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Lifetime Controlling Defects in Tool Steels

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Christian Rudolf Sohar

Lifetime Controlling Defects in Tool Steels

Doctoral Thesis accepted by
Vienna University of Technology, Austria

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Supervisor's Foreword

Tool steels are used in tools for cutting or forming processes, but also in engine components, e.g. for diesel direct injection. These applications involve cyclic mechanical stresses, very high numbers of loading cycles being encountered, e.g. during the life of an engine. These materials are high alloy steels, which after heat treatment reach a service hardness of 60 up to 70 Rockwell C hardness combined with high strength and toughness, bending strength levels being $>3,000$ MPa. The lifetime of products made of tool steel grades is mostly limited either by wear at the tool surface—e.g. blunting of cutting edges—or by fatigue processes within the tool, i.e. by fracture. In the latter case it is of decisive importance to study the relationship stress amplitude—service life cycles right up to $>10^9$ cycles, i.e. into the “gigacycle” range, and to identify the crack-initiating features within the microstructure.

The present thesis describes an interdisciplinary approach to investigate the fatigue behavior of differently manufactured tool steels in the gigacycle range, including materials science, metallurgy, chemistry, physics and mechanical engineering. An ultrasonic frequency resonance testing system was employed operating at 20 kHz, and the experimental setup was modified specially for high strength, low-ductility materials. This enabled reliable fatigue testing up to very high loading cycle numbers in reasonable time, 10^{10} cycles being attained in 5–6 days, compared to about 8 years in standard fatigue testing (for a single test!). S–N (“Wöhler”) curves, depicting applied loading stress amplitude versus loading cycle number to failure, were taken for cold work tool steels and high speed steels. All of them showed a steadily decreasing slope: thus the existence of a true fatigue limit, i.e. a stress level below which fracture does not occur at all, which is still frequently claimed to hold for steels, could be clearly disproved, at least for the tool steels studied here. However it showed that correct fatigue data are only obtained if residual stresses in the specimens are avoided; it was found that compressive stresses, which are easily introduced into the specimen surface during preparation, strongly affect the fatigue stress levels.

Furthermore, it could be shown that fatigue testing up to very high loading cycle numbers (up to 10^{10}) is well suited for identifying crack nucleating

micro-constituents in the material. In conventional wrought tool steels, produced by ingot metallurgy, large primary carbides and carbide clusters, which are regular constituents of the material and are responsible for the high hardness and excellent wear resistance of the material, were found to act as crack nucleation sites and thus to be responsible for fatigue failure. In powder metallurgy (PM) tool steels, in contrast, relatively rare nonmetallic, mainly oxidic, inclusions caused fatigue failure, since due to the special production route the primary carbides are too small ($<5\text{ }\mu\text{m}$) to initiate fatigue cracks. Generally the fatigue endurance strength for powder metallurgy tool steels was found to be at a level almost double that of equivalent steels produced by ingot metallurgy. This indicates that PM tool steel grades are particularly well suited for applications where fatigue loading dominates.

Typically, the level of the S–N curves depends only on the manufacturing route but not on the type of steel. PM tool steels show virtually the same S–N graphs as PM high speed steels of markedly higher hardness, and the same holds for ingot metallurgy steels, though at a lower stress level. This underlines that the fatigue behavior of the tool steels is defect-controlled and not matrix-controlled, which implies that in the case of gigacycle fatigue loading, tool steels show a behavior very similar to that observed for structural ceramics in static loading. Thus, the present thesis greatly contributes to the understanding of the mechanical behavior of metallic materials with complex microstructure and high hardness.

Vienna, August 2011

H. Danninger

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I dedicate this book to my family, in gratitude for their unlimited support during my school and university education.

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Chapter 1

Introduction

1.1 Tool Steels

1.1.1 Tool Steels: General Overview

Tool steels are defined as “any steel” that is “used to make tools for cutting, forming, or otherwise shaping a material into a part or component adapted to a definite use” [1]. Despite this definition, large quantities of tool steels are also used for non-tool applications, e.g. for springs, engine parts, bearings and magnetic components, since they offer excellent mechanical properties. The earliest tool steels were plain carbon steels, and only because production processes and technologies improved, it was possible to develop more and more highly alloyed steels with better properties. The history of tool steels will be covered in the subsequent section of this thesis. Nowadays, most tool steels contain large quantities of alloying elements, which range from carbide forming elements like molybdenum, tungsten, vanadium, and chromium to others like manganese and cobalt. The purpose of the alloying elements in the tool steels is the improvement of the mechanical properties in order to meet the ever increasing service demands of these steels, and to provide better dimensional control during the applied heat treatments. The influence of the main alloying elements on the steel chemistry and the resulting properties will be discussed later in more detail. The major quantity of produced tool steels are cast and wrought, i.e. ingot metallurgy products. After the casting and remelting, processes such as forging, swaging, hot or cold rolling are required in order to attain smaller inclusion and carbide sizes, which are a prerequisite for appropriate mechanical properties, and to achieve the desired dimensions of semi-finished products. These processes exhibits one major drawback: Primary carbides and impurities become elongated in the rolling direction. Thus, mechanical properties reveal strong material anisotropy.

In contrast, tool steels produced by means of powder metallurgy (PM) eliminate this major drawback of wrought tool steels. PM tool steels offer structural uniformity and also very low primary carbide sizes—below 5 μm —which influences

the mechanical properties in a very positive way. Furthermore, there is the possibility of obtaining steels with chemical compositions that are not accessible by the conventional ingot metallurgical pathway since these compositions often cause segregation-related hot workability problems in conventional ingot processes, which advantage of PM allows tuning of desired properties. The use of PM tool steels has become more and more attractive for toolmakers due to the excellent mechanical properties, despite the higher material, esp. alloying cost. However, the higher prices are compensated for by longer lives of the tools.

For success in application, attention must be drawn to four major points: First, appropriate steel has to be chosen from the numerous variants of existing tool steels in order to meet the application requirements. Secondly, the steel needs a careful production and subsequent inspection granting reliability in-service. Furthermore, the design of the tool component is a rather critical point. Inappropriate geometries of the part could lead to failure during heat treatment processes or to fatigue failure during service due to areas of high stress concentration. Finally, appropriate heat treatments, which are required to achieve the desired working properties, have to be adjusted to the type of steel and its designated application, and they have to be conducted carefully.

In the following, some remarks on the historical developments of tool steels, the different production processes, fundamentals of heat treatment processes, the effect of major alloying elements, and classification and applications of tool steels will be presented in order to support basic knowledge about tool steels.

1.1.2 Historical Aspects of Tool Steels

In contrast to nowadays, where less than 1% of the produced steels are tool steels, the earliest usage of steels was exclusively for tools [2–6]. Prehistoric forges and smelters indicate that the extraction of iron ores was practiced even then. In the Great Pyramid a fragment of an iron tool was found, which was about 5,000 years old. About 900 BC. Homer described the hardening of steel weapons, and Aristotle reported about the manufacture of “Wootz Steels” of India to be performed since 350 BC by remote mountain people in the north of India. They charged iron with finely divided wood or green leaves into a small crucible and sealed it with clay. The crucible then was heated several hours and the product was a small lump of uniform steel. The famous “Damascus Steel” was first manufactured in its name giving Syrian city around 300 AD, and was later brought to Europe by the crusaders in the eleventh to twelfth century. In Toledo, Spain, the “Damascus Steel” was then copied, which resulted in the “Toledo Steel”. These steels differed from the Wootz Steels in such a way, that thin strips of steel alternated with thin strips of soft iron was welded, twisted and wrought together, which resulted in a characteristic patterned appearance of the steel sword and knives.

Interestingly, these ancient production processes were lost during the dark ages. However, probably the reason for this disappearance was that the amounts of steel,

which could be made by these methods, were too small, and the processes too costly. In the seventeenth century the cementation process was introduced, in which iron is heated in contact with charcoal. The product was carburized iron that was hardenable. Due to the blisters on the steel surface this steel is also referred to as “blister steel”. The major disadvantage of this steel was the heterogeneity resulting from carburization.

In 1740 Benjamin Huntsman, a British spring manufacturer and clockmaker, rediscovered the melting of steel, and thus, eliminated the heterogeneity of the blister steel. It was referred to as “Crucible” or “Cast” steel. In the following the manufacture of crucible steel became an art and many secrets were handed down from father to son. The next major development in the field of tool steels was the discovery of the so-called “Mushet Steel” by Robert Mushet in the mid-nineteenth century. He suggested for the first time to add manganese to Bessemer steel, but also tried the addition of other alloys to steel. He found that the addition of tungsten resulted in a self-hardening steel. This steel, now known as Mushet Steel, contained about 2%C, 2.5%Mn, and 7%W, and is referred to as “self-hardening” steel. However, it is noted here that Mushet was not the first who conducted experiments with alloying elements for steel. Especially, chromium and tungsten were investigated in this respect. Famous Faraday and Stodart were the first investigators (1820) dealing with the addition of chromium to iron and steel. Faraday thought at this time that the addition of chromium would confer the steel beneficial hardness desired for tools and other uses due to its very hard nature. He thought that chromium might take the place of carbon, which however, was not the case since chromium per se does not confer hardness.

Berthier, animated by Faraday’s work, conducted numerous experiments in 1821. For example, he found that the presence of iron facilitated the reduction of chromium oxide. He made ferro-chromium containing up to 60%Cr. He used the ferro-chromium for the production of chromium steel containing 1 and 1½%Cr. However, the only special quality he stated was the damascening. The steel exhibited silvery whiteness. Knives and razors made of this chromium steel were reported showing good quality. It is noted that no comment was given about the carbon content of the steel, so that it is impossible to figure out the properties of Berthier’s chromium steel.

And once more it was Mushet who proposed the first commercial application of chromium to iron and steel. He suggested to heat lumps of pig, cast, or refined iron to just below of the melting point, then pulverize them along with powdered wolfram or tungsten, or powdered chrome ore, or oxide of chromium. Julius Baur (New York) introduced the manufacture of chromium steel on a practical scale in 1865 and took a patent on his invention. In the following years he introduced further improvements of the process, and also an improved method for the making of ferro-chromium. Baur found what Berthier has reported just some 50 years before, that direct alloying of chrome ore with the iron or steel was too uncertain. Instead, ferro-chromium has to be employed to achieve reliable properties of the chromium steel. It is very likely that the reason for the great success of “his”

New York Chrome Steel Company was the fact that this company produced their own ferro-chromium.

In the beginning of the nineteenth century the Spanish brothers De Elhuyar reported that tungsten with pig iron formed a dense greyish-white hard and brittle combination. First attempts to produce tungsten steel were made in 1855 by Jacob, owner of tungsten mines in Austria and Dr. Koeller at Reichraming. The two of them obtained patents in France for the production of tungsten steels in 1856. Mayr, of Leoben, Austria, produced tungsten steel on a commercial scale about this time, which was stated to be of same quality as Krupp's steel. Between 1898 and 1900 Taylor and White discovered that if self-hardening steels, such as the Mushet steel, get quenched (e.g. in oil bath) just below their melting point phenomenal cutting ability was attained. These experiments were regarded totally absurd at that time, because most researchers thought that such a treatment would ruin any tool steel. However, the Taylor steels represent the basis for the production of the so-called "high speed steels". Taylor also recommended tempering of these steels at about 600 °C, however, for a long time tempering was not performed in practice. It was also known at this time that chromium can be substituted for manganese improving the self-hardening behavior of the steels, and molybdenum for tungsten. However, the price of molybdenum was too high at this time. Thus, molybdenum was not used commercially at that time. Further improvements were achieved by the addition of vanadium, which was reported for the first time around 1905. Vanadium contents ranging from 0.15 to 0.35% improved the red hardness, the endurance of the tools and enabled higher cutting speeds. However, at this time it was found that vanadium content above 0.3% did not improve the steel. In contrast, hardness suffers a significant decrease due to high amount of ferrite, if the amount of carbon is too low, which was not known at that time. Thus, vanadium was originally used as scavenger to remove slag impurities and to reduce the amount of nitrogen through vanadium nitride formation. However, in the 1930s it was found that it is possible to add more vanadium if simultaneously the carbon content gets adjusted. In the beginning of the twentieth century high speed steels also containing cobalt were produced. It turned out that cobalt increased the cutting performance, especially at high temperatures since it enhances the red hardness of the steel. However, cobalt was very expensive at that time and, thus, not used extensively.

Further major developments were driven mainly by the two world wars. During World War I tungsten high speed steels were tried to be replaced by high chromium high carbon steels, especially in France due to the lack of alloying elements (mainly tungsten) available at the constrained market. However, these steels showed insufficient resistance against high temperatures, but offered high wear resistance and crushing strength in cold work applications, which represent the main application field of this steel type until nowadays. Similarly, during World War II molybdenum was substituted for tungsten in high speed steels in Great Britain due to the tungsten shortage during this time. However, a major problem of the usage of Mo was its tendency to decarburization during the heat treatments. This was solved in the 1930s by a coating with borax, and practical more important by the

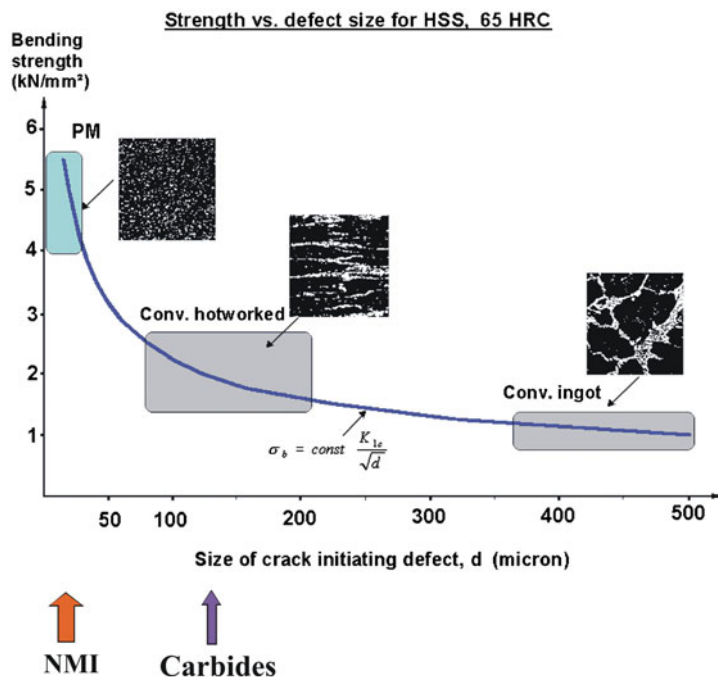


Fig. 1.1 Relationship between defect size and bending strength as obtained for as-cast, cast and conventionally hot worked and PM tool steels (with courtesy of Böhler Edelstahl GmbH&Co/ Böhler Uddeholm Powder Technology, Kapfenberg, Austria, personal communication)

introduction of salt bath and controlled atmosphere furnaces that inhibited the decarburization. This enabled the broad use of molybdenum during World War II.

More recently, steel refinement processes such as electro slag remelting (ESR) aimed to reduce the amounts of impurities in the steel, so that in today's conventionally made tool steels the defects limiting the mechanical properties are predominantly the primary carbides (Fig. 1.1).

Really significant new developments of tool steels evolved with the upcoming of the application of the powder metallurgy (PM) for tool steel production during the late 1960s. The idea behind was that the only way to reduce the segregation during solidification was to speed up the rate of cooling which implies smaller ingots, i.e. sizes of approximately 1 mm. Thus, only powder metallurgy was the key to this problem. As early as 1959 water atomized HSS powder was canned and hot extruded to bars by BSA in England. Similar experiments were performed at Allegheny—Ludlum in the USA. At IIT Research Institute in the USA HSS powder was inert gas atomized and quenched in a water reservoir, i.e. the atomization occurred in a humid atmosphere. The canned powder was heated and hammer forged and rolled. However, useful products were not attained by the

above mentioned methods, since nonmetallic inclusions detrimentally affected the mechanical properties.

The breakthrough was achieved by combining dry inert gas atomization for producing nearly oxide free powders with hot isostatic pressing (HIP) of the canned powders. This process was developed simultaneously, but independently in the USA by Crucible Material Corporation (Crucible Particle Metallurgy process—CPM) and in Sweden by Stora Kopparberg and ASEA (ASEA-Stora Process—ASP) and revealed very low amount of nonmetallic inclusions. Thus, these PM tool steels offered excellent mechanical properties.

Elimination of segregation and a very fine microstructure with a uniform distribution of primary carbides are major advantages of the PM tool steels, resulting in superior mechanical properties. Figure 1.1 underlines the significant improvement of bending strength by the PM technology compared to conventional hot worked tool steels, which directly results from the significant smaller defect sizes: the primary carbides are very small, i.e. $<5\text{ }\mu\text{m}$. Thus, instead of the primary carbides nonmetallic inclusions are the decisive factor in crack initiation. However, as described above the ASEA-Stora process reduced the amount of inclusions significantly and thus, solved this problem. This will be discussed later in more detail. Furthermore, the PM offered high flexibility of alloying, since conventional methods were severely limited in respect to carbon, nitrogen, and alloying metals content due to segregation-related hot workability problems. Thus, the production of really new tool steels with tuned properties became possible by the PM pathway, which reduced the segregation.

The presented historical development of tool steels has shown that many improvements were based on the introduction of new alloying elements and of heat treatment and production processes. Thus, in the following the influence of alloying elements on the steel properties will be discussed in detail, and basic principles of heat treatments and their impact briefly described.

1.1.3 Manufacturing Processes

1.1.3.1 Conventional Metallurgy

At first, raw materials, mostly steel scrap and pig iron have to be carefully selected in order to avoid too high contents of impurities, i.e. undesired elements such as nickel, cobalt, and copper, which cannot be oxidized out of the melt [2, 7]. Composition of the scrap should have lower percentage of the various elements than it is required in the final steel, so that the final composition can be adjusted by controlled addition of ferroalloys and carbon. Furthermore, the distribution of the alloying elements within the melt should be homogeneous, in order to avoid heterogeneous products. Commonly, the raw materials are melted in an electric arc furnace, which has usually a rather low mass capacity. Siemens invented this melting process in laboratory scale in 1878. But, due to lack of electric power

supply at that time it took another two decades until Heroult installed the first commercial electric arc melting plant for steel production in 1899. Despite the development of several other arc furnaces, the Heroult furnace remained the most important furnace until nowadays. The furnace consists of a shell including the hearth, walls, and roof, and the three working electrodes. The hearth has to be made of basic magnesite or burned dolomite in order to withstand the highly corrosive basic lime slags. The walls and the roof must withstand the enormous heat but they need not to be basic since there should be no direct contact with the slags. Thus, silica bricks can be used which is more cost efficient than the magnesite material. Locally in-between the arc and the metal bath temperatures up to 8,000 °C can be reached.

After the meltdown of the scrap, several refining stages are performed in order to lower the content of impurities and carbon, if required. First of all, removal of oxidizable elements such as carbon, phosphorus, manganese, or chromium is carried out by introducing oxidizing agents to the slag or by lancing oxygen into the furnace. In order to decrease the oxygen content of the steel melt it is deoxidized through addition of ferrosilicon, aluminum, or aluminum–silicon, calcium–silicon or calcium–magnesium alloys. Insoluble oxides such as SiO_2 , Al_2O_3 , and CaO are formed which get absorbed in the slag. Sulfur reacts with CaO and forms CaS , which gets also absorbed in the slag. Compositional adjustments are made through addition of ferroalloys and carbon through crushed graphite electrodes, carbon briquets, or carbure. However, refining and compositional adjustments can also be performed in separate refining processes such as argon oxygen decarburization (AOD), vacuum arc degassing (VAD), or vacuum oxygen degassing (VOD).

Either ingot casting or continuous casting is applied to form steel ingots. However, continuous casting is seldom applied since segregation and cracking, due to the high alloy content and wide solidification range, impose problems. In order to achieve higher cleanliness of the tool steels special refining such as secondary remelting process (e.g. electro slag remelting, ESR) can be applied. Subsequently, the (refined) ingots undergo several hot working processes, which can be forging (hammering, swaging) and/or rolling. The purposes of these operations are reduction of the ingot size, breaking up the eutectic carbide networks in the as-cast tool steel, redistributing of segregates, and to some extent welding small defects in the ingot. Due to the high alloy content and high amount of primary alloy carbides in the steel, thorough control of the forging and rolling operations is required, especially concerning the temperature, the applied forces, and probable decarburization and oxidation. The semi-finished products then have to be soft annealed, which means heating to temperature in the austenite-carbide two phase field and subsequent slow cooling. Through this, soft ferrite microstructure with dispersed, spherodized carbides is obtained that is reasonably machinable. Furthermore, annealing provides microstructural uniformity which is essential for subsequent hardening heat treatments, as will be described later in more detail.

1.1.3.2 Powder Metallurgy (PM)

PM has become a very important manufacturing route for high-performance tool steels [1, 6, 7, 8, 9]. Major advantages are the possibility of alloying high amounts of carbide forming elements, which is not accessible by conventional ingot metallurgy (IM), since on solidification of the ingot carbide segregation and formation of large networks of eutectic phases would occur. PM technology eliminated these problems since here segregation can only take place within every single particle (“micro-ingot”) when a homogeneous melt is atomized into small droplets, i.e. on an extremely limited scale. Furthermore, also the obtained primary carbide sizes are significantly smaller compared to those observed in IM tool steels. There is a large variety of possibilities for powder production, shaping, and consolidation. In contrast to pressed and sintered PM steel precision parts, tool steels must have a fully-dense, pore-free microstructure in order to yield the extraordinary mechanical properties, since every remaining pore would act as stress raiser and initiate material failure. Thus, only PM production pathways that ensure fully dense structure can be applied for PM tool steel manufacturing. Today’s commercial basic production routes are mostly based on the ASEA-Stora process [10, 11] developed in the 1960s; they start with atomization of a steel melt containing the desired amounts of alloying elements. The steel melts can be obtained in an EAF or an induction furnace. Two main possibilities for the atomization exist: In gas atomization, a melt stream is disintegrated into droplets by gas jets (commonly nitrogen) and solidify quickly in flight, while the cooling agent in water atomization is water.

Gas atomized powders, which consist of spherical particles, are further encapsulated in cans of mild or stainless steel in order to achieve the required pressure difference to consolidate the loose powder particles during the *hot isostatic pressing* (HIP). Optionally, the filled cans can be pre-pressed isostatically to increase the contact area between the particles, which reduce the costly time of preheating to HIP temperature significantly. During preheating the powder is vacuum degassed. Then, the evacuated and gas-tight welded can is inserted into the pressure vessel of a hot isostatic press (HIP unit) and consolidated for up to 3 h at temperatures between 1,100 and 1,200 °C and at pressures up to 1,000 bar. Under these conditions the powder particles pressure-sinter together and form a fully dense tool steel billet. This billet is subsequently hot worked. Before the introduction of the ASEA-Stora process, nonmetallic inclusions coming into existence during the powder production were responsible for not acceptable mechanical properties of as-HIPed tool steels, since these usually rather large inclusions, that showed sizes similar to carbide dimensions in conventional hot-worked tool steels, were sites for crack initiation. However, the ASEA-Stora process solved this problem mainly by the usage of inert gas as cooling medium. This HIP process represents the most important production pathway for PM tool steels.

Water atomized powders are usually used for the production of tools with complex shape. In water atomization the liquid metal stream is atomized by water

jets and collected in a water pool. However, major drawback of this method is the high oxygen content of the powder particles (up to 3,000 ppm). It forms an oxide shell at the particle surface of a thickness of about 200 nm. The major advantage of water atomized powders is the angular shaped particle geometry, which enables mechanically compaction and vacuum sintering, since the green parts have sufficient mechanical stability for further handling. However, the microhardness of water atomized particles ranges from 500 to 1100 HV 0.1, which depends predominantly on the cooling rate. Thus, in order to be able to compact these particles and also to remove the oxide layer to some extent, soft annealing of the powder, commonly under vacuum atmosphere, is performed prior to powder compaction. After cold pressing, the shaped green parts are consolidated by *vacuum sintering*. Sintering temperatures ($\sim 1,250$ °C), required to achieve full dense tool steel parts, are about 100 °C higher than the temperatures applied during hot isostatic pressing (HIP) due to the lack of elevated pressure. However, the sintering temperature has to be adjusted very precisely, since at too high temperatures coarsening of the microstructure would occur. In contrast, in case the applied sintering temperature is too low, undesired porosity would remain in the material. Thus, the sintering to full density represents a rather critical step in this process, in particular since the optimum sintering temperature is also affected by the carbon content, i.e. temperature control and carbon control are both essential.

Historically, “pseudo-HIP” processes, which comprise *compaction at atmospheric pressure* (CAP) or the *STAMP process* were invented in order to save cost of the HIP. However, the costs of HIP devices and procedure decreased significantly over the course of time so that the pseudo-HIP processes do not find large appliance nowadays. Nevertheless, a brief description of CAP and STAMP follows: the *compaction at atmospheric pressure* (CAP) process uses gas atomized powders, which are coated with a thin film of boric acid (H_3BO_3). It acts as sinter activator in such a way that it deactivates the oxide surface layer at the powder particles during sintering within a glass mould at about 1,200 °C under atmospheric pressure through which densities of 95–99% are reached. However, it is unclear how the sinter-activation reaction products affect the mechanical properties of the obtained steel since inclusions very likely are formed during this process. In the *STAMP process* gas atomized powders are canned, preheated and inserted into a pressure chamber of a hydraulic press, filled with a deformable, thermally stable, powdered material, which is pressurized. Full density of high speed steel billets can be obtained within 5 min time. The cost advantage of the STAMP process is a result of avoiding the expensive HIP autoclaves, which however does not apply nowadays.

An important process, for which gas atomized powders are the starting point, is the *Osprey process*, which is also referred to as *controlled spray deposition* (CSD), *spray compacting* or *spray forming* [12]. In this process, liquid metal is directly converted to a homogeneous solid of refined structure without any intermediate process step. Atomization resembles the gas atomization powder production. However, the material is collected at a rotating substrate before full solidification does occur. The obtained structure is free from macro segregation, has a fine and

uniform grain size and is potentially capable of properties equal or superior to powder metallurgy HIP products [13, 14]. The whole process is performed within the atomization chamber, where a clean atmosphere exists, which is important for the rather defect-free welding of the layer by layer deposited steel droplets. The density that can be achieved by this method ranges from 95 to 99%. Thus, hot working is required to attain full density.

Powder hot forging and *powder extrusion* are two further processes that can also be applied in tool steel production. However, powder forging is not used on commercial scale due to mainly two difficulties: powders without sufficient green strength must be canned and degassed before forging, which is a costly handling step. *Hot extrusion* can be applied to both; gas atomized and water atomized powders. The spherical powder particles, deriving from gas atomization, have to be canned and vacuum degassed during preheating. Then, the material can be extruded and the can material be removed by machining. In contrast, annealed irregular shaped water atomized powders are directly filled into polyurethane rubber moulds and cold isostatically pressed. The so-obtained “green” steel billets can then be extruded. However, due to the lack of canning, preheating and handling have to be performed under reducing or inert atmosphere in order to avoid undesired oxidation. Extrusion temperatures range from 1,100 to 1,200 °C. In order to obtain fully dense material the extrusion ratio should exceed 10/1.

