the change of sound velocity as function of the elastic strain of the samples was found to be significantly influenced by this small differences in dislocation densities as to be seen in Figure 6.

All nine second order and twenty third order constants are given as function of the thickness reduction ratio of cold rolled steel plates by Chatellier and Touratier /69/.

The experimental results indicate a significant influence of plastic deformation on the elastic constants. Hence, care must be taken if ultrasonic techniques are applied to evaluate stress states of plastically deformed components.

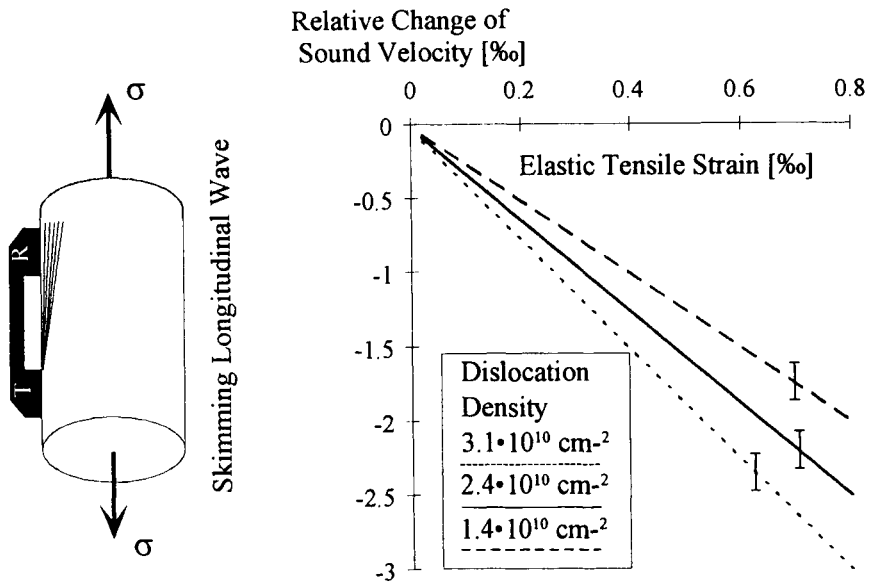


Figure 6. Relative change of wave velocity as function of elastic strain of steel samples with different dislocation densities.

Density

Whereas the density in conventional metals can usually be regarded as constant, the densities in sintered metals and in ceramics can change locally. In view of the evaluation of stresses in ceramics, the influence of density on the relevant elastic constants was investigated for Al_2O_3 specimens. Table 7 summarizes the experimental results.

Table 7

Elastic constants [GPa] of Al_2O_3 samples with different densities.

Density [g/cm ³]	Shear modulus μ	Young's modulus	l	m	n
3.915	$160 \pm 3\%$	$395 \pm 3\%$	$-68 \pm 15\%$	$-970 \pm 10\%$	$-1300 \pm 10\%$
3.90	$159 \pm 3\%$	$391 \pm 3\%$	$-69 \pm 15\%$	$-953 \pm 15\%$	$-1279 \pm 10\%$
3.87	$154 \pm 3\%$	$380 \pm 3\%$	$-76 \pm 15\%$	$-919 \pm 15\%$	$-1228 \pm 10\%$
3.5	$119 \pm 3\%$	$284 \pm 3\%$	$-70 \pm 15\%$	$-600 \pm 15\%$	$-865 \pm 10\%$

It is found that shear and Young's moduli as well as the third order constants m and n change linearly with density whereas the constants l remained unaffected [70]. Since linear changes of the ultrasonic velocities as function of the elastic strain have been found, it is assumed that the described formalism can also be applied to determine the higher order elastic constants of Al_2O_3 ceramic.

Creep and fatigue damages in metallic components cause, among others, local changes of the material density. In order to allow a quantitative ultrasonic stress analysis in damaged components, the influence of the damages on the elastic moduli and on the strain dependence of ultrasonic velocities must be systematically investigated.

The published results on the influences of microstructure on the ultrasonic velocities and especially on the strain dependences of ultrasonic velocities are of individual nature and might be used for a qualitative analysis. In view of the quantitative ultrasonic evaluation of stress states it is recommended that the material constants should be evaluated using a sample with a microstructural state which is representative for the state of the component to be tested.

4.04 **Measuring systems and setups for specific applications**

The ultrasonic techniques for the evaluation of stress states use the influence of the strains respectively stresses on the ultrasonic velocities. Since the effect is in the range of some tenths of percent, the ultrasonic path length and the ultrasonic time-of-flight have to be measured with an accuracy of some parts in tenthousand. The measurement of the path length is usually avoided either by using transmitter and receiver probes with unchanged distance fixed in a rigid frame or by the combined use of two waves propagating the same path with different sensitivities to the stress. Hence, the time-of-flight remains as the only measuring quantity.

There are basic configurations for measuring the ultrasonic time-of-flight in components as sketched in Figure 7.

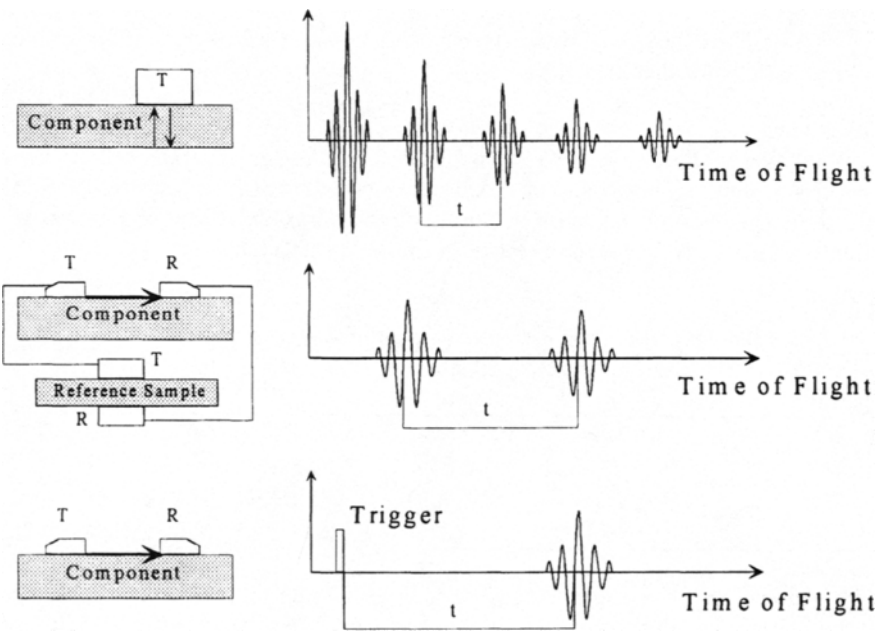


Figure 7. Ultrasonic time-of-flight measurements on components.

The upper part displays the case for evaluating bulk stresses. The time difference between the two back wall echoes corresponds to the time, the wave needs to propagate twice the thickness. Using a reference material, as sketched in the central part of Figure 7, has the advantage that e.g. temperature influences on the sound velocity can be easily discriminated. The lower part of the figure illuminates the measurement of the time between the electric trigger signal and the signal of the received ultrasonic wave.

In order to minimize measuring errors due to unstable ultrasonic coupling conditions, the use of a second receiver is recommended. The difference between the time from the trigger to the first received signal and from the trigger to the second received signal is independent of the coupling instability. In the past, considerable efforts were made in order to achieve the time-of-flight measurement with the needed accuracy. Nowadays, the updated electronics facilitate the task.

The simplest measuring setup for manual measurement consists of an ultrasonic device and a two channel oscilloscope. The ultrasonic apparatus should have a high frequency (RF) exist to allow the visualization of the high frequency ultrasonic signals on the screen of the oscilloscope. The oscilloscope should have a time measurement module with a temperature stabilized time reference enabling a resolution of the order of ns. By manual adjusting a time delay in the oscilloscope, two gated back wall echoes can be overlapped. The time delay is given in the screen of the oscilloscope.

Since higher frequencies in the short ultrasonic pulse are stronger attenuated than lower frequencies, the wave length in the succeeding echo is longer than the wave length in the preceding echo.

Hence, the most accurate time-of-flight is measured between the group of vibrations of the first signal and the group of vibrations of the second signal, yielding the group velocity of an ultrasonic pulse. This procedure can be realized by digitizing the ultrasonic echoes and applying a cross-correlation algorithm. Appropriate setups and software are available on the market. Another technique also uses the digitized form of the ultrasonic signals. The application of a Fast Fourier Transformation algorithm yields the ultrasonic amplitudes and phases in the frequency domain. A shift in the phases corresponds to a change of the time-of-flight. Since there is an ambiguity of 2π in the phase spectrum, special care must be taken.

An overview as well as a comparison of five analysis algorithms for measuring ultrasonic time-of-flight and calculating ultrasonic velocities is given in [71]. It is found that the most consistent results are provided by the overlap, cross-correlation and cepstrum algorithms.

Mostly, the time-of-flight is measured from the trigger signal to a specific part of the ultrasonic signal or from a specific part of the first to the similar specific part of the second ultrasonic signal. The possibility mentioned first is used in almost all wall-thickness gauges. The time-of-flight counter is started with the trigger signal and stopped as soon as the receiving signal reaches a predetermined amplitude threshold.

The second technique measures the time between a zero crossing of the first signal and the corresponding zero crossing of the second echo. This procedure is sufficiently unaffected by changes of the ultrasonic amplitudes. Since this technique measures the time distance between selected positions of each signal, its application cannot be recommended for time-of-flight measurements on materials with strong ultrasonic attenuation or dispersion.

Not only the ultrasonic attenuation but also the frequency dependence of the ultrasonic velocities (dispersion) cause uncertainties of the absolute time-of-flight measurements. In order to avoid such uncertainties, the application of the same ultrasonic probes with the same center frequencies for both, the evaluation of the acoustoelastic constants using the representative

sample as well as the evaluation of the stress state in the corresponding component, is the most common practice.

Ultrasonic transducers with normal incidence, as they are available on the market, are suitable for the ultrasonic stress analysis. Piezoelectric longitudinal wave transducers with center frequencies between 1 and 20 MHz and shear wave transducers with center frequencies between 0.5 and 10 MHz and diameters in the range from about 3mm to about 25mm are standard. Electromagnetic transducers for shear waves, with the advantage that no coupling medium between transducer and surface of the component is needed can be ordered from specialized manufacturers /72/. The vibration direction of the shear wave transducer is usually given by the manufacturer. It can also be determined as described in /73/.

In order to generate a surface wave (Rayleigh wave) or a skimming longitudinal wave, the normal incident longitudinal transducer is mounted onto a wedge with the corresponding angle as calculated using the Snellius law /74/. Electromagnetic transducers can be built for SH- and also for surface waves /72/.

There are different setups for measuring the time-of-flight of ultrasonic waves. Personal computers with a plugged in ultrasonic transmitter/receiver board are also available as robust analog devices suitable for in-field measurements. Since the ultrasonic techniques for the evaluation of stress states are not "just take it from the shelf and apply" techniques, the commercially available setups for evaluating stress states are optimized for specific applications. The DEBRO 30 is designed for evaluating surface stress states in components like rails and girders with a quasi one axial stress state /16,20/. Different manufacturers offer setups for evaluating strain and stress in screws and bolts. STRESS MIKE, BOLT MIKE, BOLTMASTER, BOLTGAGE and DT 500 B are the most prominent instruments for this purpose. The AUSTRA system was the first fully automated setup for evaluating the two axial surface stresses in components like rolls and rotors. The modular concept enables its application to characterize bulk stress states also /18,75/. The UER setup is built for the automated evaluation and classification of stress states in rims of monoblock railroad wheels /23/. DEBBIE is optimized for the in-field evaluation of stress states in rims of railroad wheels /76/. A different technique for measuring the time-of-flight as those mentioned above is described in /19/. A superheterodyne phase sensitive measuring system was built for the evaluation of stresses in thin plates.

The Acoustic Microscope allows the characterization of stress states in the very near surface layers with local resolutions in the range of μm . The major area of application is the characterization of stresses in electronic components and in non-metallic layers. Both the setups and the techniques are constantly being improved in terms of spatial and lateral resolutions as well as in terms of understanding and interpreting the observed images /8,9, 77,78,79,80/. The published activities deal with optimized applications for specific cases making a short description of the state of art difficult if not impossible. There are two categories of techniques to be distinguished.

One technique takes advantage of the well known velocity-strain-relationship as described above. Defocussing the microscope, the velocity of a Rayleigh wave can be determined and the surface stress state can be evaluated using the appropriate acoustoelastic constants of the part of the material propagated by the wave. The result and its accuracy depends heavily on the precision of the wave velocity and on the choice of the relevant constants. As described, the acoustoelastic constants are influenced by microstructural variations. Hence, special care must be taken if stress states in the μm -scale are calculated using the acousto-microscopic data.

The second category gathers techniques which generate interference patterns. The patterns are caused by the phases or amplitudes of two ultrasonic waves, propagating with velocities which are differently influenced by the stress state. The result is an information of the sum or difference of the principal stresses. Since the physical background of the measured effect is quite complex, a calibration using a representative sample is recommended.

The application of the acoustic microscope for characterizing or for evaluating stress states can so far only be performed by experienced operators.

4.05 Evaluation of stress states in metallic components

A broad variety of ultrasonic applications for evaluating stress states is described in the review articles and handbooks /7,8,9,10,11,73,77,81,83/. Usually, the ultrasonic stress analysis is prepared in such a way that the elastic constants are evaluated. Using these constants, the influences of any one-, two-, or three axial stress state on the ultrasonic velocities or times-of-flight are calculated. The possible influence of texture or changes of the microstructure are also taken into account. These evaluations determine which kind of ultrasonic wave or even combinations of different waves are to be applied. A-priori information concerning the stress states of the component of interest (e.g. one principal stress is negligibly small) or concerning the texture (e.g. texture is homogeneous along the measured trace) or the stress equilibrium conditions is used for simplifications of the technique and for minimization of the effort.

The ultrasonic application for evaluating stress states is not limited to metals. The acoustoelastic effect, as formulated by Hughes and Kelly /2/ in terms of the equations (10,11,12) is valid for isotropic materials with a cubic crystal symmetry only. But the same equations can be used to evaluate stress states in materials for which linear changes of the ultrasonic velocities are measured if an elastic strain or stress is applied. Furthermore, even in case of a nonlinear relation between velocities and stress state, the stress state can be characterized using the mentioned dependence for calibration. In fundamental investigations using monolithic ZrO_2 and Al_2O_3 ceramic samples, it was found that the sound velocities change linearly with the elastic strain. This effect is of the same magnitude in ZrO_2 as it is in steels. The stress or strain influence on the sound velocities was found to be much smaller in the Al_2O_3 ceramics /70/.

4.051 Determination of the principal axis of strain and stress

The basic equations describing the influence of the strain states on the ultrasonic velocities presume that the ultrasonic waves propagate and vibrate along principal axes of strain. For the ultrasonic stress analysis it is also assumed that the principal axes of strain coincide with the principal axis of stress. Hence, the determination of the principal axis of strain and stress is the first step to take for the ultrasonic application.

The use of the ultrasonic birefringence of linear polarized shear waves is straightforward for that purpose. In almost all cases of application, one principal axis is known, e.g. the thickness direction of a plate. A linear polarized shear wave, propagating the thickness is reflected at the opposite surface of the plate and a backwall echo is received. Multiple back and forth reflections yield a backwall echo sequence. Turning the probe, that is also turning the direction of vibration with respect to the two principal directions of the plane, the backwall echo

sequence shown in the upper part of Figure 8 can be seen if the direction of polarization coincides with a principal axis. In the case of coincidences of both directions, the linear polarized shear wave keeps its polarization. The amplitudes decrease due to attenuation effects. In case of no coincidence of both directions, the shear wave vibrates elliptically. The elliptical vibration can be described in terms of the superposition of two wave components, each vibrating parallel to the principal axis. The shape of the ellipse depends on the angle between the incident polarization direction and the principal axes as well as on the difference of the velocities of the components. If the angle between the incident vibration direction and the principal axes is 45° , a backwall echo sequence appears as shown in the lower part of Figure 8. The two wave components of the incident wave have not only the same frequency but also the same amplitudes. The different velocities of the two wave components cause a phase shift between the components which increases with the ultrasonic path. If this phase shift reaches $\frac{1}{2}$ ultrasonic wavelength, a destructive interference occurs.

Turning the ultrasonic probe with known polarization direction, the direction is found under which the destructive interference appears. The principal axes are under $\pm 45^\circ$ to that certain polarization direction. By adjusting the vibration direction of the incident wave parallel to each of the principle axes, the extreme values of the ultrasonic velocity or time-of-flight confirm these directions.

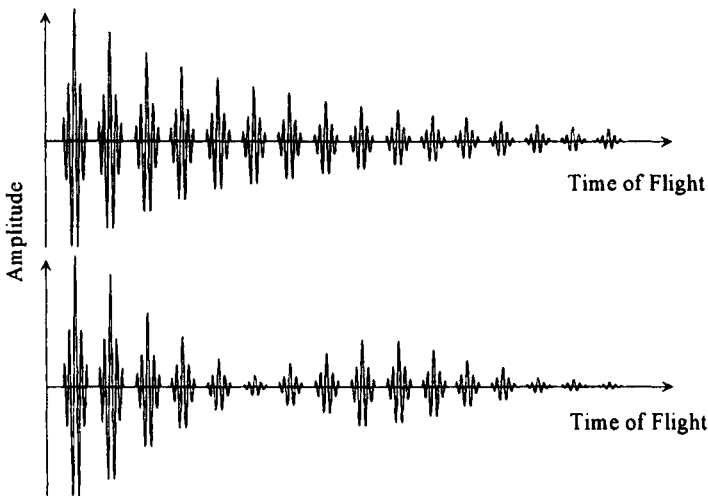


Figure 8. Ultrasonic back wall echo sequence. Vibration direction of incident shear wave parallel (top) and under 45° (bottom) to principal stress axes.

4.052 One axial stress states

In order to evaluate the strain or stress state in screws and bolts, all commercially available systems use a longitudinal wave propagating the length of the screw. The longitudinal time-of-flight is measured before and after the screw has been tightened. The simultaneous use of a radially polarized shear wave, propagating the length of the screw allows the strain and / or stress analysis also if the original length or time-of-flight in the unstrained case is not known.

The technique as well as the design of a probe for the combined use of longitudinal and shear waves are described in /82/. It is important to note that the acoustoelastic constant depends on the relationship between the tightened and the total length of the screw.

The application of skimming longitudinal wave propagating a layer of the tread of a rail can successfully be used to evaluate the longitudinal stress in new rails. In order to discriminate influences of microstructural changes along the length of the rail, the simultaneous application of a skimming longitudinal wave in the upper part of the web is recommended. The longitudinal stress is negligibly small in that part of the rail; the time-of-flight represents the value for the stress-free state. Ultrasonic results, evaluated continuously along 1 m of rail lengths are found to be in good agreement with the results of the partly destructive ring core technique, as to be seen in Figure 9. The stress state in the head of used rails cannot be regarded as one axial. Hence, other ultrasonic techniques are demanded. The state of developments is given in /24,25,26/.

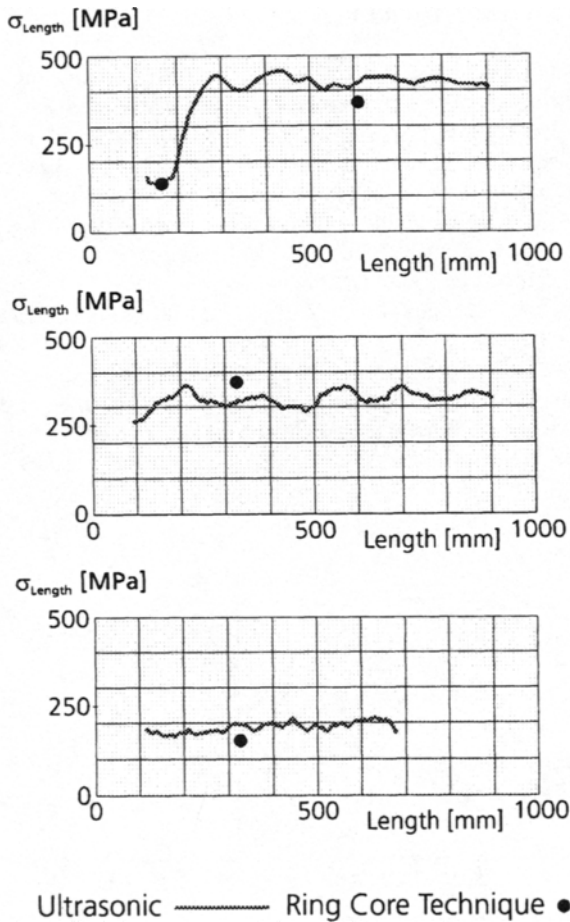


Figure 9. Longitudinal stress in the tread of new rails along the rail lengths

4.053 Surface stress states

Rayleigh waves are most often applied to evaluate surface stresses. A recent application to analyze stresses in a steel bridge is described in /84/. The advantage associated with Rayleigh waves is the possibility of generating those waves with electromagnetic transducers. The disadvantage is the sensitivity to surface conditions like roughness, paint and corrosion products, and the fact that this wave is less sensitive to stresses than e.g. a skimming longitudinal wave propagating the surface near layer. The disadvantage herewith is the need of a coupling medium. Hence, the choice depends on the conditions of application. The results of an application of a skimming longitudinal wave to evaluate the surface stresses of hardened steel rolls is chosen as Figure 10 because they also show the results of an established partly destructive technique /17/.

4.054 Two axial stress states in the bulk

The application of two linear polarized shear waves, propagating the thickness with polarization parallel to each of the two principal axes of stress yield the difference of the two stresses as a result. The advantage is that the shear modulus and the third order constant n are the only material dependent parameters needed. Furthermore, it has been found that the constant n is less effected by microstructural changes than the other higher order constants. Another advantage is the possibility of generating theses waves with electromagnetic transducers, enabling an easy manipulation along measuring traces. Successful applications include the control of the stress relief treatments of steel and Al alloy plates, the analysis of stresses in rails in the track /25/ and the evaluation of the stress state in rims of railroad wheels /21,22,23,85,86/.

In order to evaluate two axial stress states in the bulk of thin components, the application of a resonant method with an electromagnetic transducer is recommended as described in /19/.

In case of a known and constant thickness, the combined use of the two shear waves with a longitudinal wave yields the individual values of the two principal stresses.

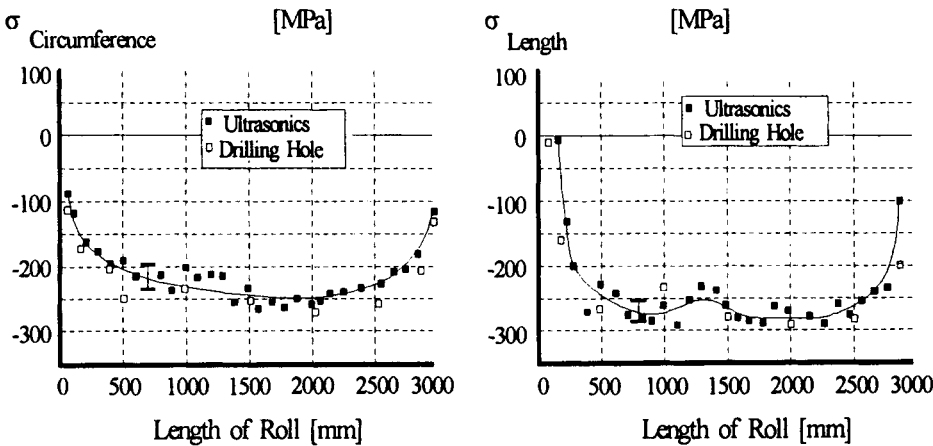


Figure 10 . Surface stress state of a hardened roll.

4.055 Three axial stress states

In case the geometry of the component allows the propagation of linear polarized shear waves along two of the principal directions, a three axial stress state can be characterized by two differences of two principal stresses. The propagation along the third axis yield the third difference of the two stresses acting along the principal directions perpendicular to the sound propagation direction.

Otherwise, only the measurement of the velocities of longitudinal and shear waves allow the quantitative evaluation of the three principal stresses. There is no publication known to the author in which the three individual stresses of a three axial bulk stress state are evaluated.

4.056 Stresses in and around weldments

The quantitative evaluation of welding stresses using ultrasonic techniques is only possible if the acoustoelastic constants are known for the material in the weld seam, in the heat affected zone and in the base material. If these constants were evaluated using representative samples, good agreements with results of established partly destructive techniques can be achieved as Figure 11 illustrates /87/.

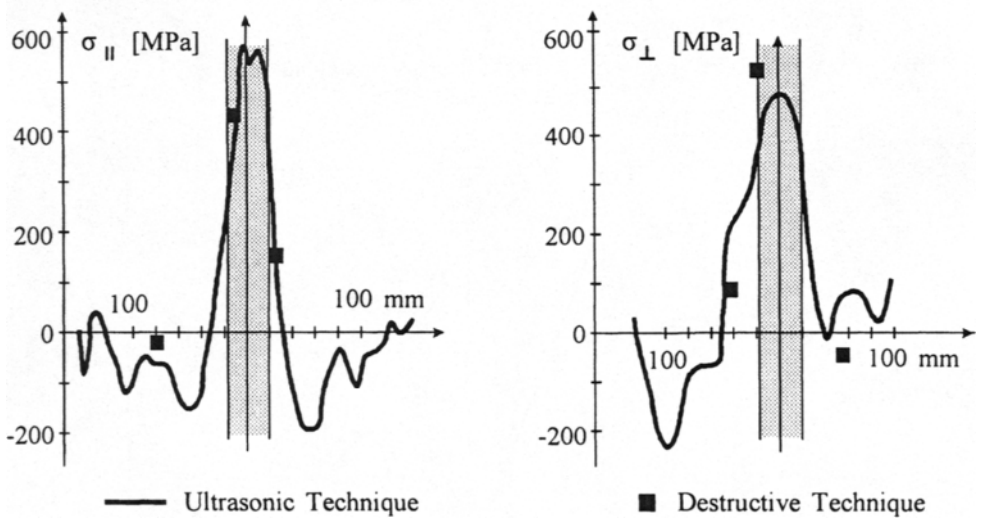


Figure 11. Stresses parallel ($\parallel$) and perpendicular ($\perp$) to a multilayered ferritic weld seam.