1.1.4 Alloying Elements in Tool Steels

Numerous alloying elements can be found in tool steels [7]. The reason for the use of—in many cases rather expensive—elements is that by the addition of these elements significant improvement of the material properties, i.e. high strength and hardness at high toughness, can be achieved. Typical microstructure of heat treated tool steel consists of a dispersion of hard carbides in a tempered martensite matrix. Many variations of this microstructure exist, depending on the concentrations of the alloying elements in the steel but also on the heat treatment processes applied. Thus, tool steels are complex microstructural systems. However, three major alterations of the steel can result from alloying: the hardening behavior, the nature and properties of the carbide phases, introduced by the addition of carbide forming elements, and finally changes in the tempering characteristics of the steel might occur.

Carbon is by far the most important alloying element in all sorts of steels, especially in tool steels, since even small changes of the carbon content have a significantly stronger impact on the thermal behavior and the resulting steel properties than any other alloying element. Figure 1.2 shows the Fe-C binary diagram for the carbon content range as observed in common plain carbon tool steels. At room temperature in equilibrium any carbon tool steel consists of body-centered cubic ferrite and orthorhombic cementite. Above the critical temperature A_{c1} , structural change to face-centered cubic austenite and (partly) carbide

Fig. 1.2 Fe-C binary diagram for C contents relevant for tool steels [7]. Reprinted with the permission of ASM International. All rights reserved (www.asminternational.org)

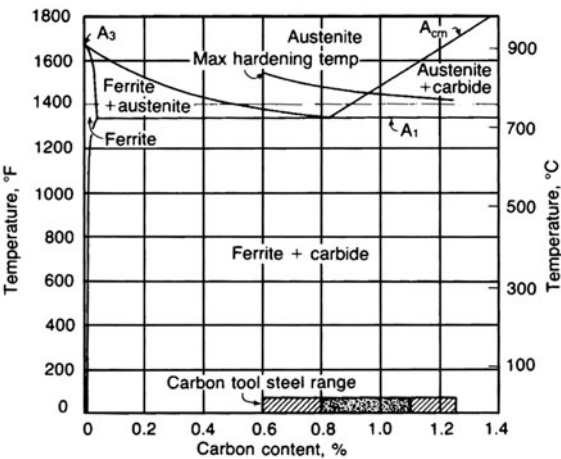


Table 1.1 General effect of important alloying elements [7]

Effects of alloying elements	Alpha-stabilizing elements	Gamma-stabilizing elements
Carbide forming elements	Chromium, molybdenum, titanium, tungsten, vanadium, zirconium, tantalum	Manganese
Elements mainly solved in the iron matrix	Silicon	Carbon, cobalt, copper, nickel

dissolution occur due to the higher potential of austenite for dissolving carbon compared to ferrite. Most of the used tool steels are hypereutectoid steels, which means that during austenitizing excess carbides remain undissolved. Thus, the three main equilibrium phases are ferrite, austenite and carbides, the existence of which can be determined by equilibrium phase diagrams. However, when heat treatments are performed, phase transformations are tied due to fast temperature change and thus, additional non-equilibrium phases such as martensite and bainite can be formed dependent on the content of carbon and other alloying elements in the steel.

For more details about the iron–carbon binary system it is referred to the literature [2, 7].

1.1.4.1 Effects of Alloying Elements on Ferrite and Austenite Stability

Table 1.1 shows the most important alloying elements widely added to tool steels, classified after their ability of influencing the ferrous phase through shifting the phase field boundaries in the Fe-C phase diagram, and after their carbide forming ability. It should be pointed out that most of the alpha stabilizing elements seem to induce their body-centered cubic structure to the iron phase. In contrast,

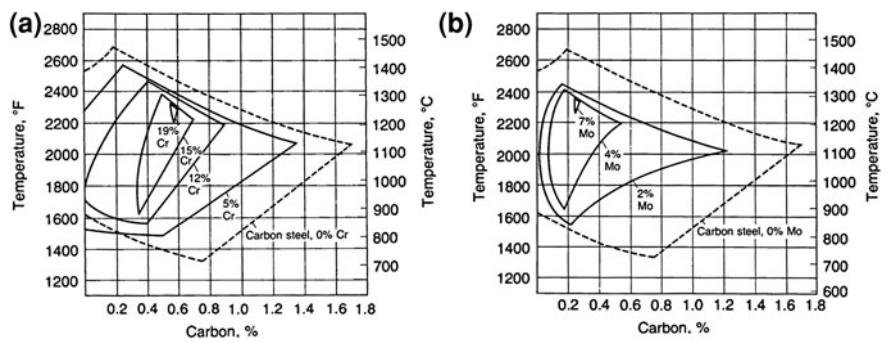
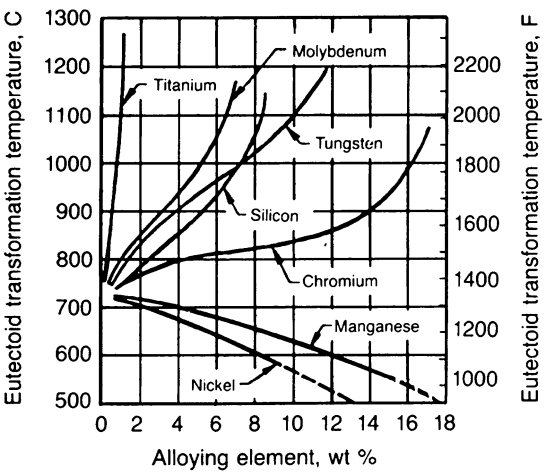


Fig. 1.3 Influence of several chromium (a) and molybdenum (b) contents on the stability of austenite [7]. Reprinted with the permission of ASM International. All rights reserved (www.asminternational.org)

Fig. 1.4 Effect of alloying elements on eutectoid transformation temperature in steel [7]. Reprinted with the permission of ASM International. All rights reserved (www.asminternational.org)



the elements that stabilize the austenite phase have themselves a face-centered cubic lattice.

For example, chromium and molybdenum, respectively, cause a contraction of the austenite phase field (Fig. 1.3). With increasing chromium and molybdenum content, respectively, the area of the austenite field shrinks considerably. Consequently, a limit of carbon, chromium, and molybdenum contents, respectively, in tool steels exists in order to attain hardenable steels. Furthermore, ferrite stabilizing elements also increase the eutectoid transformation temperature with increasing alloy content, as shown in Fig. 1.4. In contrast, gamma-stabilizers such as manganese or nickel lower this temperature and widen the austenite phase field. Cobalt, though it is mentioned in Table 1.1 being an austenite stabilizer does not have any effect on the iron structure at the cobalt concentrations used in tool steels.

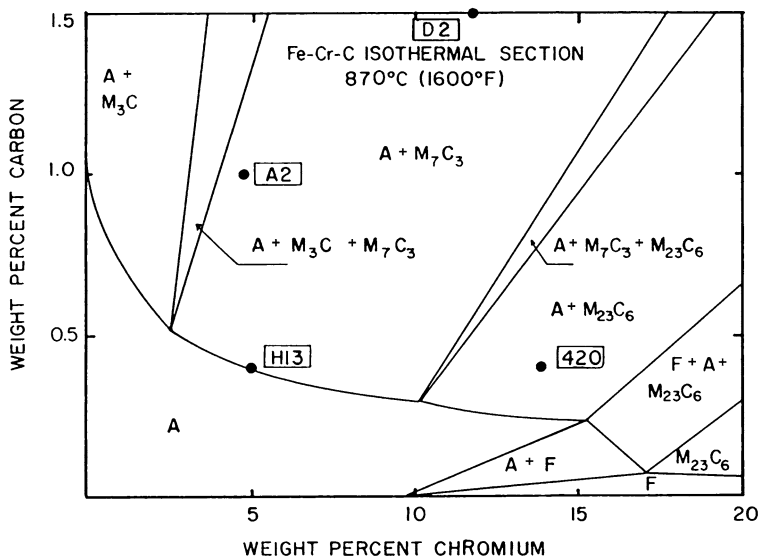


Fig. 1.5 Part of the isothermal section at 870 °C of the Fe-Cr-C system [7]. Reprinted with the permission of ASM International. All rights reserved (www.asminternational.org)

1.1.4.2 Alloy Carbides in Tool Steels

The alloying elements chromium, molybdenum, tungsten and vanadium are strong carbide forming elements and of high importance for the mechanical properties of tool steels, since the large volume fractions of carbides formed from these elements provide the high hardness and wear resistance of tool steels, however, making hot deformation, heat treatment, and also machinability of the tool steels themselves more difficult. They come into existence during solidification, hot working or heat treatment.

At the chromium concentration relevant for tool steels, chromium forms two carbide types, namely Cr_{23}C_6 and Cr_7C_3 depending on the chromium and carbon contents, respectively (Fig. 1.5). An important feature of the chromium carbides is their high hardness. Tarasov [2] measured significantly higher hardness for the chromium alloy carbide of type M_7C_3 (1820 Knoop) than for the alloy cementite $(\text{Fe,Cr})_3\text{C}$ (1150 Knoop). Combined with their usually rather coarse appearance these carbides offer high wear resistance in cold work applications.

The tungsten, molybdenum and vanadium-rich carbides are responsible for the high hardness at elevated working temperatures. Thus, tungsten and molybdenum high speed steels are widely used for cutting applications despite the considerable alloy costs. Basically, the two systems, Fe-C-W and Fe-C-Mo, are more or less the same. They form the same so-called double carbide types that are M_6C and M_2C at concentration levels relevant for tool steels, where “M” stands for either

(Fe,W) or (Fe,Mo), thus, these carbides always contain significant amounts of iron and exist only in the ternary systems. Moderate amounts of Cr, V, and Co can also be dissolved in the M_6C carbides. The carbides exhibit higher hardness (about 75 HRC) than the chromium carbides. The M_6C type carbides resist dissolving upon austenitizing, thus, remain existent as excess carbides, which are potentially responsible for high abrasion resistance and also inhibit undesired austenite grain growth during austenitizing. Only at too high temperatures the M_6C carbides partially dissolve in the iron matrix, which goes along with undesired austenite grain coarsening. However, once these carbides are dissolved they are extremely reluctant to precipitate on tempering. Only at tempering temperatures above about 510 °C they precipitate as intermediate carbides of type M_2C . This precipitation accounts for the secondary hardness and the red hardness of high speed tool steels. At temperatures over 650 °C the stable M_6C carbide type starts to reform. The effect of molybdenum and the formed carbide types are rather similar to those observed for tungsten. However, molybdenum has a lower melting point than tungsten, thus, the austenitizing is performed at slightly lower temperatures and the secondary hardening effect is more pronounced in Mo-based high speed steels compared to tungsten-based steels. The temperature range in which austenite is formed is narrower, thus, precise adjustment of the austenitizing temperature is required. The red hardness of the molybdenum steels is slightly lower than for tungsten high speed steels, which can be compensated by the addition of some tungsten or vanadium. Vanadium forms another carbide type, namely MC, where “M” stands for vanadium. It is among the hardest alloy carbides (about 84 HRC), increases the cutting efficiency of the tools through the higher wear resistance, and it is extremely slow to dissolve upon austenitizing. However, there is one important fact associated with the addition of vanadium: the carbon content has to be adjusted appropriately in dependence of the vanadium content, since the hardness would suffer a rapid decrease when increasing the vanadium content above about one percent at fixed carbon content. The reason for this phenomenon is a reasonable amount of ferrite existing in such steels, enhanced by excess of vanadium that is dissolved in the iron matrix, which stabilizes the ferrite phase. However, the higher carbon concentrations required to balance the V content lead to higher amount of retained austenite in as-quenched steel, which additionally is more stable during tempering. Thus, higher tempering temperatures, longer holding times, and multiple tempering have to be applied to vanadium tool steels in order to enable the decomposition of retained austenite.

Manganese forms with carbon no fewer than five different special carbides, of which, however, none of these carbides exist below a manganese content of 24%. Thus, since in tool steels the manganese content never exceeds 3%, manganese carbides are never found in tool steels. In high speed steels the manganese content is kept below 0.35% because it causes increased brittleness and danger of cracking on quenching. Thus, the importance of manganese as special carbide forming element is low.

1.1.4.3 Effects of Silicon, Nickel, Cobalt, Phosphorous and Sulfur

Silicon is a ferrite stabilizer and tends to destabilize the cementite and cause its decomposition into ferrite and graphite. This phenomenon is capitalized in certain free-machining tools where silicon is added to produce graphitization for sake of lubrication. In high speed steels the amount of silicon is usually kept below 0.35%, since higher contents would cause brittleness of the steel.

Nickel is mostly found in the ferrite phase, only up to 2% can be included in the cementite. The same applies to silicon and cobalt which also have a very low solubility in cementite. Nickel, like manganese, is an austenite stabilizer and lowers the critical temperature. Thus, it tends to increase the amount of retained austenite on quenching, thereby limiting the maximum hardness of as-quenched steel.

Cobalt does not have any effect on the iron structure at the cobalt concentrations used in tool steels. It is widely used in high speed steels for applications like deep cuts, and cutting hard and scaly materials, which justify the use of cobalt despite its high price and hazardous effect to human health. The main effect of cobalt in high speed steels is to increase the red hardness, thus improving the cutting efficiency. It is mainly dissolved in the iron matrix that is strengthened by the dissolved cobalt through which higher overall hardness of the steels at room and elevated temperatures can be attained. Furthermore, cobalt raises the solidus temperature of the steels, thus higher heat treatment temperatures are accessible without the negative effect of grain growth. Consequently, higher amounts of strong carbide forming elements such as tungsten, chromium, vanadium, and carbon can be dissolved in the iron matrix during austenitizing, which results in greater secondary hardness and better red hardness. Thus, higher cutting speeds are accessible with high speed steel containing cobalt without losing too much material hardness and strength. However, significantly higher amounts of retained austenite in the as-quenched steel have to be considered. Major drawbacks of cobalt are the increased brittleness, the tendency towards decarburization during heat treatment, high costs, and the health hazards.

Phosphorus and sulfur can be introduced through contamination of steel scrap. In general, the content of phosphorus is kept at very low levels so that any influence on the steels properties can be excluded. Sulfur, in contrast, can also be purposely introduced for free-machining steel qualities. The principal advantage is easier grindability and, thus better surface finish. Sulfides formed, i.e. molybdenum disulfide MoS_2 , act as lubricants, thereby these sulfur containing steels offering greater cutting performance. However, it was found for sintered steels that MoS_2 reacts with Fe during sintering at temperatures above 600 °C, forming Fe_{1-x}S sulfides [15], which compromises the beneficial effect of sulfur addition.

The major advantages of the addition of alloying elements in tool steels balancing the additional costs can be summarizing as follows:

- Higher strength
- Less distortion during heat treatment

- Better abrasion and wear resistance
- Higher toughness at same hardness levels
- Better red hardness and strength at elevated service temperatures.

1.1.4.4 Effects of Alloying Elements on Response to Quenching from the Austenitizing Temperature and on Hardenability

After austenitizing the tool has to be cooled at appropriate cooling rate to yield the desired structure and hardness. The cooling has to be performed fast enough so that martensite without intermediate products as bainite, pearlite is formed. Alloying elements influence the required cooling rate through shifting the CCT curves to longer times. Furthermore, they could shift the temperature thresholds for martensitic transformations ($M_{\text{start}} = M_s$, and $M_{\text{finish}} = M_f$) to higher or lower temperatures, thus also influencing the amount of retained austenite existing after quenching. If only carbon is present, very rapid cooling is needed to achieve fully martensitic structure. In contrast, the strong carbide forming elements chromium, tungsten, molybdenum, and vanadium shift the isothermal transformation curves to longer times, thus hardenability is improved, and slower cooling can be applied to achieve same structural properties. Assuming that cooling is correctly accomplished, then intermediate transformation products are not formed, but part of the austenite, i.e. retained austenite, would not be transformed into martensite when room temperature is reached, which effect reduces the as-quenched hardness. Also dimensional stability, structural strength, and the tool toughness can be influenced negatively. Raising the austenitizing temperature increases the amount of alloying elements dissolved in the austenite, i.e. dissolution of alloy carbides, which is beneficial for secondary hardness. This, however, lowers the M_s temperature and thus results in more retained austenite in the as-quenched steel. Thus, in high speed steels, for which secondary hardness is most important, multiple tempering is required to eliminate the retained austenite.

1.1.4.5 Effects of Alloying Elements on Tempering

In as-quenched tool steels three types of structural phases exist, namely martensite, excess carbides, and retained austenite, which influences the mechanical properties negatively, as described above. Tempering is applied to tool steels for two main objectives: Structural changes of the martensite including fine carbide precipitation and transformation of the undesired retained austenite occur. Four principal shapes of tempering curves can be identified for the four main groups of tool steels, which are presented in Fig. 1.6a. Class 1 represents carbon and low-alloy tool steel with rapid softening occurring above a certain tempering temperature, due to the precipitation of iron and alloy carbides that further grow in size. For medium to highly alloyed cold work tool steels this critical temperature for softening is found to be

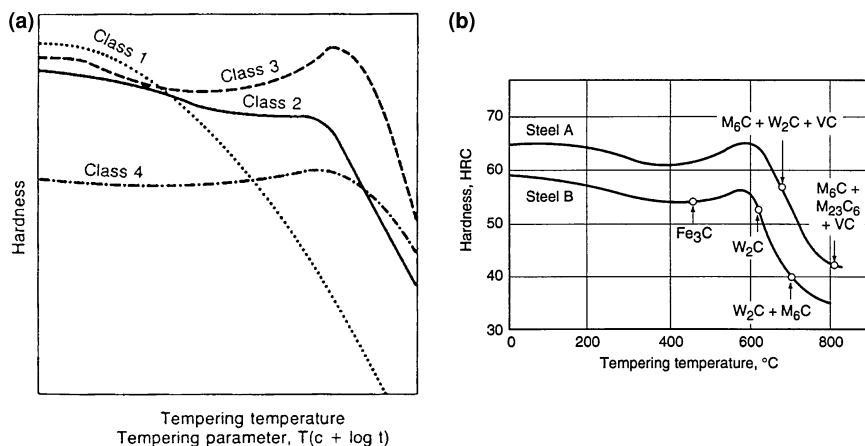


Fig. 1.6 Tempering of tool steels: **a** four major types of tempering curves, **b** characteristic tempering curves “class 3” tool steels [7]. Reprinted with the permission of ASM International. All rights reserved (www.asminternational.org)

shifted towards higher temperature as indicated by curve of class 2. The tempering behavior of highly alloyed high speed steels can be described by curve of class 3. In addition to the retarding of the softening, secondary hardening through precipitation of fine so-called “secondary carbides” of tungsten, molybdenum (both M_2C type) and vanadium (MC), as described above, takes place. Class 4 represents medium to highly alloyed hot work die steels offering a similar behavior as class 3, but starting at lower as-quenched hardness due to their lower carbon content.

The phenomenon of secondary hardening is mainly based on the following: The conditioning of retained austenite, i.e. depletion of alloy elements and carbon through secondary carbide precipitation, and its transformation to martensite on subsequent cooling from the tempering temperature. In low to medium alloyed steels austenite transforms to bainite or is partially stabilized at the low tempering temperatures applied on those steels. Secondary hardening does not take place, due to the lack of sufficient amount of carbon and alloying elements. In high alloy steels, secondary hardening is an important factor. At temperatures below 400–500 °C the retained austenite remains untransformed, but conditioning reaction occurs. The martensite that is formed through transformation of the conditioned austenite upon subsequent cooling from the tempering temperature is, of course, untempered. Therefore, often a second tempering is necessary. The more important factor contributing to secondary hardening in highly alloyed tool steels is the precipitation of submicroscopic alloy carbides usually occurring at temperatures between 450 and 600 °C. These tungsten, molybdenum, and vanadium carbides (Fig. 1.6b), are responsible for this effect that is also important for hot hardness of tool steels. Cobalt slightly enhances this effect by moving the secondary hardening peak to higher temperatures. However, the effect of the

carbide forming elements on the tempering processes depends also on the applied austenitizing temperatures, since the processes above described only can take place if sufficient amounts of alloy elements and carbon have been dissolved in the austenite upon austenitizing.

1.1.5 Classification and Application of Tool Steels

There are many types of tool steels that differ in their chemical composition and are used for different applications. There exist numerous national standards such as American Iron and Steel Institute (AISI), British (BS), French (AFNOR), German (DIN), and Japanese (JIS) according which tool steel companies produce and classify their products. These standard compositions can be produced by conventional ingot metallurgy (IM), but also by powder metallurgy (PM). However, by means of PM, also chemical compositions of tool steels can be attained that cannot be produced by IM. Since the compositions of PM tool steels rarely correspond to any standards, these products are commonly sold by their trade name. AISI designations classify the numerous tool steels in up to ten main groups either according to their hardening behavior, to their chemical composition, or according to their application field. Each of these groups has in common similar applications and similar chemical compositions at least within certain—sometimes rather broad—ranges of compositions, which gives a comprehensive and understandable overview of the principal types of tool steels. Thus, it is used in the following to provide a brief overview of the most important tool steels in commercial usage.

Due to the low-alloy content of group “W” water-hardening tool steels (e.g. “W1”, 0.6–1.4%C), formation of martensite is only accessible by fast quenching in water and only iron carbides can be observed in these steels. Addition of small amounts of chromium is used in order to increase hardenability and wear resistance. The addition of vanadium helps to maintain fine grain sizes, which enhances the toughness of the steel. Group “W” tool steels have, due to their low alloy contents, poor resistance to softening at elevated temperatures. Thus, the usage of group “W” steels is limited to cold work applications such as cold heading, striking, coining, and woodworking. Furthermore, these steels can be used as wear-resistant machine tool components and as cutlery.

Group “O” oil-hardening tool steels (e.g. “O1”, 0.9%C, 1.0%Mn, 0.5Cr, 0.5W) have higher alloy content compared to group “W”, thus, they can be hardened in oil, which represent a milder quenching medium, thus helping to prevent cracking and dimensional distortion. Although the compositions of group “O” steels vary considerably in both type of alloy and alloy content, they reveal similar general characteristics and are used for similar applications, which are dies and punches for blanking, trimming, drawing, flanging, and forming. Oil-hardening tool steels are also used for machinery components such as cams, bushings, and guides and also for gages. The group “O” steels offer high wear resistance at low temperatures due to their high carbon contents forming carbon rich martensite upon

quenching and due to undissolved alloy carbides. However, resistance to softening at elevated service temperature is low.

Air-hardening tool steels of group “A” (e.g. “A2”, 0.95–1.05%C, 4.75–5.50%Cr, 0.90–1.40%Mo, 0.15–0.50%V) contain significant amounts of alloying elements, especially chromium, molybdenum and vanadium, which improve the hardenability of the steel, so that they can be air hardened. Less dimensional distortion and very low tendency to cracking during cooling from the austenitizing temperature are important advantages of these steels. Typical usage of these steels are cold work applications such as shear knives, punches, blanking and trimming dies, forming and coining dies.

Group “D” represents various cold work tool steels having high carbon and chromium contents (e.g. “D2”, 1.50%C, 12.0%Cr, 1.0%V, 1.0%Mo). These steels offer very high abrasion and wear resistance, which are provided by the large volume fraction of primary alloy carbides that exist in addition to the high-carbon saturated martensite. The primary alloy carbides that form a eutectic network during the solidification process which is disintegrated into carbide stringers remain undissolved during austenitizing. In addition numerous finely dispersed secondary carbides precipitate from the high-carbon martensite upon tempering. The high concentration of chromium, which is also partly dissolved in the iron matrix, enables martensite formation on air cooling. Group “D” steels are used for cold work applications for which high abrasion resistance is required, such as long-run dies for blanking, powder pressing, forming, thread rolling, dies for cutting laminations, brick molds, gages, burnishing tools, rolls, and shear and slitter knives. However, machining and grinding of group “D” steel tools is rather difficult and expensive due to the high abrasion resistance of these steels. A major part of this thesis was dedicated to the fatigue investigation of AISI D2 type steel. Therefore, in the experimental part a more detailed description of this steel follows.

All the steels described above can be used for cold work applications. However, since they do not resist softening at elevated temperatures they cannot be used under such service conditions. For purposes such as hot working, die casting, cutting operations, several hot work and high speed tool steel grades exist that contain significantly higher amounts of costly alloying elements that can support high red hardness of the material. Group “H” includes three major types of hot work tool steels containing predominantly chromium, tungsten, or molybdenum, respectively.

Steels of group “T” and group “M” are high speed tool steels, which are used for high speed cutting applications. Molybdenum (“M” group, e.g. “M2”, 0.85–1.00%C, 4.0%Cr, 2.0%V, 6%W, 5%Mo) and tungsten (“T” group, e.g. “T1”, 0.75%C, 4.0%Cr, 1.0%V, 18.0%W) high speed steels show very similar service performances. However, due to the fact that half of the weight percent of molybdenum is required to achieve the same properties when molybdenum is substituted for tungsten, the molybdenum high speed steel grades offer a very significant cost advantage. Thus, most of high speed steels produced and used today are molybdenum variants, although tungsten high speed steels (group “T”)

were the first tool steels grades (Taylor and Mushet steel) that exhibited extremely high red hardness, which has been described in the part about historical aspects of tool steels in the thesis. Tungsten high speed steels contain also some amounts of chromium, vanadium, cobalt in addition to tungsten. The high amount of alloying elements, of which most are ferrite stabilizers, is responsible for good hardenability of these group “T” steels. The high amount of strong carbide forming elements provides a large number of very hard, wear-resistant carbides dispersed in the iron matrix, accounting for the excellent wear resistance of high speed steels. The combination of high red hardness and wear resistance represents the basis for high performance cutting applications such as bits, drills, milling cutters, and hobs. But these tool steels are also used for making dies, punches and in high temperature structural components such as aircraft bearings and pump parts.

The “M” grade high speed steels contain, in addition to molybdenum, some amounts of tungsten, chromium, vanadium, and cobalt. The wear resistance can be improved by increasing the carbon and vanadium concentration, which have to be adjusted as described above. The addition of cobalt has beneficial effects on the red hardness of the steel, but lowers the material toughness. Since molybdenum high speed steels tend to decarburize and can be damaged from overheating, the adjustment and control of the austenitizing temperature and atmosphere are critical issues for group “M” high speed steels. Group “M” steels comprise also some ultra-hard high speed tool steel grades (e.g. “M42”, 1.10%C, 3.75%Cr, 1.15%V, 1.50%W, 9.50%Mo, 8.0%Co), for which a steel hardness of about 70 HRC can be reached. These steels contain higher carbon contents and higher amounts of strong carbide forming elements, resulting in very high volume fractions of primary alloy carbides. In addition finely dispersed secondary alloy carbides are formed during high temperature tempering. High cobalt contents can be observed in these ultra-hard high speed steels in order to strengthen the iron matrix. However, due to the high amount of alloying elements, toughness can be a point of concern with these high speed steels. Thus, often these steels are heat treated to hardness levels below the maximum attainable hardness. i.e. by austenitizing at somewhat lower temperature compared to “normal” molybdenum high speed steels, which offer more ductility. For this thesis the fatigue behavior of M2 and M42 high speed steels was evaluated.

Furthermore, there are several other groups of special-purpose steels comprising shock resistant steels (group “S”), low-alloy special-purpose tool steels (group “L”), and mold steels (group “P”). The main alloying elements in shock resistant steels (e.g. “S1”, 0.5%C, 1.5%Cr, 2.5%W) are manganese and silicon. Carbide forming elements such as chromium, tungsten and molybdenum are also added to provide hard carbide precipitates, and thus, high strength and wear resistance. Carbon content is kept at moderate levels below 0.5%, resulting in low carbon content of martensite. This is essential for high toughness and fracture resistance in addition to good wear resistance provided by the alloy carbides required for non-tooling structural application. Low-alloy special-purpose steels (e.g. “L2”, 0.5–1.1%C, 1.0%Cr, 0.2%V) that are used for machine parts such as arbors, cams, chucks, and collets and for other special applications contain small

amounts of carbide formers such as chromium, vanadium, and molybdenum, and toughness-increasing nickel (up to 1.5%). The required high strength is provided by the high carbon content in the martensite and the finely dispersed alloy carbides. Mold steels (group “P”, e.g. “P2”, 0.10%C max, 0.1–0.4%Mn, 0.1–0.4%Si, 0.75–1.25%Cr, 0.1–1.5%Ni, 0.15–0.40%Mo) contain in addition to carbon (up to 0.3%) up to 5% chromium and up to 4% nickel. Due to the low carbon content these steels have a very low hardness in the annealed condition, which enables to perform a mold impression by cold hubbing. Subsequently, the mold undergoes carburization, hardening and tempering in order to achieve service hardness. Resistance to softening at elevated service temperatures is rather low. Mold tool steels comprise also steels with very high chromium content up to 27%Cr (e.g. 17Cr–0.65C) offering high corrosion resistance. Mold steels are used in low temperature casting dies and in molds for plastic forming processes.

There exists a large variety of commercially available PM tool steels, especially high speed steels, cold work and hot work tool steels, with compositions according to the AISI standards presented above. However, in addition many PM grades are produced that cannot be attained by conventional ingot metallurgy due to their relatively high alloy content. Thus, these grades are sold by their trade names such as Böhler high speed steel “S590” (1.3%C, 4.2%Cr, 5.0%Mo, 3.0%V, 6.3%W, 8.4%Co) corresponding to grade “ASP30”, or Böhler cold work tool steel “K390” (2.45%C, 4.15%Cr, 3.75%Mo, 9.0%V, 1.0%W, 2.0%Co), which grades have been investigated.

1.2 Fatigue of Materials

1.2.1 General Considerations

Numerous types of components are used in cars, trucks, trains, airplanes and many more machines [16]. Important structural parts are made of materials having specific properties useful for the respective application. The materials employed are usually selected according to the required service demands. These parts are exposed to mechanical and thermal stresses and strains, environmental influences such as hydrogen containing gases, aqueous environments, and high, low or often changing temperatures during service operations. All of these mentioned factors might eventually cause corrosion and/or metal embrittlement and might induce cracks in the components. Several modes of material failure such as impact and fatigue failure and creep fracture can take place. However, the reason for failure of components cannot always be found in improper materials selection or unsuitable material properties. Wrong design, inaccurate material processing—e.g. heat treatment—and also component misuse are rather common reasons for fatal failures of components. For engineers it is of utmost importance to recognize and evaluate all possible loading types and eventually involved failure modes occurring for each part.

Table 1.2 Three major groups of sources for material failure and corresponding damages

Mechanical effects	Thermal effects	Environmental effects
Yielding	Thermal shocks	Radiation damage
Rupture	Thermal relaxation	Corrosion
Wear	Creep	
Impact		
Buckling		
Fretting		
Galling and seizure		
	Fatigue	

Then, the component design has to be adapted and an appropriate material selected that provides the necessary mechanical properties. Failure analysis, prediction, and prevention during service time are critical tasks for any structural part. In order to prevent in-service failures of components, which could put human lives in jeopardy and could result in considerable economical losses, failure mechanisms of the employed materials have to be studied extensively.

Generally, mechanical failure can be defined as any change of size, shape, or material properties of a component that might lead to incapability of fulfilling the intended function of the part. There are numerous sources for occurrence of failure in a material, which can be categorized into three major groups (Table 1.2).

Failure due to mechanical stresses and strains can result in a number of damage phenomena. Ductile and brittle rupture can occur depending on the toughness of the material. Many types of wear such as adhesive, abrasive, or corrosive wear might occur, which can cause surface fatigue failure or deformation and fretting. High energy impacts can result in immediate fracture, just deformation, or also subsequent fatigue failure after certain number of loading cycles. Creep characterizes continuous deformation of a material under long term exposure to stresses below the yield strength, the effect of which increases with increasing temperature. Service at elevated temperatures might lead to material softening due to aging. of the microstructure. Both processes result in significant loss of material strength, resulting in material failure e.g. at subsequent exposure to high loads. Suddenly changing temperature might induce thermal shocks to the material, which can be responsible for microstructural changes within the material, e.g. embrittlement. Another failure mechanism of high importance in practice is corrosion due to aggressive environments. Corrosion failure can occur due to direct chemical attack by e.g. acids or hydrogen, galvanic processes, cavitation, material erosion and pitting. It can also result from biological processes or in combination with low stress loadings.

Fatigue failure represents a specialty among all of the above discussed failure modes. It can occur due to mechanical cyclic loading, but also due to cyclic thermal and environmental processes. However, it has to be mentioned that in practice, fatigue failure often occurs due to a combination of the above described failure processes and usually the effects are synergistic.

1.2.2 Brittle and Ductile Failure

Fracture of a component, which is the final result of the aforementioned processes, can be defined as the separation of a body into two or more pieces in response to mechanical stresses and strains. Basically, two fracture modes can be observed: ductile and brittle fracture. While ductile materials like most metals exhibit plastic deformation due to their ability to partial energy absorption before fracture, brittle materials like most ceramics show little or no energy absorption and plastic deformation. However, the categorization “ductile vs. brittle materials” has to be regarded with utmost care, e.g. it holds only at room temperature, since temperature and environmental effects can considerably influence the material fracture behavior. For example ceramics can behave ductile at high temperatures, since atoms have higher mobility at elevated temperatures, thus allowing at least some (micro-) plastic deformation within these otherwise brittle materials. Alike, metals also can break in a brittle way at low temperature or if they contain impurities causing embrittlement of the metal matrix. For example, titanium behaves brittle if oxygen diffused into the titanium metal.

Generally, the material failure can be divided into two processes. First, a crack has to be initiated, which can be due to some kind of very small material defect. This initial process of failure is called the crack initiation process. The so-formed crack propagates then through the material/part until total fracture of the component does occur. While in ductile materials the crack propagation is rather slow, since part of applied energy is consumed by plastic deformation in front of the crack tip, in brittle materials the crack grows extremely fast. Consequently, if a crack has been formed in a brittle material, final failure is foreseeable. Thus, ductile fracture is preferred in component design, since plastic deformation gives a warning hint before fracture. Furthermore, the slow crack growth allows that certain flaws exist in the material without fatal effect on the component. Ductile materials also can withstand higher stresses prior to failure due to the ability for absorption of large amounts of the applied energy. However, in many cases ductile materials cannot be used due to e.g. environmental conditions or high service temperatures.

1.2.3 Fatigue Failure

The discussed failure modes—ductile and brittle—usually occur under static loading. However, in this work the fatigue behavior of tool steels has been under investigation, aiming to exhibit lifetime controlling defects in such hard high strength steels. Thus, the thesis focuses on mechanical fatigue testing and fatigue fracture. Mechanical fatigue failure might take place if a material is subjected to dynamic and fluctuating stresses and strains, which is commonly the case for structural components in aviation, construction and other machines. This failure

due to so-called “cyclic loading” occurs at stress levels considerably lower than the static (yield) strength of a material. Fatigue failure takes a long time to evolve, which has been the reason for the naming of this failure phenomenon. Especially for metals, fatigue is the most important failure type, since fatigue accounts for over 90% of all metallic failures [17]. The danger of fatigue failure is its abrupt occurrence, usually without any warning. The nature of fatigue fracture is usually brittle-like even in ductile materials, since only low plastic deformation takes place due to the low stresses involved. Consequently, fatigue failure can be described using the principles of fracture mechanics for brittle materials. Fatigue failure can be divided into two phases—fatigue crack initiation and subsequent crack propagation. In the following sections, basic definitions and principles of fatigue and the history of fatigue investigations will be described in more detail.

1.2.3.1 History of Fatigue Investigations

Studies of fatigue phenomena date back to first half of the nineteenth century [18]. A German mining engineer named Albert was among the first who investigated metal fatigue. About 1830 he studied the fatigue behavior of iron chains. Poncelet is acknowledged to introduce the term “fatigue” with respect to metal failure about 1840. With the increasing use of ferrous alloys as structural components in bridges and for railways, research interests in the field of fatigue failure mechanisms of the used materials became stronger. However, as it often happens in history of applied sciences, a fatal railway accident in 1842 near Paris was the driving force for further detailed investigations of metal fatigue. Rankine, a British railway engineer, discovered distinct characteristics of fatigue failure, among which most important was the fatal influence of stress concentration. The works of August Wöhler, who performed systematic investigations of fatigue failure in railroad axles around 1860, are well known. He was the first who noted that the—nowadays referred to—cyclic strength of steel was considerably lower than its static strength. His studies led to the development of the so-called “Wöhler-curve” or “S–N-curve”, which gives the relationship of material life versus loading stress amplitude. In 1874 another German engineer, Gerber, developed methods for fatigue life calculations for different mean stresses of cyclic loading and thus, was among the first to introduce fatigue life predictions into engineer’s design consideration. In 1886 Bauschinger found the elastic limit to be different in reversed loading compared to that observed in monotonic loading. This phenomenon is now commonly known as “Bauschinger effect”. At the beginning of the twentieth century, Ewing and Humfrey were the first to explore the mechanisms of the fatigue process. For this they investigated the fatigue behavior of plain iron and were able to show that slip bands developed in many grains of the polycrystalline material. With ongoing fatigue deformation these slip bands broadened and in the end led to the formation of fatigue cracks. Around 1910 several researchers worked on questions concerning fatigue. Basquin developed empirical laws to estimate endurance limits of materials. Bairstow performed early research on the

understanding of cyclic hardening and softening. In the 1930s several books about fatigue of metals appeared. In this period, fatigue failure evolved as a major field of scientific research. Further important work was accomplished by Coffin and Manson in the 1950s, who independently found plastic strains to be responsible for cyclic damage. Both of them proposed an empirical relationship between the number of cycles to fatigue failure and the plastic strain amplitude, which is nowadays widely used and referred to as the “Coffin-Manson relationship”. In 1913 Inglis published a mathematical work about stress fields near microscopic flaws. Griffith developed an energy concept for estimation of fatigue behavior of such micro-flaws. These two researchers provided the first mathematical tool for quantitative modeling of brittle fatigue failure. The major drawback of their works was the incapability of the treatment of metal fatigue. In the 1950s Irwin showed that the amplitude of the stress singularity ahead of a crack could be expressed in terms of the so-called stress intensity factor. These works mark the beginning of the so-called linear elastic fracture mechanics. In the 1960s, Paris et al. found a relationship between fatigue crack propagation per stress cycle (da/dN) and the stress intensity factor ΔK during constant amplitude cyclic loading. In the second half of the last century, considerable research of fatigue mechanisms in respect to mechanical, structural, and environmental factors was done, supported by the introduction and advances in both optical and electron microscopy. Thompson et al. found an effect that is now known as the formation of “persistent slip bands” (PSB). At the surface of fatigued metals, roughening has been observed due to the formation of PSBs. Zappfe and Wordon documented for the first time characteristic markings at the fracture surface, now commonly referred to as fatigue striations. Correlation of the spacing between these striations and the rate of fatigue crack growth was first published by Forsyth and Ryder in 1960. Further research then concentrated on the modeling of fatigue crack growth for engineering materials. Effects retarding crack propagation such as crack closure and crack deflection were studied by Ritchie, Suresh, and co-workers. Summarizing their findings, the rate of fatigue crack growth is not only dependent on the stress intensity factor but also on prior loading history and crack size. Small cracks were observed to grow faster than longer flaws at equal far-field ΔK values. Furthermore, several crack closure phenomena were identified by these authors, including “plasticity-induced”, “oxide-induced”, and “environmental-induced crack closure” and “stress-induced phase transformations”. In 1975 Pearson described for the first time that short cracks behave completely different than estimated by theories of fracture mechanics. Nowadays, this problem is commonly referred to as “short crack problem” and merely describes the fact that if flaws of same magnitude or smaller than the characteristic microstructures of the material increase in length, the crack growth rate may diminish [19]. Thus, due to those recent findings much effort was put into research of effects of crack closure and size effects.

Very recently the fatigue behavior of metallic materials at very high cycle numbers up to the gigacycle regime, especially of high strength steels employed for structural components, became a field of interest in fatigue research. Naito et al. [20] had shown in 1984 that carburized and surface hardened steels do not

exhibit a conventional fatigue limit at 10^6 – 10^7 cycles and fail even beyond 10^7 loading cycles at fairly low applied stresses. Until then, steels were thought to have a true fatigue or endurance limit, which represents a stress amplitude level below which these steels exhibit infinite life.

Summarizing, the history of material fatigue investigations is rather short and started with the extensive use of metals and steels as structural parts for bridges and railways. This research field is a very complex scientific topic, for which it is essential to employ many different science disciplines such as physics, engineering, chemistry, and mathematics. A variety of research in fundamental, applied, and industrial research is required in order to provide sound knowledge of factors influencing the fatigue behavior of materials. Potential material improvements are extremely dependent on such fatigue fundamentals.

1.2.3.2 Fatigue: Basic Definitions

Fatigue failure is a consequence of cyclic loading of a material or component. The occurring stresses can be axial (tension–compression), flexural, (bending), and/or torsional (twisting). These stresses can basically occur in three different fluctuating stress-time modes. Figure 1.7a represents an alternating sinoidal-like cyclic loading between a maximum tensile and compression stress of equal magnitude. The second mode (Fig. 1.7b) shows similar characteristics, however, the *mean stress*, which is defined as

$$\sigma_m = (\sigma_{\max} - \sigma_{\min}) / 2 \quad (1.1)$$

is not zero.

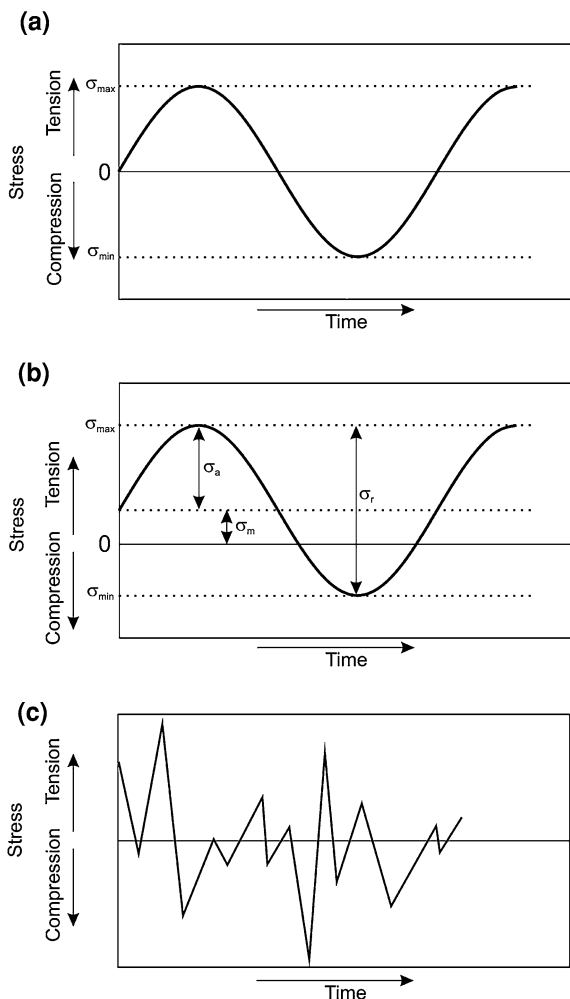
For the third stress-time mode (Fig. 1.7c), in which the stress amplitudes and the loading frequency vary randomly, often a range σ_r of maximum and minimum stress amplitude is used to describe the loading characteristic. The effective *stress amplitude* is then one half of the range σ_r . Furthermore, a *stress ratio* R , can be defined as follows:

$$R = \sigma_{\min} / \sigma_{\max} \quad (1.2)$$

1.2.3.3 The S–N Curve

The so-called S–N or Wöhler curve represents the relationship between the number of cycles to failure and the applied stress (stress amplitude, or eventually minimum or maximum stress). In Fig. 1.8 two S–N-curves are presented, which are characteristic for a material revealing a fatigue limit (1), which represents a stress level below which fatigue failure—at least theoretically—would not occur even at infinitely high N , and a material without such a limit (2), for which only a fatigue (endurance) strength can be defined. This fatigue strength corresponds to a

Fig. 1.7 Basic stress-time modes of cyclic loading [17]



stress at which for a certain number of loading cycles fatigue failure does not occur. The fatigue life of a material is the number of loading cycles until failure occurs. It is important to note here that the presented curves are best-fit curves of experimental data that, however, often reveal considerable scatter since a lot of parameters such as specimen processing, homogeneity, or testing environment affect the reproducibility. Furthermore, two basic types of fatigue can be distinguished: In low-cycle fatigue, applied stresses are relatively high, thus significant plastic strain might occur, and fatigue lives are short (up to 10^5 cycles). In contrast, at lower stresses only quasi-elastic deformation takes place and the number of cycles to material failure is much longer—usually above 10^5 loading cycles. This fatigue regime is referred to as high cycle, very high cycle, ultra high cycle or gigacycle fatigue regime.

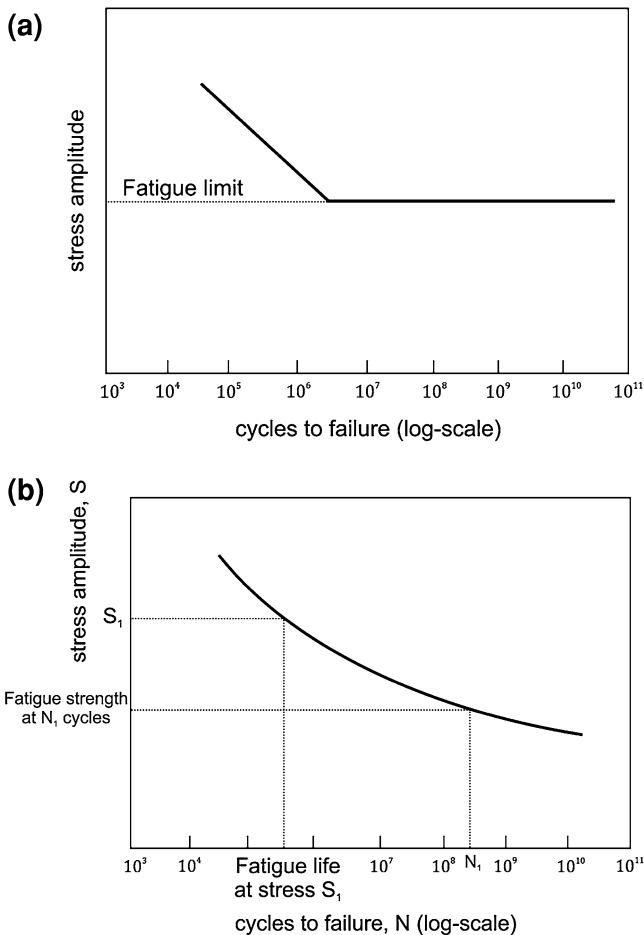


Fig. 1.8 Typical S-N curve for a material with a fatigue limit (a) and without (b) beyond 10^6 cycles [17]

The process of fatigue failure can be divided into three stages. It starts with the nucleation of a fatigue crack that emanates due to stress concentration at a microstructural feature within the material. In the following, the fatigue crack propagates at a certain growth rate that depends on the applied stress, the crack length, and eventually on environmental effects. If the crack length reaches a critical value, final fracture occurs through unstable crack propagation. Since the last stage of fracture is very fast, the total fatigue life is assumed to be the sum of loading cycles needed for crack initiation (N_i) and that required for growth to a critical crack length (N_p). At high stresses in low cycle fatigue usually the propagation is decisive for fatigue life, since a crack is virtually instantly formed

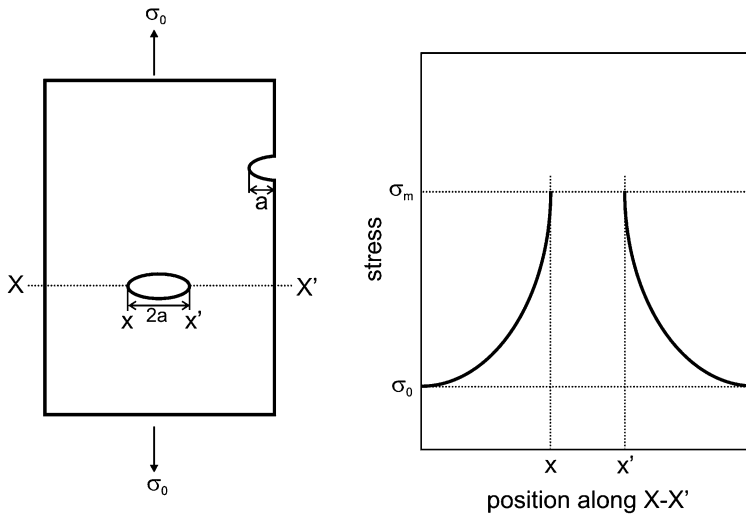


Fig. 1.9 Effect of a flaw on the stress distribution in plate [17]

due to the high load. In high cycle fatigue, which is characterized by low stress loading, the crack initiation process can be the lifetime-dominating factor.

1.2.3.4 Fracture Mechanics

In order to enable prediction of the fatigue behavior of materials and components, mathematical models are required, which are capable of describing the processes of fatigue failure from crack initiation to propagation. This field of research is commonly called “Fracture Mechanics”. In the 1920s Griffith proposed that flaws such as pores or inhomogenities within a material act as stress raisers (Fig. 1.9) enabling crack formation even at low stresses. An applied external or internal stress is amplified at such a defect, which result in a maximum stress $\sigma_{c,max}$ at the tip of the flaw. In this respect the geometry of a flaw is of importance, since sharp edges show significantly higher stress concentration than rounded flaws. The ratio $\sigma_{c,max}/\sigma_0$ is often called the stress concentration factor K_t , which describes the extent of stress amplification for a flaw of defined geometry. The effect of stress raisers is higher for brittle materials compared to ductile materials, because for ductile materials some plastic deformation occurs at the flaw tip when the stress exceeds the yield strength of the material. Consequently, a redistribution of the stress takes place, leading to somewhat reduced stress concentration. This concept however does not work for cracks, i.e. infinitely sharp notches, since in this case the stress would be infinite. Applying the K_t concept to cracks would imply that a component containing a crack would fail at the smallest load, which is in contrast to practical experience.

Griffith developed a theory for brittle fracture, for which he defined a critical stress σ_c that is required for crack propagation in brittle solids. He introduced a relationship between this critical stress and the modulus of elasticity E of the material, the specific surface energy γ_s and the half crack length a , as follows:

$$\sigma_c = \sqrt{(2 E \gamma_s / (\pi a))} \quad (1.3)$$

Obviously, the geometry of the flaw is not considered here. However, it is assumed that it has sufficiently sharp edges so that it is capable of nucleating a crack.

For ductile materials, the plastic deformation in front of the crack has to be considered as mentioned above. Thus, the specific surface energy is substituted by the sum of $\gamma_s + \gamma_p$, where γ_p represents the plastic deformation energy. Due to the usage of these energy terms the Griffith theory is commonly referred to as “energy approach” to fracture mechanics.

Furthermore, the “stress intensity factor K ” is another important parameter for fracture mechanical considerations. It is defined by the following equation:

$$K = Y\sigma \sqrt{(\pi a)} \quad (1.4)$$

in which “ σ ” represents the applied stress, and “ a ” the crack length.

The factor “ Y ” considers geometrical aspects of the crack and the loading type. It is highlighted here that the stress intensity factor K , applicable to infinite sharp edges of a flaw, i.e. cracks, and the stress concentration factor K_t , applicable to finite sharp edges, are two parameters deriving from different concepts, and thus are not equal.

1.2.3.5 Fatigue Crack Propagation

The stress intensity factor has huge importance for the prediction of fatigue crack propagation, which can be described by the crack propagation rate. It is defined by the change of crack length upon certain number of loading cycles. In order to avoid unexpected, possibly fatal in-service failures, knowledge of the crack propagation rate for a given crack length is of utmost importance since usually every material contains some kind of defects, and in many applications, e.g. aeroplanes, the formation of cracks cannot be avoided. In experimental fatigue studies, the growth of a fatigue crack as a function of the number of loading cycles can be determined. For this reason, at fatigue-exposed components data of crack length are recorded in appropriate inspection intervals and plotted against the number of loading cycles (Fig. 1.10). The crack growth rate at a given fatigue crack length can be derived by the differentiation of the curve at the corresponding point. In 1961 Paris et al. showed that there exists a relationship between the propagation rate (da/dN) and the range of the stress intensity factor ΔK as follows:

$$(da/dN) = C (\Delta K)^m \quad (1.5)$$

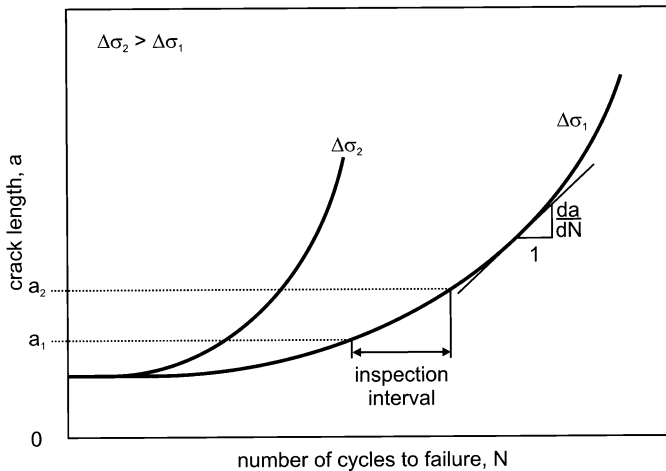


Fig. 1.10 Crack growth behavior in constant amplitude fatigue loading for two stress amplitudes $\Delta\sigma_1 < \Delta\sigma_2$ [18]

Where “C” and “m” are material constants that also depend on the environment, loading frequency, and stress ratio. Usual values for “m” range from 1 to 6. Taking the logarithm on both sides of the equation and further re-forming lead to the following linear expression:

$$\boxed{\log (da/dN) = m \log \Delta K + \log C} \quad (1.6)$$

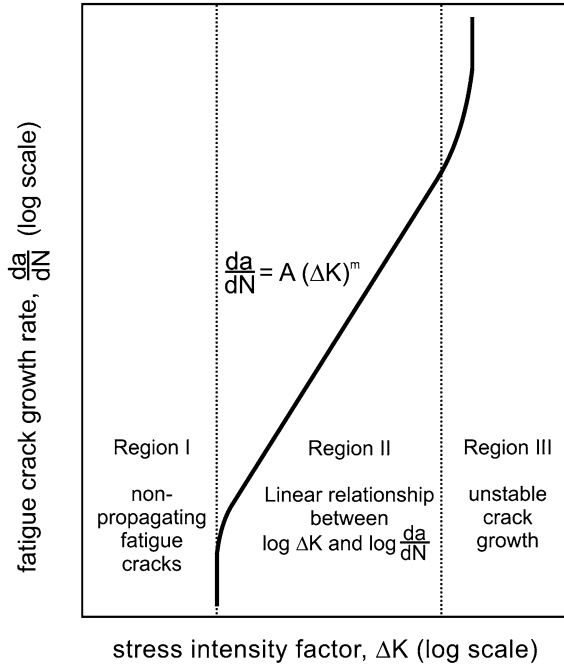
The two constants “m” and “C” can be derived from the slope and the intercept of the straight line obtained by the logarithm (Fig. 1.11). Obviously, three growth regimes can be distinguished. At low stresses and small defect sizes cracks do not grow (“region I”). The threshold value ΔK_{th} represents the lowest ΔK for which a crack can grow. Subsequent “region II” is characterized by a linear relationship between the logarithm of the crack growth rate and ΔK , which is referred to as “Paris-Law-regime” of stable crack growth. In “region III” the growth rate starts to increase at high pace just before final fracture. This information about the crack growth behavior of a material is essential for practical applications. It allows the estimation of service and inspection intervals and safe usage of components with small defects, which is important especially for the aviation and aerospace industry.