Knowing the appropriate acoustoelastic constants and taking advantage of locus continuous measurements of the ultrasonic time-of-flight, the stress states in and around weldments can be mapped. Figure 12 shows the stress parallel to the seam $\sigma_{||}$ in a welded Al-alloy plate with 4 mm of thickness /88/. The results along two traces are displayed in Figure 13.

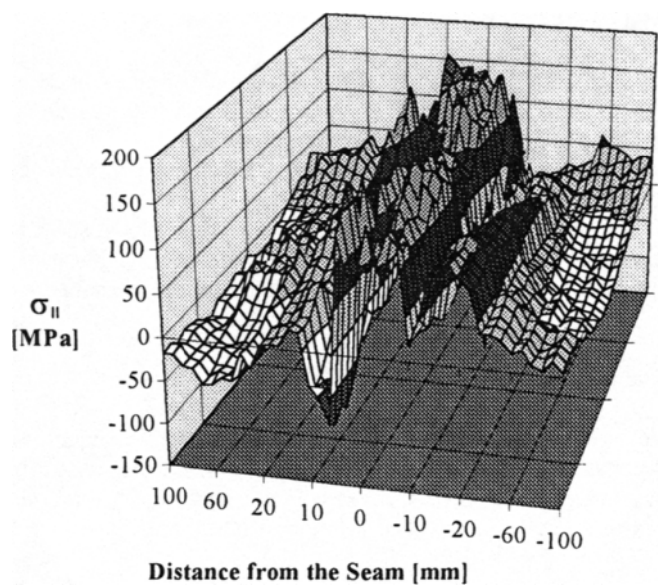


Figure 12. Map of the stress parallel to the weld seam in an aluminium sheet.

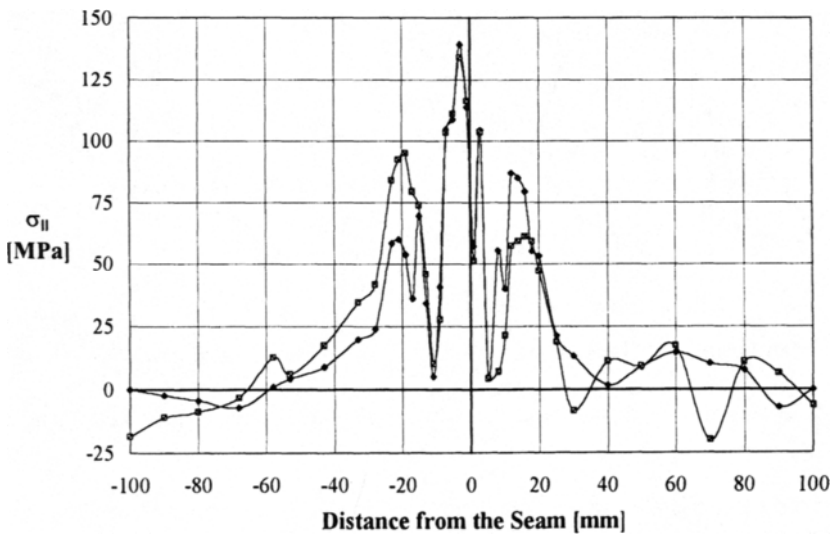


Figure 13. Stress parallel to the weld seam along two traces across the seam.

Comparisons of the stresses evaluated using skimming longitudinal and Rayleigh waves are given in /89/. The experiments are performed on a welded stainless steel pipe and a welded Al-Mg alloy plate. The application of a skimming longitudinal wave and intensive investigations using linear polarized shear waves to evaluate stress states in welded steel plates are reported in /90/ and /33/, respectively.

Since cracks in welded components are often found in the area of the heat affected zone and base material, the stress state in that zone is of particular interest. Using the acoustoelastic constants of the base material, the ultrasonic application has been proven to be a reliable tool for evaluating the surface and bulk stress states and for controlling the post welding heat treatment.

4.057 Evaluation of stress states in components with orthotropic texture

There is no procedure which can be generally applied for that purpose. Depending on the stress state to be evaluated, on the strength of the texture and on the geometry of the component, the appropriate approach has to be chosen and, if needed, adapted.

Since the influence of texture on the ultrasonic shear wave velocity remains the same if the directions of wave propagation and vibration are interchanged (equations 30,31,32), the texture independent equation is derived:

$$\frac{v_{ij} - v_{ji}}{v_{ji}} = \frac{t_{ji} - t_{ij}}{t_{ij}} = (\sigma_i - \sigma_j) / 2\mu \quad (45)$$

The application of the equation presumes that the principal axes of stress coincide with the symmetry axes of the texture. If Θ is the angle between the rolling and the propagation direction and Ψ the angle between the rolling direction and the direction of the principal stress σ_1 , the application of SH-waves allows the evaluation of the difference of the in plane stresses /91,92/.

$$(V_{SH}(\Theta) - V_{SH}(\Theta+90^\circ)) / V_T = (\sigma_1 - \sigma_2) \cos 2(\Psi - \Theta) / 2\mu \quad (46)$$

Experimental and theoretical results agree very well as shown in /8/.

The approach described in /93/ is also independent of texture. The principal axes of stress are assumed to be parallel to the texture symmetry directions. A SH wave is applied which propagates a plate not normally but under the angle φ to the surface normal. The wave propagates the thickness of the plate, is reflected at the rear side of the plate and received by a second transducer at the surface. Transmitter and receiver are adjusted parallel to the direction of the principal stress σ_1 ; the velocity V_{SH1} is determined. The velocity V_{SH2} is measured after the sensors are aligned parallel to the directions of stress σ_2 . The evaluation equation is

$$\frac{V_{SH1} - V_{SH2}}{V_{SH}} \Big|_{\varphi} = (\sigma_2 - \sigma_1) \cdot F_1(\varphi) + F_2 \cdot \cos^2 \varphi \quad (47)$$

V_{SH} is the SH velocity in the stress free case, F_1 is a function of second and third order elastic constants and F_2 is a second order constant. Measurements at two angles of φ are made

to evaluate the two unknowns F_2 and $(\sigma_2 - \sigma_1) \cdot F_1(\varphi)$. Knowing $F_1(\varphi)$ for the two angles φ by preceeding calibration experiments, the difference of the principal stresses can be evaluated.

Applying an approach given in /92,94/, a similar experimental effort yields an equation for $\sigma_2 - \sigma_1$ which is independent of the elastic and acoustoelastic constants.

The combined use of longitudinal and shear waves propagating along the same principal directions results also in a relationship (equation 38) which is independent of texture. The application allows the evaluation of the sum of the two in plain stresses. Other combinations, as given in the equations 29,36,37 are useful only if the texture is characterized by one of the expansion coefficients. The same restriction holds for the approach described in /95/.

If the texture can be assumed as homogeneous, the equilibrium conditions of the stress state can be used to evaluate the expansion coefficients and to separate the texture influence on the ultrasonic velocities. The application is restricted to components with a simple geometry and a dense coverage with measuring positions.

A further possibility of separating the effects of texture from those of stress uses the ultrasonic scattering at grain boundaries between grains with different orientations. Hence, the ultrasonic birefringence (equations 17,18,19) is a function of the ultrasonic frequency if it is caused by texture and is independent of frequency if caused by stress. The technique, described in /96/, presumes a known relationship between two constants, containing the single crystal elastic constants and the expansion coefficients of the fourth and sixth order. Numerical calculations using the data of cold rolled α -brass and cold rolled recrystallized steel samples show a linear correlation between the two mentioned constants. Experimental investigations on other cold rolled steel samples confirm the correlation /97/. This approach has the advantage that the actual degree of texture is taken into account.

All other published approaches known to the author presume a slight anisotropy in order to simplify the possibilities described above.

4.058 Evaluation of dynamic stresses

Although there is no systematic investigation or application known to the author, it is pointed out that ultrasonic techniques render possible the evaluation of changing stress states. Updated measuring techniques take up to 200 measurements per second. The mechanical strength and electrical stability of both, piezoelectric and electromagnetic probes can be designed to meet the requirements for a long term application in rough environments.

4.059 Resolution and accuracy

The result of the ultrasonic stress analysis is always a mean value of the stress influencing the velocity of the applied ultrasonic wave while it is propagating a section of the component. Hence, the resolution depends on the sound beam geometry given by the probe diameter, the ultrasonic wavelength and the path length. The smallest diameter of probes, still easy to handle, is about 6 mm, the diameters of the defocused ultrasonic beam in acoustic microscopes is in the range of 10 μ m.

Wavelengths cover a range from typically 6 mm to about 0.3 mm and may be as small as about 3 μ m if a 1GHz acoustic microscope is used.

The path length is either the transmitter / receiver distance in case of the surface stress analysis or the thickness of the component or an even multiple of the thickness, depending on the used time-of-flight measuring technique. In case of acoustic microscopy, the path length is about the diameter of the defocused beam.

Often the local resolution is regarded as increased by measuring along a trace with step widths smaller than the probe diameter and by taking the measured value to the coordinate of the central point of the probe. In doing so, the local resolution depends on the used scanning system.

The accuracy in its true meaning depends on a variety of parameters. Here, the comment is limited to the reproducibility of a measurement.

The measurements of ultrasonic time-of-flight can be made reproduceable within ± 1 in 10^{-4} . The reproducibility of AEC values is in the range of $\pm 3-5\%$ and the evaluated stress values are reproduceable within the same range. Reproduceable measurements performed at other positions of the (quasi) homogeneous component may scatter. Hence, the appropriate averaging technique should be applied.

There are two aspects to be considered with respect to the result of the ultrasonic stress analysis: One is the inaccuracy associated with the stress free reference, the other is the inaccuracy associated with the AEC's evaluated using a representative sample. The accuracy of the stress value is usually about 15 - 20 MPa and $\pm 10\%$.

Comparing ultrasonic results with those of the established ring core or hole drilling techniques, agreements within the error bars of both techniques are found. The results of an extensive round robin test of destructive and nondestructive techniques are given in /98/.

4.06 Recommendations

Complementary to the established nondestructive x-ray diffraction and to the partly destructive hole drilling and ring core techniques, which are the most frequently applied techniques, the ultrasonic techniques permit the evaluation of surface and bulk stress states of components. The major advantages of the ultrasonic techniques are the fast data acquisition, the possibility of locus or time continuous measurements and the low costs per measuring point. However, in order to get quantitative stress values, the ultrasonic techniques require evaluations of the elastic properties which are not needed for the application of the established techniques. Hence, it is important to precisely know the task of the stress state analysis.

In some cases, it is sufficient to measure the ultrasonic time-of-flight along traces before and after a specific treatment of the component. The homogeneity of the stress distribution or the stress relief after thermal or mechanical treatments can be tested that way. The decrease of time-of-flight can be interpreted as decrease of tensile or increase of compressive stresses, the change of stress can be approximated using the relative change of time and the appropriate acoustoelastic constants given in earlier paragraphs.

If quantitative results are requested, the evaluation of the material dependent acoustoelastic constants is needed. Since the inaccuracy of the final result is dominated by the inaccuracy associated with the acoustoelastic constants, it is strongly recommended to use samples which are as representative as possible for the component to be stress analyzed. The use of the same ultrasonic sensors for both, the evaluation of the needed acoustoelastic constants and the evaluation of the stress state in a component also reduces the inaccuracy.

Since the ultrasonic sensors are aging and hence change their properties, it is important to have a reference sample with constant properties. Measuring the time-of-flight of ultrasonic waves propagating the sample, the conditions of the ultrasonic sensors should be documented. A similar procedure is recommended to take different temperatures of the component into account.

The general procedure of the ultrasonic stress analysis as well as the different techniques to evaluate the stress states in components is given in chapter 4.05. The described cases can be regarded as examples for a beneficial application of ultrasonic techniques.

On the other hand, the evaluation of stresses in monolithic ceramics, except ZrO_2 , or in ceramic heat protection layers and the stress evaluation in hardened surfaces with hardness and stress gradients are certainly not cases for a successful ultrasonic application. Beside the established techniques, the micromagnetic techniques are recommended if the material is ferromagnetic.

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5. Micromagnetic techniques

W. A. Theiner

5.01 Historical review

The interaction of stress fields with ferromagnetic materials can be recognised in macroscopic ferromagnetic parameters as well as in their micromagnetic behaviour. The changes in macroscopic ferromagnetic parameters as a result of stresses are studied in measuring e.g. hysteresis curves, anhysteresis, susceptibilities, permeability, coercivities, remanence and magnetostrictive parameters /1-3/.

If a ferrous material is magnetostrictive the hysteresis curve becomes sheared under the influence of stress fields: if the material is stressed, it will change its size in characteristic dependency of the magnetic field strength. From these ferrous solid state properties different stress sensitive parameters can be derived if a full magnetisation reversal is recorded. Many basic research and development studies have been performed since the beginning of this century. Beside the better understanding of why materials become ferromagnetic the discovery of new ferrous materials led very soon to early industrial applications: permanent magnets, soft magnetic materials with small loss factors, magnetostrictive filters, magnetic resonance circuits, electromagnetic transducers, magnetostrictive units for actuators and mechanical damping devices.

With the ongoing understanding of ferromagnetic properties /1,2,4,5,6/ - especially micromagnetic theory - one becomes able to explain interactions between lattice imperfections and internal stresses with Bloch-walls. Although most of these studies have been performed on single crystals and poor polycrystalline materials, results describing macroscopic magnetic parameters as well as microscopic remagnetisation processes up to now in the best quantitative approach. Since that time micromagnetism has been the most successful tool to design new magnetic nondestructive techniques. Since the 1950's and 60's when the theoretical foundations of micromagnetism were laid, these nondestructive methods for residual stress evaluation have been derived from irreversible Bloch-wall movements, reversible magnetisation processes, from magnetostrictive active Bloch-walls (BW) and from rotation processes (RP) occurring in the higher magnetic field range. Theoretical works have also shown that the understanding of magnetic anisotropy - crystal, elastic, magnetostrictive - in the micromagnetic description was helpful for the development of stress dependant nondestructive techniques as well as for stress and microstructure dependent micromagnetic parameters.

Most micromagnetic nondestructive techniques started in the sixties. The evaluation of published works as registered in data banks of mechanical engineering, applied physics and nondestructive testing shows that since 1985 macroscopic magnetic parameters (hysteresis in the set-up technique, eddy current, time-of-flight measurements influenced by ΔE effect, ...),

irreversible micromagnetic (magnetic Barkhausen, acoustic Barkhausen, incremental permeability, mechanical excited Barkhausen, dynamic magnetostriction) and reversible micromagnetic parameters (reversible permeability, susceptibility and dynamic magnetostriction) have been used to measure stresses in ferrous materials.

In these two decades devices for measuring residual stresses were built up. Barkhausen effect units, devices using dynamic magnetostriction and incremental permeability set-ups are in use. Developments since 1980 use the multi-parameter micromagnetic NDE-approach. This technique enables unambiguous stress measurements to be carried out even in disturbed situations. Disturbance means that additional parameters as microstructure, texture and gradients are influencing micromagnetic stress measurements. Therefore it is not surprising that micromagnetic techniques have become increasingly dominant for stress measurements. This can be deduced from published papers between 1976 - 1996.

64 % of published papers have used micromagnetic parameters and 36 % macromagnetic and eddy current techniques (Figure 1). The most frequently investigated and applied micromagnetic techniques are derived from Barkhausen signal parameters (Figure 2). Further assessments can be deduced from the state of the art of used methods: Is ND method in industrial use? Are prototypes available? Is the technique able to perform quantitative stress measurements? Can the method evaluate stresses in real time? Influence different microstructure states quantitative stress measurements? Ability of the system to measure different parameters simultaneously? Analysis shows that most micromagnetic stress measurements use one measuring parameter and only a minority of published works are using multiparameter micromagnetic strategies. Many papers are concerned with basic research: in these papers mechanical, X-ray, neutron diffraction and further physical methods are used to assess sensitivity of micromagnetic methods; to calibrate parameters with well known techniques, e.g. x-ray; to measure different stress types; to evaluate stress-gradients; etc. (Figure 3).

The developments of the last 20 years have shown that many micromagnetic stress measurements have been made in R&D laboratories and only few industrial applications have become known. The main reason for this „technology gap“ are influencing factors and interference signals which in many cases under real-testing situations prevent quantitative ND stress measurements. Greatest demands on ND is set however in the case of real time measurements in production lines. Therefore new nondestructive stress testing units using a set of testing parameters which can be used for appropriate computer codes for getting unambiguous stress results. These units are tailored to the needs of customers and using *Nondestructive Intelligent Stress Testing* (NIST) probes as well as NIST hard- and software devices.

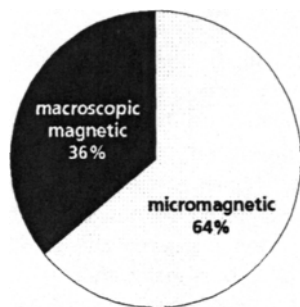


Figure 1. Percentage of papers in which micromagnetic and macroscopic magnetic parameters are used for stress and residual stress measurements within the period between 1976 and 1996

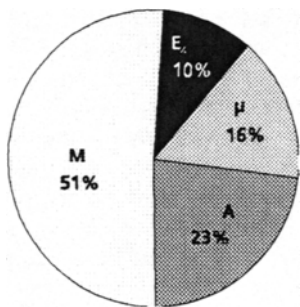


Figure 2. Micromagnetic stress measurements published between 1976 and 1996. E: dynamic magnetostriction, μ : permeability / incremental permeability, A: acoustic Barkhausen noise, M: magnetic Barkhausen noise.

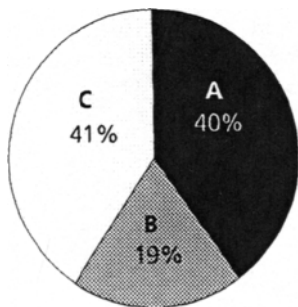


Figure 3. Nondestructive stress measurements within the period between 1976 and 1996 which are using: A: one micromagnetic parameter, B: multiparameter micromagnetic methods, C: different methods like acoustic, magnetic and other techniques.

5.02 Symbols and abbreviations

BW	Bloch-wall
BW1	Bloch-wall of 1 st kind
BW2	Bloch-wall of 2 nd kind
RPrev	Reversible rotation process
RPirr	Irreversible rotation process
E	Amplitude of excited magnetostrictive electromagnetic ultrasonic mode
EC	Eddy Current
H	Magnetic field strength
H_{Δ}	Amplitude of alternating magnetic field
ΔH_M	Width of MB amplitude curve versus H at x% of maximum
H_t	Tangential magnetic field strength
H_E	Exciting magnetic field strength of yoke
H_C	Coercivity
H_{CM}	Coercivity derived from H-position of M_{MAX}
$H_{C\mu}$	Coercivity derived from H-position of $\mu_{\Delta MAX}$
H_{CO}	Coercivity, derived from upper harmonics analysis of H_t
J	Magnetic polarisation
K	Distortion factor, derived from upper harmonics analysis of H_t
MB	Magnetic Barkhausen noise
AB	Acoustic Barkhausen noise
M_{MAX}	Maximal amplitude of rectified Barkhausen signal
f_A	Analysing frequency of Barkhausen signal
f_E	Magnetic exciting frequency
f_{Δ}	Exciting frequency of incremental permeability
RS I, II, III	Residual stresses of first, second and third kind
ND	Nondestructive
NDE	Nondestructive evaluation
$r_M, \Delta r$	Anisotropy parameter
$\lambda(H)$	Magnetostriction as function of magnetic field
λ_L	Longitudinal magnetostriction
$\mu_{\Delta}(H)$	Incremental permeability as function of magnetic field strength
$\mu_{\Delta MAX}$	Maximal value of $\mu_{\Delta}(H)$
ΔH_{μ}	Broadening of $\mu_{\Delta}(H)$ -curve at a fixed μ_{Δ} -value
σ_Y	Yield strength
σ_+	Tensile stress
σ_-	Compressive stress
σ_{ND}	Nondestructively determined residual stress values

5.03 Physical fundamentals

5.031 Interactions of stress states with micromagnetic parameters

Micromagnetic theory explains the existence of Bloch-walls; describes why ferrous materials have a domain structure; describes the interaction of Bloch-walls with lattice imperfections and internal stresses. The magnetostrictive and electromagnetic interactions between Bloch-walls and the interactions between regions where local disturbances of the magnetic lattice properties are sources for electrostatic and stress fields. For nondestructive micromagnetic stress measurements the different kinds of BW's are the most important „intrinsic sensor“. As defined by [4]:

Bloch-walls of the 1st kind (BW1) are walls which separate magnetic domains in which the magnetostrictive residual stresses do not vanish in adjoining domains (Figure 4). Therefore these BW1-type, in iron e.g. all (100)-90° BW's, have great interacting volumes. Whenever one BW1 type is moved the elastic energy density will be changed in dependence of their local position. This means that BW1 can only be moved from the actual position if local magnetic field strength or elastic energy density will be changed by external energy supply and that macrostresses will interact with this Bloch-wall type in a direct manner.

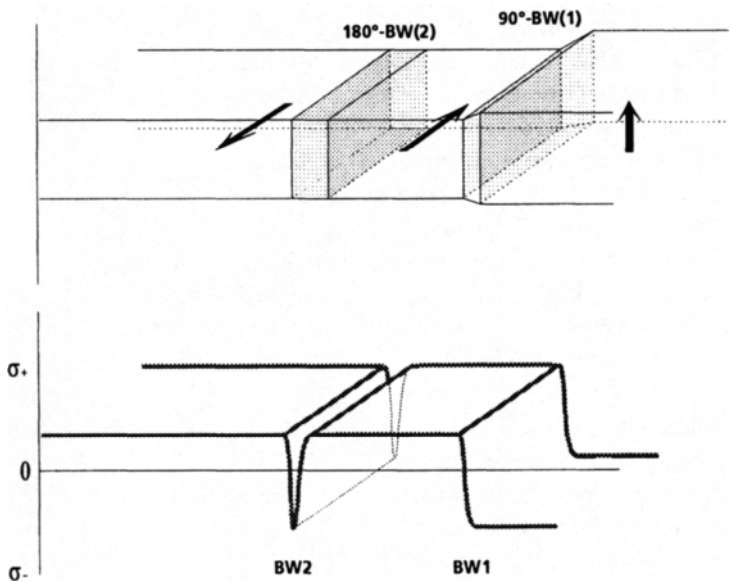


Figure 4. Magnetostrictive stresses caused by BW1 and BW2 Bloch-walls in adjoining domains.

Bloch-walls of the 2nd kind (BW2) only produce residual stresses within the Bloch-wall itself. In iron all 180° BW's and the (110)-90° walls are of BW2 type. Because BW2's separate areas with the same magnetostrictive behaviour no elastic energy will be changed during their

movement. BW2 do not interact with macrostresses (RSI) and microstresses e.g. RSII stresses within grains which are larger than BW2 dimension. These walls only sense steep stress gradients in the dimension of the Bloch-wall thickness as produced e.g. by dislocations /1-4/ or in an indirect way, by electromagnetic and magnetostrictive interactions between different volume areas and BW's to minimise electromagnetic and elastic energy density /5,6/.

Beside BW1 all rotation processes (RP) will change the magnetoelastic energy density and therefore all these processes are stress sensitive and. magnetostrictive active.

Rieder's classification of Bloch-wall types is the key for the development of stress sensitive and nondestructive testing methods. Together with the micromagnetic theory of magnetic demagnetisation processes one can derive different micromagnetic nondestructive parameters which are different from their principal reaction upon stresses and microstructure parameters.

5.032 Nondestructive micromagnetic parameters

ND parameters can be derived from macroscopic magnetic and macroscopic magnetostrictive parameters. In this case ferrous material will be described as a continuum by which volume areas much greater than domain dimension contribute to the measured magnetic quantity. These values are e.g. the magnetisation J , the magnetic flux density B or the longitudinal magnetostriction λ_L . Table 1. Quantitative results can only be obtained if test specimens have a defined geometry and specimen is excited by a constant magnetic field strength over the whole cross section. Because of these restrictions these tests are mainly performed in R&D laboratories and not in field applications.

Stresses which are measured with sensors in the set-up technique are derived from reversible (rev), irreversible (irr) and rotation (RP) processes. Most of these techniques are using electromagnetic transducers for magnetic field excitation and signal detection. If different micromagnetic processes determine ND parameters these quantities record independent physical information about ferrous state and offer therefore the possibility for designing multiparameter micromagnetic probe systems Table 2.

Table 1

Macroscopic ND methods which can be applied to test specimens with defined geometry. Techniques are distinguished with regard to magnetisation and micromagnetic processes.

ND method	magnetisation process	micromagnetic process	sensor
magnetisation	rev	BW1	electromagnetic
	irr	BW2, RP	
magnetic flux	rev	BW1	electromagnetic
	irr	BW2 RP	
magnetostriction	rev	BW1	electric
	irr	RP	optic

Table 2

ND methods which are used in the set up technique. Techniques are distinguished with regard to magnetisation and micromagnetic processes.

ND method	Magnetisation process	Micromagnetic process	Sensor
Magnetic Barkhausen signal	irr	BW1 BW2 RPirr	electromagnetic
Acoustic Barkhausen noise	irr	BW1 RPirr	acoustic
Eddy current	rev irr	BW1 BW2	electric electromagnetic
Incremental permeability	rev irr	BW1 BW2 RP RPirr	electric electromagnetic
Time signal of tangential magnetic field strength	rev irr	BW1 BW2 RP RPirr	electromagnetic
Dynamic magnetostriction	rev irr	BW1 RP RPirr	acoustic electromagnetic

These different physical properties as described in Table 2 allow:

- the design of multiparameter micromagnetic devices;
- to measure different nondestructive targets simultaneously;
- to distinguish between different influencing parameters to suppress disturbances.

The last point is in fact the most important one in determining whether nondestructive stress techniques will be successful in industrial applications or not.

Which reversible and irreversible nondestructive parameters can be derived from micromagnetic properties? Micromagnetic theory and micromagnetic phase theory predict that BW1, BW2 and RP are contributing at different magnetic field strengths to the magnetisation process. In polycrystalline ferrous materials BW2 contribute to remagnetisation in a dominant

way around coercivity field strength H_C . Outside of this field region BW1 becomes more and more dominant. If magnetisation becomes saturated BW density decrease and further remagnetisation processes take place by RP's Figure 5. Because of this micromagnetic rules interactions with microstructure parameters can be recorded with one set of micromagnetic quantities whereas stresses are measured by values mainly determined by BW1 and RP. In polycrystalline materials reversible and irreversible BW2 demagnetisation processes become largest around H_C . This can be verified by differential and reversible permeability measurements recorded over the tangential magnetic field strength H_t /1,2/. This behaviour can be used to determine coercivity values by the H-field position of the maximum whenever BW2-type is involved (see Table 2). Outside of the H_C field region BW1 and RP contribute as described above to magnetisation. For getting all this different information the most important of the measuring variables is the appropriate chosen amount of the exciting magnetic field strength H_E .

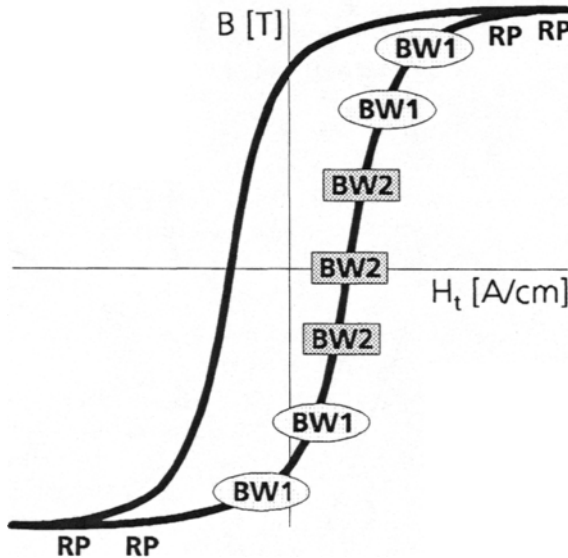


Figure 5. Magnetic field depended remagnetisation processes by Bloch-wall type 1 (BW1) and 2 (BW2) and rotation processes (RP). BW1: stress sensitive, BW2: no interaction with macrostresses, RP: stress sensitive.

If magnetoelastic or magnetostrictive ND parameters are measured the H_C -region is marked out by minimum or maximum values. This can be seen from the longitudinal magnetostriction plot. Because only BW1 and RP contribute to elongation and contraction of ferrous materials the minimum at $d\lambda/dH=0$ indicates for polycrystalline stress free materials the H_C -value.

Table 3
Micromagnetic parameters which can be used for stress measurements, measuring variables and influencing factors.

ND method	ND parameters	Measuring variables and influencing factors
Magnetic Barkhausen signal (MB)	MB events versus time rectified MB amplitude versus H rectified MB power spectrum of MB pulse high distribution of MB events MB recorded versus exciting current of yoke coercivity H_{CM} derived from maximum of rectified MB signal MB recorded versus exciting current of yoke width of MB amplitude curve versus H at x(%) of maximum ΔH_M	frequency of yoke excitation f_E frequency range in which MB signals are measured f_A amplitude of exciting field strength of yoke H_E <i>lift off of sensor</i> <i>design and electrical properties of probe system</i> <i>properties of electronic units (filter, amplifier, signal to noise ratio)</i>
Acoustic Barkhausen signal (AB)	AB events versus time rectified AB versus H frequency spectrum of AB	frequency of yoke excitation f_E frequency range in which AB signals are measured f_A amplitude of exciting field strength H_E <i>design and electrical properties of probe system</i> <i>properties of electronic units (filter, amplifier, signal to noise ratio)</i> <i>coupling of acoustic transducer</i>
Eddy current (EC)	impedance value versus time real and imaginary impedance values	frequency range in which EC signals are measured f_A amplitude of HF-exciting field strength H_A

Table 3 (continued)

ND method	ND parameters	Measuring variables and influencing factors
Eddy current (continued)	Phase of signals	<i>lift off of sensor</i> <i>design and electrical</i> <i>properties of probe system</i> <i>properties of electronic units</i>
Incremental permeability (μ_Δ)	impedance value versus time impedance value versus magnetic field strength width of incremental permeability curve versus H at $x(\%)$ of maximum real and imaginary impedance values Phase of signals coercitivity $H_{C\mu}$ derived from maximum of impedance signal	frequency of yoke excitation f_E frequency range in which EC signals are measured f_A amplitude of exciting field strength of yoke H_E amplitude of HF-exciting field strength H_Δ <i>lift off of sensor</i> <i>design and electrical</i> <i>properties of probe system</i> <i>properties of electronic units</i>
Time signal of tangential magnetic field strength (H_t -T)	coercivity distortion factor amplitude of upper harmonics phase of upper harmonics	frequency of yoke excitation f_E amplitude of exciting field strength of yoke H_E <i>lift off of sensor</i> <i>design of probe system</i>
Dynamic magnetostriction (E)	EMAT-amplitude versus magnetic field strength H_t EMAT-phase versus magnetic field strength H_t coercivity H_{CE} derived from minimum of EMAT- amplitude versus magnetic field strength H_t	frequency of yoke excitation f_E frequency range in which EMAT signals are measured f_E amplitude of exciting field strength of yoke H_E amplitude of HF-exciting field strength H_Δ <i>lift off of transducer</i> <i>design and electrical</i> <i>properties of probe system</i> <i>properties of electronic units</i>

5.033 Stress dependency of ND parameters

Quantitative micromagnetic stress measurements are influenced by different parameters. In this chapter most important influencing factors as principal stresses, microstresses, temperature and microstructure are discussed briefly whereas the influence on ND stress measurements by microstructure gradients, texture and different ferrous materials are not further taken into account.

Principal stresses

All direct to stress related ND parameters use BW1 and RP magnetisation processes. Macroscopic stress fields interact on BW1 and RP in such a way that BW1 movements take place to reduce elastic energy density. By this process, the mean averaged orientation of magnetic domains is aligned parallel or perpendicular to the direction of the acting stress field. For ND stress measurements one has to recognise that the probabilities for magnetisation directions parallel and antiparallel to the σ -direction are occupied with the same probability [1-4]. This means that demagnetised ferrous materials remain in this state and therefore no change in e.g. longitudinal magnetostriction or magnetisation parameters will be observed in a macroscopic scale. Whenever magnetic fields are superimposed in σ -field direction this direction becomes a preferred magnetisation direction, if material to be tested has a positive magnetostriction. In this case strains parallel to the exciting H-field will support remagnetisation processes and will become a magnetic „easy“ direction; if compression stresses are acting in H-field direction one needs greater magnetic-field strengths to achieve e.g. the same magnetic flux value as for tensile case; direction becomes a „poor“ magnetisation direction (Figure 6). This behaviour can be observed for all macroscopic and micromagnetic parameters being used for stress measurements in a more or less dominant way (Figure 7). This stress induced magnetic anisotropy is used to determine the axis of principal stresses. For this procedure only the exciting magnetic field direction H_E must be rotated during the measurement of stress related parameters. From the minimum-maximum figure of recorded signals the principal axis can be determined immediately.

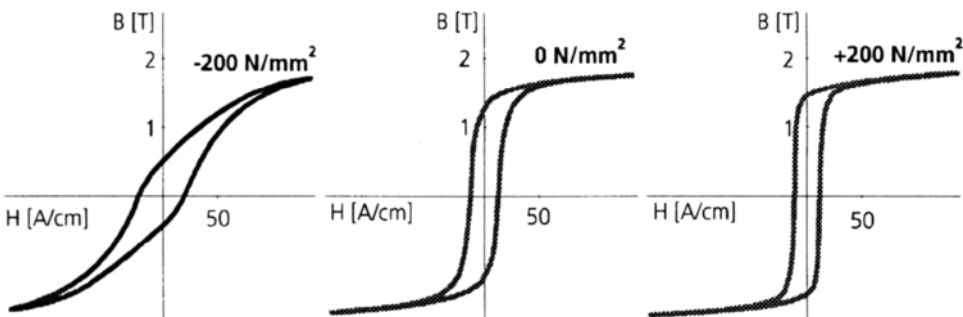


Figure 6. Shape of hysteresis of steel for different stress levels.

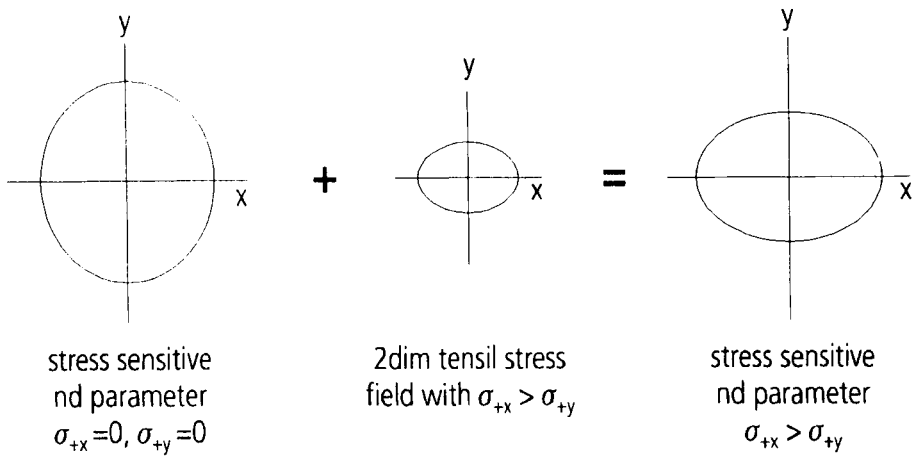


Figure 7. Determination of principal stress axis by micromagnetic parameters

Macrostress

The distance between BWs measured in steel grades is between approximately 0.5 and 5 μm ; grains have dimensions in the range of 2 and 200 μm . If macrostresses are applied on a body BWs within grains will see only the stress field which acts in this single crystal. This means that only microstress-states are responsible for the BW arrangement. Macrostress will influence the domain arrangement in such a way that averaged values of microstresses get field character. Therefore macrostresses can only be measured by micromagnetic parameters in accordance with the field character of stress fields and appropriate dimensions of the sensor.

Non-linear properties of ferrous materials

A difficulty for getting unambiguous stress values remains from non-linear magnetostrictive properties. Polycrystalline iron has in the low magnetic field region, H-field strength smaller than approximately 30 A/cm, a positive longitudinal magnetostriction which is caused by (100)-90° BW's. At greater field strength values magnetostriction becomes negative because of the dominant contribution of (110)-90° and (111)-90° BW's.

From this one can see that different kinds of stress-supported micromagnetic processes take place in dependence of H-field strength (Figure 8). This must be kept in mind when stress measurements are performed. The non-linear behaviour can be seen from micromagnetic parameters if they are recorded versus the magnetic field strength. This property of the ferrous state is the reason why one will obtain ambiguous results in nearly all applications whenever only one quantity is used.

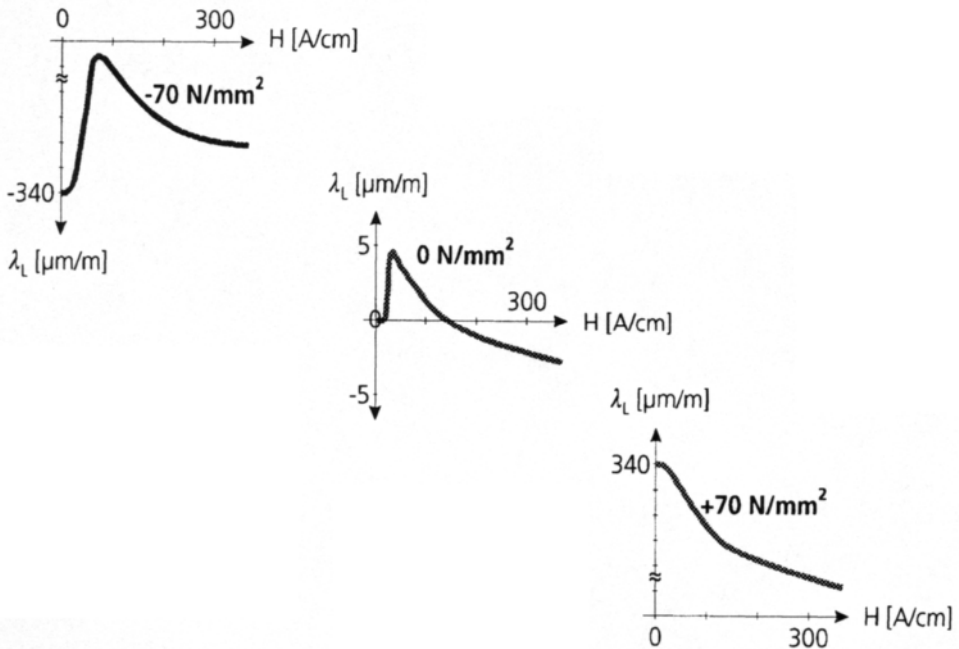


Figure 8. Shape of longitudinal magnetostriction versus magnetic excitation for different stress levels.

Microstructure influences

An important point which must be recognised for micromagnetic stress measurements is the influence of microstructure on micromagnetic ND properties [1,2,3]. In hard materials, e.g. martensitic steels, BW1 type can hardly be moved by stresses because high defect densities acting as strong pinning centres and high stress levels within subgrains decrease the BW1 density. Remagnetisation processes can only take place over a large range of H-field. Many ND parameters became therefore more linear. This can be seen from the time signal of the flux density, the tangential field strength and from longitudinal magnetostriction measurements (Figure 9). In contrast to magnetic hard materials annealed ferritic steels with coarse grains and a small defect density (precipitation, dislocations, second phases, dissolved atoms) are very stress sensitive. The BW rearrangement induced by stresses can easily be performed at low energy input and hence for low stresses. All ND parameters show non-linear behaviour versus stresses and magnetic field strength.

Temperature influence

If measurements are performed at different temperatures one has to recognise that the Bloch-wall orientation and hence local magnetisation is mainly determined by crystal anisotropy K . This anisotropy is temperature dependant and energy density decreases with increasing temperatures [1-4]. This means that stress anisotropy becomes more and more dominant and ND stress measurements more sensitive at elevated temperatures.

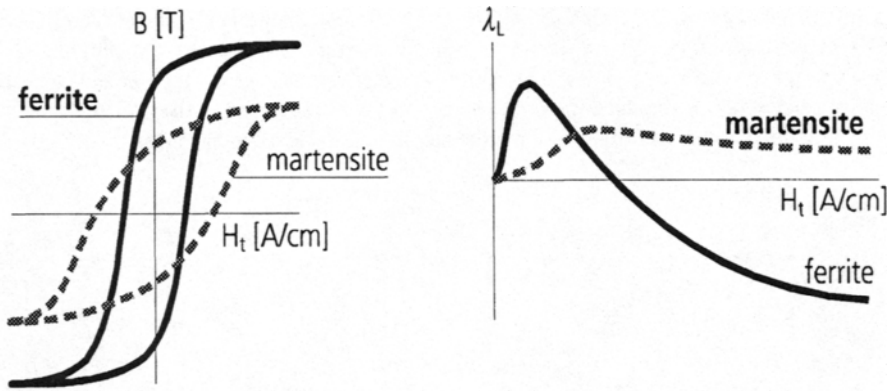


Figure 9. Soft and hard magnetic materials which show different sensitivity if stress levels are changed.

5.04 Micromagnetic residual stress measurements

5.041 Testing units and sensors

As outlined in chapter 5.1 most electromagnetic stress tests are using one ND parameter respectively different methods and matched sensors (Figure 3); only a minority of application are using the multiparameter approach with one sensor. All units have their own characteristics concerning the signal to noise ratio, the frequency range where signals are detected, the filter characteristics etc. (Table 3). The design of sensors is matched to the needs of the customers. In the normal case the design is restricted to the capability of the laboratory which has manufactured the device. By this situation and by the main difficulties to build up sensors with equal electromagnetic characteristics no standards or codes could be developed up to now for nondestructive electromagnetic residual stress tests.

5.042 Calibration procedures

The calibration of micromagnetic parameters (Table 2) can be performed using either tensile or bending tests; x-ray / neutron diffraction methods can be used, if calibration must be performed on the component itself. Combinations of different nondestructive methods, like ultrasonic and micromagnetic techniques, can also be useful in different disturbed applications because of the different information content of each method. If microstructure states and residual stresses are superimposed one has to use at least two independent ND parameters to avoid ambiguous results.

One procedure using Barkhausen signals for residual stress measurements is shown in Figure 10. Both parameters M_{MAX} and H_{CH} are measured in the set up technique. M_{MAX} - the maximum value of the rectified Barkhausen signal shows a non-linear dependency upon stresses for the annealed state (A) and a poor more linear reaction for the hard martensitic

states (B). This behaviour is always observed ferromagnetic polycrystalline materials with positive magnetostriction. The coercivity H_{CM} shows the same stress dependency as the macroscopic coercivity value H_C evaluated from hysteresis measurements. Namely, a more or less linear behaviour for the hard material and a non-linear reaction for the soft material state. Both information can be used to determine the stress state or/and the hardness value.

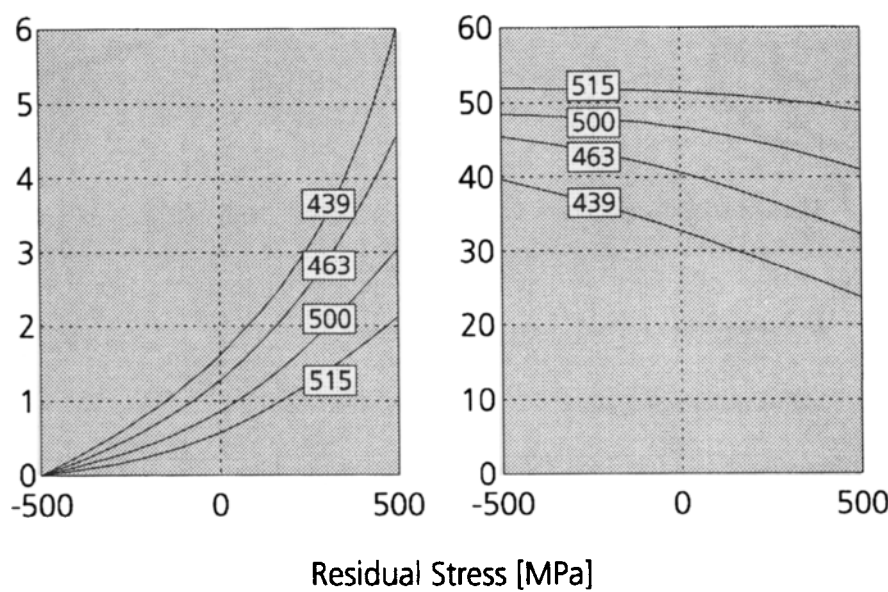


Figure 10. Stress calibration of Barkhausen noise amplitude M_{Max} , and coercivity H_{CM} versus stress for microstructures with different Vickers hardness H_V (H_V -values indicated in Figures).

After calibration quantitative ND stress measurements are obtained. Because steel grades have a non-linear stress dependency unambiguous values can only be achieved by using multiparameter testing devices with at least two independent parameters. The calibration procedure for these multiparameter units is the same as described for the Barkhausen method. Appropriate approximation function for quantitative ND-tests are usually obtained by multiple regression or by neural net analysis.

5.043 Applications

Residual Stress and Hardness Tests in Low Pressure Turbine Blades /9/:

Turbine blades of the last stage row in low-pressure (LP) turbine sections must operate in regions of high moisture. This leads to water droplet erosion of the leading edge. To increase the resistance of the blades to this type of erosion, a flame hardening process is employed. This

process, if not properly applied, increases the susceptibility of the blade material, X 20Cr 13 to stress corrosion cracking.

For stress corrosion cracking to occur, sufficiently high tensile stresses must be present. The amount of the total surface stress present in the hardened leading edge of the blade is determined, to a large extent, by the residual stresses from the flame hardening process. If there are large residual tensile stresses in this area, cracking can be expected after a certain operating period as a result of stress corrosion. The presence of chlorides or other cycle impurities aggravates the situation.

In order to carry out quantitative hardness and residual stress tests on low pressure turbine blades, stress and microstructure sensitive parameters M_{MAX} and H_{CM} are used. After the calibration (see chapter 5.042 and Figure 10) of the micromagnetic parameters in a bending machine using bars of appropriate geometry quantitative ND hardness and residual stress tests are possible. The probe system, the pick up sensor for Barkhausen signals as well as the yoke are designed in such a way that the transducer system has always an intimate surface contact. To assure that absolute residual stress values correlate well with micromagnetic results, X-ray measurements are performed on some testing points on the bars as well as on the turbine blades. By this procedure one can verify that blade geometry has no influence on ND results, even if calibration is performed on the flat bar geometry. Results are shown in Figure 11. This micromagnetic technique is applied in the factory for quality inspection after flame hardening and for blade inspection in power plants. The critical residual surface stresses can be accurately determined using either X-ray or micromagnetic testing equipment. Further electromagnetic applications and prototype-devices are described in /10, 11/.

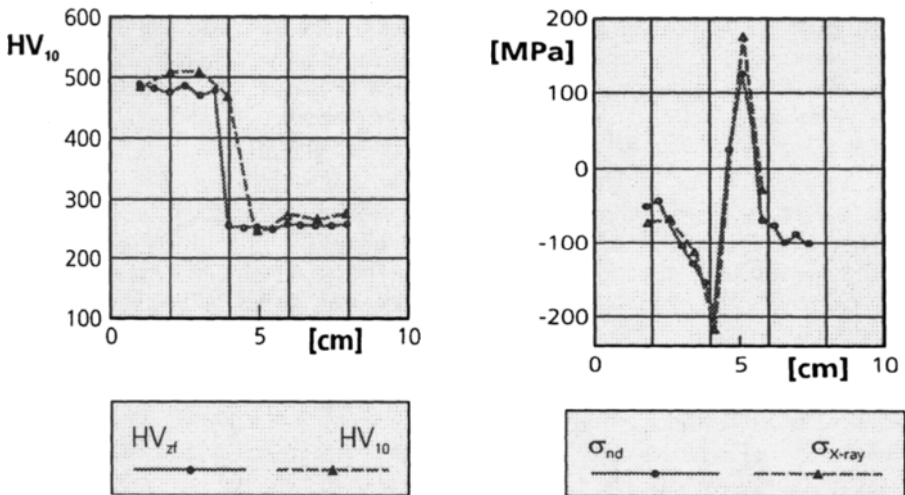


Figure 11. Hardness and residual stress measurements by Vickers, x-ray and micromagnetic devices

5.044 Micromagnetic stress tests of machined surfaces

Microstructural parameters and residual stress states in near surface zones determine the functional behaviour of components. Very different dimensions of the affected zone are yielded depending on the applied treatment. In the case of inductive hardening, stress and microstructure parameters are influenced up to several millimetres of depth. On the other hand finishing procedures will influence the surface integrity in the order of micrometers only.. Because all used ND parameters (Table 3) are influenced by microstructural parameters and stresses in a more or less sensitive way, one has to evaluate first which ND parameters

- are the most sensitive to measure surface stresses
- must be used to determine additional surface characteristics e.g. retained austenite, hardening depths
- are necessary to perform quantitative residual stress measurements under the consideration of main influencing factors as lift-off, microstructure, and temperature which can disturb stress tests.

This information will be used to evaluate approximation functions for different objectives. This functions will be determined by neural net or multiple regression analysis.

Residual Stress in Laser Hardened Components /12/

Micromagnetic techniques developed during the last years have been found to be most appropriate for a nondestructive evaluation of the machining quality of laser-hardened components. Combining the complementary information from the various microstructure and stress sensitive measuring parameters, a fast estimation of relevant quality parameters can be simultaneously achieved. Using a multiple regression analysis, approximation functions are obtained to determine the hardness, hardening depth and residual stresses.

Nondestructive evaluation of residual stresses is demonstrated for laser-hardened components using multiparameter sensor systems. As a consequence of a laser-hardening treatment tensile and compressive residual stresses are created in the surface and the surface near microstructure is transformed to martensite. The separation of these superimposed residual stress and microstructure gradients implies that several independent measuring parameters have to be used. To get quantitative results micromagnetic parameters, must be calibrated as described in chapter 5.042. By performing a bending stress experiment on the hardened and on the non-hardened surface a stress calibration of the testing parameters can be achieved as demonstrated in Figure 12 at a laser-hardened track of a 50 NiCr 13 specimen. The nondestructive techniques combined in this calibration are magnetic Barkhausen signal and H_1 -upper harmonics analysis. The micromagnetic parameters are measured as a function of applied bending stress. The data are modelled by least square fitting - in this case, by a multiple regression analysis. Linear as well as non-linear models have been used to evaluate multidimensional approximation functions. In this case, the approximation function for residual stress σ is obtained as a function of the micromagnetic parameters M_{MAX} , H_{CM} , K and H_{CO} .

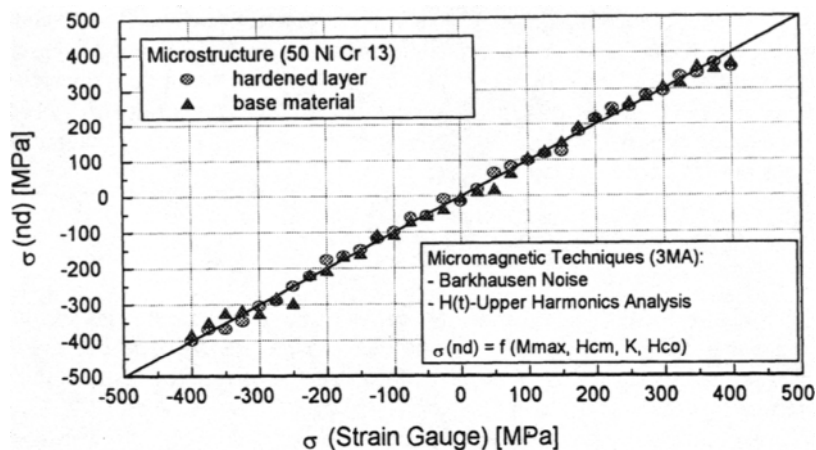


Figure 12. Micromagnetic versus conventional measurement of stress by strain gauge, Correlation Coefficient 99 %, mean standard error 15.4 MPa

As can be seen in Figure 12 the residual stress values $\sigma(\text{ND})$, calculated from the micromagnetic measurement correspond exactly to the values measured by a strain gauge. Both, the hardened and the bulk states, can be measured by one and the same approximation function. The advantage of the applied *Micromagnetic Multiparameter Microstructure and stress Analysis (3MA)*-technique: residual stresses, hardness and hardening depth are measured simultaneously.