1.2.4 Fatigue Behavior of High Strength Bearing and Spring Steels

1.2.4.1 General Aspects

Until recently, steels were thought to have a true fatigue or endurance *limit*, which represents a stress amplitude level below which these steels exhibit infinite

Fig. 1.11 Typical relationship between logarithm of the crack growth rate and of the logarithm of the stress intensity factor exhibiting three growth regimes. [18]



life (Fig. 1.8a). This concept was introduced first by Wöhler in 1860. Under constant amplitude loading, such materials typically showed a plateau in the S–N-curve starting at 10^6 cycles and beyond. However, the fatigue behavior beyond 10^6 cycles became a focus of scientific and engineering world after Naito et al. [21] had shown in 1984 that carburized and surface hardened steels do not exhibit a conventional fatigue limit at 10^6 – 10^7 cycles and fail even beyond 10^7 loading cycles at fairly low applied stresses, as schematically indicated in Fig. 1.8b. This phenomenon was observed predominantly for high strength bearing and spring steels, for which numerous studies on the fatigue behavior [22–49] have been published during the last two decades. Material failure beyond the conventional fatigue life, especially of such high strength steels employed for structural components, represents an important issue in today's fatigue research, since the required design lifetime of such parts often exceeds 10^8 – 10^9 loading cycles e.g. in gas turbine disks, car engines, and (high speed) railways. The number of data about the fatigue behavior above 10^7 loading cycles is rather limited compared to the data available in the low-to-medium cycle fatigue regime, due to time and thus, financial constraints at usual test frequencies. Very frequently, fatigue testing has been and still is carried out to $N_{\max} = 2 \cdot 10^6$ cycles only, based on the unfounded notion that above this N level the fatigue endurance strength is independent of N . Also the range of high strength steels that have been under investigation so far is rather small and is limited to AISI grades SAE-52100, and to JIS grades -SUP7, -SUP12, -SCNM439, -SCM435, -SCM440, -SUS304, -SUS316,

-SUS405, -SUJ2, -SWOSC-V, and -SMn443. In order to overcome the time constraints of usual testing setups such as rotating bending machines or servo-hydraulic tension compression devices, ultrasonic fatigue testing at frequencies of 20–30 kHz, which can provide fatigue data up to 10^{11} cycles at reasonable costs, became popular in order to solve the mentioned constraints. This fatigue testing technique will be discussed later in more detail.

The most important conclusion of all the above cited studies on the very high cycle fatigue behavior of high strength steels is that in the long life regime above 10^7 loading cycles these steels fail also below the conventional fatigue limit. Sonsino claimed that the existence of a real fatigue limit is a fiction [50]. Usually the material failure of these high strength steels in the very high cycle fatigue regime is associated with internal crack initiation and so-called fish-eye patterns at the fracture surface. Mughrabi [51] provided a comprehensive discussion of conditions at which internal fish-eye fatigue failure can occur at low load levels below the conventional fatigue “limit”. He assessed two major types of shapes of the so-called stepwise, duplex, or multistage S–N curves that can be observed for high strength steels. Figure 1.12a presents a so-called “twofold” S–N curve revealing two fatigue “limits”—one for surface failure and another for internal fish-eye failure. Figure 1.12b shows an S–N curve with a plateau starting at 10^6 cycles, which represents the conventional surface “fatigue limit”. However, due to internal defects within the material, such as nonmetallic inclusions, it fails also above 10^7 loading cycles at lower stresses. It should be noted that in that case a fatigue limit definitely *does not exist*. Mughrabi [51] performed statistical considerations in order to predict the probability of occurrence of internal fatigue failure. He concluded that if the volume density of internal defects such as non-metallic inclusions exceeds a critical value significantly, which depends on the specimen geometry and defect size, then the relative probability of occurrence of internal fatigue failure decreases while that of surface-induced failures increases. The occurrence of fatal cracks originating from the surface can only be ignored if the initiation of fatigue cracks at the surface is slower than in the interior. In this respect, Mughrabi [51] correctly also mentioned that other parameters such as the nature of the potential defects or residual stresses within the material might influence the fatigue behavior significantly and have to be considered.

1.2.4.2 Fatigue Crack Origins in High Strength Steels

Fatigue crack initiation in high strength bearing and spring steels was found to occur at the specimen surface at stress levels above the conventional (surface) fatigue limit up to 10^5 – 10^6 loading cycles. Surface crack initiation takes place at machining flaws, surface slips, nonmetallic inclusions located at the surface or at holes generated by breaking-out of inclusions. Beyond 10^6 – 10^7 cycles, life-controlling cracks tend to originate in the interior of the specimen, forming so-called internal “fish-eye” patterns at the fracture surface. In most cases nonmetallic inclusions, such as Al_2O_3 , TiN, SiO_2 , MgO, and CaO or sulfides, are observed in the center of the fish-eye,

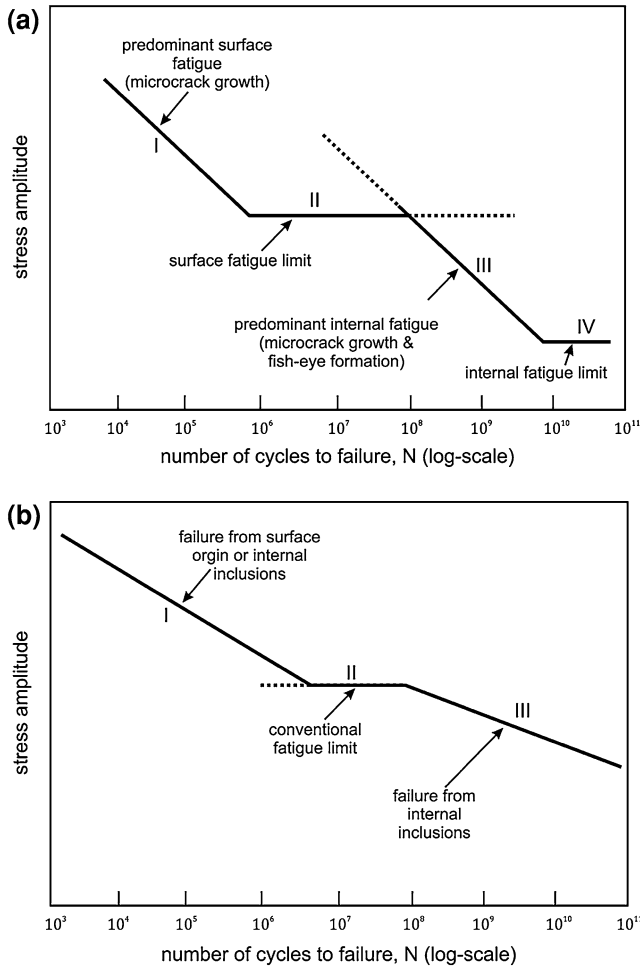


Fig. 1.12 Schematic illustration of two types of S–N curves described in the literature for high strength steels [37, 39]

acting as microcrack nucleation sites. Some studies [26, 27] have revealed so-called “matrix facets” or “internal facets”, i.e. crack origins where a nonmetallic inclusion of other defect was not observed, to be responsible for crack initiation. The majority of the studies showed internal origins to be responsible for the failure for the investigated high strength steels in the very high cycle regime. But it is noted that there are also works [42, 48] that revealed another crack initiation mode in addition to the internal fish-eyes, referred to as surface contact fish-eyes, which seemed to depend, amongst others, on the loading type. There, the crack origin was located close to the specimen surface, and smaller defects suffice for crack initiation, supposedly due to the stress field interaction of the defect particle and the free surface.

It has been shown that the area around the internal crack origin plays a critical role in the fatigue mechanism. Murakami et al. [36] introduced the term “optically dark area” (ODA) for this zone since it appeared dark in the light microscope. They reported that the specimen life time is the longer the larger the size of the ODA. Murakami et al. also claimed [35] that ODA crack growth is a synergistic effect of cyclic loading and hydrogen environment. Ochi et al. [41] called this zone ‘rough surface area’ (RSA) and obtained also RSA size-specimen life relationship, while the size of the inclusion proper did not reveal such a correlation. Sakai et al. [42] claimed the formation of microcracks or vacancies around the defect particle due to its stress field to be responsible for the formation of “fine granular area” (FGA) around the defect. Shiozawa et al. [44, 46] also observed a very rough granular morphology—a “granular bright facet” (GBF). Furthermore, those authors [46, 52] reported numerous spherical carbide particles with diameters smaller than 1 μm within the GBF, thus, relating the formation of the GBF to these carbide particles, which they proposed to be generated during the fracture process (although no description of the process is presented). They [46, 52] proposed a model for the formation of the GBF—“dispersive decohesion of spherical carbides”. There, they claimed that numerous microcracks are formed within the surroundings of the defect particle by decohesion of the small spherical carbides (a diameter of about 900 nm being given) from the matrix, resulting in the formation of a short fatigue crack. Tanaka et al. [47] called this special morphology zone around the crack origin “facet” (FCT). Summarizing, it can be argued that even if defects are smaller than the size required for fatigue crack propagation, a process exists during which microcracks can be formed and propagate even at stress intensities below the threshold value. Finally these microcracks forming this characteristic surface morphology join to form a small propagating fatigue crack.

1.2.4.3 Influence of Loading Type and Test Frequency on the Fatigue Data

Unfortunately, a variety of testing systems were employed in the studies published until now, operating at different loading ratios and test frequencies. Especially upon high frequency (ultrasonic) fatigue testing there are three major reasons why frequency effects are of concern: First, damping effects cause significant temperature increase [53], which might result in differences of the S–N data [54]. Furthermore, dislocation movements are slower compared to the fast loading–unloading at high frequency testing. Third, Murakami et al. [36] claimed that hydrogen embrittlement around crack initiating internal nonmetallic inclusions is the reason for internal fish-eye failure of high strength steels at very high cycle numbers. Thus, time might play a decisive role for hydrogen embrittlement. However, Furuya et al. [28] performed comparative measurements at 100, 600 and 20,000 Hz for a high strength low-alloy steel (JIS SNCM439). These authors [28] could not find any influence of the different test frequencies on the obtained S–N data and fracture surface structures. Marines et al. [55] observed “no noticeable

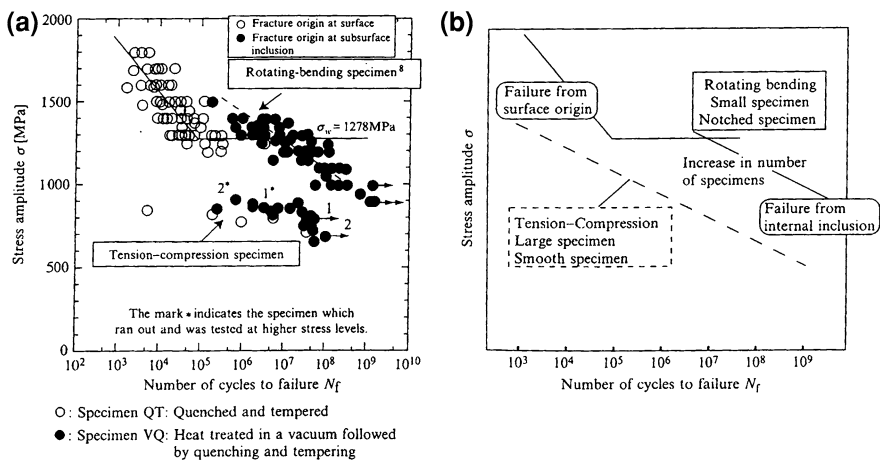


Fig. 1.13 Effect of the loading mode on the S-N characteristic: **a** for a high carbon chromium steel (JIS SUJ2), and **b** schematic variation of the S-N curve after Murakami et al. (reprinted from [34] with permission from Wiley)

frequency effect” on S-N data of cast iron in the tested cycle number range of 10^5 – 10^{10} , when loaded at 20 kHz and at 25 Hz, respectively. Similarly, Marines et al. [22] showed for an AISI-SAE 52100 bearing steel that testing at 20 and 30 kHz vs. 35 Hz caused only “very small” effects on the obtained S-N data. Spoljaric et al. [56] investigated the effect of testing frequency on the fatigue life of PM alloy steels reporting no significant influence on the obtained fatigue data. Weiss et al. [57] found the test frequency having only very limited influence on the obtained S-N curves for various metals and alloys.

Thus, while the frequency effect on fatigue behavior seems to be negligible, the influence of the loading mode cannot be denied: Rotating bending fatigue tests usually exhibit so-called ‘duplex’ or ‘multi-step’ S-N curves while in tension-compression tests such a stepwise shape was found by Wang et al. [23] only. Murakami et al. [35] compared fatigue data of the same high carbon chromium steel (JIS-SUJ2) obtained in rotating bending and tension-compression test, respectively (Fig. 1.13a). Obviously, the observed fatigue strength was lower in tension-compression mode and also the multistage shape of the S-N curve disappeared. Murakami et al. [35] attributed this different appearance of the S-N curves to the smaller test volume and to the stress gradient occurring in rotating bending tests (Fig. 1.13b). Accordingly, for a similar steel (NF 100C6) Marines et al. [22] observed a continuously decreasing shape of the S-N curve without any indication of a step and thus also proposed that the stepwise S-N curve rather represents the rotating bending behavior than the properties of the steel. These authors [22] also suggested that the specific appearance of the S-N curve originates from the stress gradient occurring in rotating bending tests. These authors

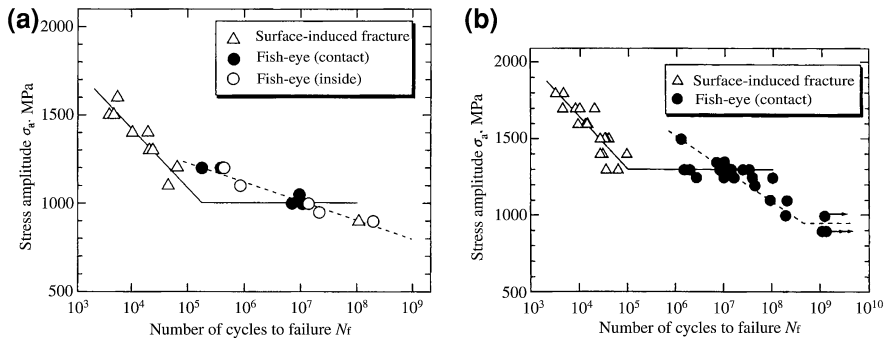


Fig. 1.14 S–N data of JIS-SUJ2 bearing steel under: **a** rotating bending, **b** axial loading after Sakai et al. (reprinted from [25] with permission of Wiley)

performed a stress correction of the rotating bending S–N data, i.e. considering that the maximum stress at a given crack nucleating inclusion depends on its distance to the surface and correlating the rotating bending data with tension-compression data of the steel, which led to a continuously decreasing curve, as obtained for tension-compression tests.

Another indication for the discrepancy of fatigue data between rotating bending and tension-compression tests is given by the observations of Sakai et al. [42] for a JIS-SUJ2 bearing steel. The origins of fish-eye failures obtained in rotating bending tests were exclusively located in surface contact (Fig. 1.14a). However, on axial loading in addition also fish-eye failure due to origins located inside the specimen were observed (Fig. 1.14b). Furthermore, again in the case of axial loading the shape of the S–N curve appeared to be rather continuous than stepwise as in rotation bending. The authors [42] attributed this behavior to the stress gradient present in rotating bending, with an additional comment that in rotating bending also the loaded volume is far smaller than in axial loading. Thus, in the latter case the number of critical defects, such as inclusions, in the loaded volume is significantly higher, which on the one hand makes crack origins in the interior of the specimen more probable, and on the other hand reduces the fatigue strength considerably compared to rotating bending loading.

Results of Nishijama et al. [40] further support the claim of Murakami et al. [35], since these authors [40] did not find a sharp, stepwise shape of the S–N curve although employing a rotating bending test system. However, Nishijama et al. [40] investigated also the effect of temperature on the fatigue response. Interestingly, at temperature of 300–400 °C a sharp stepwise S–N curve was obtained in contrast to experiments at room temperature and at 200 °C. These authors [40] attributed this observation to the formation of a hard oxide layer at the specimen surface at elevated temperatures that inhibits slip deformation, and thus restrains the formation of surface cracks. Consequently, the transition stress from surface-induced failure to internal failure is higher than for specimens without such a hard surface layer.

1.2.4.4 Effects of Surface Residual Stresses on the Fatigue Behavior

The observations of Nishijama et al. [40] clearly underline that differences of microstructure and hardness in the surface layer, possibly induced by different surface preparations of the specimens, might have a strong impact on the obtained fatigue properties. A good example is the occurrence of surface residual stresses, which are unfortunately not mentioned in many of the fatigue studies, which does not improve the comparability of the existing investigations on fatigue of high strength steels. The strong influence of residual stresses on the fatigue crack growth was shown by Berns et al. [58], who claimed that crack initiation is delayed in zones where compressive residual stresses are present, since they change the mean stress by reducing the loading stress and consequently the crack growth rate. On the other hand high tensile stresses below the compressive surface zone affect the mean stress by increasing the loading stress and the crack growth rate there.

Ochi et al. [41] investigated the effects of residual stresses, induced by specimen grinding, on the fatigue properties of a high-carbon chromium bearing steel (JIS-SUJ2) and a nickel–chromium–molybdenum steel (JIS-SNCM439). For specimens without surface compressive stresses (electro-polished) these authors [41] observed at the lower stress amplitudes both types of crack origins—internal and surface-related. In contrast, for ground specimens, which showed high compressive residual stresses at the surface, surface-induced failures occurred only at higher stress amplitudes. Masaki et al. [59] studied the effect of shot peening on the fatigue behavior of AISI 316L stainless steel and observed a significant improvement of the fatigue strength within the entire range of cycle numbers tested (10^4 – 10^8). Furthermore, these authors [59] noticed a partial relaxation of the initial compressive residual stresses during the rotating bending test. The extent of this relaxation seemed to depend on the loading cycle number.

Shiozawa et al. [45] investigated the fatigue behavior of shot-peened JIS-SUJ2 bearing steel. Two different variants of shot peening were performed: While in case of “SP” bombardment with high speed steel particles with a size of 500 μm was applied, for “WPC” treatment particles with a size of only 55 μm were used. The shot peening induced high compressive residual stresses at the specimen surface in both cases, however, by the WPC procedure relatively high tensile stresses were induced in the subsurface region. Figure 1.15 presents the corresponding fatigue data. Obviously, the shot-peening surface treatment suppresses the surface-induced failure at higher stress amplitudes, thus significantly improving the fatigue strength. However, due to the higher surface roughness induced by shot-peening procedure “SP” (larger particles), surface failure occurs at very high stress amplitudes, in contrast to the “WPC specimens”, which exclusively showed internal failure. At lower stresses only marginal differences were detected, since in this stress range only internal fatigue failure is relevant for the studied steel due to the low probability of occurrence of inclusion located at the surface.

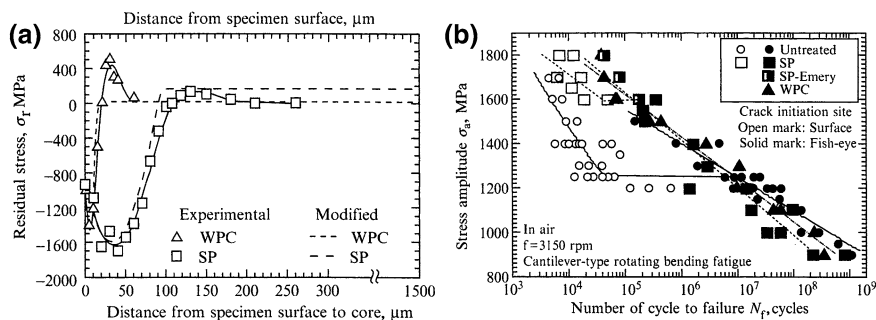


Fig. 1.15 **a** Residual stress distribution for the different surface treatments, **b** corresponding S–N curves obtained for shot-peened and ground bearing steel, respectively (reprinted from [44] with permission of Wiley)

Regarding the fact that in practice, residual stresses are virtually always present in the surface layer of a machined workpiece and that these stresses, induced by the applied machining operations, represent a crucial parameter of the surface integrity of the machined part, detailed investigations of the effect of residual stresses on the fatigue behavior, especially in the gigacycle fatigue regime, are required.

1.2.5 Fatigue Behavior of Tool Steels

1.2.5.1 General Aspects

The studies presented above described investigations into high strength bearing and spring steels only strengthened by a martensitic matrix, but containing a low amount of intrinsic defects, mostly non-metallic inclusions. In contrast to these steels, tool steels contain numerous primary carbides required for high abrasion resistance, often of the same size or even larger than the aforementioned inclusions. Primary carbides may also act as crack initiating “defects”. Thus, the amount of potential defects in tool steels might be several orders of magnitude higher than for the steels discussed above, which represents a major difference to the high strength structural steels with martensitic microstructure. Investigations [60–65] of fatigue behavior of tool steels, especially up to the gigacycle regime, are scarce. Berns et al. [60] were among the first to investigate fatigue fracture of tool steels. In rotating bending and compact tension tests of AISI D2 type tool steel, those authors found that fatigue cracks definitely started at the edges of primary chromium carbides if tempered below 200 °C. At higher tempering temperatures the carbides were fractured themselves, thus nucleating fatigue cracks. Fukaura et al. [62], who tested a JIS-SKD 11 (AISI D2 type equivalent) tool steel in a rotating bending system up to 10^7 cycles, also found primary chromium carbides as nucleation sites for fatigue cracks, resulting in internal

fish-eye type fracture at lower stress amplitudes. However, those authors did not take into consideration possible residual stresses, which play a critical role in crack initiation [58, 63, 64]. Berns et al. [61] compared powder metallurgically (PM) and conventionally produced (ingot metallurgy) high speed steels: In vacuum sintered PM steels, cracks started at pores and, after hot working, at nonmetallic inclusions mostly in the subsurface region. In contrast, for conventional cast and wrought tool steels those authors [61] observed that the primary alloy carbides located in the surface layer nucleate fatigue cracks, since they represent the largest discontinuity and have a high frequency of occurrence. Marsoner et al. [63] claimed that two types of crack starting modes occur during fatigue of PM tool steels—internal type at nonmetallic inclusions and surface type at nonmetallic inclusions or at primary carbides (aggregates). Those authors [64] investigated also one PM high speed steel in two different purity grades, obtaining similar results as those mentioned above. But, in addition to the surface and internal crack initiation modes, subsurface crack nucleation at nonmetallic inclusions was observed, and for the cleaner grade (lower content of nonmetallic inclusions) some internal crack origins were identified as carbide clusters. Furthermore, Marsoner et al. [64] claimed that “low residual stresses apecimen surface favor the subsurface crack nucleation” and that “the number of failures caused by surface carbide clusters increased significantly”. Those authors [64] also argued that there is no significant influence of the residual stresses on the surface and the internal fatigue limit; however, fatigue testing has been done only to 10^6 cycles in this case, and it is highly doubtful that a fatigue “limit” can be attained at these fairly low cycle numbers. Marsoner’s findings are supported by Meurling et al. [65], who did not find any significant difference in fatigue strength at 2×10^6 loading cycles for four surface conditions and corresponding amounts of surface residual stresses; however, once more the low maximum loading cycle number has to be considered. The studies of Meurling et al. [65] also revealed internal oxide inclusions and carbides as crack initiation sites for specimens with high surface residual stresses, and in absence of these compressive stresses, also carbides at the surface caused fatigue cracks.

Summarizing the existing literature on fatigue of tool steels, crack origins were found to be nonmetallic inclusions, but also primary carbides and carbide aggregates (primary carbide clusters), which were located in the interior or within the surface layer of the specimen. These studies investigated the fatigue behavior of tool steels up to 10^7 cycles. However, studies of the fatigue behavior of tool steels up to the gigacycle regime are missing until now. Furthermore, surface residual stresses might play an important role in fatigue behavior of tool steels, similar to high strength bearing steels as described above, which also has to be taken into consideration.

1.2.5.2 Development of Surface Residual Stresses

Since the investigation of the effect of residual stresses on the fatigue behavior of tool steels represented an important part of this work, in the following section the

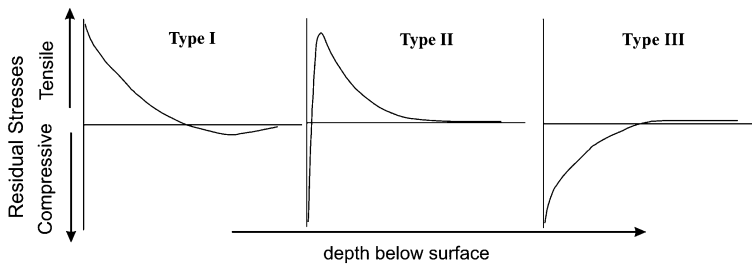


Fig. 1.16 Three types of surface residual stresses caused by machining operation, according to Parrish [66]

impact of machining operations on the evolution of surface residual stresses, especially in tool steels, is discussed in more detail. Parrish [66] defined three types of surface residual stresses, schematically presented in Fig. 1.16 which are caused by machining operations due to the combination of mechanical and thermal effects.

During type I machining, excessive friction heat development generated by shearing of the workpiece and rubbing of the machined surface against the clearance face causes local expansion of the workpiece, often accompanied by compressive plastic deformation. During the subsequent cooling the material is reduced to the original volume, thus inducing tensile stresses [67]. Type II represents a machining process where heat was generated, thus, tensile stresses result. However, also plastic deformation occurred at the surface which created compressive stress at the surface since heat generation was much lower there compared to type I machining. Type III represents the ideal machining where only surface work hardening takes place, resulting in compressive residual stresses at the surface and down to a certain depth to which plastic deformation has occurred, which prevents crack formation. However, below the compressive zone slight tensile stresses exist, balancing the compressive residual stresses. New trends of machining, such as hard turning or high speed milling (HSM), aim on inducing this beneficial type III residual stresses at the workpiece since these stresses improve the fatigue behavior. Ordás et al. [68] investigated the residual stresses in F-521 tool steel (AISI D2 equivalent) resulting from hard turning. Those authors found significant compressive residual stresses at and below the surface. They claimed that the amount of induced residual stresses is dependent on whether a new tool or a worn one was used, which was attributed to the difference of the sharpness of the cutting edge, thus a difference of friction area. Ordás et al. claimed that when a new cutting tool was used the magnitude of the induced compressive residual stresses was much lower and the penetration depth much shallower. Axinte and Dewes [69] evaluated the effect of HSM on the surface integrity of AISI H13 hot work tool steel. It was shown that high compressive stresses were induced dependent on the machining parameters such as cutting speed, feed per tooth, workpiece angle. For example, increasing the cutting speed was found to reduce the amount of compressive stresses, probably due to thermal effects. Poggie et al. [70] investigated the

influence of surface finish on surface residual stresses, friction and wear behavior for AISI A2, D2, and PM variant tool steels. Those authors [70] claimed that grinding with 62 grit aluminum oxide caused high compressive residual stresses at the surface. They also found that the surface residual stresses were reduced through unlubricated sliding wear. The extent of the reduction was dependent on the volume of the material removed. Poggie et al. [70] claimed that the decreased residual stresses “result from subsequent layers of material being removed in a non-abusive fashion”. Thus, underlying material without significant residual stresses is then exposed. Wear was not found to induce residual stresses. However, in this case the wear rates were found to be too low to remove all material deformed by surface finishing. Thus, considerable compressive residual stresses were present even after the wear experiments, which could be of high practical interest considering that the fatigue behavior of a material can be significantly influenced by surface residual stresses [64, 65], as described above. Another crucial factor for the formation of surface residual stresses is the type of lubricant that is used during machining or grinding. Xiao and Zhang [71] revealed that vegetable oil is more effective than either grinding fluid or cold compressed air in reducing the friction between tool and workpiece, which results in a good surface finish and increases the subsurface compressive residual stresses. Hence those authors [71] concluded that at small depth of grinding vegetable oil can replace conventional fluids in finish grinding, leading to superior properties in respect to surface integrity and reduce cost and environmental impact.