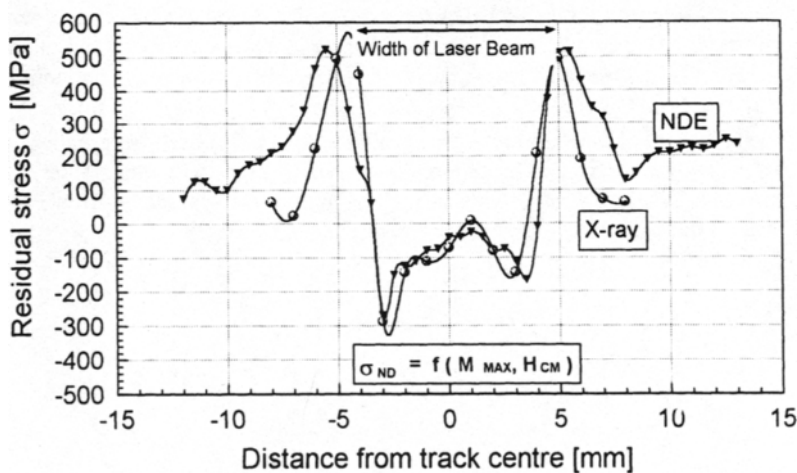


Figure 13. Residual stress profile across a laser-hardened track of a 50 CrMo 4 specimen

This is achieved by a multiparameter unit with parameters derived from the magnetic Barkhausen signal, the time signal of tangential field strength, the incremental permeability and eddy current parameters. Figure 13 shows the micromagnetic stress measurements across a laser hardened track in comparison with x-ray results. Both results do not fit exactly because of different penetration depths and because of two dimensional stress states which are acting in different ways on x-ray and magnetic parameters.

Shot peening

Figure 14 and Figure 15 show how parameters derived from Barkhausen noise are influenced by work hardening and by microstresses of the second kind (RS II). One can see that two different shot peened states - C1: shot peened with 1 bar, C7: shot peened with 0.36 bar - can be characterised by x-ray as well as by the coercivity profile method (Figure 14). The reason for the increase of measured values in the near surface zone is caused by dislocations which are produced by work hardening of the ferritic phase. The depth profile of residual stresses of second kind shows that the RS II values can be evaluated from x-ray and bending arrow etching measurements and by Barkhausen parameters as described in /14/. The used micromagnetic parameter is the ΔH_M -value. ΔH_{M0} represents the stress free bulk material state, whereas ΔH_M represents the value of the shot peened state.

The nondestructive parameter (Figure 15) decreases with decreasing compressive stress levels. As outlined above one can see that micromagnetic parameters sense residual stresses of second kind and that this information can be obtained in a nondestructive way without etching.

Grinding

Quality characteristics of machined surfaces are: compressive residual stresses, hardness, values and microstructure parameters. Up to now quality inspection use x-ray, hardness and nital-etching tests. ND methods in the last ten years apply electromagnetic and micromagnetic techniques /13-16/. If several targets must be determined simultaneously multiparameter procedures must be used.

First tests have shown /17/ (Figure 16) that residual stresses, caused by grinding, as well as surface hardness values and case depths can be determined by this multiparameter technique. Method use the 3MA-technique with modules: multifrequency-eddy-current, incremental permeability and the time signal of tangential magnetic field strength.

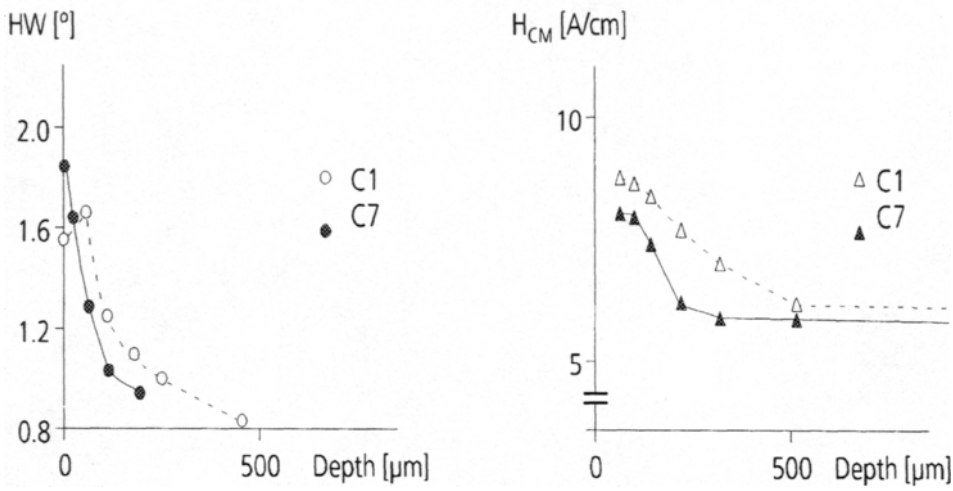


Figure 14. Depth profile curves for two different shot peened states (C1, C7) recorded by X-ray half width (HW) and coercivity (HCM) measurements.

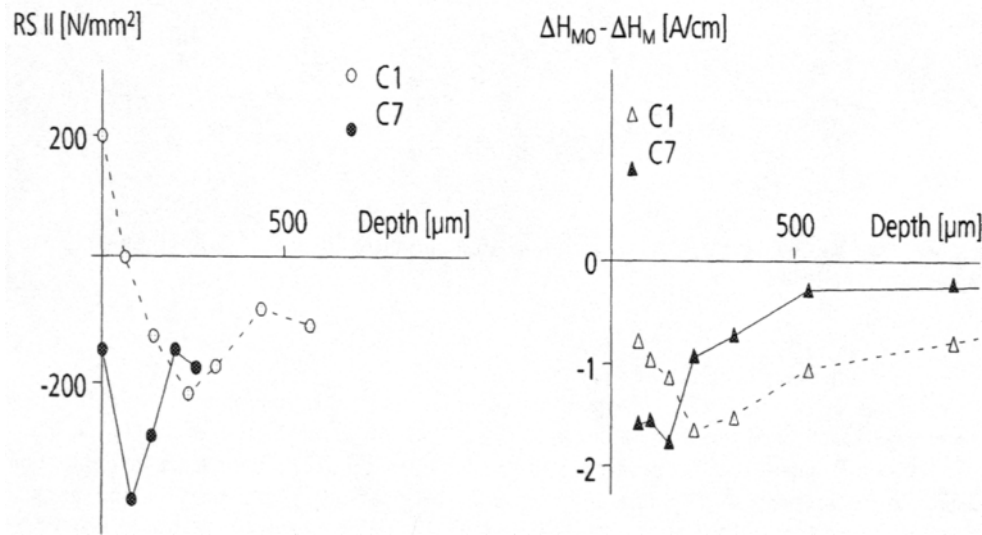


Figure 15. RSII depth profile curves determined by X-ray and deflection etching method and micromagnetic profile curves. Both Figures are for the same shot peened states shown in Figure 14.

electromagnetic

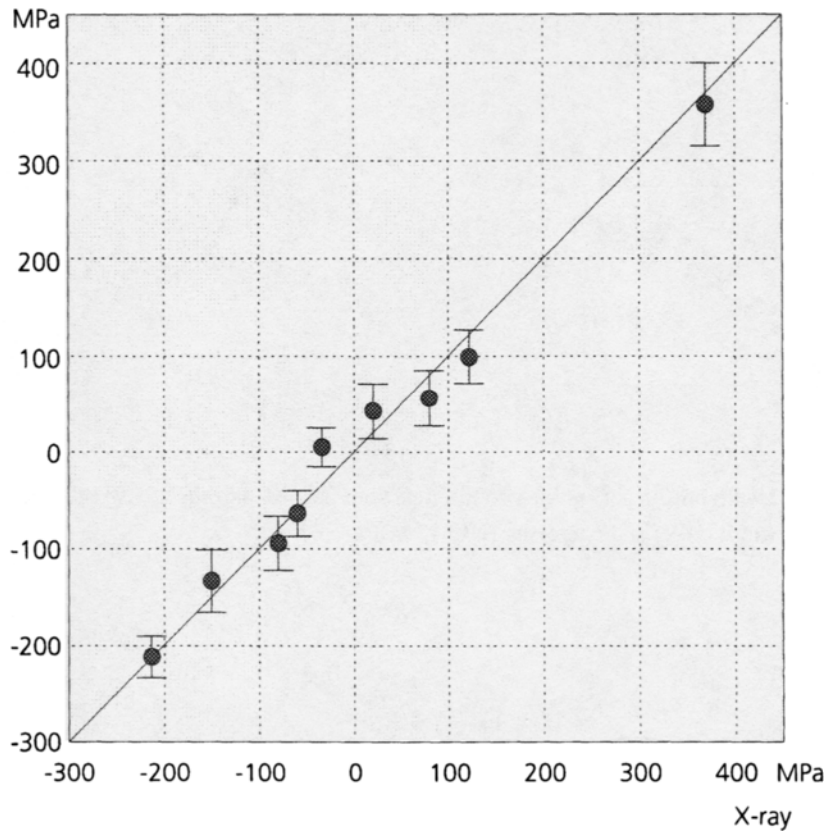


Figure 16. Residual stress measurements after grinding by x-ray and multiparameter micromagnetic techniques.

Stress measurements in textured steel sheets

These steel sheets are used in the automotive industry. Quality inspection is performed on specimens taken from the ends of the coil. Quality characteristics of these sheets are: yield strengths (σ_Y), anisotropy values (Δr , r_m), visual inspections and Erichsson tests. One pilot unit uses ultrasonic time of flight and incremental permeability parameters for the process integrated inspection of the sheet quality /18/. How Barkhausen parameters are influenced by texture and stresses was examined in /19/. If one uses the electromagnetic multiparameter approach-multi-frequency-eddy-current, incremental permeability, time signal of magnetic field strength - one can measure as demonstrated recently /20/ r_m , Δr and σ_Y values simultaneously with one electromagnetic, micromagnetic probe system (Figure 17, 17', 18).

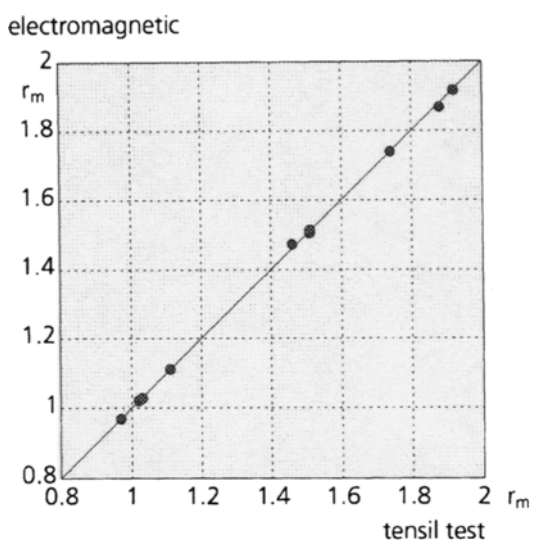


Figure 17. Nondestructive electromagnetic determination of anisotropy parameter r_m in thin steel sheets

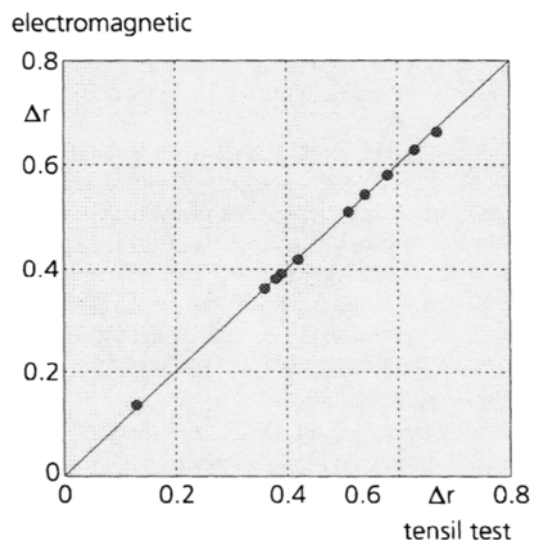


Figure 17'. Nondestructive electromagnetic determination of anisotropy parameter Δr in thin steel sheets

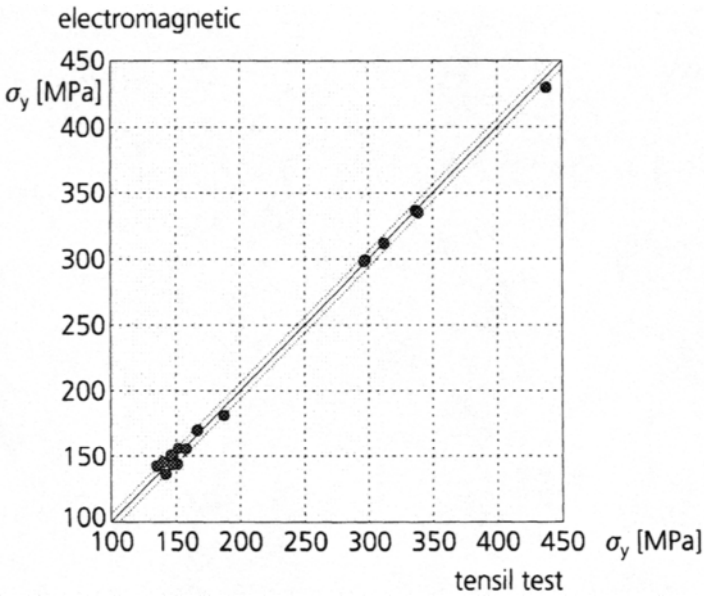


Figure 18. Nondestructive electromagnetic determination of yield stress σ_y in thin steel sheets. Approximation function uses 9 coefficients for least square analysis. Mean standard error $1\sigma = 5.09$ MPa.

5.045 **High resolution stress measurements**

To perform stress measurements with a high local resolution one can use different micromagnetic methods [13, 21-24]. Key elements for these tests are miniaturised probes [21,22]. In the case of Barkhausen and incremental permeability one can use modified video-heads. To detect e.g. irreversible Bloch-Wall movements, the head must be positioned very precisely with regards to the most sensitive receiving area. The head resolution which has been gained is approximately 10 μm . The accuracy for the sensor position at the sample surface should be about one μm . The measuring time, including positioning time, is approximately about one second. By scanning the sensor across the surface of the test specimen one gets two-dimensional images of micromagnetic parameters.

One result of a high resolution micromagnetic stress device [22] is shown in Figure 19. Barkhausen signals have been detected with a video-head across a stress spot structure, which has been produced by focused laser beam treatment. The energy input was chosen to make sure that no α - γ transition occurs and therefore no rehardening zones must be kept in mind. The measured dependencies of the Barkhausen signal could thus be completely attributed to the residual stresses induced as a result of the thermal treatment. As it had been expected, the highest residual tensile stresses occurred in the middle position of the laser focus.

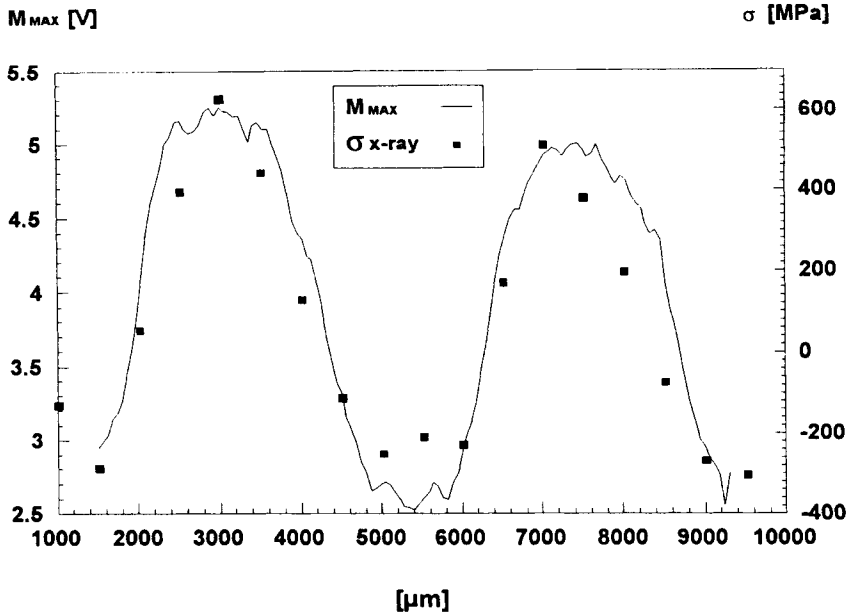


Figure 19. X-ray and micromagnetic (Barkhausen) residual stress measurement across stress spots which have been produced by laser treatment

5.05 Recommendations

Micromagnetic methods should be used to measure surface characteristics like stresses, microstructure parameters and texture.

To achieve quantitative results one has to use calibration procedures and multiparameter techniques.

Electromagnetic and acoustic disturbances of ND-signals can avoid quantitative tests. This key point must be kept in mind for further developments and industrial applications.

Standardised test parts and test procedures are necessary for the development of general rules. Standard tests should be used to compare the sensitivity of micromagnetic devices which are offered on the market.

Nondestructive electromagnetic techniques can perform tests fast and without surface contact. Therefore these devices should be used for *Post-Process-Inspections* (PPI) and *Process Integrated Nondestructive Tests* (PINT).

Nondestructive micromagnetic and electromagnetic methods should be applied if *Failure Mode and Effect Analysis* (FMEA) require such tests.

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6 Assessment of residual stresses

B.Scholtes

6.01 Historical review

It is now well established that on principle no technical materials, components or structures are available completely free of residual stresses. Since the first indications of residual stress states during the last century, (see e.g. [1-4]), the number of papers and presentations dealing with evaluation of residual stresses has considerably increased. Despite this fact, the assessment of existing residual stress states still today is often controversial. The reason is that quite a number of specific aspects have to be taken into account in measuring residual stresses making such analyses sometimes troublesome and doubtful and that the notation „residual stress“ is not always used in an unambiguous way.

In general, the statement that residual stresses can both have a favourable or detrimental effect on the behaviour of technical parts or components is valid. Very often, the ignorance of existing residual stress states and their consequences is used to explain unexpected failure.

In the past, progress in machine building and mechanical engineering as well as in the improved understanding of materials microstructure and the resulting properties was closely correlated with advances in assessment of residual stresses. The development was of course markedly influenced by improvements of measuring techniques. It is interesting to note that often residual stress effects on the materials behaviour have been discussed long before first quantitative proof of existing residual stress states has been made. And even then, in most cases, considerable time elapsed before first successful attempts were made for a quantitative assessment of residual stresses.

In fact, articles and components, using residual stress effects to improve performance and reliability have been produced at all times (see e.g. [5]). Probably the first correct description of a residual stress state in a technical component was given by A. Wöhler [2] in 1860, describing residual stress states of plastically bent bars. Important milestones of residual stress assessment were efforts made to understand the consequences of mechanical surface treatments on the fatigue behaviour of components. The historical development in this field is described in detail in [6]. Significant contributions came from the work of O. Foeppl [7,8] and A. Thum [9,10] as a consequence of their discussion on residual stress effects after surface rolling to improve the fatigue behaviour of metallic components. Also during this time, first hints were made at the positive consequences of compressive residual stresses in the case of corrosion fatigue [10,11]. W. Ruttman [12] was the first to clearly demonstrate the consequences of machining residual stresses for fatigue loaded parts. The application of autofrettage processes to increase the strength of tubes under alternating internal pressure was suggested by [13]. Shot peening residual stresses were quantitatively measured by E.W. Milburn not before 1945 [14]. One of the earliest examples to take residual stresses of fatigue loaded parts quantitatively into account is described in [15], where flat specimens with holes bearing different

quenching residual stresses were investigated. Because of their technological importance, casting residual stresses belong to the earliest subjects of residual stress research [4,16]. The same is valid for joining residual stresses. First quantitative analyses of welding residual stresses are described in 1934 [17-19]. However, their origin and consequences remained uncertain until recently.

Also the importance of heat treating residual stresses has been recognized early and attempts were made to measure existing stress distributions [20-22]. The origin and the controlling factors of heat treating residual stresses were only gradually classified step by step [23]. Certain aspects, e.g. transformation plasticity effects, although already mentioned in the last century [24], are still under discussion.

Very early, also attempts were made to calculate residual stress distributions occurring as consequences of technological processes. First attempts started in the 1920's in the case of heat treating residual stresses (see e.g. [25, 26]). Practical consequences, however, remained small until introduction of finite element methods (see e.g. [27-30]) which allowed detailed insight into the residual stress determining mechanisms and a realistic modelling of the processes.

As a result of the rapid introduction of fracture mechanic principles in the field of materials science and engineering, also the consequences of residual stress states have been intensively studied in this domain. Residual stress distributions at crack tips were theoretically predicted by [31] and experimentally verified by [32]. A survey about important contributions to residual stress effects on fracture mechanics and crack propagation is given in [33].

Finally, experimental and theoretical micro residual stress analyses should be mentioned, which considerably improved knowledge about the relations between materials microstructure and its macroscopic mechanical behaviour (see e.g. [34-37]). Considerable time elapsed before research succeeded in quantitative analyses of residual stress states within single grains of polycrystals [38, 39]. The classification of micro and macro residual stresses has a long history [40] and was finally established in [41]. Nowadays, measurement and assessment of micro residual stresses still and again is under discussion and an exciting research area.

6.02 General remarks

Residual stresses are mechanical stresses acting alone or in addition to applied stresses in materials and components. Consequently, if they are not quantitatively known, they may cause considerable uncertainty in designing components correctly. In some cases, a marked improvement of behaviour of components can be achieved compared to residual stress free states, if appropriate residual stress distributions have been introduced. On the other hand, detrimental residual stress states may cause unexpected failure of machine parts. Obviously, as a consequence, residual stresses are one of the most interesting phenomena for materials scientists as well as for engineers and machine builders. Knowledge about origin, determination and assessment of residual stresses is strongly increasing. However, in most cases, consequences of residual stresses on operation behaviour of components are only qualitatively evaluated taking existing experiences into account. Often this is due to a lack of quantitative data about existing residual stress distributions and their stability under loading conditions.

The stress state acting in a given point of a body is described by the stress tensor σ_{ij} with respect to a system of coordinates $\underline{s}^i$ (see Figure 6.1). Very often this system of coordinates correlates with symmetry axes or marked machining directions of the components under in-

vestigation. From Figure 6.1 also the significance of the tensor components can be seen. Elastic deformations resulting from acting stresses can be calculated using Hooke's law

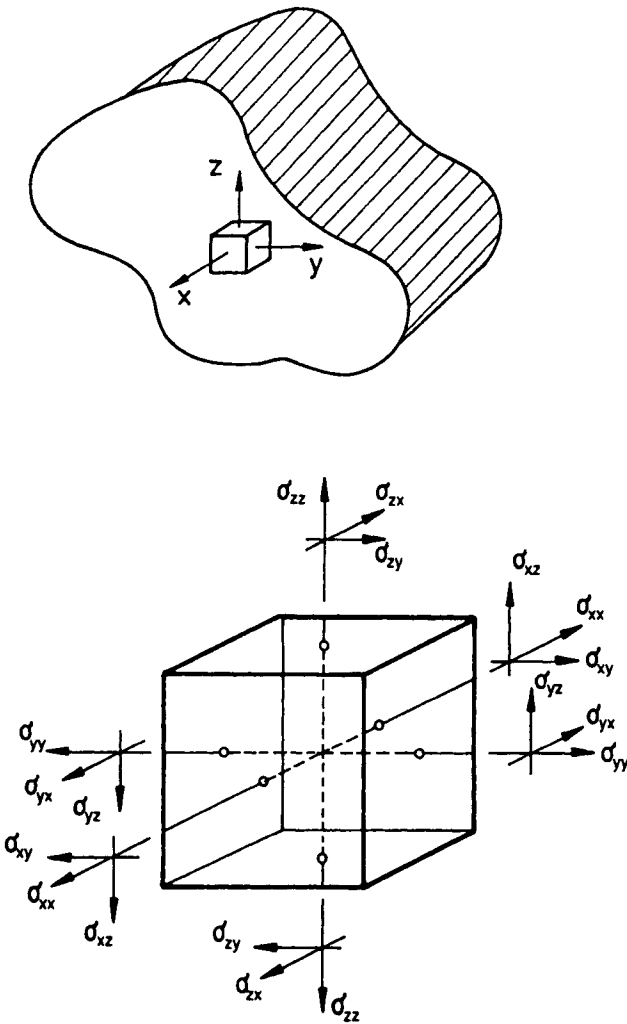


Figure 6.1. Significance of stress tensor components.

$$\varepsilon_{ij} = \{(v+1)/E\} \cdot \sigma_{ij} - \delta_{ij} \{v/E\} \cdot (\sigma_{xx} + \sigma_{yy} + \sigma_{zz}) \quad (6.1)$$

with $\delta_{ij} = 1$ for $i = j$ and $\delta_{ij} = 0$ for $i \neq j$. E is Young's modulus and v Poisson's ratio of the material under consideration. If an appropriate coordinate system is used, shear stress components disappear, and stress states are described by principle stresses σ_i ($i = 1, 2, 3$). If no external forces or moments are acting, stress tensor σ_{ij} is identical with the residual stress state σ_{ij}^{rs} in a point.

If loading stresses as well as residual stresses exist, they can be summarized to the stress tensor of total stresses σ_{ij}^{ts} . This is achieved by simply adding the respective tensor components, if the stress tensor of loading stresses σ_{ij}^{ls} and of residual stresses σ_{ij}^{rs} is described with respect to the same coordinate system s^i . More detailed information about the description of stress states can be found in many textbooks (see e.g. [42,43]).

In all practical cases residual stresses resulting from technological treatments are superpositions of macro und micro residual stresses. Definition and separation of the stress types is described in chapter 2.06. To assess a given residual stress state correctly, it is mandatory to decide, whether residual stresses σ_{ij}^I of the first, σ_{ij}^n of the second or σ_{ij}^m of the third kind or superpositions of different stress types are acting. Problems arising in this context are due to the fact that measuring techniques for residual stresses are in different ways sensitive to the three types of residual stresses mentioned on the one hand and that consequences of residual stress states for strength and lifetime of components have to be assessed quite differently for residual stress states of the I, II or III kind. Only residual stresses of the first kind σ_{ij}^I can be looked upon as stresses equivalent to loading stresses resulting from external forces and/or moments [41].

To assess the consequences of residual stresses on strength and lifetime of components, it is convenient to differentiate between (quasi-) static loading and fatigue loading. In both cases, it has to be considered whether cracks exist. This has separately to be taken into account. Residual stress effects on relaxation and creep will not be treated further here, because residual stress relief can be assumed for these loading conditions.

The following chapters have the aim to give a short survey about the present knowledge of assessment and consequences of residual stresses in materials science and engineering. For a detailed discussion of individual cases, it is referred to the references cited.

6.03 Residual stress effects on components under static loads

6.031 Plastic deformation and fracture of components without cracks

In the case of static loading, different types of failure modes have to be distinguished. Failure may occur as a consequence of onset of plastic deformation, of exceeding a certain amount of plastic deformation or of fracture. Furthermore, elastic or plastic instability may be regarded as failure criterion.

The basis for a quantitative assessment of statically loaded components is the determination of equivalent stresses σ_e corresponding to given loading stresses, which take into account the consequences of multi-axiality of stress states on strength and failure. Equivalent stresses consider different effects resulting from multiaxial residual stress states, e.g. the resulting

principal normal stresses, principal shear stresses, principal normal deformations or distortion energy. It is assumed that in case of multiaxial stress states failure occurs, if the appropriately chosen equivalent stress σ_e has the same effect as the critical uniaxial stress which, itself, leads to failure of the materials state under consideration. This leads to different equivalent stress values, depending on the underlying effect chosen. A detailed description of significance and determination of equivalent stresses is given in [44]. A necessary prerequisite for the calculation of the decisive equivalent stress of a component therefore is the correct choice of the appropriate equivalent stress type and the determination of locally occurring stress states to find out location and amount of maximum σ_e -values. If this is achieved, the basic equation to design components can be written as

$$\sigma_e \leq \frac{R}{s}$$

(6.2)

where R is the appropriate materials resistance against failure and s is a safety factor. R has to be chosen according to the type of failure expected.

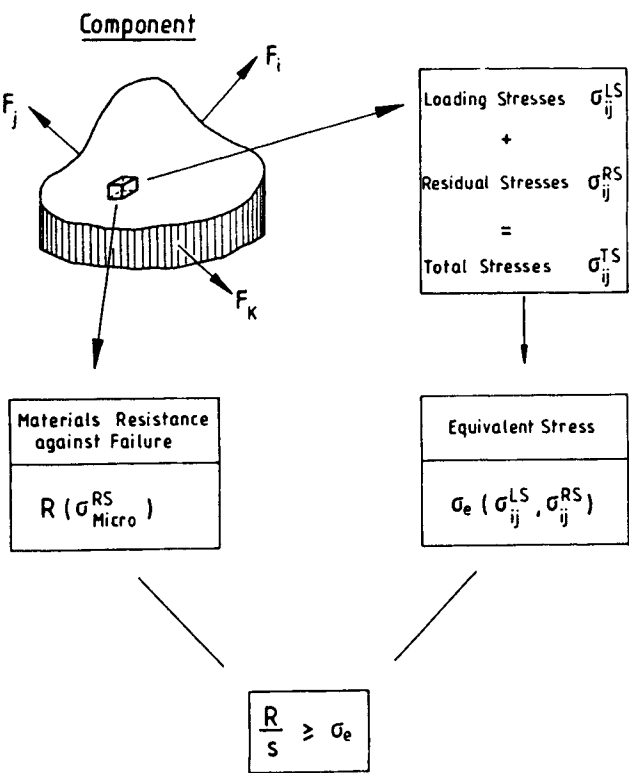


Figure 6.2 Design of statically loaded components with macro and micro residual stresses.

If failure occurs as a consequence of plastic deformation, yield stress R_{es} is decisive. If small amounts of plastic deformation are allowable, proof stresses, e.g. $R_{p0.2}$, have to be used. If failure occurs by fracture, R is identical with ultimate tensile strength R_m (Figure 6.2). A summary of the procedure is given in Figure 6.2. One can see that only macro residual stresses are treated equivalent to loading stresses, whereas micro residual stresses are assumed to influence materials resistance R .

It has to be pointed out, however, that there are remarkable differences between loading and residual stresses [45]. An important fact is that loading stresses are clearly determined by amount and sign of loading forces, whereas residual stresses may change amount and sign during loading. As a consequence, residual stress states have to be assessed differently for brittle and for ductile materials states in the case of the failure modes mentioned above. Only for failure by „onset of plastic deformation“, residual stresses have to be considered for all materials states. If, however, plastic deformations take place, residual stress distributions may be more or less changed or even relaxed before failure occurs. Therefore, in the case of fracture, only for brittle materials states, residual stresses have to be taken into consideration. In many cases, where no quantitative knowledge about residual stress states exists, they are only taken into consideration by the application of appropriate safety factors s .

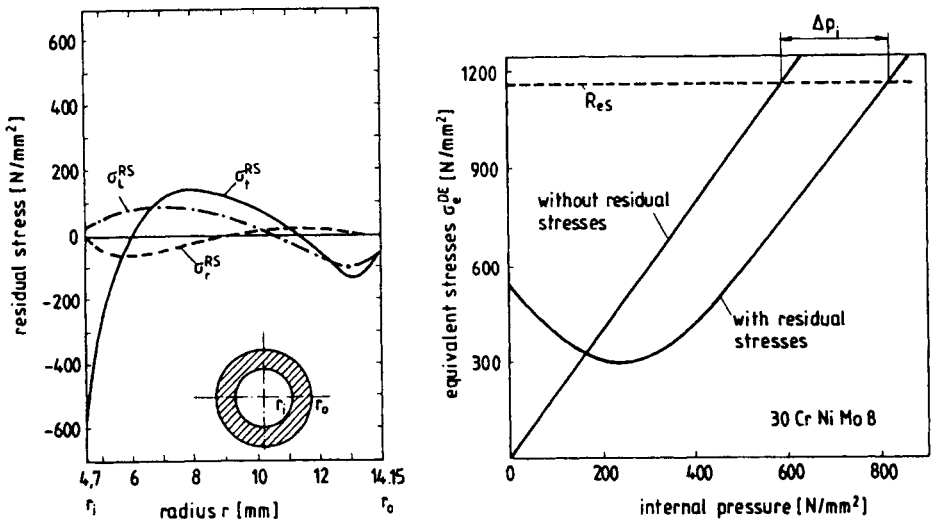


Figure 6.3. Residual stresses in an autofrettaged steel tube (left) and consequences on equivalent stress state at the inner surface (right).

Residual stresses, depending on their distribution and sign, may diminish as well as increase locally acting equivalent stresses. An example is given in Figure 6.3. It deals with an autofrettaged steel tube, where triaxial residual stresses have been introduced by applying an overpressure (see Figure 6.3, left). For the inner surface, equivalent loading stresses for a residual stress free tube following the distortion energy (DE-) hypothesis are given by the straight line as a function of internal pressure. If autofrettage residual stresses are taken into account, for internal pressures $p_i \geq 170 \text{ N/mm}^2$ lower equivalent stress values result compared to residual stress free starting conditions. In this case, an excess internal pressure $\Delta p_i \approx 225 \text{ N/mm}^2$ is allowable, if onset of plastic deformation is taken as failure criterion (see right part of Figure 6.3).

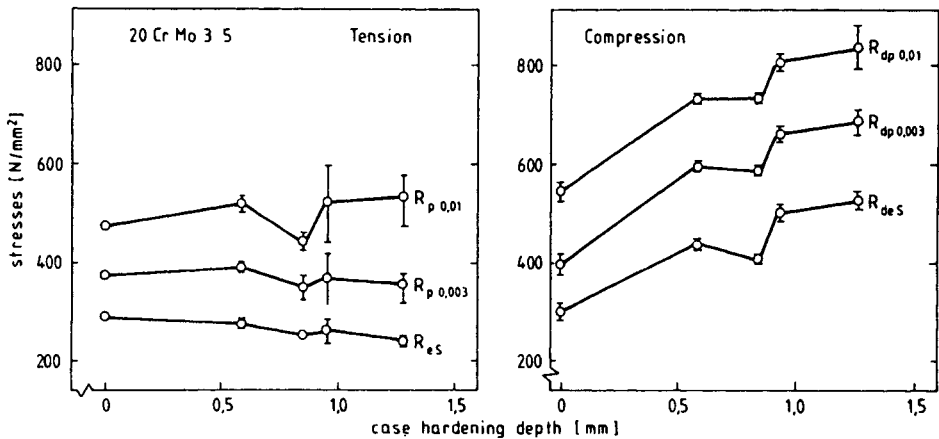


Figure 6.4. Influence of case hardening depth on yield and proof stresses resp. of tension (left) or compression (right) loaded 20 CrMo 3 5 [46].

In Figures 6.4 and 6.5 two examples are given which experimentally verify the influence of residual stresses on statically loaded parts. Figure 6.4 deals with 20 Cr Mo 3 5 specimens with a diameter of 5mm, which have been case hardened to different case hardening depths and then tested in uniaxial tension or compression tests [46]. In all cases, first plastic deformations occur in the softer core of the specimens and, consequently, yield stress values and proof stresses for small plastic deformations are influenced by tensile residual stresses, which occur in the core region as a consequence of the case hardening process. Because the amount of tensile residual stresses increases with case hardening depth, this leads to decreasing strength values R_{eS} , $R_{p0.03}$ and $R_{p0.01}$ in tension tests and increasing values R_{deS} , $R_{dp0.03}$ and $R_{dp0.01}$ in compression. However, the situation is complicated by a continuous rearrangement of residual stresses with plastic deformations. This explains, together with strain hardening effects, that

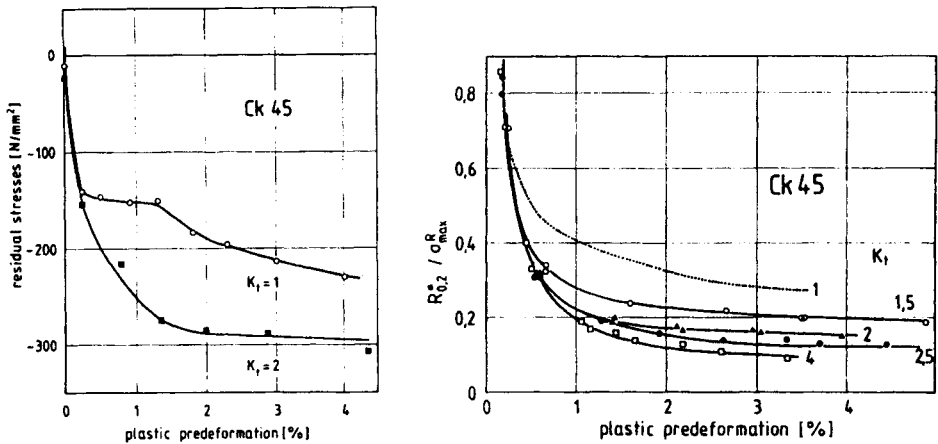


Figure 6.5. Residual stresses at the surface and in the notch root resp. of bending bars made of steel Ck 45 as a function of tensile predeformation (left) and consequences on proof stress ratio $R_{0.2}^* / \sigma_{\max}^R$ measured after changing of deformation direction (right) [47].

$R_{p0.01}$ increases with case hardening depth. However, for the materials states under consideration, in addition, a strength differential effect is observed, which leads to higher stresses in compression compared to tension.

From Figure 6.5 (left), one can see that plastic bending introduces compressive residual stresses on the tensile side of the bars, which increase with predeformation and are considerably higher for notched ($K_t = 2$) than for smooth ($K_t = 1$) bars [47]. On the right hand side of Figure 6.5, the influence of predeformation and hence residual stresses on proof stresses $R_{0.2}^*$, measured for loading in a direction opposite to predeformation and related to maximum predeformation stresses $\sigma_{\max}^R$, is shown. It is obvious that $R_{0.2}^* / \sigma_{\max}^R$ decreases with plastic predeformation as well as with notch factor K_t . This can be attributed to the macro residual stresses developed in the outer fibre and in the notch root of the bending bars as a consequence of inhomogeneous plastic straining during predeformation and which act in the same direction as loading stresses, if the direction of deformation is changed.

However, to fully describe the materials behaviour in detail, Bauschinger effect [48], which is related to micro residual stresses and also changes of macro residual stresses with deformation history, have to be taken into account.

A further example to consider the influence of residual stresses on plastic deformation of statically loaded parts is given in [49]. Here, consequences of compressive residual stress states introduced by shot peening operations on the onset of plastic deformation for tension or

compression loading is successfully modelled. Finally, it should be pointed out that, as a consequence of multiaxial residual stress states, the risk of brittle fracture can considerably be increased compared with residual stress free components. This is also considered in the next paragraph on the basis of fracture mechanics concepts.

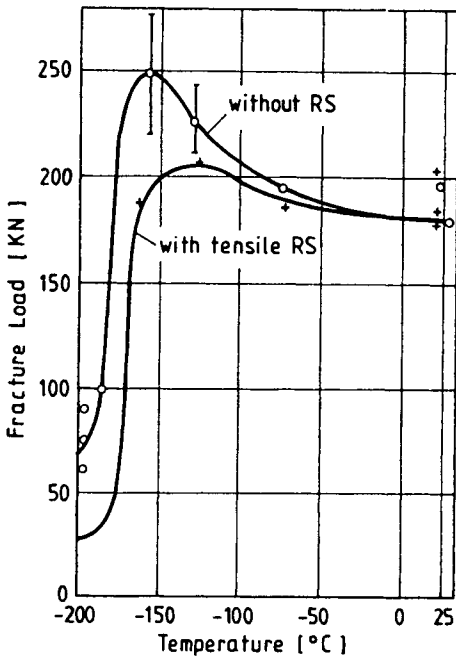


Figure 6.6. Consequences of tensile residual stresses on fracture loads as a function of test temperature [55].

6.032 Fracture of components with cracks

The design of components with flaws or cracks, which includes the determination of admissible loads as well as admissible crack lengths and flaw dimensions resp. to avoid unexpected failure is based on the concepts of fracture mechanics. These concepts describe fracture of materials with defects relating fracture strength to macroscopic stress distributions resulting from applied loads and geometries of components. In these concepts it is assumed that influences of existing stress fields on crack opening and stable as well as unstable propagation can be described in terms of characteristic parameters. These are the stress intensity factor K and crack extension force G for (mainly) elastic cases and J-integral as well as crack tip opening displacement δ taking into account large scale plastic deformations. Failure occurs, if critical

values of these parameters are attained. Details can be found in many textbooks (see e.g. [50 - 52]). A good survey is given in [53].

If cracks exist in residual stress fields, crack opening as well as propagation behaviour will be influenced in case by existing residual stress components as well as by applied stresses. Different aspects have to be taken into consideration. At first, absolute values of locally acting stresses are influenced by the combined effect of loading and residual stresses. Consequently, resulting effective fracture mechanics parameters have to be determined, which control crack initiation. Crack propagation may also be affected depending on the amount of plastic deformation and the associated redistribution and relaxation of residual stresses. An important aspect is the influence of residual stresses on the degree of multiaxiality of stress distributions. More or less ductile materials may exhibit brittle fracture under the influence of multiaxial residual stress states. Finally crack opening mode may be influenced. Mode I - loading stresses, together with multiaxial residual stress states lead to mixed-mode crack opening behaviour. Experimental evidence for the consequences of residual stresses on fracture has been given for a number of cases (see e.g. [54 - 58]). A typical example is shown in Figure 6.6 [55].

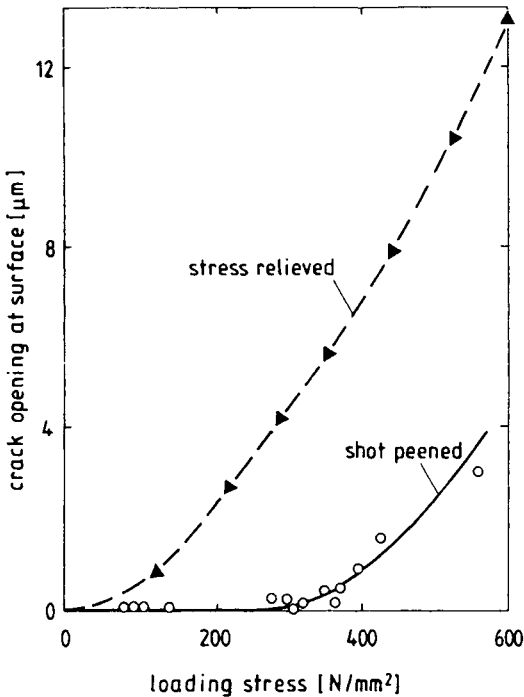


Figure 6.7. Crack tip opening of a shot peened and a residual stress free Ti-6Al-4V specimen [58].

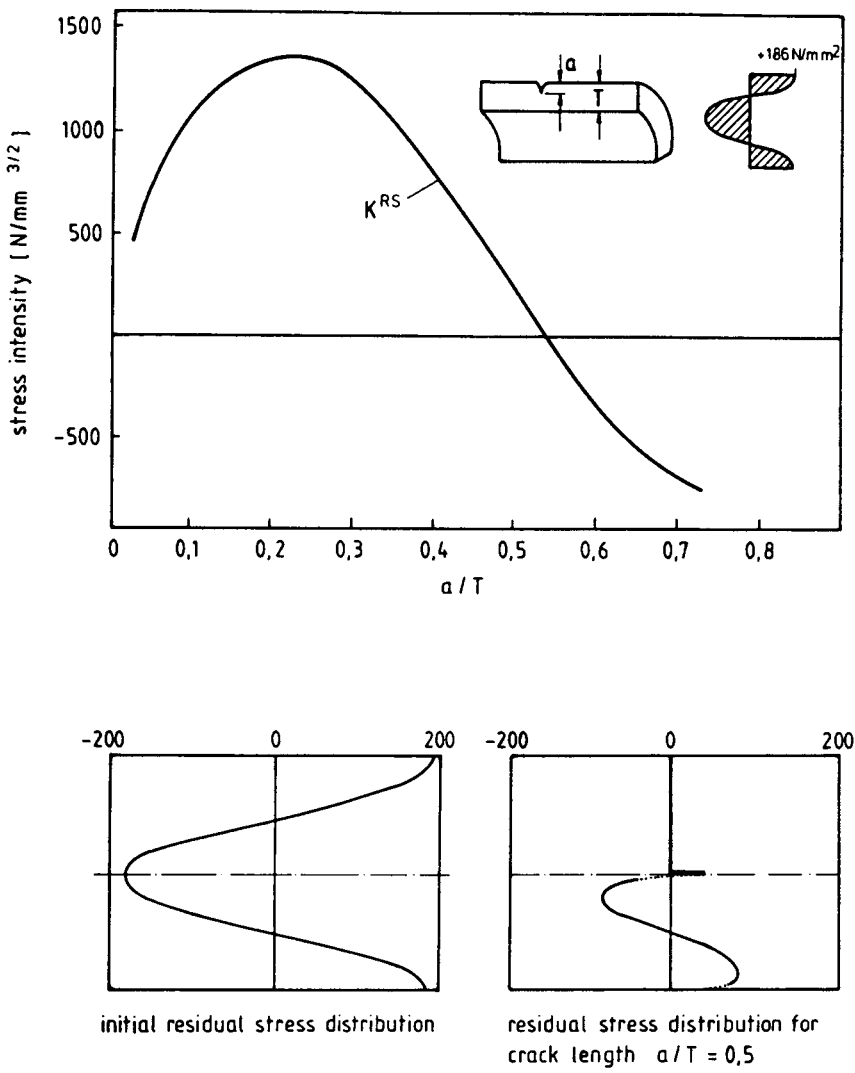


Figure 6.8. Stress intensity factor (above) and residual stress distribution as a function of crack propagation across the wall of a cylinder tube (below) [64].

The influence of tensile residual stresses, introduced by rapid local heating, on fracture load of centre cracked specimens was investigated over a wide range of temperatures. This was done to take brittle as well as ductile materials conditions into consideration. The experimental results clearly show that in the range of low to medium toughness values, tensile residual

stresses considerably reduce fracture loads. For a shot peened Ti - 6 Al - 4 V - alloy, [58] could demonstrate by the aid of sophisticated experimental technique, that shot peening compressive residual stresses considerably increase the threshold value for crack opening and diminish crack opening values for given loading stresses compared to the stress relieved state (see Figure 6.7). Also in the case of ceramics, which are typical examples of brittle materials, residual stress effects on fracture have been studied. A clear positive influence of compressive residual stresses on strength is stated in many cases [59].

In the case of linear elastic fracture mechanics, it is assumed that an effective stress intensity factor K^{tot} describes the loading state of a cracked component with residual stresses and that

$$K^{\text{tot}} = K^{\text{LS}} + K^{\text{RS}} \quad (6.3)$$

is valid. (K^{tot} : total stress intensity factor, K^{LS} : loading stress intensity factor, K^{RS} : residual stress intensity factor). Appropriate methods to calculate K^{RS} -values for given geometries and residual stress fields have been developed [60-69]. These methods are based on FE-calculations and so called „weight functions“, which have been derived for different crack geometries [60,66]. Also crack closure integrals are used [64,67] as well as simple superpositions of residual and loading stresses [68,69]. A number of interesting examples and references are summarized in [60].

An important question in connection with residual stress related stress intensity factors and crack propagation is, how residual stresses redistribute during crack propagation and whether they relax partly or completely. Different cases are reported in literature, with K^{RS} increasing as well as decreasing, while cracks propagate through existing residual stress fields. Sufficient basic knowledge in this field is essential to predict whether a crack propagates instable or crack arrest occurs. Interesting cases dealing with this problem exist for pipe or vessel weldments [60, 64, 68 - 70]. An example is shown in Figure 6.8 [64]. A sinusoidal residual stress distribution is assumed across the thickness of a tube wall with tensile residual stresses at the inner and outer surfaces resp. If a crack starts propagating from the outside, residual stress redistribution is calculated assuming linear elastic behaviour as a function of crack length and the effective residual stress intensity factor is determined. For the case investigated crack propagation is connected with an increase of K^{RS} up to a crack length $a/T \approx 2.25$. For longer cracks, K^{RS} decreases. Similar results are described in [68]. They demonstrate that, for materials states with low fracture toughness, fracture mechanic concepts are applicable to adequately model the behaviour of components with residual stresses.

From Figure 6.6 one can see that with increasing toughness, influence of residual stresses tends to vanish. Only few examples exist to assess quantitatively the consequences of residual stresses in the ductile-brittle-transition region. While in the case of full scale yielding, as a consequence of large plastic deformations and associated stress relaxation, residual stress effects can be ignored completely, fracture in the transition region must be studied more in detail. In this context, a number of high strength structural steels have been investigated by [71]. A typical result of these experiments is plotted in Figure 6.9. In the upper part, three types of transversal residual stress distributions, produced by different thermal and mechanical treatments are shown. Below, fracture resistance J-values are plotted for specimens with compressive or tensile residual stresses resp. as well as for nearly residual stress free ones. A clear tendency can be stated that compressive residual stresses increase and tensile residual stresses decrease J-values measured. The same could be stated for crack tip opening displacement

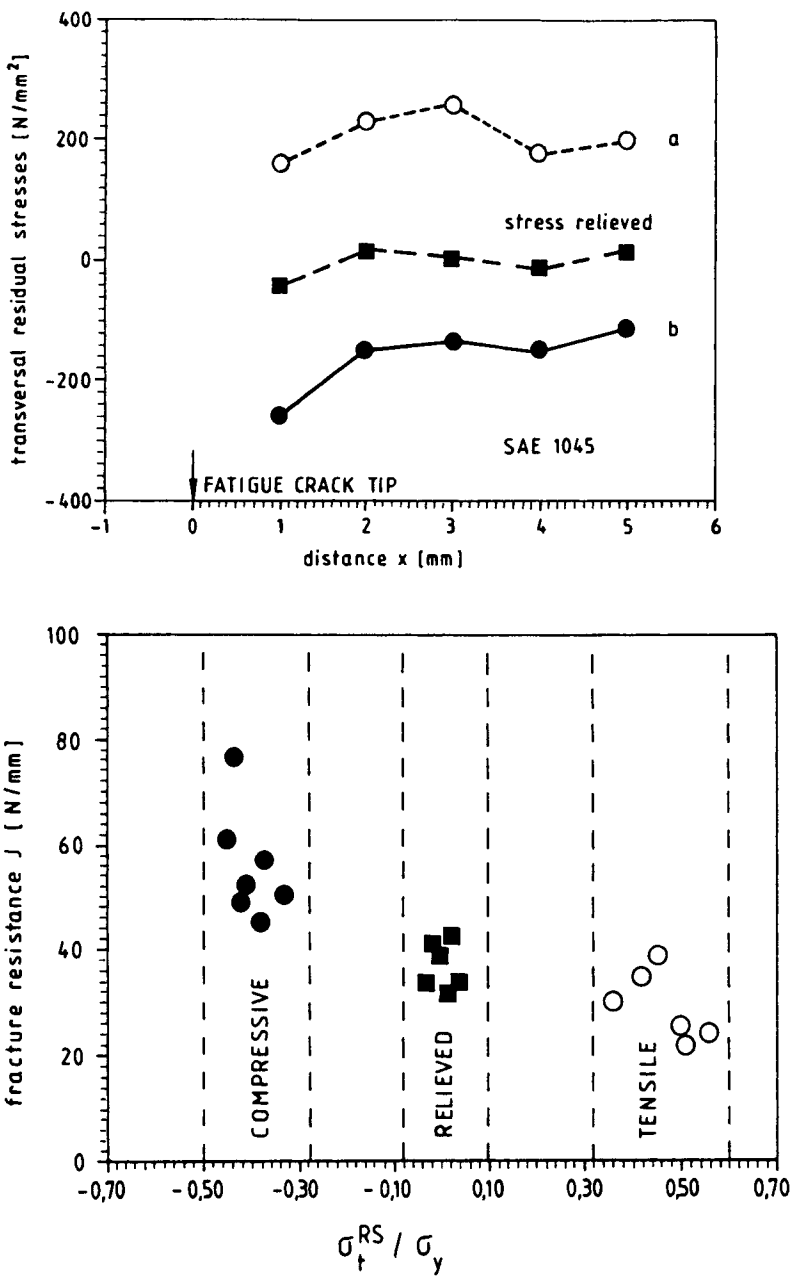


Figure 6.9. Residual stress distributions (above) and resulting influences on fracture resistance J (below) [71].

values δ .