1.2.5.3 Anisotropy Effects on Fatigue Behavior of Cast Tool Steels

Since primary carbides definitely can nucleate fatigue cracks—as has been described above—any anisotropic distribution of these carbides in the steel might significantly affect the fatigue behavior, which has not been studied for tool steels until now. In bearing and spring steels, for which the occurrence of nonmetallic inclusions is decisive for fatigue behavior of the material, recent investigations revealed a strong influence of material anisotropy. Furuya et al. [24] showed for 1800 MPa-class spring steels that differences of fatigue strength were insignificant for specimens prepared from bars with different degree of deformation (hot working), which was attributed to the fact that effective inclusions were of similar small size in both cases. However, specimens with axis transverse to the rolling direction revealed significantly reduced fatigue strength [24]—about 50% of those parallel to the rolling direction. This effect is due to large, elongated MnS inclusions—definitely a fatigue anisotropy effect. Transverse specimens from bars with lower degree of deformation showed worse fatigue behavior due to larger inclusions. Temmel et al. [72] recently observed a similar influence of MnS inclusions on the fatigue behavior for low-alloyed structural steel (42CrMo4). Steel variants with high sulfur content revealed more pronounced anisotropy, which was attributed to the higher MnS population. Kaynak et al. [73] observed strong

anisotropic behavior of short cracks in Mn alloyed mild steel En7A with high content of elongated MnS inclusion.

In the present thesis the gigacycle fatigue behavior of wrought medium-carbon high chromium tool steel (AISI D2 type), containing numerous primary carbides, was investigated also with respect to possible anisotropy effects.

1.3 The Aims of this Thesis

The investigations presented in this thesis were performed in the framework of a 4 year termed joint research project of Vienna University of Technology (Institute of Chemical Technologies and Analytics; project leader: Prof. Dr. Danninger) and University of Vienna (Faculty of Physics; project leader: Prof. Dr. Weiss).

Over the past 30 years, numerous studies concerning the fatigue behavior of (PM) metallic materials, especially in the very high cycle fatigue regime employing the ultrasonic fatigue testing method, have been jointly conducted by the two project leaders. For example in the 1970s Weiss et al. studied the high cycle fatigue behavior of PM-Mo and Mo alloys [74, 75]. Danninger et al. [76, 77] evaluated experimental correlations between microstructure and mechanical properties of sintered iron. Spoljaric et al. [78, 79] showed the strong effect of singular defects and the influence of the production parameters on the fatigue behavior of low alloy PM steels in particular at high N. In Refs. [80–82] the microstructural features limiting the fatigue life of PM steels and structural parts are extensively discussed and the existing literature is thoroughly reviewed. Summarizing the cited articles, it can be stated that fatigue crack initiation in PM materials is always a combined effect of existing stress raisers such as inclusions, slip bands, precipitates, and the occurrence of pores, which result in superposition of the corresponding stress fields causing fatigue crack initiation. Especially the pore size, structure and geometry, which are affected by the production parameters play hereby an important role. Fatigue failure was frequently obtained beyond 10^6 loading cycles and a real fatigue limit was not obtained up to 10^9 cycles.

Based on the mentioned investigations, here the transition was done to studying the very high cycle fatigue behavior of fully dense PM materials, as PM tool steels are, and comparing their fatigue response to that of conventional cast and wrought (ingot metallurgy) tool steels. In principle, the reasons why the fatigue behavior of tool steels, also up to very high loading cycle numbers, is of high interest are threefold: First, for scientific purposes, providing fatigue data up to the gigacycle is necessary in order to contribute to the ongoing discussion about the shape of S–N curves for such high strength steels and about the question whether a real fatigue limit does exist for these materials or not. Secondly, during service operations, tools are repeatedly exposed to stresses and strains due to the contact between tool and workpiece, possibly leading to failure of the tool as a consequence of wear, but also due to fatigue fracture of the tool material [83, 84].

Furthermore, tool steels are also used for structural components e.g. in engine or aerospace industry, for which cyclic loading is a critical task.

Finally, fatigue testing at low stresses up to the gigacycle fatigue regime represents a reasonable tool for identifying defects, especially singularities, in the studied material. Furuya et al. [85] proposed that fatigue testing employing a 20 kHz ultrasonic fatigue testing system is a novel method for the inspection of inclusions in low-alloy spring steels. These authors [85] argued that this inspection method is superior to a conventional scanning of polished surfaces. For their purpose they had to conduct the fatigue tests in the region of low stress levels, for which predominantly internal fish-eye failures occur for the studied JIS-SUJ2 and JIS-SCM440 steel. Of course, such tests can also be performed at conventional fatigue test machines at lower test frequency, which however, would consume significantly more time. The major drawback of the high frequency method is given by the heat development in the specimens due to damping effects, which has to be taken into account. Probable frequency effects on the fatigue data have been found to be insignificant for high strength bearing steels, as discussed above. Once a 20 kHz ultrasonic fatigue testing setup including appropriate specimen design is established and adapted for the material type to be studied, this method is a reliable and fast way to identify the most detrimental defects within a material.

Since users of tool steels require continuing improvements of the material properties, especially with respect to reliability and life time, closer tolerances and finer surface finish, several processes to improve the steel quality have been introduced in the recent decades. Special refining processes such as electro slag remelting are nowadays state of the art in the production of ingot metallurgy tool steels, which decreases the amount of slag impurities significantly. However, in absence of these impurities larger carbides and carbide agglomerations, which are formed during the solidification process, represent potential failure origins. This problem was eliminated by introducing powder metallurgy tool steels, which showed significant smaller carbides. Consequently for PM steels the existence of nonmetallic impurities turned out to be the limiting factor. Improved atomization processes [86, 87] reduced the amount of nonmetallic inclusions and once more turned the focus on carbide aggregates as crack initiating species. According to [87, 88], less and finer carbides seem to be the appropriate way to improve the properties of powder metallurgy tool steels. However, it is obvious that detailed knowledge of potential crack raisers is required for further improvement of tool steel performance. Thus, in the present work an optimized and computerized 20 kHz ultrasonic fatigue testing system, operating in fully-reversed push–pull mode, was installed and adapted for the fatigue testing of hard, high strength tool steels, since this method, as described by Furuya et al. [85], offers the possibility of fast and accurate detection of potential material defects, especially for microstructural singularities such as inclusions.

The aims of the research presented here, which was principally conducted by the author of this thesis (Vienna University of Technology) in cooperation with Dipl.-Ing. Agnieszka Betzwar-Kotas (University of Vienna, Faculty of Physics),

on the fatigue behavior of tool steels in the gigacycle regime can be summarized as follows:

- Installation of a computerized ultrasonic frequency resonance fatigue testing system optimized for testing of such high strength steels, comprising a newly acquired ultrasonic generator, the acoustic horn, coupling piece, and test specimen cooling system, as well as configuration of appropriate fatigue test specimen geometries.
- Identification of potential crack origins in both conventional ingot metallurgy and powder metallurgy tool steels in the gigacycle fatigue regime. In this respect statistical considerations on the location of potential crack initiation sites will be made based on the investigation of the steel microstructure with regard to size of carbides and their probability of occurrence, and the respective literature will be discussed.
- Determination of the shape of the corresponding S–N curves and comparison to the types that can be found in the literature, which were discussed above. Furthermore, the existence of a true fatigue limit should also be assessed.
- Evaluation of material anisotropy effects in ingot metallurgy tool steels, since it has been shown for spring and low-alloy structural steels that anisotropic MnS distributions significantly affect the fatigue data, as described above. However, here—for tool steels—not MnS inclusions, but alloy carbides are responsible for any material anisotropy.
- As described above, surface residual stresses might have a considerable impact on the fatigue behavior of a material. Thus, the effect of surface residual stresses resulting from specimen grinding after the applied heat treatment was studied.
- Detailed analysis of the obtained fracture surfaces might provide information about the crack initiation and propagation process.
- Furthermore, fracture mechanical considerations based on fracture surface inspections and also on fatigue crack growth experiments could be accomplished.

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Chapter 2

Experimental

2.1 Investigated Materials

In the present work the fatigue behavior of five commercially available tool steels has been studied. The steels were acquired from Böhler Edelstahl GmbH (Austria) in annealed condition, also referred to as the “as-received” material. Two cold work tool steels (Böhler trade names K110 and K390) and three high speed steels (Böhler trade names S500, S600 and S590) were used. Steels K110, S500 and S600 are conventional (ingot metallurgy) tool steels, while K390 and S590 are produced by powder metallurgy (inert gas atomization + HIP). Table 2.1 shows the chemical composition of the studied steels, which was determined utilizing X-ray fluorescence analysis, except for the carbon content which was measured by a C, N, S-analyzer (LECO CS-230).

In the following sections the investigated steels will be presented in more detail together with specimen preparation and applied heat treatment procedures, while a comprehensive material characterization will be presented in Chap. 3.

2.1.1 Cold Work Tool Steel (Böhler K110)

The studied wrought cold work tool steel, Böhler trade name K110, is a member of *AISI class 430 D2* medium carbon–high chromium steels [1, 2]. The German standard classifies this steel as *X155CrVMo12.1* and by the steel DIN number 1.2379. The Japanese denotation is *G4404 SKD11*. Chromium forms numerous hard carbides, which are responsible for excellent wear resistance and remarkable deformation resistance. The high chromium content is not sufficient to provide the level of corrosion resistance characteristic for stainless steels since most of chromium is incorporated in the alloy carbides. However, the chromium concentration in the matrix is high enough so that these steels are very resistant against oxidation and staining, especially in as-hardened and polished condition.

Table 2.1 Chemical composition (weight %) and AISI classification of the investigated tool steels

Steel	AISI	C	Si	Mn	Cr	Mo	V	W	Co	S	P
K110	D2	1.55	0.32	0.32	13	0.85	0.89	0.12	0.12	0.006	0.014
K390	–	2.45	0.38	0.41	4.8	4.8	11	1.4	2.1	0.005	0.015
S500	M42	1.10	0.52	0.23	4.2	11	1.0	1.9	8.1	0.002	0.018
S600	M2	0.90	0.25	0.30	4.1	5.0	1.8	6.4	0.13	Not determined	
S590	–	1.32	0.51	0.33	4.5	5.8	3.5	7.8	9.2	0.007	0.017

The moderate carbon content of about 1.5% results in acceptable machinability and less brittleness compared to the early high carbon–high chromium cold work steels which had a high carbon content ranging from 2.00 to 2.50%. Molybdenum improves hardenability, especially air hardening ability, since it suppresses the formation of pearlite, and it improves toughness without having a negative effect on austenite grain size and retained austenite content. The influence of vanadium is somewhat ambivalent. Vanadium can be predominantly observed within the carbide phases and tends to inhibit grain growth. Thus, finer grain sizes are obtained, which however, comes along with a decrease in hardenability for vanadium contents >0.8% resulting in higher austenitizing temperature required for through hardening. The usual working hardness range for this cold work tool steel type is between 58 and 64 HRC, depending on the applied heat treatment temperatures. Broad usage of chromium alloyed ledeburitic tool steels dates back to the time of World War I. During this time these steels replaced tungsten high speed steels for cutting operations, especially in France due to the lack of alloying elements available at the war-restrained raw material market. However, it soon turned out that these steels showed insufficient resistance against high temperatures, but offered high wear resistance and crushing strength in cold work applications like blanking, forming, pressing, and shearing processes, which represent the main application field of this steel type until today.

This cold work tool steel is one of the most popular grades in usage and is produced by conventional ingot metallurgy. As mentioned above, the main alloying element in this steel is chromium, which is a strong carbide-forming element. According to the Fe-C-13%Cr phase diagram (Fig. 2.1) three different chromium carbides can exist in these high carbon–high chromium steels, depending on the ratio of chromium to carbon content. If this ratio is less than 3, the predominant carbide species is the alloy cementite $(\text{Fe,Cr})_3\text{C}$. If the chromium content is increased, or the carbon concentration lowered, the mentioned ratio exceeds 3. Thus, the two chromium enriched carbides M_7C_3 or M_{23}C_6 are observed. Molybdenum and tungsten tend to stabilize the M_{23}C_6 carbide [1].

In the annealed condition the steel consists of the ferritic matrix and different carbide types depending on the carbon content. At low carbon concentration only the carbide type $(\text{Cr,Fe})_{23}\text{C}_6$ exists. With increasing carbon concentration the carbides $(\text{Cr,Fe})_7\text{C}_3$ additionally appear, which replace the M_{23}C_6 entirely with increased carbon content. At even higher carbon concentration alloy cementite

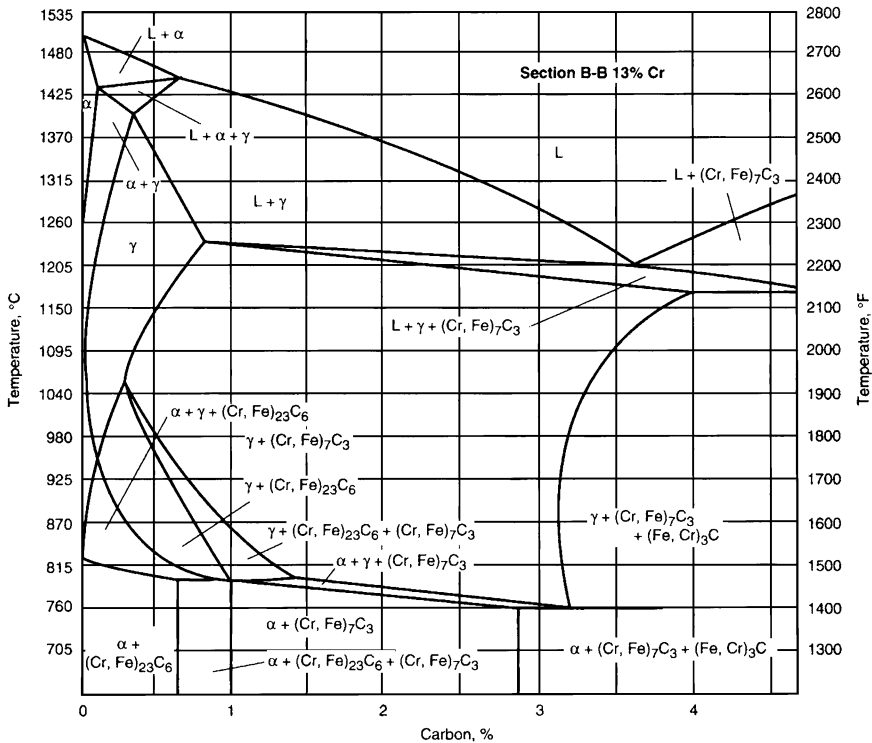


Fig. 2.1 Vertical section of Fe-C-Cr diagram at 13% Cr [2]—Reprinted with the permission of ASM International. All rights reserved (www.asminternational.org)

(Fe,Cr)₃C is formed in addition to the (Cr,Fe)₇C₃, both containing lower stoichiometric amounts.

During austenitizing AISI D2 type tool steels (about 1.5%C and 13%Cr) at 1000 °C, which is rather close to the usually applied austenitizing temperatures, and subsequently applying fast cooling, the following phase transformations take place (Fig. 2.1): Upon heating at about 780 °C, austenite starts to form, which is slightly above the critical temperature for plain carbon steels, since chromium is a ferrite stabilizer. At temperature above 1000 °C all of ferrite successively transforms to austenite. In addition to the austenite excess carbides of type (Cr,Fe)₇C₃ exist, which are not completely dissolved in the matrix below 1200 °C. Consequently, quenching of this steel from 1000 °C would result in a martensitic matrix, a certain amount of retained austenite, and undissolved excess carbides of type (Cr,Fe)₇C₃. The carbide type present in the as-quenched steel does not change during quenching and subsequent tempering, and can dissolve large amounts of iron. Tarasov [1] claimed for this carbide significant higher hardness (1820 Knoop) than the alloy cementite (1150 Knoop).

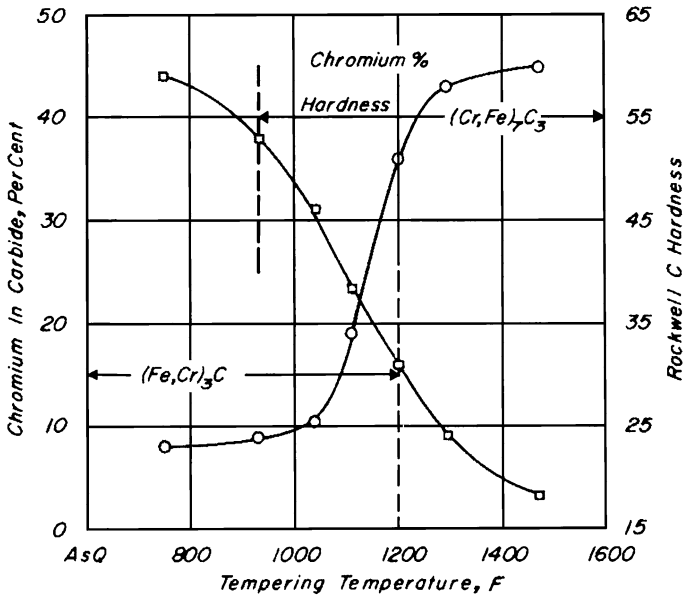


Fig. 2.2 Chromium content of carbide precipitated on tempering and the corresponding steel hardness [1]—Reprinted with the permission of ASM International. All rights reserved (www.asminternational.org)

However, the tempering temperature influences the nature of the carbides that precipitate upon tempering of chromium tool steels (Fig. 2.2). At tempering temperatures below about 530 °C predominantly alloy cementite precipitates from the martensite grains. However, with increasing tempering temperature also $(\text{Cr,Fe})_7\text{C}_3$ carbides emerge accompanied by an abrupt decrease of steel hardness. The chromium content of the secondary carbides increases with increased tempering temperature.

2.1.1.1 K110 Specimen Processing

The steel was supplied in form of annealed cylindrical bars with a diameter of 15.5 (bar A) and 106.5 mm (bar B), respectively. Hour-glass shaped fatigue specimens with their axis parallel to the rolling direction of the steel bars were machined out of both bars (designated as K110L and K110LL). In addition, fatigue specimens with axis in transversal direction (K110TT-M and K110TT-A) were manufactured out of the bar with the larger diameter. Specimens designated “K110TT-M”, were machined from the inner core of the 106 mm bar and specimens “K110TT-A” from “outside” were distinguished, as indicated in Fig. 2.3, which shows the schematic of specimen preparation from billet BB. First rectangular bars were milled and cut from the initial cylindrical rods, as presented in Fig. 2.4.

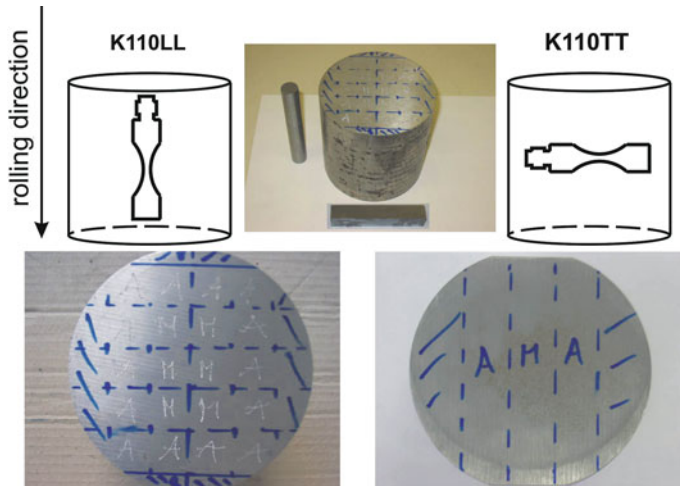


Fig. 2.3 Schematic of specimen preparation out of bar BB

High friction energy was generated during machining, which resulted in blue to violet colored chips (Fig. 2.4a). Subsequently, the obtained bars were turned to cylindrical bars of desired geometries, which were then machined to the fatigue specimen geometry.

All K110 fatigue specimens were machined to the desired geometry, which is shown in Fig. 2.5, and their surface longitudinally ground in annealed condition before the heat treatment. The grinding and polishing setup is presented in Fig. 2.6. The specimen rotates slowly while it is polished parallel to the specimen axis by a rapidly rotating disk. The rotating disk is spring mounted in order to avoid undesired pressure on the sample, which might induce stresses in the material. Vegetable oil was used as coolant and lubricant.

After pre-grinding the samples were cleaned in n-heptane in order to remove the lubricant vegetable oil. The steel samples were then austenitized at 1040 °C for 25 min in high purity N₂ (N₂ 5.0 = 99.999%) and quenched in oil. Tempering was done at 530 °C for 2 h in high purity N₂ with subsequent slow cooling of the specimens. A relatively high tempering temperature was chosen in order to lower quench-induced residual stresses and minimize thermal stresses in the workpiece, respectively. The heat treatment conditions for all of the studied steels are summarized in Table 2.2. After the heat treatment all samples were polished to mirror-like finish in the longitudinal direction. Two procedures have been applied: Specimens of Series I (K110L-I) were polished using a 600 mesh SiC paper and 12 μm chromium oxide paste. For specimens of series K110L-II, K110LL, and K110TT, after polishing with 240 mesh alumina abrasive paper, material removal of 40–150 μm depth was accomplished in the narrowest section of the samples using 15 μm diamond suspension, in order to remove surface residual stresses induced by grinding with abrasive paper. Details about residual stresses will be

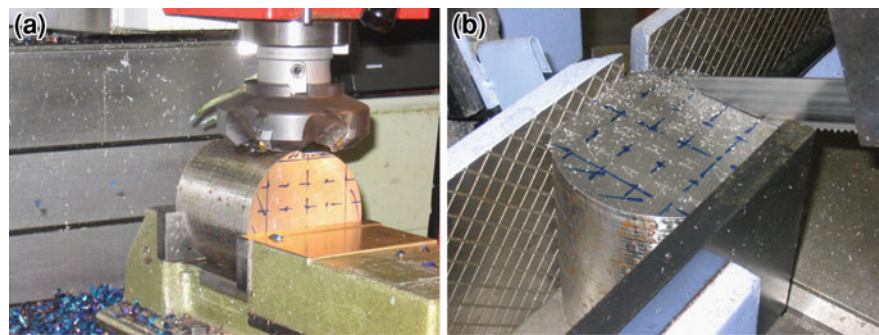


Fig. 2.4 Machining of specimens K100LL and K110TT. **a** Milling of K110 bar B. **b** Cutting of rectangular bars using a band saw

Fig. 2.5 Fatigue specimen geometry for wrought cold work tool steel K110

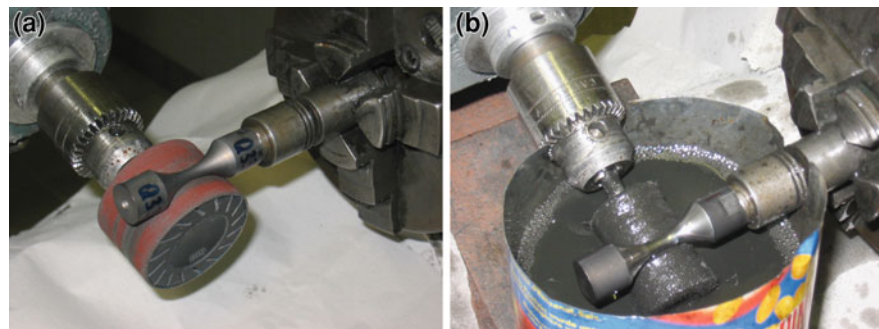
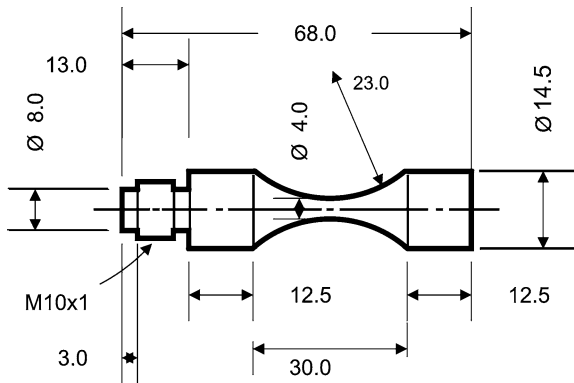


Fig. 2.6 Grinding and polishing setup. **a** Grinding with emery paper. **b** Polishing with diamond suspension

extensively discussed later. Subsequent polishing to mirror-like finish was performed using 6 μm diamond suspension. The diamond suspension polishing setup is shown in Fig. 2.6b.

Table 2.2 Heat treatment conditions for the five studied tool steels

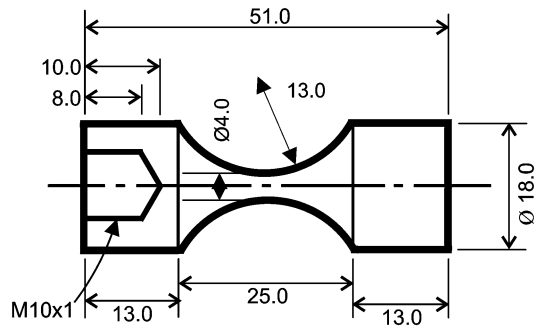
Steel	Test series	Specification	Initial bar diameter/ mm	Austenitizing temperature (°C)/ time (min)	Quenching medium	2 h tempering at temperature (°C)
K110	K110L-I	Axis = RD, high RS	15.5	1,040/25	Hardening oil	1 × 530
	K110L-II	Axis = RD, low RS				
	K110LL	Axis = RD	106.5			
	K110-TT	Axis ⊥ RD				
K390			20.5	1,040/25		3 × 560
S500			15.5	1,190/25		2 × 610/ 1 × 570
S600			18.0	1,200/30		2 × 610/ 1 × 570
S590			18.5	1,170/25		3 × 560

2.1.2 PM Cold Work Tool Steel (*Böhler K390 Microclean*)

In order to examine the effect of powder metallurgy production on the fatigue behavior, PM cold work tool steel K390, usually used at similar working hardness levels as the conventional variant K110, was chosen. The predominant carbides in steel K390 are the V-rich MC type carbides, in contrast to the Cr-rich M_7C_3 type carbides of steel K110. (A PM steel similar to K110, the former grade Böhler K190, is no longer available). There is no international classification comprising this highly V-alloyed tool steel. It has a high carbon content (2.45%), and a medium chromium and molybdenum content of about 5%. The dominant alloying element is vanadium (8%), which predominantly forms very hard MC type carbides. The high carbon content is required to balance the high amount of V, since the steel hardness would suffer a rapid decrease if high amounts of vanadium exist within the matrix. The reason for this phenomenon is that vanadium is a strong alpha-phase stabilizer. However, if the V content is appropriately balanced by the addition of sufficient carbon, the VC carbides improve the cutting performance and the red hardness of the steel significantly. In addition, the cobalt content of about 2% improves the hardness of the steel at elevated temperatures.