For welding residual stresses in a mild steel, it is shown in [72] that crack opening displacement as well as plastic zone size are larger for crack tips in tensile residual stress zones compared to those in compressive residual stress zones.

Also in the case of elastic plastic fracture mechanics, consequences of residual stresses are theoretically taken into account by a superposition of residual and loading stress effects [73]. This allows the elaboration of adequate procedures for safe constructions with residual stresses in cases, where linear elastic fracture mechanics procedures are no more applicable [74, 75].

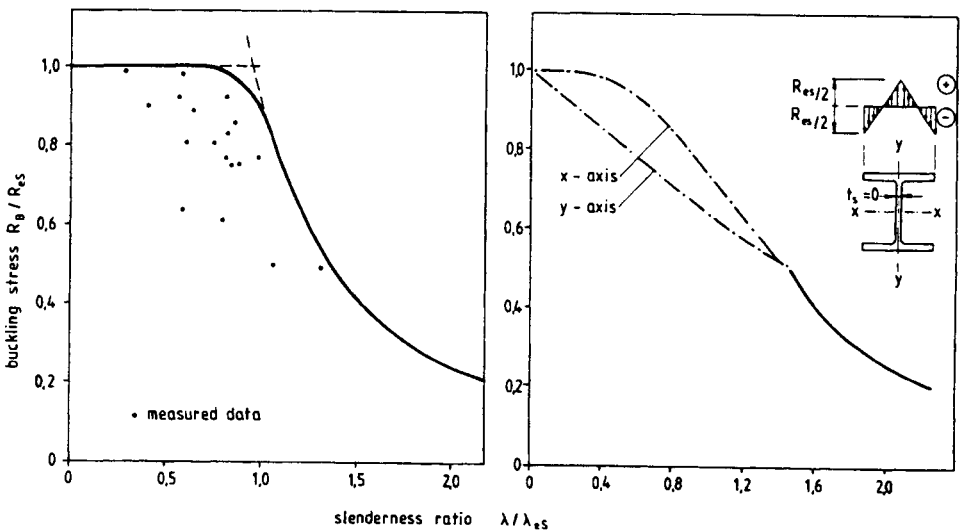


Figure 6.10. Experimentally determined buckling stresses (left) and calculated values, taking the residual stress distribution indicated into account (right) [77].

6.033 Instability

A very important consequence of residual stresses on statically loaded components is their influence on instability or ultimate collapse loads of compression loaded bars, rods, plates or shells [76-79]. A summary of important results in the field of steel constructions can be found in [79]. Most important origins of residual stresses in these cases are manufacturing processes, straightening operations and especially welding processes. Different consequences of residual stresses on instability are possible. First of all, residual stresses are additional internal loads which may diminish bearable external collapse loads. A change of instability failure mode is possible as a consequence of the complex and inhomogeneous loading situation resulting from individual residual stress states. E.g., instead of elastic instabilities, plastic instabilities may occur. Finally, residual stresses can produce geometrical imperfections which, on their part, influence collapse loads.

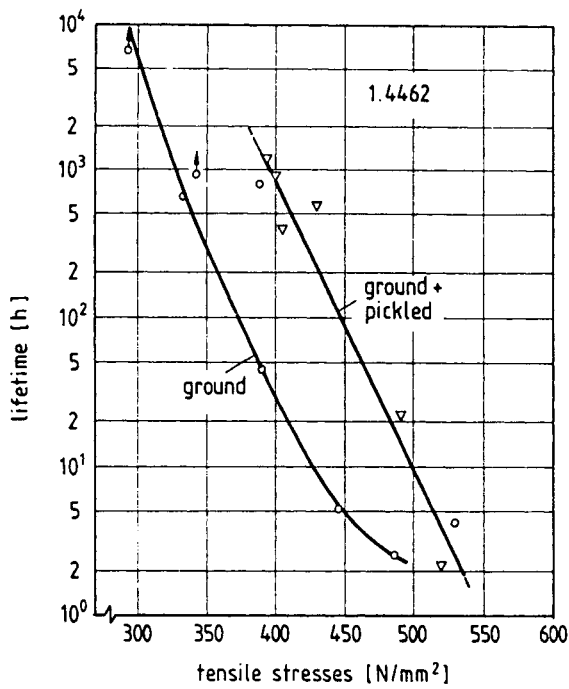


Figure 6.11. Influence of tensile residual stresses on liefetime of differently treated specimens made of steel 1.4462 in MgCl_2 - solution of 125.5° C [85].

There is no general rule for the consideration of residual stress states in the case of instabilities and the assessment of consequences of residual stresses is quite different ranging from negligible to a considerable reduction of collapse loads. In some cases, a great scatter of collapse loads is reported, which leads to undesirable high security factors in design of components with residual stresses. Figure 6.10 gives an example [77]. Different estimations exist to take residual stresses quantitatively into account. This is shown on the right hand side of Figure 6.10. If a simplified residual stress distribution, as indicated, is taken into consideration, satisfying agreement between theory and experiment is found.

6.034 Stress corrosion cracking

In corrosive environments metallic materials like austenitic stainless steels or aluminium base alloys, but also some nonmetallic materials develop cracks as a form of localized corrosion, which is called stress corrosion cracking (SCC). A prerequisite for SCC is, beside the influence of appropriate corrosive media and sensitive materials, the action of tensile stresses, which can be loading stresses, residual stresses or a superposition of both. Basic principles and

different aspects of SSC are outlined in several textbooks (see e.g. [80,81]). Quite a number of cases are reported, where tensile residual stresses together with corresponding environments and materials were responsible for catastrophic failure of components [82]. Therefore, such stress systems have carefully to be avoided, if stress corrosion cracking is anticipated. On the other hand, because all three conditions mentioned have to be fulfilled for SCC, by introducing compressive residual stress fields, the risk of failure can considerably be diminished or even avoided. A great number of examples is given in literature demonstrating especially the positive effects of appropriate mechanical surface treatments in the case of steels and aluminium base alloys [83 - 87]. An example of the behaviour of austenitic/ferritic steel is shown in Figure 6.11 [85]. Ground specimens with surface tensile residual stresses have a considerable shorter lifetime than ground and pickled specimens, where surface layers with tensile residual stresses had been removed by the pickling process.

6.04 Residual stress effects on components under fatigue loading

6.041 Introductory remarks and characteristic observations

There is no doubt that in the case of fatigue loading, consequences of residual stresses are most pronounced. First hints on residual stress effects on strength and lifetime of fatigued components date back to the first third of this century. Because of the outstanding importance of near surface materials states for fatigue loaded components, especially consequences of surface treatments and manufacturing processes were studied [6-12, 88, 89]. A survey about this subject is given in [90].

Characteristic examples, demonstrating the influence of near surface residual stress states on fatigue strength and lifetime are summarized in Figures 6.12 - 6.15. In Figure 6.12 and 6.13 it is clearly shown that in the case of hardened steel, as well as of aluminium base alloy, near surface compressive residual stresses considerably increase fatigue strength compared to specimens with tensile residual stresses or nearly residual stress free ones. For hardened steel Ck 45, this was achieved by different grinding processes using conventional corundum grinding, CBN-grinding as well as CBN-grinding of specimens prestressed in tension (see Figure 6.12) [91]. In this way, extreme differences of longitudinal surface residual stresses of approximately 1600 N/mm^2 could be realized. The consequences on the bending fatigue behaviour, shown in the lower part of Figure 6.12, are obvious. Wöhler curves for 50% failure probability are quite different for the respective grinding conditions and fatigue strengths of specimens with surface tensile and highest surface compressive residual stresses differ by about 350 N/mm^2 . The situation is similar for the aluminium base alloy AlSi 7 Mg in the as cast condition (see Figure 6.13) [92]. The near surface residual stress distributions, resulting from milling and shot peening finishing operations, show small compressive residual stresses for the milled state, but a thick surface layer with considerable compressive residual stresses for the shot peened state. For this condition, highest fatigue strength values were observed in bending fatigue tests.

Also in the case of corrosion fatigue, compressive surface residual stresses have a beneficial effect on lifetime and strength. For shot peened compared to ground, quenched and tempered Ck 45 this is demonstrated in Figure 6.14, together with the appertaining residual stress distributions [93]. Shot peened specimens with a relatively thick surface layer of considerable compressive residual stresses have a higher fatigue strength than ground ones with only small

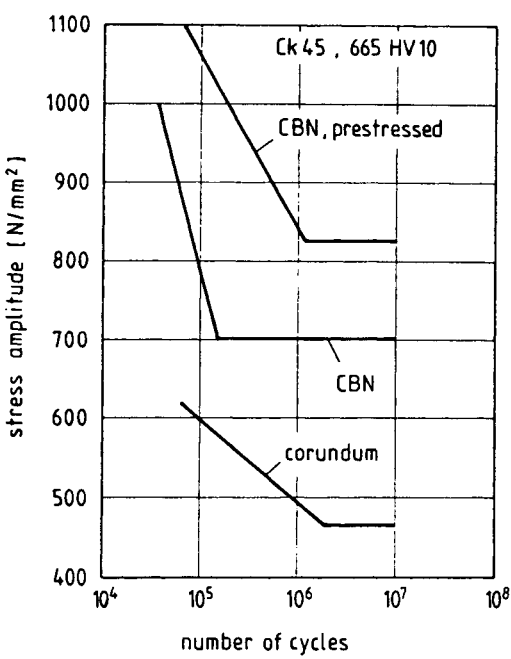
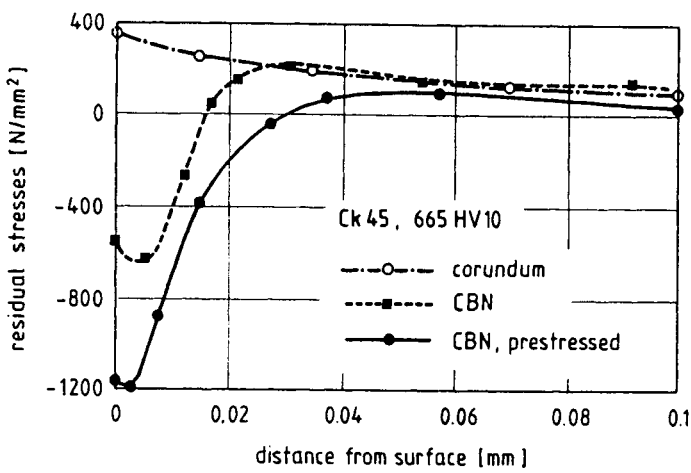


Figure 6.12. Depth distributions of longitudinal residual stresses of differently ground hardened steel Ck 45 (above) and appertaining bending fatigue Wöhler curves (below) [91].

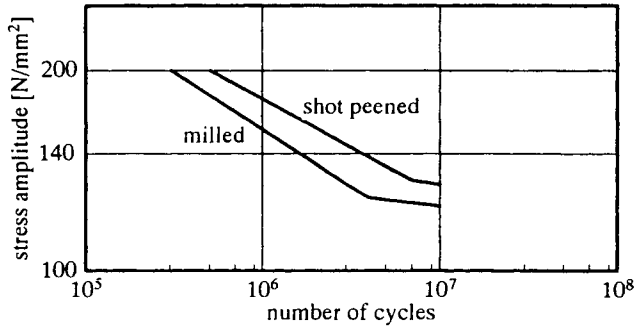
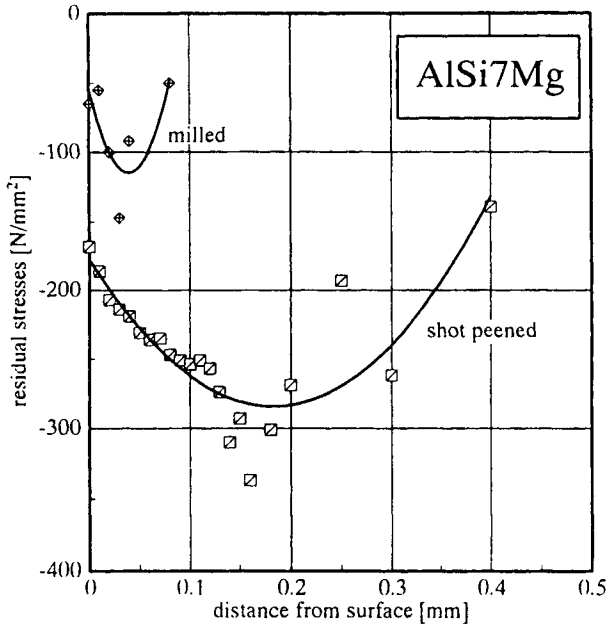


Figure 6.13. Depth distribution of longitudinal residual stresses of milled or shot peened cast Al Si 7 Mg (above) and appertaining bending fatigue Wöhler curves (below) [92].

compressive residual stresses in a thin surface layer.

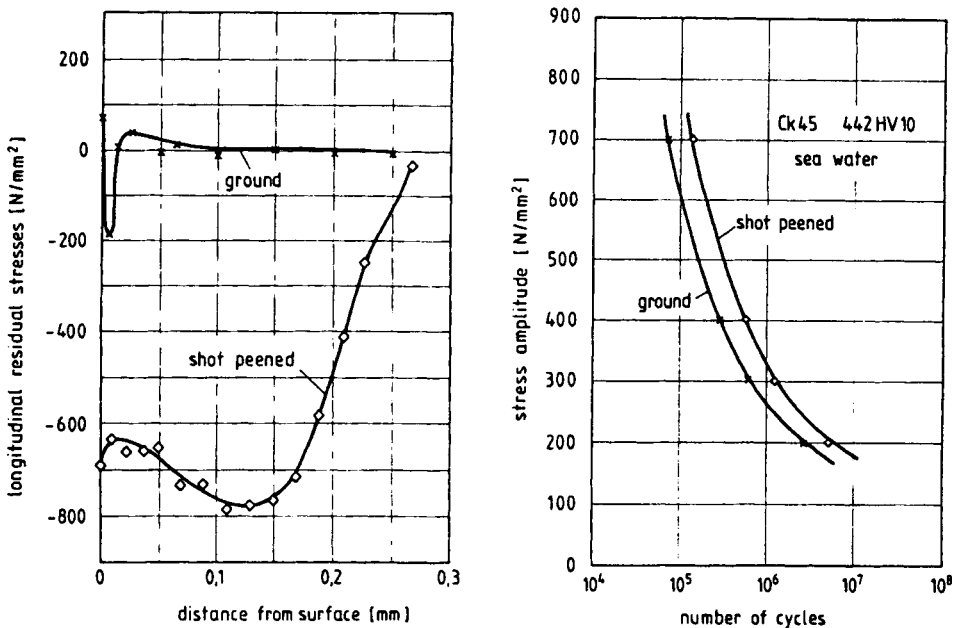


Figure 6.14. Bending fatigue Wöhler diagrams of quenched and tempered Ck 45 tested in sea water and appertaining distributions of longitudinal residual stresses [93].

However, the influence of residual stresses does not always appear as pronounced as shown in Figures 6.12 and 6.13. A classical example is presented in Figure 6.15, which clearly demonstrates that also cases exist, where residual stress influence on fatigue life is negligible [94]. For hardened and differently ground specimens with the surface residual stresses indicated, a clear positive influence of compressive and negative influence of tensile residual stresses on fatigue strength can be stated. However, for normalized materials states, milling residual stresses of different signs do nearly not have any influence on the resulting Wöhler diagrams.

A systematic survey about existing results concerning the influence of residual stresses on fatigue life leads to the following statements: In most cases, compressive residual stresses have a beneficial influence on fatigue strength of components. A maximum improvement of fatigue strength, however, requires optimized residual stress distributions, which can only be achieved, if volumes with highest compressive residual stresses coincide with highest loaded component volumes. This explains, why the influence of near surface residual stress states is e.g. more pronounced for bending fatigue than in the case of tension-compression-loading.

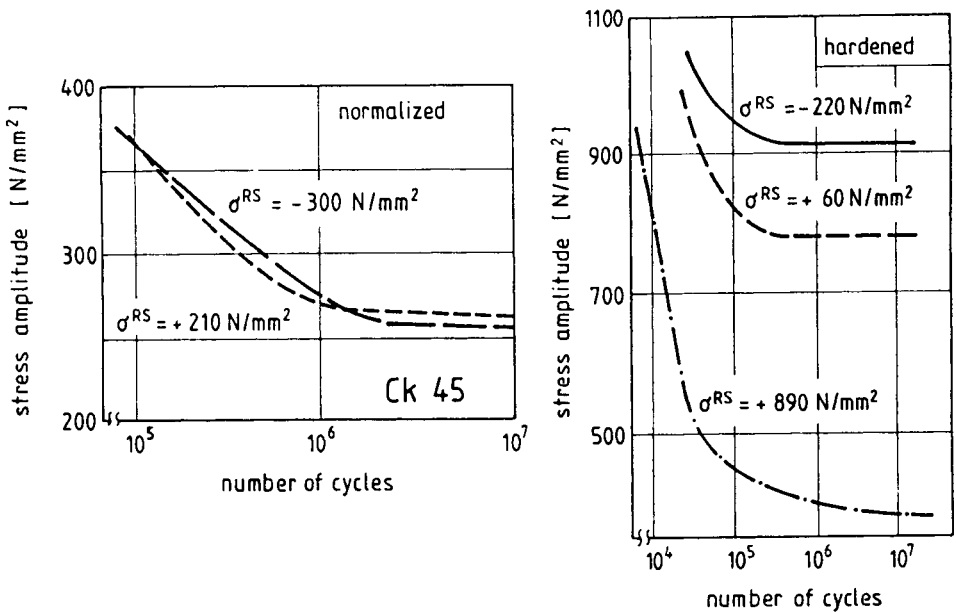


Figure 6.15. Wöhler diagrams of normalized (left) and hardened (right) specimens made of steel Ck 45 with machining surface residual stresses indicated [94].

Positive residual stress effects are, in addition, most pronounced, if detrimental starting conditions exist in near surface layers, such as decarburization or oxidation in the case of hardened steels. It is an important observation that residual stress effects also depend on the materials state under investigation. As one can see from Figure 6.14 for steels, the positive influence of compressive residual stresses on fatigue strength is much more pronounced for hardened materials states than for normalized ones.

In this context the stability of residual stresses is of central importance. It is quite obvious that residual stress effects are the more pronounced, the less stress relaxation effects during fatigue loading take place. Of course, there are two limiting cases: the effect of residual stresses on fatigue strength is negligible, if they relax at the very beginning of cyclic loading. On the other hand, their effect must be fully taken into account, if they remain stable during the whole fatigue life. The stability of residual stresses in fatigue loaded components is determined by the amount of loading stresses, the type of material and materials state, the amount and depth distribution of residual stresses and service temperature. Typical examples are summarized in Figures 6.16 and 6.17. Figure 6.16 shows that, as a function of number of cycles, shot peening residual stresses behave quite different for quenched and tempered compared with hardened steel Ck 45 [95]. In the last mentioned case, for loading stress amplitu-

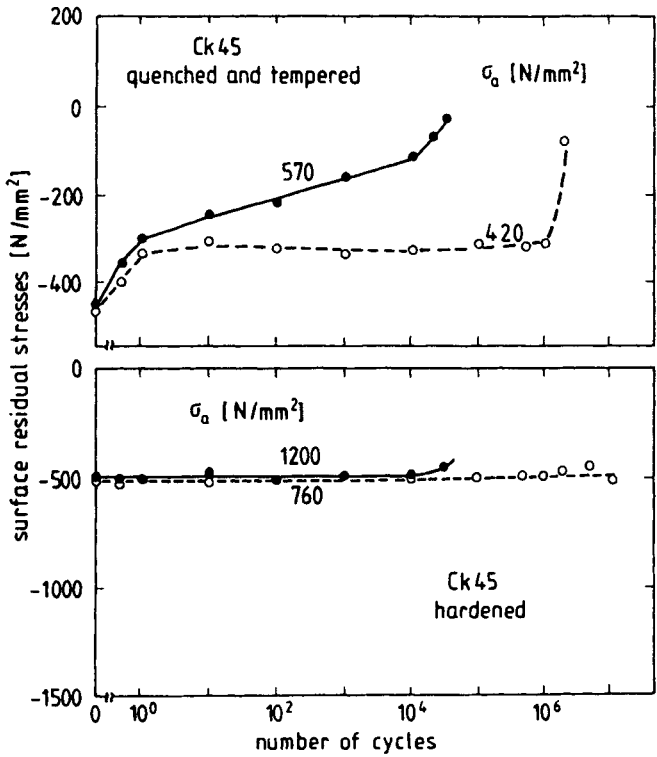


Figure 6.16. Surface residual stresses of quenched and tempered (above) and hardened (below) shot peened steel Ck 45 in bending fatigue with the stress amplitudes indicated [95].

des, leading to numbers of cycles to failure of about 10⁴ and of more than 10⁶, surface residual stresses remain stable, whereas in the quenched and tempered state a remarkable stress relaxation, especially during the first loading cycles, occurs. Similar observations can be made for aluminium base alloys (see Figure 6.17) [92]. While for stress amplitudes leading to numbers of cycles to fracture of 10⁶ and more, residual stresses relax only slightly, stress relief is more pronounced for higher loading amplitudes.

Residual stress relaxation is a consequence of micro and/or macro plastic deformations, replacing elastic strains associated with residual stresses. Consequently, it is of importance, whether the superposition of residual stresses, loading stresses and mean stresses exceeds the monotonic or cyclic yield strength. The fatigue limit of high strength materials states is in general considerably lower than the cyclic yield strength. In the case of low strength materials

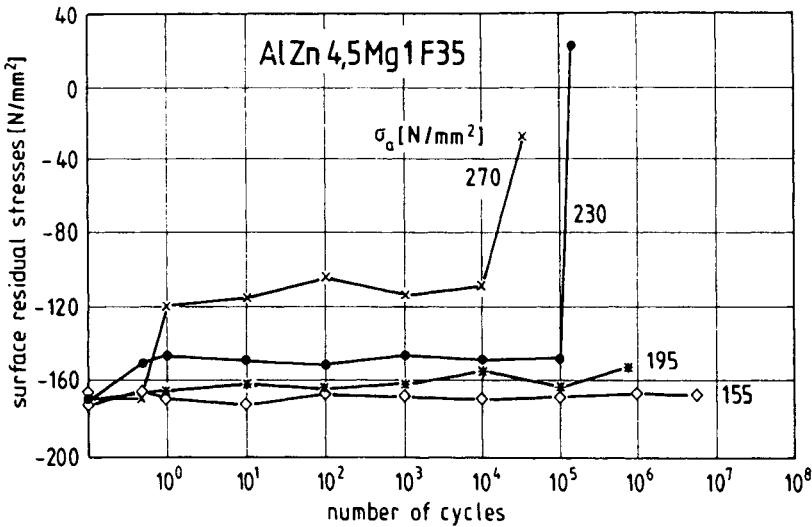


Figure 6.17. Surface residual stresses of shot peened Al Zn 4.5 Mg 1 F 35 in bending fatigue tests with the stress amplitudes indicated [92].

states, both quantities nearly coincide. This explains the fact that residual stresses do not relax in high strength materials states subjected to load amplitudes in the range of the fatigue limit.

Different attempts have been made to model residual stress relaxation processes in fatigue (see [90]). In some cases a linear relationship holds between residual stress relaxation rate and the residual stress amount of the starting condition, except for the first loading cycles and the final stage of life. An example is shown in Figure 6.18 [96,97]. Here, for surface residual stresses in the notch root of a quenched and tempered steel Ck 45, residual stress relaxation rate is plotted for two different notch factors K_t . To a first approximation, machining induced residual stresses are reduced independently of their sign proportional to their magnitudes.

The assessment of the influence of residual stresses on fatigue strength of components is complicated by the fact that other additional influencing parameters have to be taken into consideration. These are changes of local strength, phase content and surface roughness, which are also typical consequences of residual stress producing processes. In a pragmatic way, the influence of a specific manufacturing process on fatigue strength can be estimated quantitatively using a simple additive superposition of individual effects assuming

$$\Delta R_f = \Delta R_f^H + \Delta R_f^{RS} + \Delta R_f^{Ph} + \Delta R_f^R. \quad (6.4)$$

ΔR_f is the entire change of fatigue strength as a consequence of a given manufacturing process with respect to a reference starting condition. ΔR_f^H represents changes of fatigue strength,

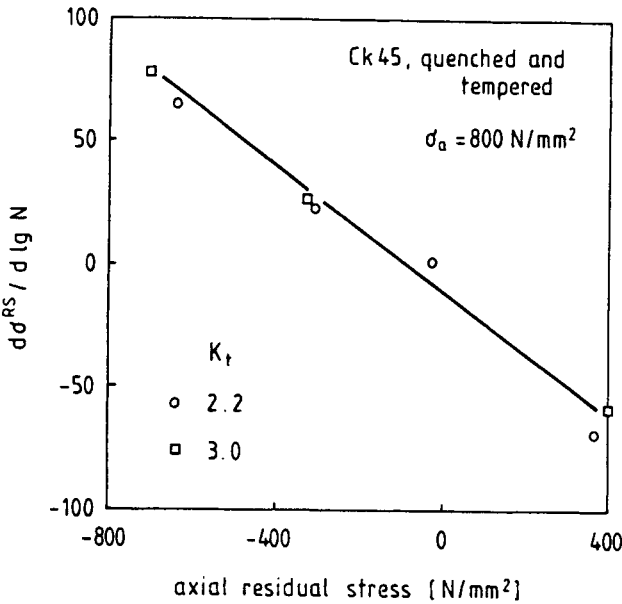


Figure 6.18. Residual stress relaxation rate in tension-compression tests with a stress amplitude of $800 N/mm^2$ in the notch root for quenched and tempered Ck 45 with notch factors $K_t = 2.2$ and 3.0 as a function of axial residual stresses. [96].

attributable to manufacturing induced hardness changes, ΔR_t^{RS} those due to residual stresses, ΔR_t^{Pn} those due to phase changes and ΔR_t^R those due to roughness changes. It has to be pointed out that the extent of each individual effect markedly depends on the state of material under consideration. The influence of surface roughness e.g. is much more pronounced for hardened compared to normalized steels. A detailed survey is given in [90].

Because of the complex consequences of residual stresses on fatigue strength of components, a differentiated consideration of all aspects is necessary. Therefore, in the following paragraphs, consequences of residual stresses on cyclic deformation behaviour, on crack initiation and crack propagation will be discussed separately. Finally, a quantitative assessment of residual stress effects on fatigue strength will be given.

6.042 Cyclic deformation

The cyclic deformation behaviour of components is characterized by a typical development of plastic strain amplitudes in stress controlled tests and of stress amplitudes in strain controlled tests as a function of number of cycles. In stress controlled tests, e.g., stress amplitude and mean stress are kept constant and the evolution of total and plastic strain amplitudes resp. as well as of mean strains is observed. Plots of these quantities versus number of cycles yield

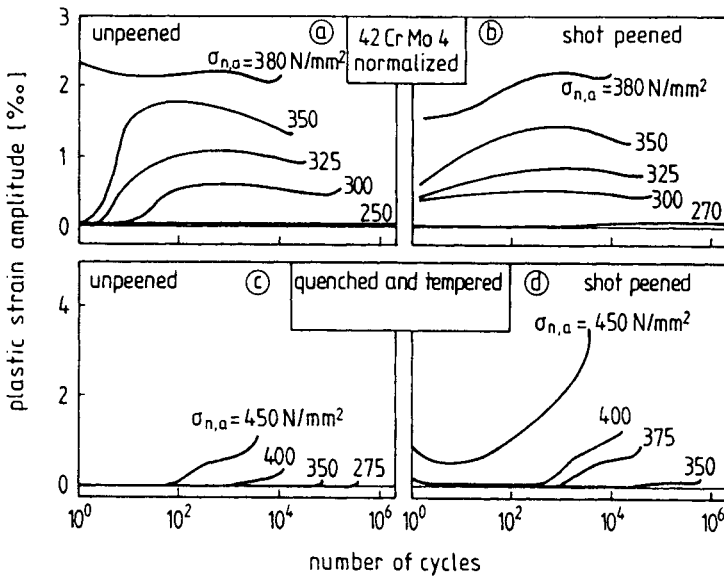


Figure 6.19. Comparison of cyclic deformation curves of normalized (a,b) as well as quenched and tempered (b,c) steel 42 Cr Mo 4 in a shot peened and unpeened condition [98].

cyclic deformation curves and mean strain curves, which allow to detect and assess cyclic hardening or softening effects. Typical examples of cyclic deformation curves are shown in Figures 6.19 and 6.20. In Figure 6.19 cyclic deformation curves of smooth 42 CrMo 4 (SAE 4140) specimens in the unpeened and the shot peened condition for push-pull loading are compared [98]. Obviously, only small differences exist between the unpeened and the shot peened state of the normalized specimens. The shot peened specimens reveal cyclic softening from the first cycle and with the exception of the stress amplitude $\sigma_a = 380 \text{ N/mm}^2$ higher plastic strain amplitudes during the first cycles. For numbers of cycles $N \geq 10^3$ the opposite tendency can be detected and the plastic strain amplitudes of the shot peened specimens are somewhat smaller than those of the unpeened ones. For quenched and tempered 42 CrMo 4 in the unpeened state the characteristic cyclic deformation behaviour occurs with a quasielastic incubation period followed by cyclic softening until crack initiation. After shot peening, onset of cyclic softening is shifted to smaller numbers of cycles. Furthermore, it is interesting to note that for identical stress amplitudes and comparable numbers of cycles, the higher plastic strain amplitudes are always measured for the shot peened specimens. In Figure 6.20 cyclic behaviour of notched specimens in the notch root area is shown [96]. Normalized states of steel Ck45 with notch factor $K_t = 3.0$, different machining induced residual stresses and nominal stress amplitudes $\sigma_{n,a} = 150 \text{ N/mm}^2$ and 220 N/mm^2 have been investigated in push-pull-

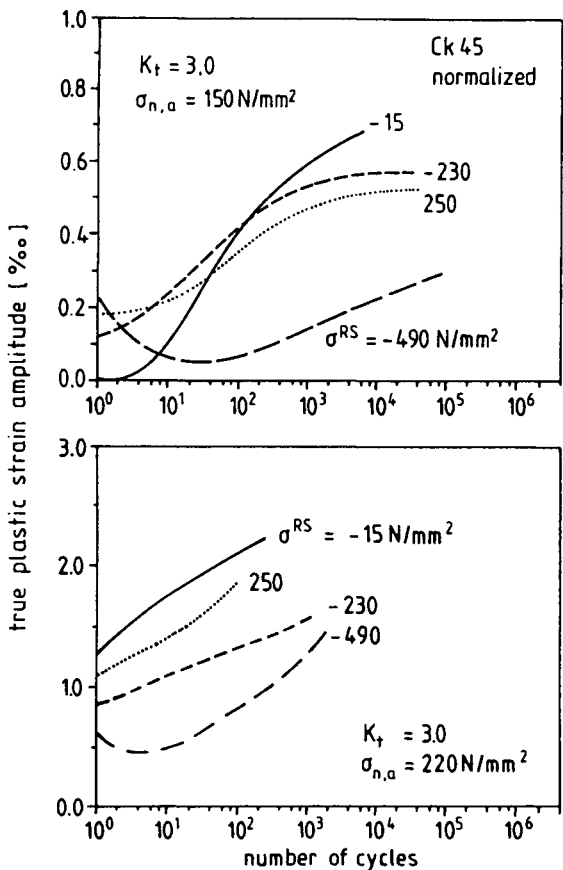


Figure 6.20. Cyclic deformation curves for push-pull tests of notched steel Ck 45 measured in the notch root area for nominal stress amplitudes of $\sigma_{n,a} = 150 \text{ N/mm}^2$ (above) and 220 N/mm^2 (below) [96].

tests. The upper diagram shows that for the nearly residual stress free state as well as for specimens with machining residual stresses, already during the first loading cycles plastic deformations occur, because yield stress is exceeded. It is interesting to note that for compressive residual stresses of -490 N/mm^2 stress relaxation to such an amount occurs that in the following loading cycles only small plastic strain amplitudes are observed. For a nominal stress amplitude of $\sigma_{n,a} = 220 \text{ N/mm}^2$ already during the very first loading cycle considerable plastic deformations occur in the notch root which are the smaller, the higher the amount of machining induced residual stresses is.

One possibility to study residual stress effects is to simulate them by applied mean stresses. If, however, consequences of residual stresses and of mean stresses on cyclic deformation behaviour are compared, it comes out that in cases, where significant plastic deformations and hence residual stress relaxation occur, effects of residual and mean stresses are absolutely not comparable in stress controlled tests. In such cases, simulation of residual stress effects by mean stresses can be achieved more realistically in strain controlled tests with mean strains of a magnitude equal to elastic strains corresponding to the respective residual stresses.

6.043 Crack initiation

Crack initiation occurs as a consequence of microstructural alterations in materials during fatigue loading. Different crack formation mechanisms are discussed [99]. However, only a few cases deal with the influence of existing residual stress fields on crack initiation. This is, of course, also due to problems connected with the detection of very small cracks and the difficult observation of their propagation. In addition, the assessment of the consequences of residual stresses on crack initiation becomes more complicated, because, in most cases, formation of residual stresses is connected with changes of other properties, such as surface topography or local strain hardening effects, which on their part have an impact on crack formation.

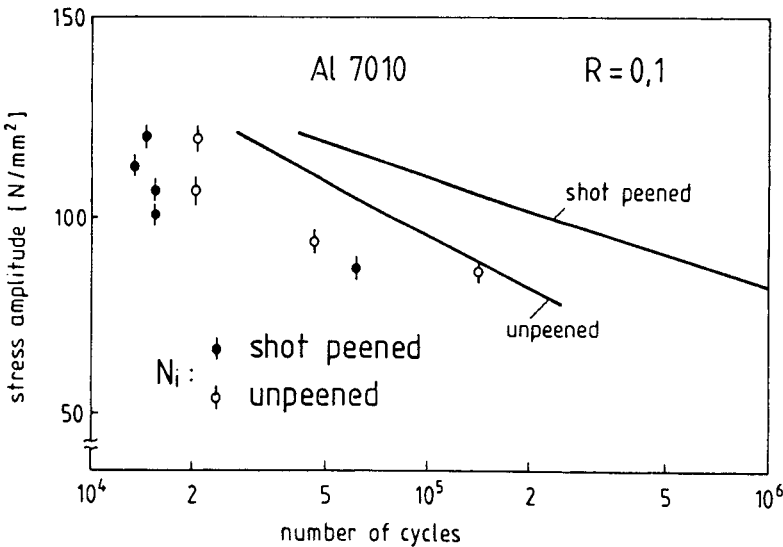


Figure 6.21. Numbers of cycles to crack initiation and to failure for untreated and shot peened Al Zn Mg Cu in bending fatigue tests [100].

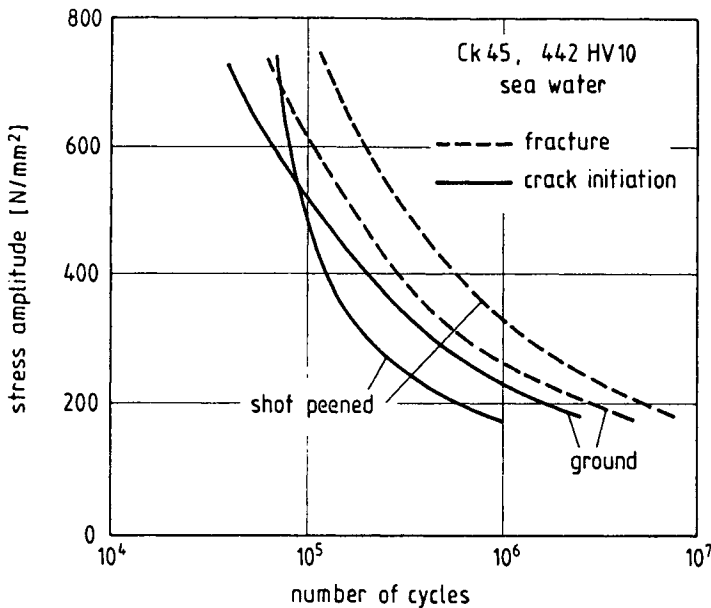


Figure 6.22. Numbers of cycles to crack initiation and to failure for quenched and tempered Ck 45 under bending fatigue loading in sea water [93].

Consequences of residual stress states, both on crack initiation area as well as on crack initiation lifetime have been investigated and discussed. Residual stresses may have a remarkable influence on the location of crack initiation. As will be discussed more in detail in paragraph 6.045, location of crack start can be influenced in a defined way by appropriate residual stress distributions. In the case of bending fatigue e.g., cracks may start below the surface, if sufficient amounts of compressive near surface residual stresses exist. On the other hand, influence of residual stresses on crack initiation lifetime is not so obvious. Typical examples of experimental observations are given in Figures 6.21 and 6.22. In Figure 6.21, numbers of cycles to crack initiation N_i and numbers of cycles to fracture N_f are compared for unpeened and shot peened specimens of the light-weight alloy Al 7010 [100]. Crack initiation times of peened specimens are shorter than those of unpeened ones. Similar observations were made for Ti-6Al-4V in rotating bending loading [101] and also for quenched and tempered steel Ck 45 in the case of bending fatigue tests in sea water [93], (see also Figure 6.14). This is shown in Figure 6.22 for shot peened specimens, where except for high stress amplitudes, cracks start earlier than for ground ones. In all cases mentioned, however, lifetime of shot peened specimens is longer than of unpeened or ground specimens. These observations are attributed to an enhanced crack formation at micro-notches, resulting from the shot peening process, which is supported by corrosion pittings in the case of sea water environment. On the other hand, in-

creased lifetime of shot peened specimens is explained by crack propagation retardation due to shot peening compressive residual stresses. There exist, however, other investigations, where processes accompanied by increased near surface residual stresses lead to increased numbers of cycles to crack initiation, compared with untreated specimens. Such examples can be found in [102-104] for mechanically surface treated steels. Obviously the influence of residual stresses on crack initiation cannot be discussed without taking into account other important parameters. These are surface topographies, in case existing manufacturing induced microcrack distributions and also strain hardening effects and resulting dislocation distributions.

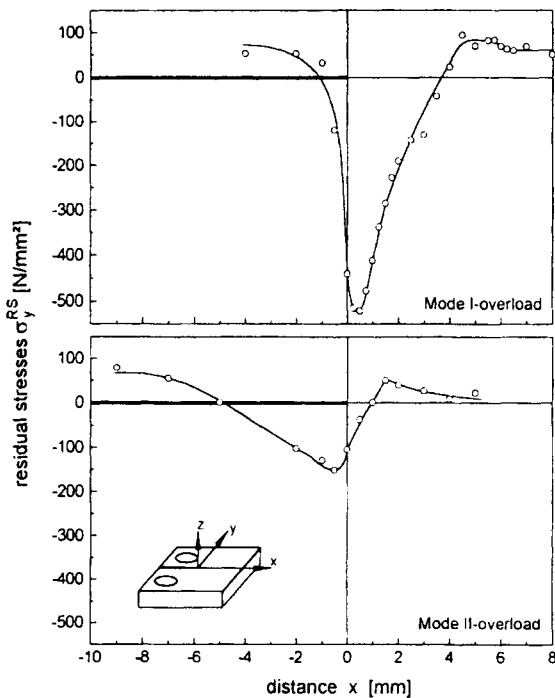


Figure 6.23. Distribution of transversal residual stresses near the crack tip of steel StE 690 after a mode I-base load of $\Delta K = 1500 \text{ N/mm}^{3/2}$ and a mode I-overload of $3000 \text{ N/mm}^{3/2}$ (above) and a mode II-overload of $3000 \text{ N/mm}^{3/2}$ (below) [106].

6.044 Crack propagation

Crack propagation, especially in the case of acting residual stresses, may be the determining factor for the lifetime of components. Propagation of fatigue cracks is treated using

fracture mechanics principles as discussed in paragraph 6.032. Crack velocity da/dN can, except for crack initiation and near fracture stage, be described by

$$da/dN = c (\Delta K)^m. \tag{6.5}$$

Taking mean stress effects and the final crack propagation state into account, among others, also

$$da/dN = c \Delta K^m / [(1-R) K_c - \Delta K] \tag{6.6}$$

is used (ΔK : stress intensity range; c, m : const., K_c : stress intensity factor for plane stress, R :

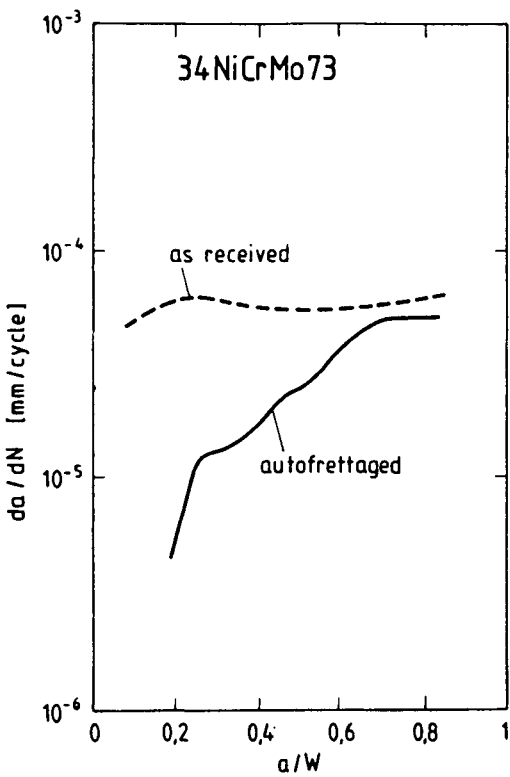


Figure 6.24. Crack propagation rates in an autofrettaged and an untreated tube made of steel 34 Ni Cr Mo 7 3, loaded with $\Delta K = \text{const.} = 632 \text{ N/mm}^{3/2}$ [110].

stress ratio) [105]. Both equations are valid for macro residual stress free materials states. However, it is implicitly included that a propagating crack itself has a characteristic residual stress field around the crack tip. Typical examples are shown in Figure 6.23 for mode I and mode II loaded cracks [106]. In both cases, cracks were produced by cyclic loading with a base load $\Delta K = 1500 \text{ N/mm}^2$. Then, twenty mode I- or mode II-overloads with $\Delta K = 3000 \text{ N/mm}^{3/2}$ were applied. Compressive residual stresses at the crack tip resulted, which are considerably higher for mode I compared with mode II-loading.

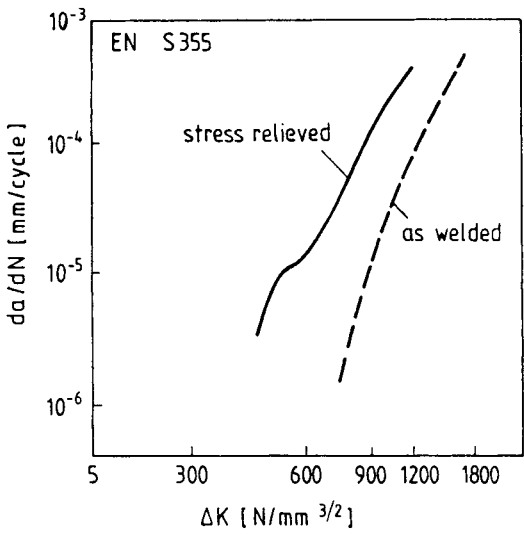


Figure 6.25. Crack propagation rates in heat affected zone of steel E 36 in as welded state and after stress relief heat treatment [109].

There can be found many cases in literature, demonstrating clearly that cracks propagating through existing residual stress fields are markedly influenced by amount and distribution of residual stresses (see e.g. [33, 60, 90, 97, 107-109]). Two examples are given in Figures 6.24 and 6.25. In Figure 6.24 crack propagation rates of an autofrettaged steel tube are compared with those of the untreated base material [110]. In agreement with equ. 6.5, loading with a constant stress intensity range results in a constant crack propagation rate of the residual stress free state. However, if the crack propagates through the triaxial residual stress field resulting from the autofrettage process, considerably smaller crack propagation rates are observed. Figure 6.25 deals with crack propagation in the residual stress field of a weld seam in comparison with that of the stress relieved material [109]. For the welded state, due to the distribution of welding residual stresses, crack propagation in the heat affected zone is considerably lower than in the residual stress relieved state and comparable with that of the

base material. Both examples clearly demonstrate the importance of residual stress fields for crack propagation and, hence, lifetime of fatigue loaded parts with cracks.

Many investigations have been conducted in this field and considerably knowledge has been gathered. The basic principle to understand fatigue crack propagation in residual stress fields and to predict and model the behaviour of real parts is to superimpose stress intensity factors of loading stresses and residual stresses. Following this idea, an effective stress intensity range

$$\Delta K_{\text{eff}} = K_{\text{eff, max}} - K_{\text{eff, min}} \quad (6.7)$$

is valid, if a crack propagates across a macroscopic residual stress field. Here, $K_{\text{eff, max}}$ and $K_{\text{eff, min}}$ resp. denote the maximum and the minimum stress intensities acting during one loading cycle, taking residual as well as loading stresses into account. On this basis, fatigue crack propagation can be predicted using eqs. 6.5 or 6.6. However, as already discussed in the case of quasistatic loading in paragraph 6.032, the possibility of redistribution or relaxation of residual stresses due to crack propagation has to be considered. In addition, crack propagation path can be influenced by existing residual stress distributions. Following eq. 6.7, different cases have to be distinguished.

For

$$K_{\text{eff, min}} = K_{\text{min}}^{\text{LS}} + K_{\text{min}}^{\text{RS}} > 0 \quad (6.8)$$

it follows that

$$\Delta K_{\text{eff}} = K_{\text{max}}^{\text{LS}} - K_{\text{min}}^{\text{LS}} \quad (6.9)$$

Then, an influence of residual stresses can only be expected because stress intensity ratio

$$R_{\text{eff}} = \frac{K_{\text{eff, min}}}{K_{\text{eff, max}}} = \frac{K_{\text{min}}^{\text{LS}} + K_{\text{min}}^{\text{RS}}}{K_{\text{max}}^{\text{LS}} + K_{\text{max}}^{\text{RS}}} \quad (6.10)$$

is also influenced by residual stresses.

If, however, $K_{\text{eff, min}} < 0$ is valid, residual stresses determine ΔK_{eff} as well as R_{eff} . In this case, residual stress effects are more pronounced than in the preceding one. This explains, why for a given R-value, tensile residual stresses may have a less pronounced effect on fatigue crack propagation than compressive ones of the same amount. Usually, it is assumed that

$$\Delta K_{\text{eff}} = K_{\text{eff, max}} = K_{\text{max}}^{\text{LS}} + K_{\text{max}}^{\text{RS}} \quad (6.11)$$

is valid for $K_{\text{eff, min}} < 0$ because negative K-values are neglected with respect to their consequences on crack propagation. There exist, however, some hints that this is not correct in all cases [105]. To successfully apply this method to predict crack growth rates in residual stress fields, the correct crack propagation law of the residual stress free material as well as

correct K^{RS} -values as a function of crack length and also the respective residual stress distributions have to be known.

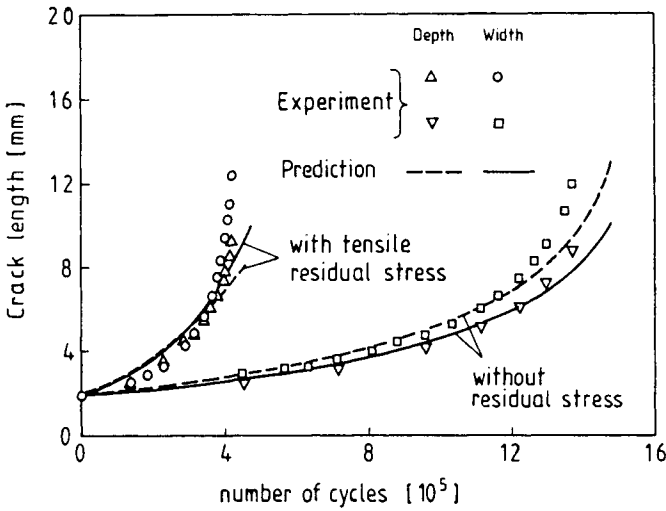


Figure 6.26. Width and depth of cracks propagating in residual stress free and tensile residual stress areas of a low carbon steel ($\Delta\sigma = 188 \text{ N/mm}^2$, $R = -0.5$) [108].

Another way to take residual stress effects in crack propagation of fatigue cracks into account is to study their effects on crack tip opening stress intensity values K_{op} . It is well known that for several reasons, crack opening behaviour is rather complex and correlated in a complicated way with applied loads. A typical observation is that cracks do not open until a certain load level is reached, which is called crack opening load [111]. One reason are residual stress fields, associated with crack tip plastic zones (see Figure 6.23). It is assumed that only that portion of loading cycle, where the crack is open, contributes to fatigue crack growth. Hence, an effective stress intensity range

$$\Delta K_{eff} = K_{max} - K_{op} \quad (6.12)$$

can be determined. If residual stress influence on K_{op} is known, ΔK_{eff} can be used to predict fatigue crack growth. A detailed discussion of problems associated with ΔK_{eff} - determination can be found in [107]. A successful application of this method to predict crack depth and crack width development in a low carbon steel under fatigue loading is shown in Figure 6.26 [108]. If the crack propagates within a tensile residual stress field, crack growth rate is much higher compared to the residual stress free state. In both cases, prediction coincides quite well with experimental observations. Of great practical importance is the fact that, as a conse-

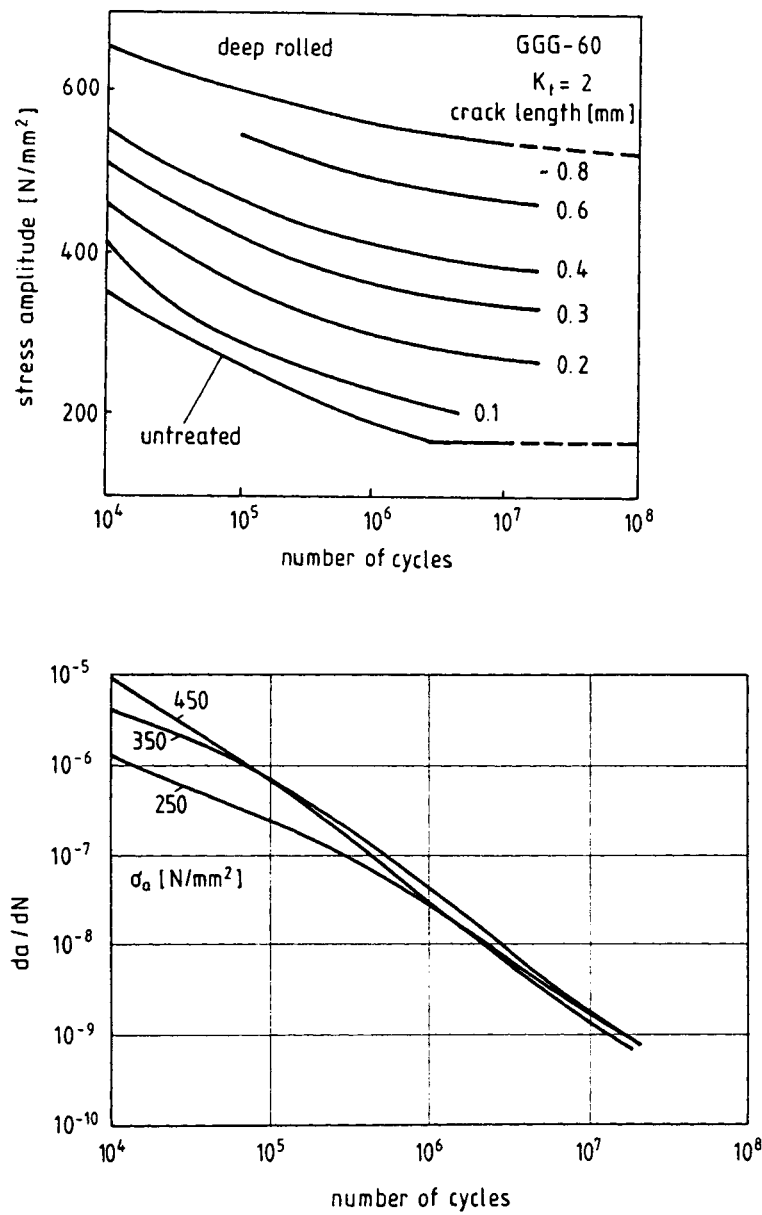


Figure 6.27. Wöhler-curves for fracture and different crack lengths of untreated and deep rolled cast iron GGG-60 and appertaining crack propagation rates for $250 \text{ N/mm}^2 \leq \sigma_a \leq 450 \text{ N/mm}^2$ in bending fatigue tests [112].

quence of existing residual stress fields, ΔK_{eff} can be diminished to such an amount that crack arrest occurs. This is e.g. the case for deep rolled components, where increase of fatigue strength usually is accompanied with nonpropagating cracks. Figure 6.27 shows Wöhler-curves, valid for different crack lengths of deep rolled notched cast iron GGG-60 in comparison with untreated specimens [112]. One can see that a remarkable strength increase due to deep rolling is only achieved if cracks longer than approximately 0.3 mm are admissible. Crack propagation rates are very low, decrease with increasing crack lengths and are more or less independent of stress amplitude for the loading conditions mentioned (see Figure 6.27, below).