The steel was acquired as cylindrical bars with a diameter of 20.5 mm in annealed condition. Fatigue specimen preparation was performed in a similar way as described above for the K110 samples. Only specimens with their axis parallel to the rolling direction of the bar were machined, since in these steels anisotropy does not occur [3]. The specimens were machined to the desired geometry according to Fig. 2.7. It should be noted that the ratio of shoulder diameter to the diameters of the narrowest section is significantly larger here than for K110 fatigue specimens. This was required due to the higher strength of the PM material. Thus, higher stress amplification at the specimen was required to enable ultrasonic fatigue testing of the K390 cold work tool steel up to fracture. All fatigue specimens were machined

Fig. 2.7 Fatigue specimen geometry for PM cold work tool steel K390



to the required geometry and longitudinally ground prior to heat treatment. Austenitizing was performed at 1040 °C in N₂ (N₂ 5.0 = 99.999%), and subsequent quenching was performed in oil. After cooling to room temperature, the specimens were cleaned in n-heptane. Three times tempering was accomplished at 550 °C for 2 h soaking time each in high purity N₂. Then the specimens were cooled down slowly. After the heat treatment the samples were ground with 240 mesh alumina abrasive paper, and subsequently material removal to a depth of 80–100 µm was performed in the narrowest section of the samples using 15 µm diamond suspension. Then polishing to mirror-like finish was performed using 6 µm and also 1 µm diamond suspension here, since intrinsic defects in the PM material are assumed to be smaller than in the conventional ingot metallurgy steels, and thus the surface finish was expected to be more crucial for PM steels. The employed grinding and polishing setup is the same as that described above for K110 specimen processing. A detailed description of the microstructure of the steel will be presented later.

2.1.3 Conventional Ingot Metallurgy High Speed Steels (Böhler S500 and S600)

In order to evaluate the fatigue behavior of tool steels with significantly higher service hardness and higher strength compared to the cold work steels presented above, also highly alloyed high speed steels were investigated. These steels contain numerous alloying elements, some of which at quite high concentration levels. Characteristic for high speed steels are higher contents of tungsten and/or molybdenum. These two strong carbide formers are responsible for the extraordinary (red) hardness of these steels. Due to the fact that Mo shows the same impact as tungsten at half of the mass, i.e. at half of the costs, nowadays Mo-based high speed steels dominate the world market, despite the fact that early high speed steels were tungsten-rich, as described in the introduction. The two conventional

ingot metallurgy high speed steels studied here differ in the carbon, tungsten and molybdenum contents.

Steel S500 is a Mo-rich high speed steel of AISI type M42/DIN HS 2-10-1-8, containing a considerable amount of cobalt, which improves the steel red hardness. Furthermore, some Cr, W and V are also present in this steel. The carbide types that occur in the steel S500 are Mo-rich M_6C , which also contains W, small V-rich MC type carbides, and in the annealed condition Cr-rich $M_{23}C_6$ that however, dissolve at the high austenitizing temperatures applied. Furthermore, steel S500 contains about 8% cobalt, which enhances the red hardness. During austenitizing the M_6C carbides tend to dissolve more rapidly than the MC type carbides. Both of them are never completely dissolved, which inhibits undesired austenite grain growth. After quenching from the austenitizing temperature multiple tempering is necessary for high speed steels in order to eliminate the retained austenite existent in as-quenched steel. Furthermore, during tempering secondary hardening takes place, i.e. precipitation of finely dispersed M_2C and MC carbides. The M_2C type carbides are replaced by $M_{23}C_6$ and M_6C type carbides with increasing tempering temperature.

Steel S600 is an AISI type M2/DIN HS 6-5-2 Mo-W-based high speed steel, which contains similar amounts of these two strong carbide-forming elements. The carbon content is slightly lower than in S500 while it contains a somewhat higher content of vanadium, but no cobalt. The carbides contained are more or less the same as in S500. Also the fatigue specimen geometry was similar to that of steel S500.

Fatigue specimens were designed similar to the geometry proposed for the conventional cold work steel K110 (Fig. 2.5) and machined prior to the subsequently applied heat treatment. For S500 specimens, austenitizing was performed at 1190 °C for 25 min in N_2 (N_2 5.0 = 99.999%). The specimens were then quenched in oil. After cooling to room temperature the specimens were cleaned in n-heptane. Subsequently the samples were tempered two times at 610 °C, and a third time at 570 °C for stress relieving, both in high purity N_2 , followed by slow cooling. S600 specimens were austenitized at 1200 °C for 30 min in high purity N_2 . Tempering was performed as for the S500 specimens. The employed grinding and polishing setup and procedure was similar to that described above for K110 specimen processing. For the elimination of residual stresses at the surface, material removal of about 150 μm by the use of 15 μm diamond suspension was performed. A detailed description of the microstructures of the two steels will be given later.

2.1.4 Powder Metallurgy High Speed Steel (Böhler S590 Microclean)

Finally, a W-Mo-based powder metallurgy high speed steel was studied since PM steels show higher strength at the same hardness and contain smaller primary

carbides than the conventional ingot metallurgy variants such as the S500 and S600 steels presented above. The steel S590 has a high concentration of W, and a somewhat lower content of Mo, which form the very hard carbides as described above. Due to the higher W content, which slightly raises the A_{c1} temperatures, somewhat higher austenitizing temperature is required. Furthermore, the steel S590 contains 3.5% V, which is balanced by a higher carbon content compared to the two conventional high speed steels. As described in the introduction, the VC is a strongly covalently bonded, and thus extremely hard, carbide that accounts for high abrasion resistance and red hardness. Another remarkable feature of steel S590 is the very high cobalt concentration of about 9%, which improves the resistance against softening at elevated temperatures [4].

S590 fatigue specimens were prepared in the same way as K390 samples. Standard austenitizing was performed at 1170 °C for 25 min with subsequent quenching in oil. Three times tempering was done at 560 °C for 2 h. The specimens were slowly cooled from the tempering temperatures. Grinding and polishing of the as-heat treated specimens was performed as described for PM cold work tool steel K390.

Table 2.2 summarizes the heat treatment conditions used for the five investigated steels. The appropriate heat treatment conditions were elaborated with small cylindrical test samples up to a height of 10–20 mm. Austenitizing temperatures were selected to achieve medium working hardness for each of the steels. After austenitizing the samples were quenched in oil, which was applied to all studied steels, although some of the steels are even air hardenable grades. This was done in order to apply identical conditions. Generally, relatively high tempering temperatures were applied to reduce internal stresses within the fatigue specimens and to provide some ductility to avoid undesired fractures, e.g. at the screw of the samples. Cooling down from the tempering temperature was performed slowly. Slow cooling at a rate of about 20 K/min after tempering was accomplished in order to minimize residual and thermal stresses within the samples. All heat treatment procedures were performed in flowing high purity nitrogen gas atmosphere (N_2 5.0 = 99.999%).

2.2 Equipment Used and Procedures

2.2.1 Furnace Used for Heat Treatment

All heat treatments were carried out in a push-type laboratory furnace (“AHT Neu”, *Austria Heiztechnik GesmbH*, Austria) with gas-tight ODS superalloy retort (tube, internal diameter 75 mm) in high purity nitrogen with 2 l/min flow rate. For better temperature control an additional NiCr–Ni thermocouple was inserted into the furnace within a specially designed probe.

2.2.2 Dilatometry

For studies of the tempering behavior of cold work steel K110, dilatometric experiments were performed on a “*Netzsch DIL 402 C*” pushrod dilatometer with Al_2O_3 measuring system. The investigation focused on the transformations occurring during tempering, especially the behavior of retained austenite. Two procedures have been applied: Samples (about $55 \times 9 \times 5$ mm) were heated from room temperature at a rate of 5 K/min up to either 750 or 600 °C, followed by cooling to room temperature at the same rate. The change of specimen length was recorded in the course of temperature change. For better visibility of the effects, the dimensional changes are graphically depicted in the following as “coefficient of thermal expansion” (although of course e.g. transformation of retained austenite is not at all related to the CTE). Therefore, the absolute level of this virtual CTE is not of relevance here, but the critical temperature ranges can be clearly identified.

2.2.3 Determination of Mechanical Properties

2.2.3.1 Transverse Rupture Strength

The determination of transverse rupture strength (T.R.S.) was performed on cylindrical samples with 6 mm diameter and a length of 80 mm with surface finish using 600 mesh SiC abrasive paper. A *Zwick 1474* mechanical tester was used. Three specimens were tested for each steel test series presented in Table 2.2. Three-point-bending, for which the maximum bending momentum $M_{b, \max}$ occurs at half sample length (at the point where the load is applied), was used.

The load was continuously increased and recorded until fracture—at the maximum load—occurred. The maximum load was then used for calculation of the bending strength or TRS according to the following equation:

$$\sigma_B = \frac{F * L_S}{4 * W} \text{ (N/mm}^2 \text{ = MPa)} \quad (2.1)$$

where F is the applied load in N, L_S is the width between the two supports, and W represents the section modulus.

In case of cylindrical specimens the section modulus W is defined according to:

$$W = \frac{\pi * d^3}{32} \text{ (mm}^3\text{)} \quad (2.2)$$

where d stands for the diameter of the specimen.

For the round tool steel specimens tested the bending strength can be calculated according to the following:

$$\sigma_B = \frac{8 * F * L_S}{\pi * d^3} \text{ (N/mm}^2 \text{ = MPa)} \quad (2.3)$$

It has to be mentioned here that, due to the extremely high strength of the investigated steels, the hard metal support rods did not withstand and fractured. Thus, in place of hard metal, high speed steel supports were used, which however, suffered slight deformation during testing. The resulting systematic error can be up to 300 MPa depending on the strength of the material, and in any case has to be considered.

2.2.3.2 Hardness

The hardness of a material is commonly defined as the resistance against the penetration by a harder material. Usually, Rockwell hardness testing is applied to hardened steels such as tool steels. In the present work *Rockwell C* testing was used, for which a diamond cone is the penetrator at 150 kg load.

The Rockwell C hardness tests were performed on an *EMCO hardness tester (M4U-025)*. Five to seven indentations were performed on cylindrical test specimens used for the evaluation of appropriate heat treatment conditions and metallographic investigations. At fatigue samples usually two to three Rockwell C hardness measurements were performed for sake of quality control. It has to be mentioned at this point that Rockwell C hardness provides an integral material hardness since the indentation is significantly larger than the microstructural features, especially for tool steels.

2.2.3.3 Dynamic Young's Modulus

The elastic behavior of a material is extremely important in engineering since service is usually limited to stress levels at which only elastic, thus reversible deformation occurs. According to Hooke's law the occurring strain ε (which corresponds to the deformation) is linearly proportional to the applied stress σ (load):

$$\sigma = E * \varepsilon \quad (2.4)$$

where E represents the modulus of elasticity or Young's modulus.

There are two experimental ways for the determination of the elastic modulus: First, it can be derived from the linear behavior of the stress–strain curve obtainable in a static tensile test, which represents the rather classical method. However, for high strength materials this method is often difficult to perform since the clamping of the samples is tricky and the high loads required impose critical demands on the testing machine design. Generally, the static Young's modulus is

prone to experimental errors. Thus, a suitable, easy alternative is given by the determination of the dynamic modulus of elasticity based on the impulse-excitation technique. Here, the material object is subjected to an initial—elastic—deformation by means of a light mechanical impulse. Immediately, the object will act as a spring-mass system and produce a transient mechanical vibration. The frequency f of the occurring vibration is characteristic for the type of material. It depends on the mass, and the dimensions of the specimen, and on the modulus of elasticity. A piezo-electric detector, which is simply brought into contact with the sample, is used to record the vibrations, and to convert it into an electrical signal. Through this, the resonance frequency f of the fundamental oscillation (the other harmonics are hereby not considered) is measured, and the Young's modulus can then be calculated according to the following equation based on ASTM standard testing procedure ASTM E1876-99:

$$E = 0.9465 * f^2 \frac{m}{w} \left(\frac{L}{t} \right)^3 \quad (2.5)$$

where f is the resonance frequency of the material, m the mass of the sample, and w , t , and L the specimen width, thickness and length, respectively.

Rectangular ground specimens (100 x 10 x 10 mm) were utilized for measuring the dynamic Young's modulus. This was done on a *Grindosonic* resonance frequency measurement system, which consisted of a piezo-electric sensor and subsequent signal amplifier, and a frequency analyzer and readout device. The specimen was supported by two elastomere pads in order to minimize support effects on the resonance frequency of the sample object. Ten repeating measurements were performed for each test series. Results will be presented together with the other determined mechanical properties in [Chap. 3](#).

2.2.3.4 Microhardness

In order to determine the hardness of the matrix, and of some carbide species such as the chromium carbides, which were large enough to indent, microhardness measurements were performed on test samples on a Leco LM 100 testing system at a penetration load of 0.25 N. At least five parallel measurements were done for each test series.

2.2.4 Metallographic and Fractographic Investigations

Metallographic investigations were performed on test samples in order to evaluate the appropriate heat treatment conditions, and for material characterization in general. Steel sections were ground with emery paper (SiC grades) and finally polished using 6 and 1 μm diamond pastes in lubricant. In order to reveal the

microstructural features of the steels etching of the as-polished test samples were performed using the following etchants: 5% Nital (5 mL HNO_3 and 100 mL CH_3OH) was used to reveal prior austenite grain boundaries, Murakami etchant (10 g KOH /100 mL H_2O and 10 g $\text{K}_3[\text{Fe}(\text{CN}_6)]$ in H_2O) was used for carbide investigations. Diluted Adler reagent (100 mL H_2O , 200 mL HCl , 60 g $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$, 12 g $(\text{NH}_4)_2[\text{CuCl}_4] \cdot 2 \text{H}_2\text{O}$) and Picral (0.35 g picric acid in 10 mL Nital) were applied for revealing the microstructure after tempering.

Etched surfaces were investigated by means of light optical microscopy (*Olympus light microscope GX51*) and scanning electron microscopy (*Fei Quanta 200* and *Zeiss DSM 962*), the former one equipped with an energy dispersive X-ray analyzing system (EDAX). Image analyses were performed using a commercial software package (*analysIS Vers. 5.0 from Soft Imaging System GmbH, Germany*) and an open source software (*ImageJ 1.37 v from National Institute of Health, USA*).

Fracture surfaces obtained by fatigue and bending tests were also examined by means of scanning electron microscopy, and in some cases by light optical microscopy. The surfaces were ultrasonically cleaned in CH_3OH but not gold sputtered.

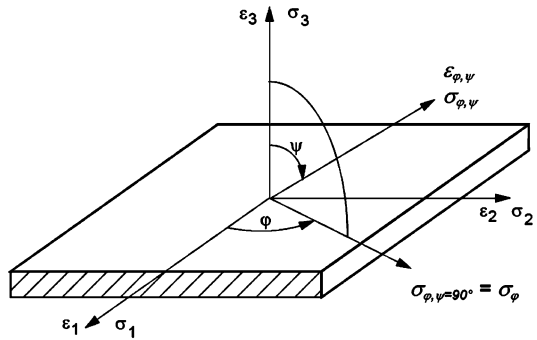
2.2.5 Residual Stress Measurements

Since it is known that grinding can cause considerable residual stresses at the surface, and since the fatigue test specimens used here have to be ground and polished after the applied heat treatment, it was decided to evaluate these—probably existing—stresses at the narrowest section, i.e. the gauge length, of the fatigue specimens. The determination of the residual stresses was performed at the *Institut für Werkstofftechnik und Metallische Werkstoffe, Universität Kassel* by X-ray diffraction, which is a state-of-the-art technique for non-destructive determination of residual stresses [5, 6].

Figure 2.8 shows schematically the stresses and strains occurring at a free surface. The stress σ_φ corresponds to the stress component parallel to the sample surface (at $\psi = 90^\circ$), which is most interesting in most cases. In the present study the residual stress σ_φ was determined in axial direction of the fatigue specimen, since stresses in this direction can definitely affect the applied stress, especially the mean stress, during fatigue testing, which will be described later together with the fatigue results. For some fatigue samples, measurements were also performed in tangential direction of the fatigue specimen. For some specimens axial residual stress depth profiles were also determined, i.e. the stresses at the specimen surface and at depth of 10–30, 50, and 70 μm , using electrolytic material removal and repeated X-ray diffraction measurements for each depth.

The measuring strategy for the residual stress determination using X-ray diffraction can be described as follows. According to the Bragg equation [5] the lattice parameter d can be calculated through determination of the diffraction angle 2θ , at which interference is observed. The lattice parameter d is affected by the

Fig. 2.8 Occurring stresses and strains at a surface [7]



presence of mechanical stresses, i.e. internal residual stresses. Thus, also the angles 2θ of the observed peaks, i.e. where interference is obtained, are changed. However, these changes $\Delta\theta$ are in the range of $0.01\text{--}0.5^\circ$, and thus, rather small. Differentiation of the Bragg equation leads to:

$$\boxed{\Delta\theta/\Delta d = -1/d * \tan \theta * 180^\circ/\pi} \quad (2.6)$$

Considering that

$$\boxed{\Delta d/d = (d - d_0)/d_0} \quad (2.7)$$

with d_0 being the lattice parameter in stress free material, corresponds to the strain ε in the direction orthogonal to the surface, the strain can be calculated as follows:

$$\boxed{\varepsilon = -\Delta\theta / (\tan\theta * 180^\circ/\pi)} \quad (2.8)$$

Thus, through determination of the diffraction angles 2θ the strain ε , and using Hooke's law, the corresponding stress σ_φ (see Fig. 2.8) can be calculated.

However, it has to be considered that the observed peak results from a number of crystallites within the volume penetrated by the X-ray. Thus, the measured interference represents always a mean of lattice parameter d in polycrystalline sample. Also the penetration depth of X-rays has to be considered in this respect. Furthermore, it is not sufficient to determine the lattice parameter, or the strain, respectively, in one direction only. The measurement procedure has to be repeated in several directions, which is often performed by conducting the diffraction experiment at constant azimuth-angle φ and varying the angle ψ , as schematically shown in Fig. 2.9. Subsequently, the determined lattice spacings d are correlated to the corresponding square of the sinus of angle ψ , which is commonly referred to as $\sin^2(\psi)$ -method. The slope of this linear relationship is proportional to the residual stress σ (Fig. 2.10).

A more detailed discussion of the determination of residual stresses using X-ray diffraction, especially of the mathematical aspects, can be found elsewhere [1].

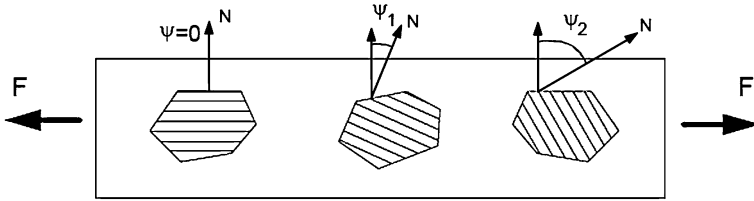


Fig. 2.9 Diffraction measurements under several angles ψ (here: by rotating the sample) for the determination of the residual stress component σ_ϕ by the $\sin^2\psi$ -method [5]

Fig. 2.10 Correlation of the measured lattice distances d with $\sin^2(\psi)$ [5]

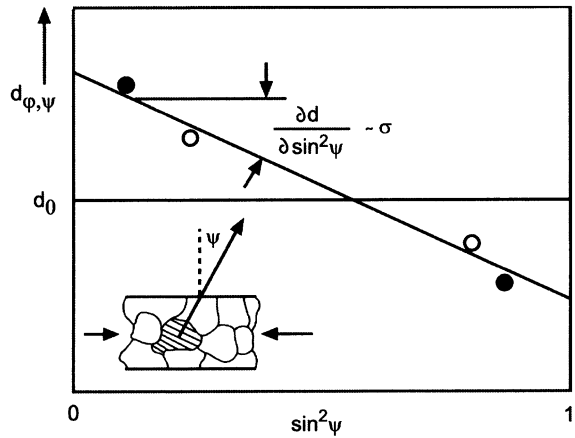
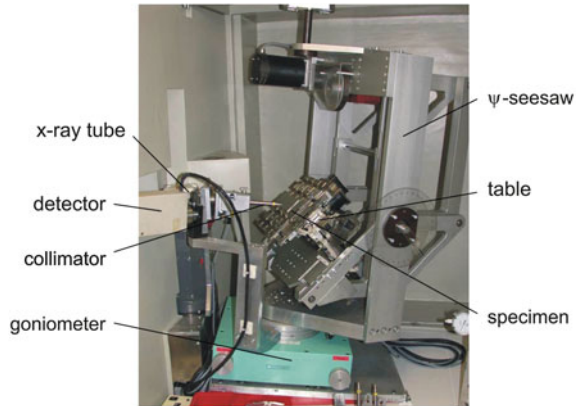


Fig. 2.11 Siemens type “F2” diffractometer for residual stress measurements (Institut für Werkstofftechnik und Metallische Werkstoffe, Universität Kassel, Internal Communication)



The employed diffractometer was a stationary ψ -diffractometer, i.e. the sample can be tilted in order to measure at different angles ψ , from Siemens, Germany of type “F2” (Fig. 2.11). The X-ray tube and the collimator are stationary in this device; the detector can be moved through the goniometer. Only angles ψ in the range of $-\theta < \psi < \theta$ are accessible, otherwise grazing incidence of the primary

Table 2.3 XRD measuring parameters for residual stress determination

Diffractometer	Siemens F2
X-ray radiation source	Cr K _α
Lattice plane	{211}
Tilt angle ψ	0, ± 18 , ± 27 , ± 33 , ± 39 , $\pm 45^\circ$
Measuring range of diffraction angle	148–164°
Diffraction angle step size	0.1°
Primary beam aperture	1.0 mm

Table 2.4 Diffraction data used for the residual stress determination

	Ferrite	Austenite	Aluminium
Lattice plane	{211}	{220}	{222}
Diffraction angle 2θ of undistorted lattice	156.07°	128.78°	156.71°
Measuring range of diffraction angle 2θ	147–163°	123–132°	150–163°
Voigt constant $\frac{1}{2}S_2$	6.09×10^{-6} mm ² /N	6.05×10^{-6} mm ² /N	18.56×10^{-6} mm ² /N

beam or shadowing by the sample would occur. Thus, interferences can be measured only at diffraction angles $2\theta > 90^\circ$, as shown in Table 2.3, where the measuring parameters are presented. High intensities at these rather high diffraction angles are achieved through using line focus instead of point focus. For the calculation of the residual stresses, data shown in Table 2.4 has been applied.

It has to be discussed at this point that during residual stress determination by X-ray diffraction three main systematic errors [5] can occur: (1) Adjustment errors of diffractometer axis, (2) non-linear $d\text{-}\sin^2(\psi)$ relationship deriving from texture, gradients or plastic deformation within the investigated material, (3) inaccurate X-ray elastic constants (e.g. Voigt-constants), which usually have an uncertainty of more than 5%.

Unsystematic errors results mainly from counting statistics and statistics of the microstructure of the investigated material, i.e. spatial inhomogeneity of the microstructure or stress distribution. Since it is not possible to account for all the occurring errors, commonly the following way for the estimation of the error of measurements is applied [5]:

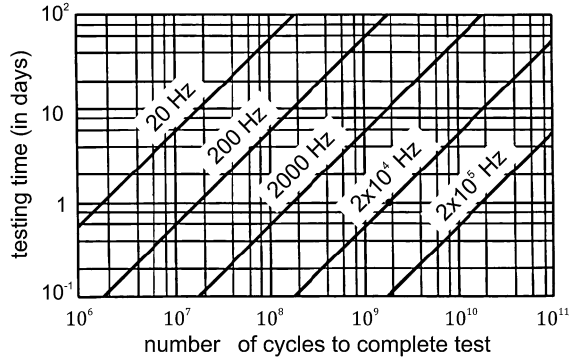
The sum S_q of the square of the deviations from the regression line of the $d\text{-}\sin^2(\psi)$ relationship is used to determine a standard deviation of the single measurements v_d that is given by:

$$V_d = \sqrt{(S_q/(n-2))}, \quad (2.9)$$

with n = number of measurements performed for the determination of the $d\text{-}\sin^2(\psi)$ -relationship.

Through error propagation calculation using the standard deviation of single measurement v_d , confidence intervals for the axis intercept and the increase of the $d\text{-}\sin^2(\psi)$ -line can be determined, from which then the error for the stress can be evaluated.

Fig. 2.12 Testing time as function of cycle number for several testing frequencies [8]



The errors resulting from the residual stress measurements performed for this thesis were in the range of 5–20%, relatively, which has to be considered when discussing the residual stress results presented in [Sect. 3.1](#).

2.2.6 Ultrasonic Fatigue Testing

2.2.6.1 Theory and Background

Ultrasonic fatigue testing is usually associated with cyclic loading of material at very high frequencies in the range of 15–30 kHz. A comprehensive description of this fatigue testing method can be found in Ref. [8]. The major advantage of this fatigue testing method is the possibility of performing fatigue tests very fast, and thus, at reasonable costs. Especially for today's high strength engineering materials, which have to withstand up to 10^{10} – 10^{11} loading cycles, ultrasonic fatigue testing represents an important alternative to conventional methods such as rotating bending or servo hydraulic systems. It has to be considered that a fatigue test up to 10^{10} cycles using a conventional system operating at 1 Hz would take 320 years. In contrast, at 20 kHz such cycle numbers can be reached within 6–7 days. Figure 2.12 shows the required testing times as function of cycle numbers for several testing frequencies. This enables to investigate more test conditions and to perform more parallel tests compared to conventional systems, which makes the results statistically more meaningful. However, it should also be considered that cycle numbers below 10^5 are not accessible by ultrasonic fatigue testing due to the high frequency (10^5 cycles are 5 s!).

The development of high frequency testing methods dates back to the beginning of the twentieth century. Example in 1911 Hopkinson [9] introduced the first electrodynamic resonance system operating up to 116 Hz, and in 1929 Jenkin and Lehman [10] used a pulsating air resonance fatigue testing system, which enabled testing at frequencies up to 10 kHz. However, as recently as in the mid-twentieth

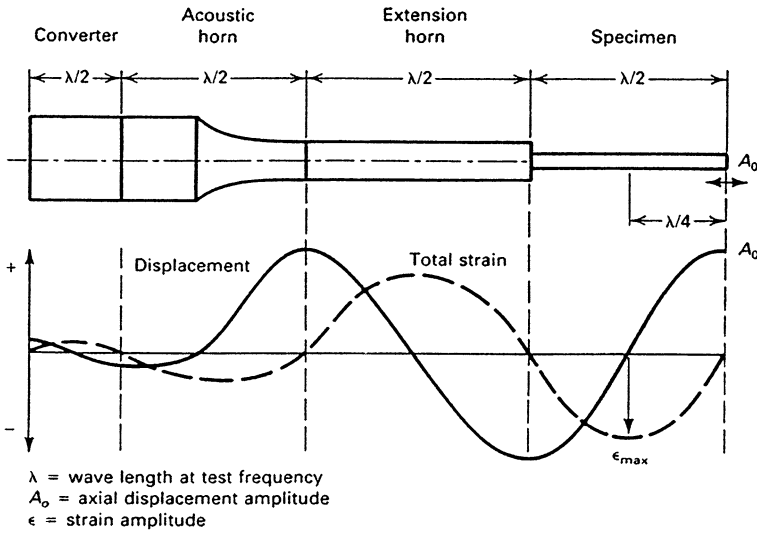


Fig. 2.13 Schematic of ultrasonic frequency resonance fatigue system and variation of the displacement and strain amplitude [19]

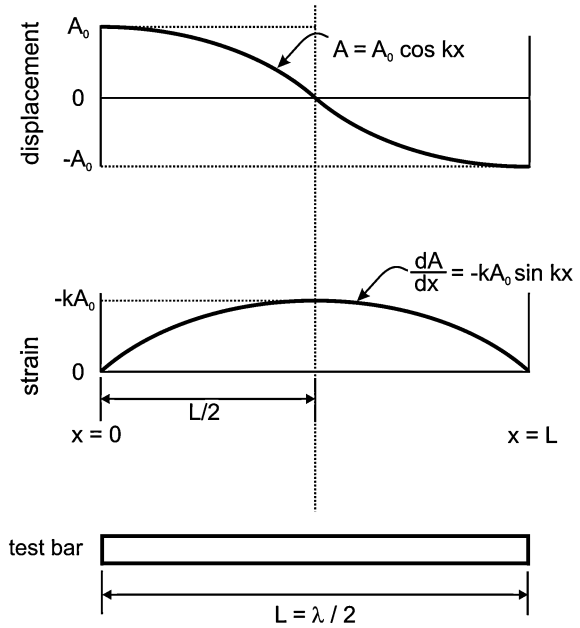
century Mason [11] introduced an ultrasonic frequency testing system operating at 20 kHz.