6.045 Fatigue strength of components with residual stresses

If consequences of macroscopic residual stresses on fatigue strength and lifetime of components have to be assessed, it is fundamental to treat residual stresses in the same way as mean stresses. Thus, residual stress influence can be expressed in a pragmatic way by

$$R_t^{rs} = R_t - m\sigma^{rs} \quad (6.13)$$

with R_t^{rs} : fatigue strength of component with residual stresses; R_t : fatigue strength of component without residual stresses, m : specific residual stress sensitivity. If residual stresses have identical consequences as mean stresses, m is identical with mean stress sensitivity M and can be estimated using the Goodman-relation

$$R_t^m = R_t (1 - \sigma_m/R_m) \quad (6.14)$$

with R_t^m : fatigue strength of component taking mean stress effects into account. Then

$$M = R_t/R_m \quad (6.15)$$

is valid. In [113], the simple relation

$$M = HV/1000 \quad (6.16)$$

between hardness HV of the material under investigation and mean stress sensitivity M is proposed. In Figure 6.28 the scatter band of experimentally observed mean stress sensitivities M for steels of different hardness is shown, together with relations proposed by [113 - 115]. Similar diagrams exist for aluminium base alloys demonstrating that their mean stress sensitivity in general is somewhat higher than that of steels.

Despite the fact that residual stresses have to be treated as mean stresses, residual stress sensitivity in fatigue is quantitatively not identical with mean stress sensitivity. The main reason for that is the different stability of residual stresses in the respective cases and has been described in paragraph 6.041. In addition, it has to be kept in mind that distribution of existing residual stress fields in general is quite different than that of applied mean stresses. Therefore, individual residual stress sensitivities have been determined for different cases, reflecting especially the respective stability of residual stresses, which depends on materials and loading conditions. A survey about existing data in literature shows that for metallic materials

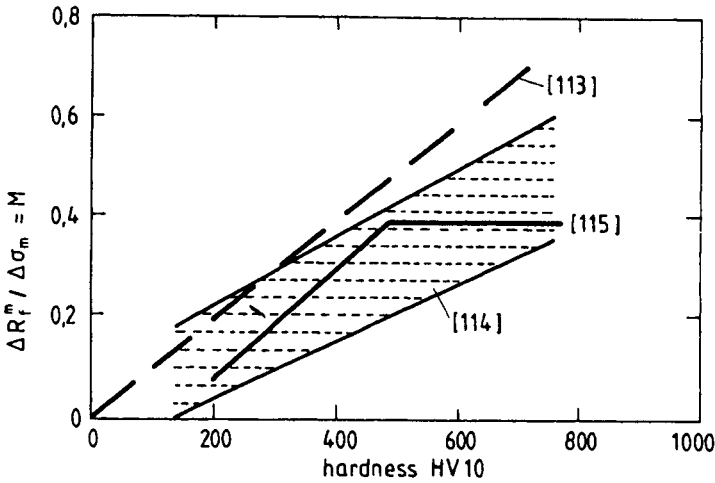


Figure 6.28. Mean stress sensitivity M for steels of different hardness.

influence of materials properties on residual stress sensitivity m is similar to that on mean stress sensitivity M . However, a clear tendency exists that m is smaller than M . Figure 6.29 shows data valid for bending fatigue tests of steels with different hardness, taken from [114]. Also data determined by [91] are plotted. In addition, residual stress sensitivity, following [113], is plotted, which is identical with mean stress sensitivity in Figure 6.28. Obviously, this tends to be an upper limit of experimentally observed values.

The use of an appropriate residual stress sensitivity allows to take residual stress effects on fatigue strength and lifetime quantitatively into account. It has, however, to be pointed out that in this way the complicated individual processes preceding fatigue failure are summarized in a very pragmatic way. For a quantitative prediction of residual stress effects on strength and lifetime of fatigue parts, especially the influence of surface topography and of strain hardening effects have to be separated from residual stress effects. This has, up to now, only been achieved in a few cases (see e.g. [94, 95]). Even then, individual distribution and relaxation behaviour of residual stresses is only globally reflected by m -values. A remarkable progress would be achieved, if well founded correlations between residual stress depth distributions, residual stress relaxation and residual stress sensitivity values m could be established. This needs, however, further research work.

From the fact that residual stresses can be treated like mean stresses, it follows that their consequences can be represented analogous as mean stress diagrams. As an example, in Figure 6.30, a classical Haigh-diagram of a quenched and tempered steel Ck45 with 420 HV 10 is shown. In addition, experimentally determined fatigue strength values are plotted as a function of the respective acting longitudinal surface residual stresses [91]. Differently ground as well as shot peened states were taken into consideration. As expected, due to stress relaxation and

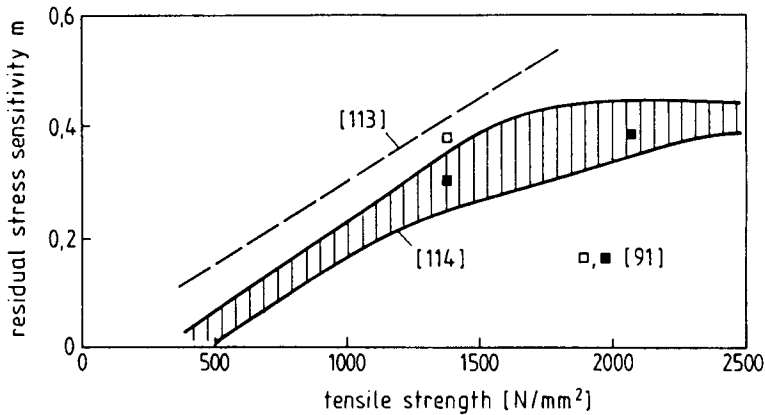


Figure 6.29. Residual stress sensitivity m for steels of different ultimate tensile strengths.

and also due to surface roughness effects, values observed and also residual stress sensitivity is smaller than predicted by the Goodman relation. This explains also why the ground specimen with highest compressive residual stresses yields a data point well beyond the limit of the Haigh diagram.

Considerable effort has been made to theoretically predict fatigue life and strength of components quantitatively, taking also residual stress effects into account. The basic principle is the assumption that total fatigue life is composed of a fatigue crack initiation period and a fatigue crack propagation period, which are treated separately. Crack initiation period is treated by modern concepts using accurate descriptions of local elastic-plastic cyclic materials behaviour in critical component volumes, e.g. in notch root area. The cyclic stress-strain relationship of the material under consideration has to be known for that purpose. Using appropriate assumptions or FE-calculations, the stress-strain path of critical volume elements is calculated. Finally, by the aid of correct damage considerations, crack initiation lifetimes or Wöhler-curves can be found. If an appropriate hypothesis is used, also cumulative damage for variable amplitude loading can be determined. A survey about the concepts used is given in [116-118]. Existing residual stress distributions are taken into account by attributing appropriate residual stress values to characteristic volume fractions of the components under consideration. For surface treated components, in [119, 120] appropriate models are described. The applicability of the models is demonstrated for nitrided as well as for carburized specimens.

An interesting variant of the concepts mentioned above is the local fatigue strength concept, which allows to predict quantitatively the consequences of residual stress distributions on crack initiation areas as well as on fatigue strength. It has particular advantages in the case of thermally, mechanically or thermo-chemically surface treated

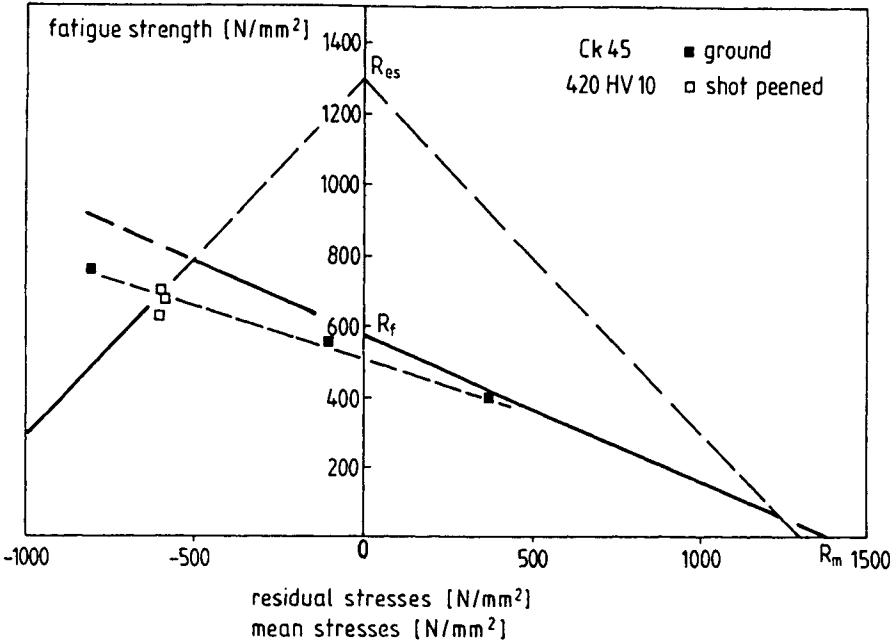


Figure 6.30. Haigh-diagram of a quenched and tempered steel Ck 45 with 420 HV 10, together with experimentally determined fatigue strength values as a function of surface residual stresses.

components. The starting point of the considerations is a modification of equ. 6.13 or 6.14 taking the depth-dependencies of all quantities into account. This yields

$$R_i^{RS}(z) = R_i(z) \cdot (1 - \sigma^{RS}(z) / R_m(z)) \tag{6.17}$$

or

$$R_i^{RS}(z) = R_i(z) - m\sigma^{RS}(z). \tag{6.18}$$

Depth distributions of ultimate tensile strength $R_m(z)$ and fatigue strength of residual stress free state $R_i(z)$ can be estimated using appropriate correlations with measured hardness depth distributions (see e.g. [113]). In this way, the locally effective fatigue strength, resulting as a consequence of inhomogeneous materials properties and characteristic variations of residual stresses, can be determined. In Figure 6.31 for a quenched and tempered shot peened and a ground steel resp. the resulting depth distributions of fatigue strength are plotted [91]. The scatter bands result from assumed variations of residual stress sensitivity. As a consequence of

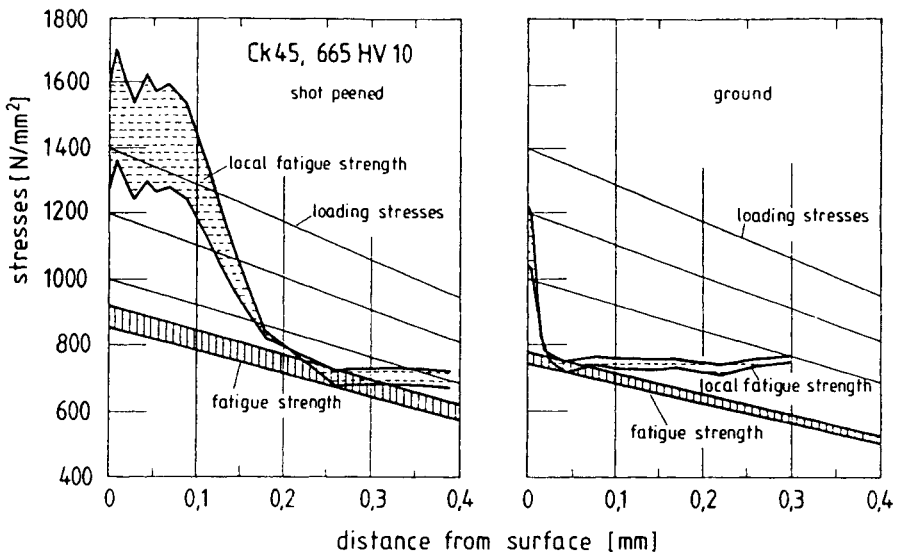


Figure 6.31. Local fatigue strength as well as loading stress distributions for shot peened (left) and ground (right) hardened steel Ck 45 [91].

the thick surface layer with shot peening compressive residual stresses, high local fatigue strength values up to a surface distance of about 0.1 mm result, which then decrease to values characteristic for materials volumes, which have not been affected by the shot peening process. On the other hand, for the ground state, only a very thin surface layer with increased fatigue strength results. In both diagrams, for the case of bending fatigue loading with different loading stress amplitudes, loading stress distributions are plotted. It is assumed that fatigue strength of the material under consideration is determined by the maximum loading stress amplitude which in any cross section reaches the local fatigue strength values. The corresponding distributions are also plotted in Figure 6.31. An important observation is that in both cases loading stresses exceed local fatigue strength values below the surface when the stress amplitude is increased. That means that crack initiation should occur in these areas, which has meanwhile experimentally been observed in many cases (see e.g. [90]). Another important consequence, which can be drawn from the observations in Fig. 6.31 is that the amount of surface compressive residual stresses is not decisive for the resulting fatigue strength, when crack initiation below the surface occurs. Maximum fatigue strength values, however, are only achieved, when residual stresses are introduced into components in such a way that their depth distributions correspond to the depth distributions of loading stresses.

6.05 Residual stresses and failure analysis

Because of the enormous impact of manufacturing processes and loading history on the resulting residual stress states, they have also to be taken into account in the case of failure analyses. Investigating residual stress states of used parts in many cases allows to assess and to control the type and quality of manufacturing processes applied and to detect defects introduced. To some extent, the loading history and the failure process itself can be determined, if measured residual stress distributions are correctly assessed [123-125].

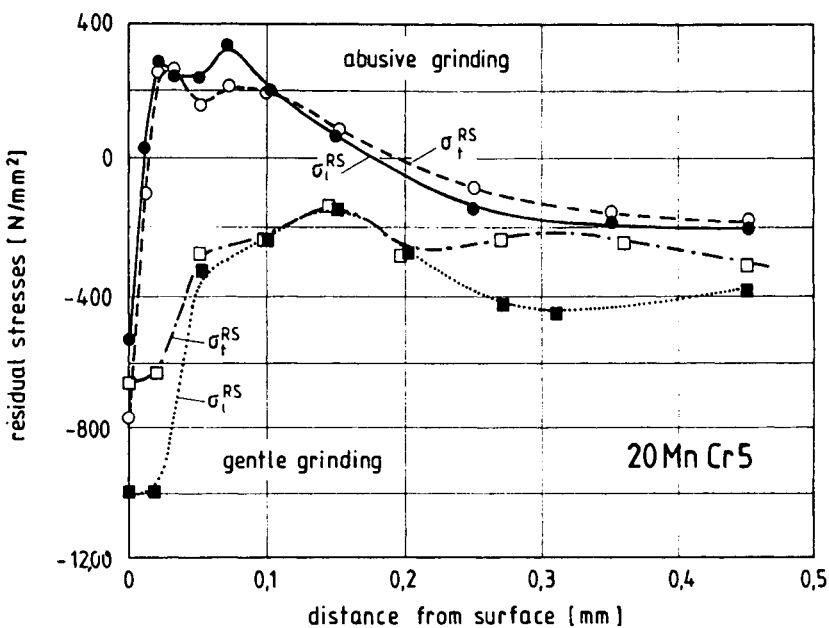


Figure 6.32. Depth distributions of grinding residual stresses for abusive and gentle grinding of case hardened steel 20 Mn Cr 5.

About the clear correlations between manufacturing processes and parameters applied on the one hand and resulting residual stress distributions on the other hand, considerable knowledge has been worked out in the past and numerous investigations exist which can be referred to (see e.g. [90, 125, 126]). Very often, residual stress analyses allow to detect manufacturing induced defects which reduce the strength or lifetime of components. A typical and important example is abusive machining, in particular abusive grinding. This can occur, if unfavourable process parameters are used or in the case of poor cooling conditions. As a consequence, considerable near surface tensile residual stresses can be created, sometimes together with a thin annealed or nearly hardened surface layer. In Figure 6.32 depth

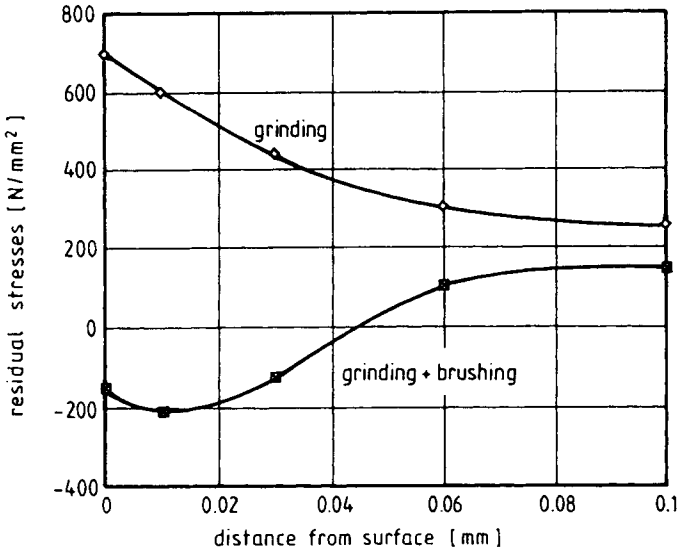


Figure 6.33. Depth distribution of residual stresses after different mechanical surface treatments of a low carbon steel.

distributions of residual stresses of a correctly and an abusively ground tooth of a gear are shown. It is important to note that below the surface residual stress distributions are considerably shifted towards tension, whereas immediately at the surface, in both cases, compressive residual stresses occur.

Sometimes residual stress measurements allow to detect unknown manufacturing steps originally not planned in the manufacturing sequence. For strength and lifetime of components consequences of such additional or wrong manufacturing steps very often are not known or incorrectly assessed. Figure 6.33 shows that a manual grinding process with a portable grinding machine, which is e.g. used as a refinishing treatment of weld seams, can introduce considerable tensile residual stresses in the near surface layers. In this case, an additional shot peening or power brushing treatment is recommended to avoid tensile and introduce compressive residual stresses in near surface layers.

In the case of heat treatments, unexpected near surface residual stress distributions may indicate microstructural anomalies, such as oxidation or decarburization effects in relatively thin surface layers. Typical examples can be found in [127].

In many practical cases, residual stress measurements are used to get indirect information about loading state and loading history of component. In this case, existing knowledge about origin and causes of residual stress is used to draw conclusions about loading history and processes applied during the lifetime of the component under consideration. Typical examples can be found in the field of bearing components. Because of the extensive knowledge existing

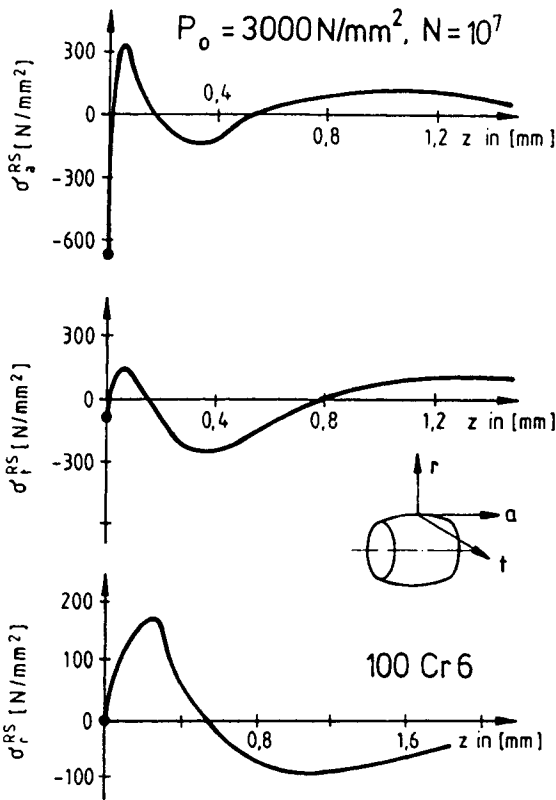


Figure 6.34. Depth distributions of axial, tangential and radial residual stresses in steel 100 Cr 6 after 10^7 rolling cycles with $p_o = 3000 \text{ N/mm}^2$ [130].

about the amounts and distributions of the multiaxial residual stress states resulting from rolling or sliding contacts under well defined contact conditions, experimentally determined depth distributions of residual stresses allow statements about the loading conditions during service of rolling elements [128,129]. A typical example of uniaxial residual stress distributions in bearing rollers after 10^7 rolling cycles is shown in Figure 6.34 [130].

Also straightening processes, which are very often applied during the manufacturing procedure of e.g. axles, shafts etc. to compensate distortions during heat treatments can be detected by appropriate residual stress analyses. The same is valid for other plastic deformation processes, such as presetting of springs.

As a final example, it should be mentioned that residual stress values measured on crack surfaces of fatigue cracks can provide useful indications about the cyclic loading conditions.

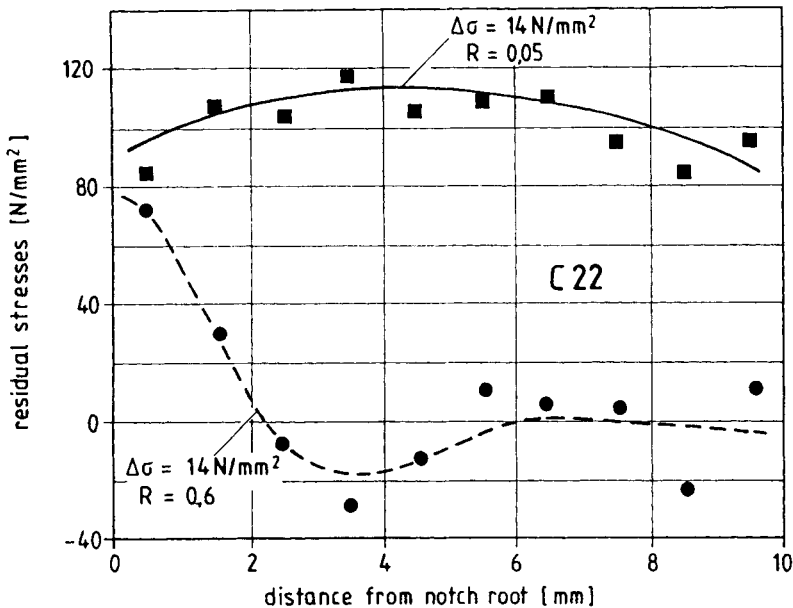


Figure 6.35. Residual stresses measured on the surface of fatigue cracks, produced with steel Ck 22 and the loading conditions indicated [123].

As can be seen in Figure 6.35 for CT-specimens cyclically loaded with $\Delta\sigma = 14 \text{ N/mm}^2$ and stress ratio $R = 0.05$, tensile residual stresses exist on the crack surface, whereas for $R = 0.6$ nearly residual stress free crack surfaces are observed due to the different closure behaviour of the cracks during the loading cycles [123].

6.06 Recommendations

Despite the fact that knowledge about origin and assessment of residual stresses has enormously increased in the past, there remain quite a number of open questions and unsolved problems. In many cases, clear correlations between production processes and process parameters on the one hand and resulting residual stresses on the other hand have been worked out. Therefore, residual stress analysis might be an excellent tool for quality control. A great number of examples is gathered in literature on this subject and can be studied. However, residual stress analyses are time consuming and methods to allow in-process measurements are missing or not sufficiently reliable. Continuous efforts are therefore necessary to improve measurement techniques to allow for quick and precise measuring procedures.

From the examples shown in this chapter, it becomes obvious that not only surface values, but complete depth distributions of residual stresses are necessary for correct assessments. In ad-

dition, it has properly to be determined what type of residual stress exists, because consequences of macro and micro residual stresses have to be quite differently estimated for the behaviour of materials and components. In this context, much effort is necessary to elaborate standard procedures to allow for a correct comparison of residual stress data determined by different measuring methods.

While a great amount of knowledge exists about manufacturing induced residual stresses, the stability of micro and macro residual stress distributions for given temperature cycles or under load as a function of loading history can only unsatisfactorily be described. This knowledge, however, is a necessary prerequisite to assess correctly the consequences of residual stress states for the behaviour of materials and components. Some promising estimations exist. But, especially for mechanically loaded parts, it would be desirable to have clear correlations between the cyclic behaviour of the material under investigation and the development of residual stress distributions in the course of fatigue loading.

Finally, the quantitative assessment of residual stress distributions with respect to their consequences on strength and lifetime of components has to be further elaborated. In this context, additional theoretical work is necessary to incorporate residual stresses in design codes, but also experimental experiences are desirable for the validation of theories. In all cases a close collaboration is recommended between research groups dealing with measurement or modelling of residual stresses and correlated aspects of materials science and technology as well as manufacturing, production and quality engineers.

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| - influence on micro- | 584f. | | |
| magnetic parameters | | | |
| - ODF | 53,296f.,303 | | |
| - polefigure | 53,168,219 | | |
| - rolling | 169f. | | |
| thermal | | | |
| - expansion coefficient | 62,273,533 | | |
| - stress | 273 | | |
| time-of-flight technique (TOF) | 500f.,534f. | | |