The core of this system consists of a kHz-generator and a magnetostrictive and/or piezoelectric transducer that transform the electrical signal into a displacement wave, which then is amplified by an acoustic horn and transduced to the test specimen. Every part of the system acoustic horn—optional extension horns—test specimen has to be in resonance, in order to achieve the required strain amplitude in the center of the specimen. In 1959 Neppiras [12] presented basic principles and developed mathematical equations for the design of the resonance system.

In the period from 1955 to 1980 various groups in Austria, Japan, Russia and the USA developed ultrasonic fatigue testing systems for sake of S–N curve determination up to very high cycle numbers, fatigue crack growth measurements, and corrosion fatigue experiments. Vidal et al. [13] described ultrasonic fatigue testing at 92 kHz in 1959. In 1973 Kromp et al. [14] developed an ultrasonic fatigue testing system operating at 20 kHz. Similar systems have been described later by others [15, 16]. Purushothoman et al. [17, 18] presented a high power ultrasonic fatigue system in 1973. In 1981 Stickler and Weiss [19] gave a comprehensive review of their development work and the application of the ultrasonic fatigue testing method.

Schematically, the experimental setup, which remained principally unchanged until today, is shown in Fig. 2.13.

Fig. 2.14 Displacement wave and corresponding strain wave over the length of a resonant test bar with uniform diameter [8]



2.2.6.2 Basic Principles of Resonance Fatigue Testing

In 1959 Neppiras [12] developed the equations that are helpful for determination of the resonance of the specimen length for different specimen cross sections. The basic principles that remained nearly unchanged till today are described in the following:

The displacement wave is generated by small periodic stimulus at the resonance frequency of the specimen, which depends on the material properties, since wave velocity C , which represents the speed of sound through the material, is defined for a test bar with a uniform diameter and length L (Fig. 2.14) according to:

$$C = \sqrt{(E/\rho)} \quad (2.10)$$

where E and ρ are the Young's modulus and the density of the material, respectively.

The length L of a resonant bar has to be $\lambda/2$. Considering also that

$$C = \lambda * f \quad (2.11)$$

with λ and f corresponding to the wave length and frequency, respectively, the bar length of a uniform test bar can be calculated as follows:

$$L = 1/(2f) * \sqrt{(E/\rho)} \quad (2.12)$$

Obviously, the material density and its elastic modulus are decisive for defining the test specimen length.

Furthermore, the development of the displacement and corresponding strain amplitudes are of importance, since at the point of minimum displacement, maximum strain is observed, as will be shown in the following:

The variation of the displacement amplitude A at a point x along a test specimen with uniform diameter is:

$$A(X) = A_0 * \cos(kx) \quad (2.13)$$

with $k = 2\pi/\lambda$.

As illustrated in Figs. 2.13 and 2.14, at half length of the test specimen, which corresponds to $\lambda/4$, the minimum displacement amplitude can be observed, since at $x = \lambda/4$ $\cos(kx)$ equals zero. In contrast, the strain amplitude $\varepsilon(x)$ reveals a maximum value at this point, since it corresponds to the first derivation of the displacement amplitude:

$$\varepsilon(X) = dA(X)/dx = k * A_0 * \sin(kx) \quad (2.14)$$

Thus, at $x = \lambda/4$ —at half length of test specimen— $\sin(kx)$ equals unity.

The stress acting at each point along the specimens axis can be calculated by using Hooke's law:

$$\sigma(X) = E * \varepsilon(X) \quad (2.15)$$

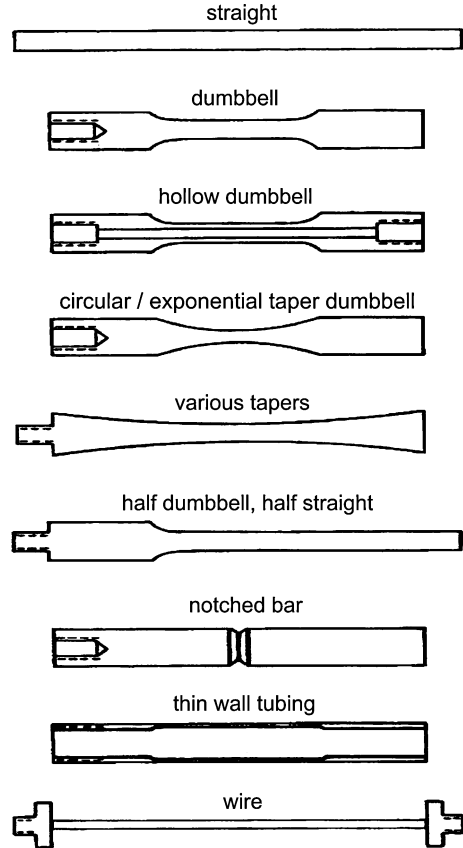
with E corresponding to the *dynamic* Young's modulus.

Due to the linear relationship between stress and strain it is obvious that maximum stress occurs at the point of maximum strain, thus, at half length of the test bar. An important consequence of the fact that loading of the ultrasonic fatigue specimen occurs only in the center of the specimen is that the requirements for fixing the specimen are rather tolerant. However, most important is a well-defined and good contact in order to transmit the displacement wave from the acoustic horn to the test bar and the other way round. The fact that hereby only one end of the sample has to be in contact with the signal source represents a major advantage of this fatigue testing method, since this enables the testing of thin or very tiny specimens such as microchips, wires, and sheets without the danger of buckling of these specimens. Also the axiality of the specimen is less critical than e.g. with servohydraulic testing.

2.2.6.3 Acoustic Horns and Specimen Geometries

The function of so-called acoustic horns is the transmission of the resonant wave from the converter to the test specimen. Usually, these horns also act as amplifiers for the wave amplitudes due to typical narrowing of the horn diameter, as

Fig. 2.15 Various geometries of ultrasonic fatigue test bars [8]



illustrated in the scheme of Fig. 2.13. As every part of the resonant system, also the acoustic horn must be of resonant length, thus its length should be $\lambda/2$. The reduction of the cross sectional area along the horn length induces an amplification of the wave amplitude, since in order to maintain the requirement of continuity of particle velocity an increase of the amplitude is required, according to:

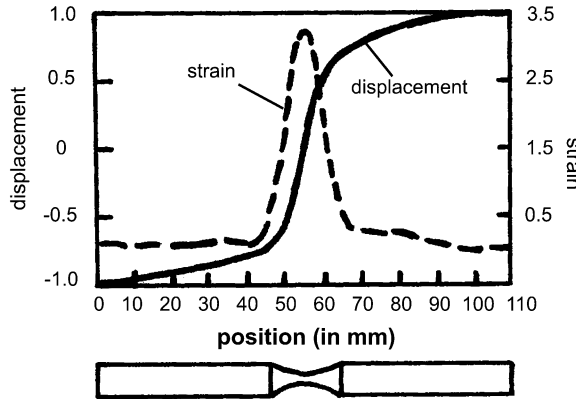
$$A_{\text{output}} = \text{area}_{\text{input}} / \text{area}_{\text{output}} * A_{\text{input}} \quad (2.16)$$

with $\text{area}_{\text{input}} > \text{area}_{\text{output}}$.

Of course, a decrease of the cross sectional area induces an increase of the wave amplitude, thus, causes the amplification. Gradual reduction of the cross sectional area (as illustrated in Fig. 2.14) is preferred to stepped horns, since high stress concentrations occurring at the steps might cause failure of the horns.

Above, the principles of ultrasonic frequency fatigue testing were explained based on test bars showing uniform diameter along their entire length. However, in praxis special geometries are used as presented in Fig. 2.15. Example dumbbell specimens are used to achieve additional amplification of the wave amplitude at

Fig. 2.16 Distribution of displacement and strain amplitudes for a dumbbell specimen [20]



the sample. This can, for example, avoid failure at the coupling point between test bar and horn or coupling piece, and can enable testing at higher stress levels with the same acoustic horn. But also the amount of material available and minimum diameter at the gage length in order to guarantee enough stiffness and also a reasonable test volume have to be considered in this respect.

However, each of these geometries, which differ from a straight uniform test bar, has an impact on the development of the displacement amplitude along the specimen length, and thus has to be evaluated. As an example, Fig. 2.16 shows the evolution of the strain and displacement amplitude for a dumbbell specimen. The most highly stressed region is limited to about ± 5 mm from the center of the bar. The strain at both ends of the specimen is negligible, which is positive for the clamping of the specimen.

2.2.6.4 Fatigue Specimen Design

In the present work two different geometries (Figs. 2.5, 2.7) have been used for the conventional ingot metallurgy and powder metallurgy tool steels, respectively. Due to the higher strength of the powder metallurgy tool steels, higher amplification of the wave amplitude at the specimen itself was required. Furthermore, inside thread was used for these steels in order to avoid stress concentration at the outer thread, which caused early failure of specimens with external screw thread. The geometries were derived according to the consideration presented above based on the principles introduced by Neppiras [12]. The dynamic Young's modulus was measured (results are presented in Chap. 3 together with the other mechanical properties determined for the investigated steels), and according to an equation for an hour-glass shaped specimen (Weiss et al. 1980, University of Vienna, Vienna, Austria, internal communications and others [21]), the fatigue sample geometries showing the desired amplification factor were estimated.

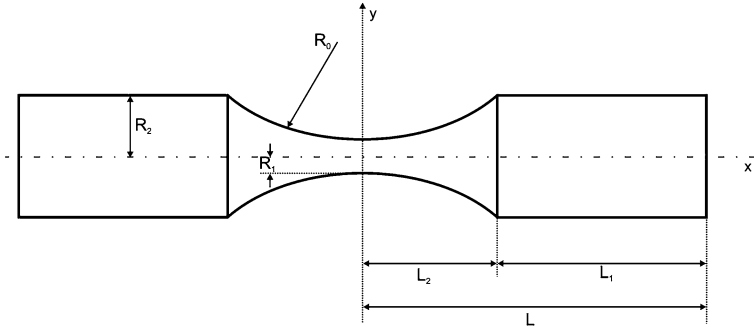


Fig. 2.17 Definition of dimensional parameters for fatigue test specimen design [21]

Table 2.5 Fixed dimensional parameters of fatigue test specimens for wrought and PM tool steels and corresponding steel densities

Dimensional parameters [mm]	Conventional ingot metallurgical tool steels K110/S500/S600	PM tool steels K390/S590
R_0	23.0	13.0
R_1	2.0	2.0
R_2	7.25	9.0
L_2	15.0	12.5
Steel density (g/cm^3)	7.7/8.3/8.1	7.6/8.1

In the following the calculation of the resonant length of the test specimens is described briefly. Figure 2.17 shows the definition of the dimensional parameters as used in the equations below. The radii R_0 , R_1 , and R_2 and the length L_2 were fixed for conventional ingot metallurgical and PM tool steels, respectively (Table 2.5). Young's moduli were determined as described in Sect. 2.2.3.3, and corresponding results are presented in Chap. 3.

Through the length L_1 the total specimen length has to be adjusted so that the sample shows resonance oscillation. The length L_1 can be calculated as follows:

$$L_1 = \frac{1}{K} \arctan \left\{ \frac{1}{K} \left[\frac{\beta}{\tanh(\beta L_2)} - \alpha \tanh(\alpha L_2) \right] \right\} \quad (2.17)$$

(Weiss et al. 1980, University of Vienna, Vienna, Austria, internal communications, and others [21]) with

$$K = 2 \pi f \sqrt{\frac{\rho}{E_d}} \quad (2.18)$$

The parameters α and β can be derived by:

$$\alpha = \frac{1}{L_2} \arccos h(R_2/R_1) \quad (2.19)$$

Table 2.6 Estimated and actual length L_1 of the fatigue test specimen

Steel	Estimated length L_1	Actual length $L_{1, a}$
K110/S500/S600	13.7/12.5/13.0	12.5
K390, S590	15.6/15.1	13.0

and

$$\beta = \sqrt{\alpha^2 - K^2} \quad (2.20)$$

Application of these equations gave the following length L_1 for the two test specimen geometries (Table 2.6):

Comparison with the actual length $L_{1, a}$, which was obtained by fine-tuning of the test samples through iterative adaptation of the specimen length in order to receive resonance oscillation at 20 kHz, shows that slightly shorter samples were used for fatigue testing. Furthermore, it has to be considered that by grinding and polishing of the fatigue sample, especially for the removal of surface residual stresses, the inner diameter of R_1 was about 0.2 mm smaller than the 4 mm used for the numerical estimation. Also the variation of the radius of curvature R_0 has to be taken into account, which is not considered by Eq. 2.17. However, principally the calculation delivered a rather good estimation.

2.2.6.5 Specimen Cooling During Testing

Due to the high frequency movements at 20 kHz the amount of energy transformed into heat, as a consequence of anelastic (damping) effects, is considerable, especially at high applied stresses. Thus, significant heating of the test bar might occur, depending on the stress level and material properties such as thermal conductivity. High temperatures can induce changes of the microstructure in the tested material as well as introduce residual stresses within the material due to temperature gradients from the surface to the core of the specimen. Both effects are capable of influencing the material properties considerably. Consequently, the generated heat has to be removed by some kind of cooling, which can be air jet cooling or a liquid cooling system. Another way for the removal of heat can be a hollow specimen design, which however, is not always feasible. The type of cooling that can be applied depends mostly on the material, i.e. its damping effects and thermal conductivity. However, in case of liquid cooling, which of course exerts a significantly higher cooling effect than compressed air, several important points have to be considered: The liquid has to flow over the specimen surface, since total and static immersion in liquid bath would influence the resonance system and, far more important, would cause erosion damage at the specimen surface. Furthermore, the employed liquid coolant has to be a non-aggressive, non-corrosive agent.

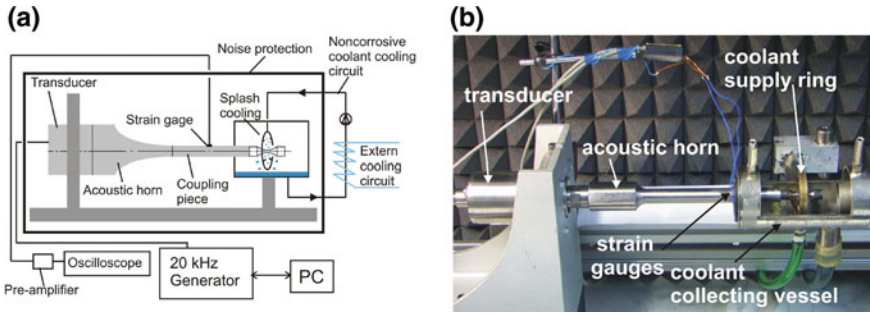


Fig. 2.18 **a** Schematic diagram of the newly installed ultrasonic fatigue testing system. **b** Photo of the vital parts of the resonance system

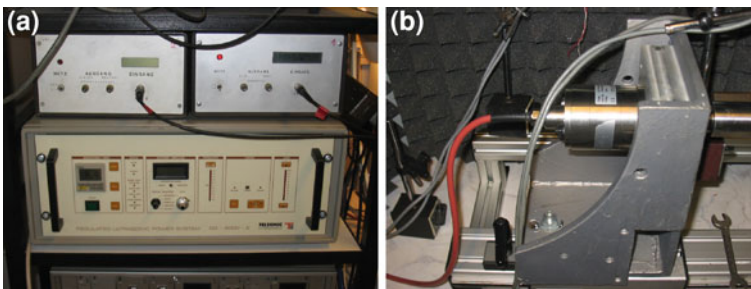


Fig. 2.19 **a** 20 kHz ultrasonic frequency generator. **b** Piezoelectric transducer

2.2.7 The Ultrasonic Frequency Resonance Fatigue Testing System

Based on the experience of more than 40 years of ultrasonic fatigue testing at the University of Vienna (Prof. B. Weiss et al.) a new ultrasonic frequency fatigue testing system was installed, of which a schematic is shown in Fig. 2.18a. The installation of this fatigue testing system, which was fully computerized and optimized for testing of high strength and very hard materials such as tool steels, represented a major part of this joint research project. The installation of the testing setup was jointly performed by Dipl.-Ing. A. Betzwar-Kotas (University of Vienna) and the author of this thesis.

The system consists of a new 20 kHz generator (Fig. 2.19a) operating within a power range of 0–2000 W, by which the electronic signal for the excitation wave is generated, and a piezoelectric transducer (Fig. 2.19b), which converts the electric signal into a standing resonant displacement wave. Both components, the generator and the transducer were acquired in the framework of the joint research project between the University of Technology (Prof. H. Danninger) and the University of Vienna (Prof. B. Weiss) were acquired from *Telsonic-Ultrasonics*,

Fig. 2.20 Acoustic horn and coupling piece

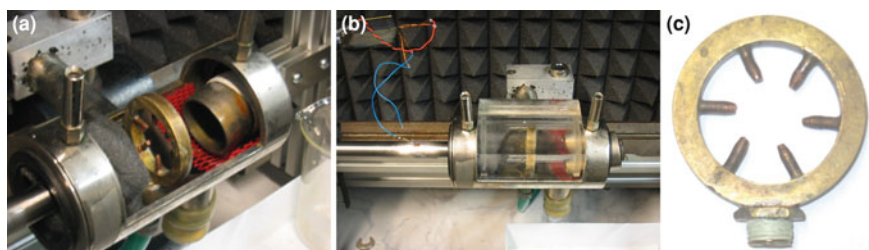
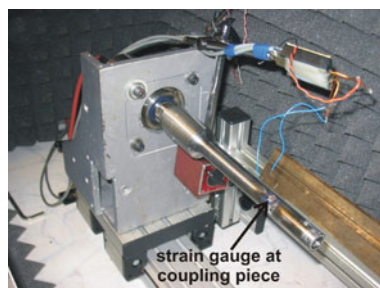


Fig. 2.21 Adapted liquid cooling setup used in principle for more than 15 years by the research group [19, 22 and others]: **a** opened coolant collecting vessel, **b** closed vessel, **c** coolant supply ring splashing coolant at six points onto the sample

Switzerland. Furthermore, the system consists of an acoustic horn for stress and strain amplification and a coupling piece (Fig. 2.20), which are both made of Ti6Al4V titanium alloy. The extension horn or coupling piece was required due to testing setup reasons, i.e. for cooling of the test specimen, which was required due to high heat generated through damping effects at the high testing frequency and due to the relatively high stresses applied.

Liquid cooling of the specimen was carried out during the fatigue tests using a noncorrosive coolant (3% *Aral Multrol* in distilled water), which was splashed onto the specimen surface as schematically illustrated in Fig. 2.18. Photos of the cooling system are presented in Fig. 2.21. From the supply ring (Fig. 2.21c) the coolant is splashed onto six points of the specimen surface in order to avoid cavitation effects, which, however, are highly improbable also since the mechanically loaded surfaces are oriented parallel to the direction of the longitudinal waves. The coolant then drops to the bottom of a collecting vessel, from which it is recovered and re-circulated, according to Fig. 2.18. In principle, this cooling method has been successfully used by the research group at the University of Vienna for more than 15 years [19, 22 and others].

The applied cooling granted constant testing temperature below 30 °C, as will be shown in Sect. 3.2.3. Temperature and cavitation effects on fatigue behavior can thus be excluded, but will be discussed later along with results of some comparative experiments, which were performed under compressed air cooling. Possible influence of corrosion on crack initiation—which however, seems unlikely since

Fig. 2.22 Schematic of the calibration procedure required for the measurement of the actual strain

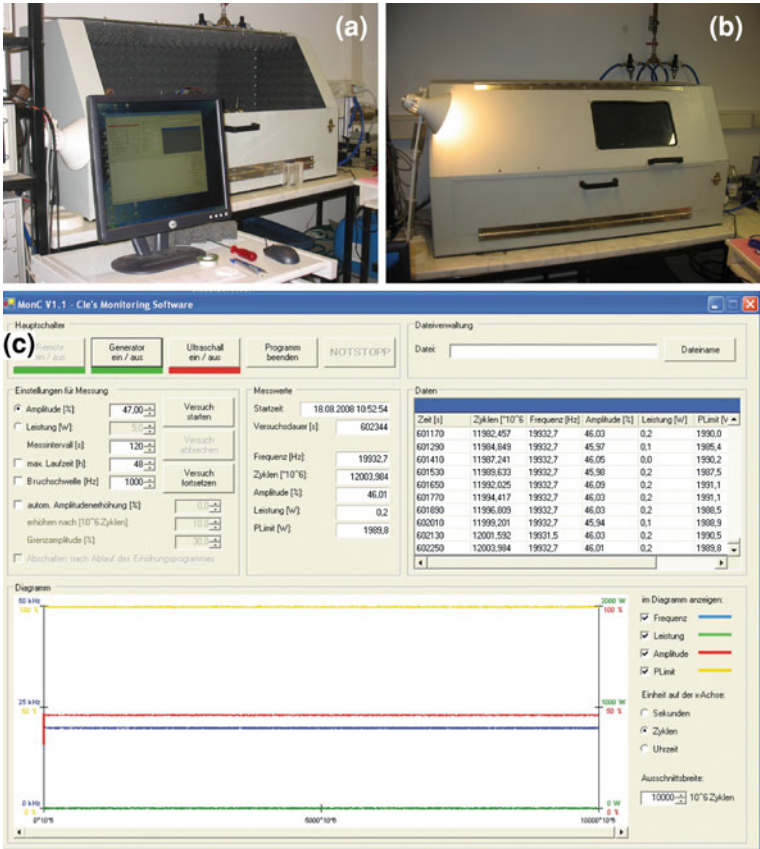
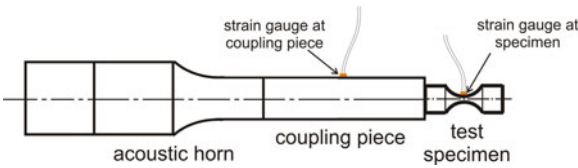


Fig. 2.23 a and b Overview images of newly installed ultrasonic frequency fatigue testing system; c user interface of proprietary monitoring software

noncorrosive coolant and short testing times were applied—will also be discussed later in more detail.

Direct strain measurement at the tested specimen was not possible due to the occurrence of high strains at the gauge length that would destroy any strain gauges and due to the necessity of specimen cooling. Thus, calibration was performed in such a way that miniature strain gauges (Hottinger Baldwin, Type 1.5/120LY11) were attached to the coupling piece and to a calibration specimen (inserted in place

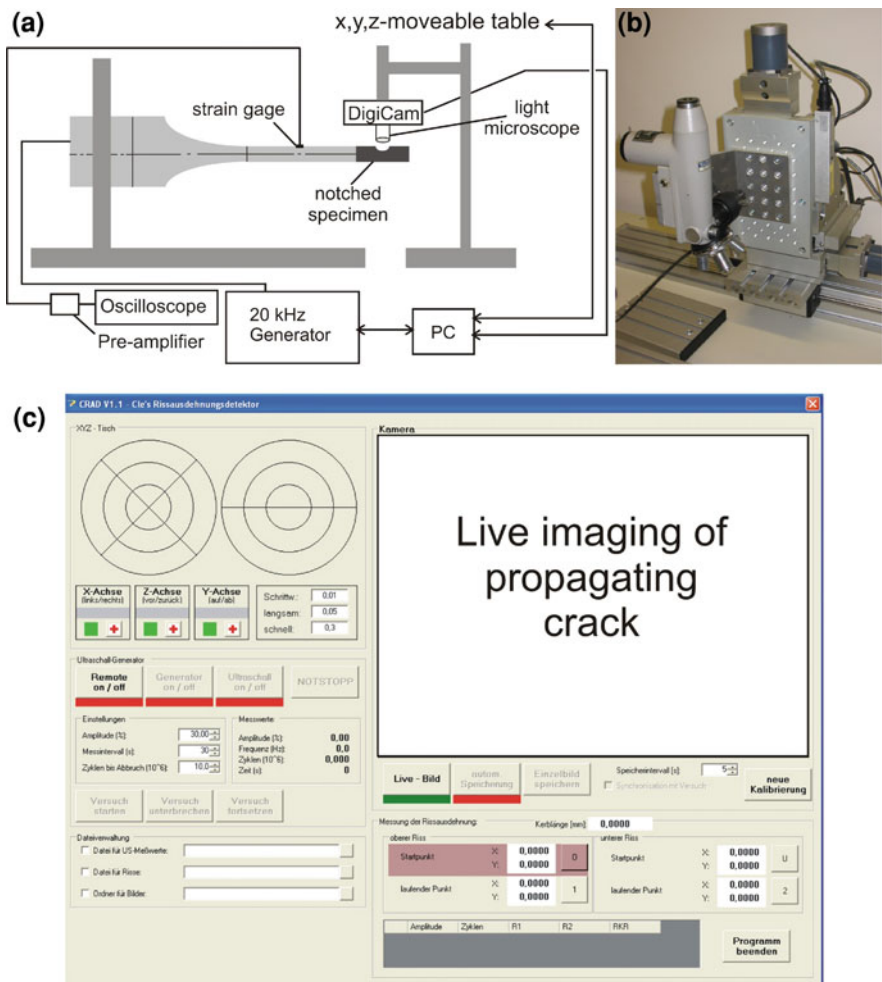


Fig. 2.24 Schematic diagram **a** of ultrasonic frequency fatigue crack growth system; **b** photo of the x, y, z-movable table with light microscope; **c** user interface of proprietary monitoring software

of the test specimen, consisting of the same material and having the same geometry), and the corresponding values of both gauges were recorded (see also Fig. 2.22). Then, during fatigue experiments the strain at the coupling piece was measured and the sample strain calculated using the calibration data. Multiplying the actual strain at the sample with the Young's modulus of the steel according to Eq. 2.15 gives the loading stress amplitude.

The generator is fully controlled by a personal computer using specially developed proprietary monitoring software, which also allows continuous

recording and online surveillance of measuring data. Figure 2.23c shows the user interface of the software. The oscillating parts of the testing system comprising the transducer, acoustic horn, coupling piece, specimens and cooling system are encased in a noise protection hood (Fig. 2.23a, b), which can be closed during testing, in order to minimize possible hazardous influence of ultrasound.

Furthermore, investigations of the crack growth behavior of the presented tool steels were aimed to be accomplished. Hereby, the ultrasonic frequency fatigue testing system was adapted according to the schematic in Fig. 2.24a principally based on the setup developed by Stickler and Weiss [19]. In addition to the oscillating system a movable x, y, z-table equipped with a light microscope and a digital camera (Fig. 2.24b) was installed in order to follow the growing crack. A specialized software routine was developed (Fig. 2.24c), which allowed controlling the US generator, the x, y, z-table and enabled the readout of the digital camera images and subsequent online determination of crack length in the course of time. Instead of round hour-glass shaped specimens flat, notched specimens were used for crack growth measurements.

The installation of the fatigue crack growth testing system and specimen design and preparation was more time-consuming than expected. Preliminary measurements were performed; detailed investigations of the crack growth behavior of tool steels will be a focus of interest of future projects.

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Chapter 3

Results and Discussion

3.1 Residual Stresses

In the introduction, the potential effect of residual stresses (RS) on the fatigue behavior has been discussed extensively (Sect. 1.2.4.4). In this work, systematic residual stress investigations were performed evaluating RS depth profiles, tangential and axial stresses, homogeneity around the specimen circumference, mechanical removal of highly stressed layers and relaxation phenomena.

3.1.1 Residual Stresses at the Surface and Depth Profiles

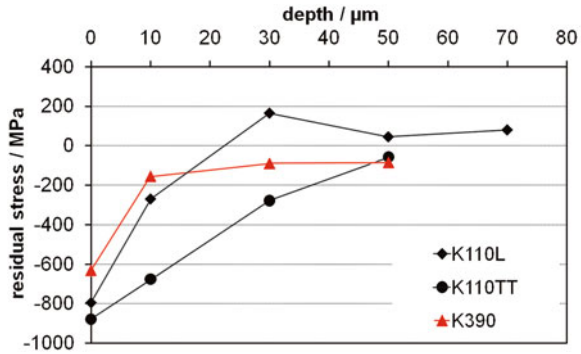
3.1.1.1 Ground and Polished Specimens

For the ingot metallurgy tool steel K110 it showed that specimen grinding and polishing to mirror-like finish after the applied heat treatment, even though fine SiC paper was used, caused relatively high compressive residual stresses at the surface, as data in Table 3.1 show. Obviously, the level of compressive surface stresses is similar for series K110L and K110TT. However, the penetration depth in case of the specimen with axis transversal to the rolling direction (K110TT) is significantly larger (Fig. 3.1), which can probably be attributed to different wear behavior due to the alignment of the elongated primary carbides. Grinding of K390 fatigue specimens induced significantly lower compressive stresses, likely due to the more homogeneous microstructure and finely dispersed, very small carbides ($<5\text{ }\mu\text{m}$), which also allows easier machining and causes less machining tool wear. The better grindability of PM tool steels compared to the IM variants has been known for a long time [1].

Table 3.1 Axial compressive residual stresses in MPa (at narrowest section) at surface and at varying depths after grinding and polishing of the as-heat treated fatigue specimens

Steel and test series	K110L	K110TT	K390
Internal sample number	K110-L49	K110-TT-Q8 ^a	K390-KP2
Depth from surface (μm)	Residual stress in MPa		
0 (=surface)	-795 ± 40	-878 ± 93	-630 ± 30
10	-270 ± 15	-676 ± 104	-155 ± 10
30	165 ± 20	-278 ± 155	-90 ± 10
50	45 ± 10	-58 ± 133	-85 ± 10
70	80 ± 5		

^a Mean of three measurements at one sample. Measurement by XRD ($\sin^2 \psi$)

Fig. 3.1 Residual stress depth profiles observed at ground and polished specimens of K110L, K110TT and K390 determined by XRD

3.1.1.2 Residual Stresses and Depth Profiles After Removal of the Compressive Stresses

Figure 3.2 shows the comparison of the existing residual stresses in representative as-ground and polished specimens and in specimens after removal of the surface residual stresses through material removal by polishing with $15 \mu\text{m}$ diamond suspension, i.e. material removal to a depth of $40\text{--}150 \mu\text{m}$ depending on the material, as described in Chap. 2.

The grinding, even though fine emery was used, caused considerable compressive stresses in the surface layer to depths of about $20 \mu\text{m}$, as described above. However, removal of the affected surface layer by polishing with $15 \mu\text{m}$ diamond suspension (K110L test series II) was an appropriate way for reducing the existing residual surface stresses considerably to about -140 to $+123 \text{ MPa}$ for K110L specimens (Fig. 3.2a, Table 3.2). Furthermore, the penetration depth of these relatively low stresses was much smaller than in as-ground and polished specimens. The other investigated tool steels showed similar stresses at the surface after the material with high compressive stresses had been removed by polishing with $15 \mu\text{m}$ diamond suspension (see Table 3.2).

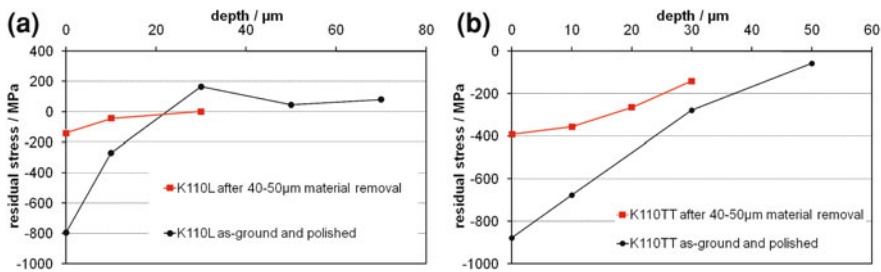


Fig. 3.2 Comparison of residual stress profiles at as-ground and polished specimens and at samples after removal of the surface compressive stresses through material removal: **a** test series K110L, **b** test series K110TT

However, for the transverse fatigue specimens of steel K110 (K110TT samples) it was not possible to virtually remove the residual stresses (Fig. 3.2b). Compared to the K110L and LL specimens (Fig. 3.3) relatively high compressive stresses were measured after material removal of 40–50 μm depth. Even increasing the depth of the removed layer to 150 μm still resulted in compressive stresses of -268 ± 82 MPa. However, obviously at least a partial reduction of the stress level was possible. The remaining compressive stresses affected the material at least to a depth of about 30 μm (Fig. 3.2b). This phenomenon was also attributed to the different microstructure-induced wear behavior that seemed to cause compressive stresses even when applying the 15 μm diamond suspension. This observation has to be considered for interpretation and comparison of the fatigue data obtained with this material.

Concluding, grinding caused high compressive stresses at the surface down to a depth of some tens of microns in each of the investigated tool steels. The removal of the surface zone, where the high compressive stresses were present after grinding, using 15 μm diamond suspension definitely turned out to be a suitable way to reduce these internal stresses to a relatively low level.

3.1.2 Axial and Tangential Residual Stress Profiles

In addition to the axial stresses, for some K110 fatigue specimens also the tangential stresses at the narrowest sections were determined, even though it can be noted that in respect to the direction of the fatigue loading the axial stresses are most relevant. These investigations showed that the tangential direction tended to show higher compressive stresses than the axial one (Fig. 3.4), however in most cases the trend was the same. It is noted here that the tensile stresses, which always balance the compressive stresses in the surface zone, often were not encountered at the depth measured. However, it can be expected that slight tensile stresses exist just below the layer with compressive stresses.

Table 3.2 Axial residual stresses (in MPa, at narrowest section) at the surface and at various depths of fatigue specimens after material removal using 15 μm diamond suspension

Steel and test series	K110L ^a	K110LL	K110TT	K390	S500	S600	S590
Internal sample number	K110L-55, K110L-69, K110L-70	K110-LL6	K110-TT-Q6 (material removal of 80–100 μm)	K110-TT-Q7, K110-TT-Q9 (material removal to a depth of 150 μm)	S500-S2	S600-S56	S590-PS26, S590-PS27
Depth from surface (μm)	Residual stress in MPa			Residual stress in MPa			
0 (=surface)	-140 ± 123	-165 ± 10	-297 ± 158^a	-268 ± 82	-161 ± 79	-130 ± 10	-103 ± 57
10	-43 ± 53		-355 ± 20	^a _b			
20	-10 ± 127		-265 ± 15				
30	3 ± 14	-25 ± 5	-140 ± 15				
50		100 ± 10					

^a Mean of three measurements at three samples
^b Mean of two measurements at two samples
Errors include error of measurement and eventually standard deviation of mean value calculation

Fig. 3.3 Residual stress depth profiles at fatigue specimens of steel K110 after material removal by polishing with 6 μm diamond paste

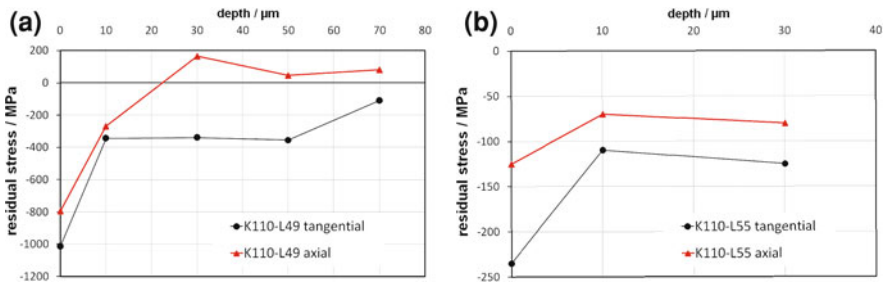
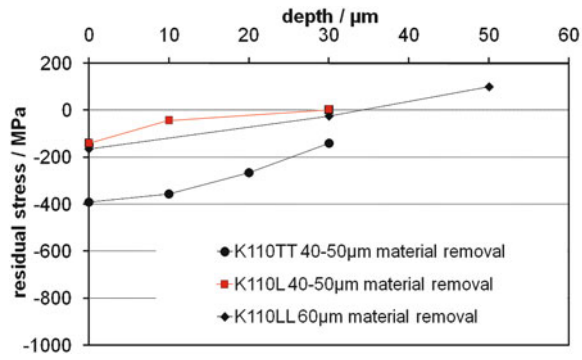


Fig. 3.4 Comparison of axial and tangential residual stress profiles obtained for K110 fatigue specimens: **a** as-ground and polished, **b** after 40–50 μm material removal

3.1.3 Homogeneity of the Residual Stresses Over the Specimen Circumference

At four specimens the residual stresses were determined at several points along the specimen circumference (0° , 45° , 90° , 180° , 270°) at the narrowest section of the specimen (Fig. 3.5) in order to determine any orientation dependence of the residual stresses, caused by the grinding and polishing process or due to material anisotropy.

The investigation of residual stresses on as-ground and polished fatigue specimen K110TT—with high compressive residual stresses at the surface—showed that the stresses were quite similar at the three points of measurement (Fig. 3.5) at the surface. At 30 and 50 μm depth a variation of the measured stresses of about 200 MPa was realized. However, a severe influence of the material anisotropy or the grinding process was not found.

Furthermore, similar investigations have been performed on specimens for which the high compressive residual stresses had been largely removed by polishing with 15 μm diamond suspension. It turned out that there was some variation of the observed stresses (Fig. 3.6) at the surface. These variations however were in the range of those measured at a depth of 30–50 μm in as-ground and polished

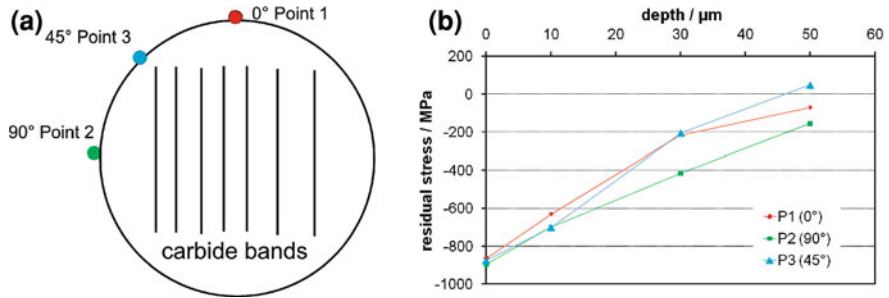
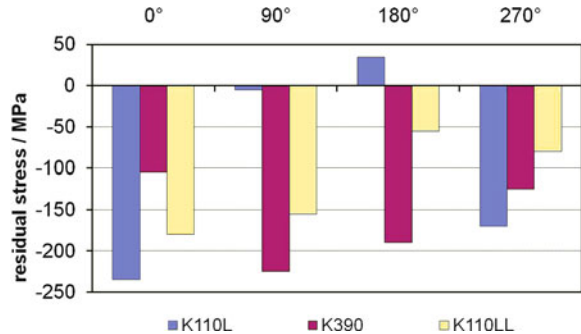


Fig. 3.5 Residual stress profiles at the circumference in the narrowest section of ground and polished (as described for test series K110L-I in Chap. 2; without removal of compressive stress layer) K110TT-Q8 fatigue specimen

Fig. 3.6 Residual stresses at the circumference in the narrowest section of K110-L94, K110-LL7 and K390-KP1 specimen, for which the compressive stresses have been largely removed by polishing with 15 μm diamond suspension



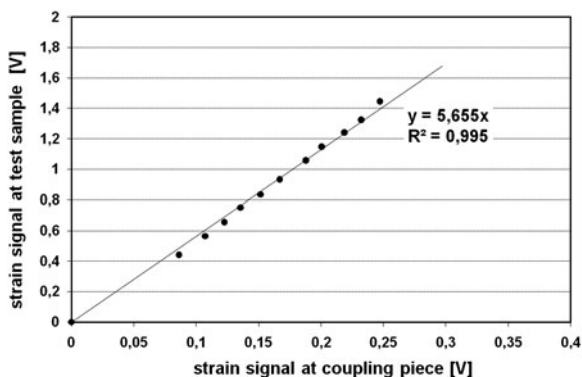
specimens, as presented above (Fig. 3.2a). Thus, it can be speculated that these variations of the stresses existent after material removal come into existence during the prior grinding process. Furthermore, Fig. 3.6 shows that the PM tool steel K390 revealed the lowest scatter of the stresses measured around the narrowest circumference, which can be a direct consequence of the isotropic PM material, especially the isotropic carbide distribution. In contrast, steel K110 showed higher variations of the measured stresses at the surface, probably resulting from the anisotropic distribution (which will be discussed later in more detail) of the primary carbides in this tool steel.

3.2 General Evaluation of the Fatigue Testing Method

3.2.1 Calibration of Fatigue Testing

Since direct strain measurement at the tested specimen was not possible due to the occurrence of high stresses and strains, which would cause failure or decohesion of

Fig. 3.7 Calibration for test series K110L-I (which will be described later) under liquid cooling. The calibration factor here was 5.655



the strain gauge from the sample, and necessary specimen cooling, a calibration procedure has to be performed. This is a commonly accepted way for determining the actual applied strain and stress, which has been successfully used for more than 35 years.

Miniature strain gauges were attached to the coupling piece and to a calibration specimen (inserted in place of the test specimen, see also Fig. 2.22), and the corresponding signals of both gauges were recorded for several strain levels (Fig. 3.7). Through this a linear relationship was determined, the slope of which gave the calibration factor k_C . Then, during fatigue experiments the strain at the coupling piece was measured and the sample strain was calculated by multiplying by the corresponding calibration factor k_C . The calibration was performed for each test series separately, which was necessary due to the fact that the material removal by polishing resulted in slightly changed specimen diameters at the gauge length.

The measured signal of the strain gauges was in volt, from which the actual strain was then calculated using a factor supplied by the strain gauge manufacturer.

Possible effects on the calibration factor exerted by the cooling medium and differences between individual strain gauges were assessed exemplarily for steel K110.

Figure 3.8a shows the calibration factors determined for three K110 test series under compressed air and liquid cooling. Any significant difference between the calibration factors for the two cooling media was not observed. Thus, the determination of the calibration factor can be performed in air, which is easier, even if the real fatigue experiments are then performed with liquid cooling. Furthermore, the difference of the calibration factors for the three test series turned out to be in a range of k_C of about 5.5–6.1 for a given measuring setup, i.e. for the same strain gauges during the comparative measurements. The reason for this variation can be found in the fact that the specimens for which material removal, i.e. removal of the compressive residual stresses, was performed (K110L-II and K110TT), had somewhat smaller diameter at the gauge length compared to the as-ground and polished specimens (K110L-I). Thus, the K110L-II samples showed a higher amplification of the displacement wave, i.e. a higher amplification factor.

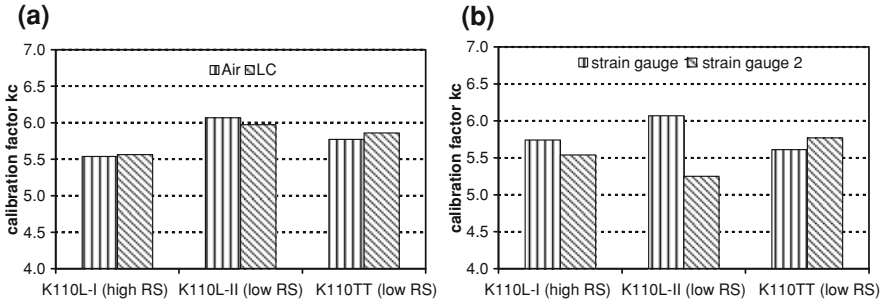


Fig. 3.8 Determined calibration factor k_C for three test series: **a** comparison of cooling by compressed air versus liquid; **b** evaluation of the error induced by strain gauges (and their attachment)

However, the calibration factor can be assessed as being constant for the entire test series.

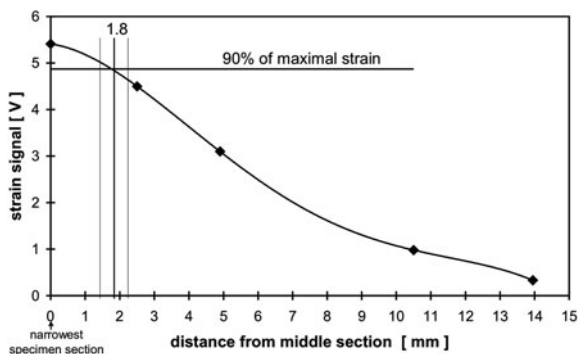
Figure 3.8b reveals the variations that occurred if the miniature strain gauges at the specimen and/or at the coupling piece (reference) were changed. These differences are due to the fact that the gauges and also the point of attachment are never completely the same. Obviously, the resulting variations are of same magnitude or slightly larger than that the differences shown above between the three test series or the differences derived from the cooling media. The observed variations for the measured strains, i.e. in the worst case (test series K110L-II—low RS; Fig. 3.8b) are about 13% maximum, relatively, which implies at a stress amplitude level of about 1200 MPa (highest test amplitude applied) a maximum error of 160 MPa, which is quite high. However, it is noted here that this represents the worst case scenario. The average error derived from the calibration procedure for test series K110L-I and K110TT was about four times smaller, i.e. relatively about 3–4%. This results in an error at a stress level of about 1200 MPa of about ± 40 MPa, which is a quite acceptable value. Consequently, at the obtained fatigue endurance strength levels, errors of about 10–15 MPa have to be considered.

Concluding, it was shown here that a comprehensive evaluation of the calibration procedure is essential for obtaining reliable fatigue data using the ultrasonic frequency fatigue testing method. The errors deriving from the calibration cannot be neglected, and their careful investigation is indispensable.

3.2.2 Determination of the Test Volume

The effective test volume, in which the nominal stress acted, was experimentally determined by measuring the strain at different points along the axis of a K110 sample. Five miniature strain gauges were attached at different distances from the narrowest section from the specimen along its axis. The strain signal was measured

Fig. 3.9 Determination of the test volume at K110 fatigue specimen: strain as a function of the axial distance from the narrowest section of the specimen



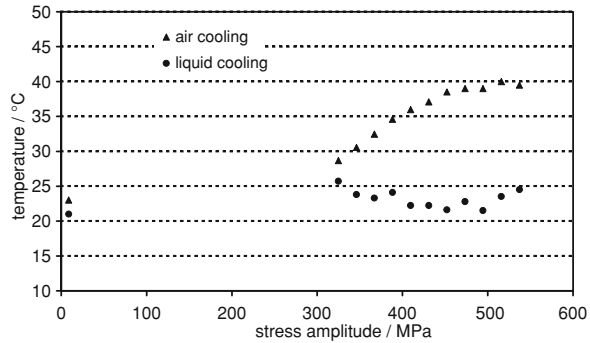
at three different stress amplitudes. Figure 3.9 shows the obtained data and the polynomial fit for one stress amplitude. The measurements at the other two stress amplitudes revealed similar results. The test volume was arbitrarily defined as the volume for which the strain is above 90% of the maximum observed strain, which threshold is marked by the horizontal line. The 90% line intersects the polynomial fit at a distance of 1.8 mm from the narrowest section of the specimen (central section). The error of this determination was assumed to be ± 0.4 mm, as indicated by the two parallel vertical lines.

The test volume, which was approximately calculated using the volume equation for a cylinder with a radius equal to the specimen radius (4 mm) and a height corresponding to the gauge length (twice the determined length = 3.6 mm). It turned out to be $45 \pm 10 \text{ mm}^3$ for the K110 specimen geometry. For the second specimen geometry used for the PM tool steels, the test volume can be assumed to be similar, since the geometrical differences close to the narrowest section of the specimen are negligible.

3.2.3 Specimen Temperature During Cyclic Loading

As already mentioned, heat generation within the specimen is one major problem during ultrasonic frequency fatigue testing. Thus, specimen cooling is essential. In this study, liquid cooling was applied, as described in the experimental section, since the stresses for testing of tool steels are extremely high, as is heat generation through damping effects. The efficiency of the applied liquid cooling compared to air cooling was evaluated by measurements of specimen temperature versus the applied stress amplitudes for test series K110L-II in such a way that the stress amplitudes were increased in small steps and corresponding temperature values were recorded for each stress amplitude, as soon as constant temperature was reached. The temperature was measured by a NiCr–Ni thermocouple (measurement error ± 1 °C)—which however is not the optimum type of thermocouple at the low temperatures observed. It was inserted through a bore axially drilled into

Fig. 3.10 Temperature in the specimen axis during cyclic loading with air and liquid cooling, respectively



the fatigue specimen from its free end to the center, i.e. the test volume, of the specimen.

Figure 3.10 shows that the specimen temperature increases slightly with increasing stress amplitude but seems to reach a plateau at about 40 °C in case of compressed air cooling. In contrast, liquid cooling showed rather constant specimen temperature in the investigated stress amplitude range slightly below 25 °C. The measured specimen temperature is not high during air cooling, however, the liquid cooling definitely provides stable conditions for a wide range of stress amplitudes due to better heat transfer. Thus it was decided to use a liquid medium, i.e. water with corrosion inhibitor, as described in the experimental chapter.

3.2.4 Cavitation and Corrosion

The occurrence of fatigue crack origins at or near to the surface at very high loading cycle numbers raised the question if there are any other reasons than microstructural defects that cause fatigue cracks to start there. Cavitation imposes a well known risk to parts under vibration loading and liquid contact. It is noted that in the present case the cooling water is splashed from six points of the supplying onto the specimen (Fig. 2.21). Thus, the sample is not immersed in the cooling medium at any time. In contrast, cooling water is delivered continuously onto the specimen surface, then dropping down into the collecting vessel, which makes formation of stable water droplets rather improbable. However, detailed investigations were performed evaluating probable cavitation effects.

Figure 3.11a shows an overview of the surface of a runout specimen (10^{10} cycles at 580 MPa), for which the cooling system has not yet been optimized. Obviously, close to the shoulders of the dumbbell heavy cavitation has occurred, causing a roughened, grayish surface structure (Fig. 3.11b). However, the affected zone is rather narrow. Figure 3.11c shows the transition region from cavitated to non-cavitated surface. In the high magnification SEM image of the specimen surface at the gauge length (Fig. 3.11d) no marks of damage, such as pits, holes or

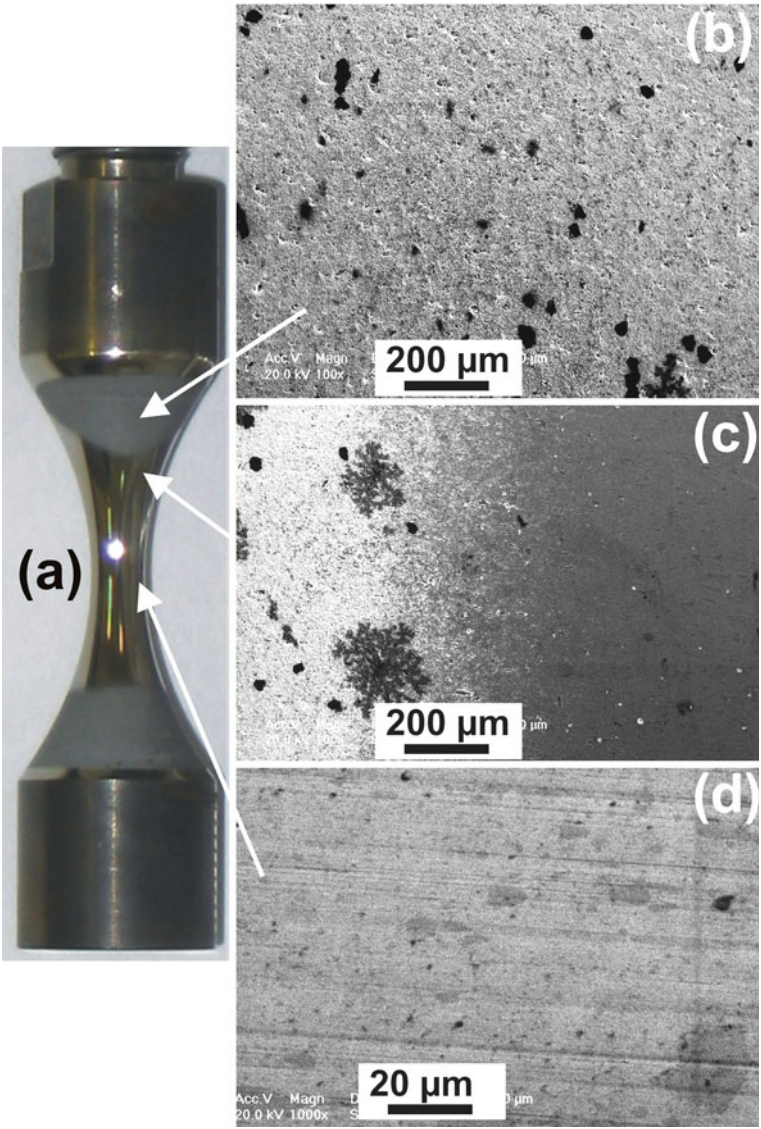


Fig. 3.11 Surface of runout specimen at the gage length after loading at 580 MPa for 10^{10} cycles without failure: **a** overview image; **b** highly cavitated surface; **c** transition area from cavitated to non-cavitated surface **d** surface at gauge length (high magnification)

other surface anomalies are visible, which also suggests that stress corrosion being responsible for crack initiation can be excluded.

Concluding, these investigations definitely showed that cavitation and corrosion did not take place, at least not in the critical, nominally loaded surface area. It has to be mentioned also that the cooling system was subsequently optimized so that

cavitation as shown in Fig. 3.11 did not occur any more. This optimization aimed on the way of applying the coolant onto the specimen through the cooling ring.

Stress corrosion can definitely be a point of concern when using water cooling, as shown by Tokaji et al. [2]. However, there are several factors here that make corrosion improbable. A noncorrosive coolant—distilled water mixture containing a corrosion inhibitor—was used. Secondly, in contrast to the study by Tokaji et al. [2], an ultrasonic fatigue testing system was employed, which consequently means that the testing times were several orders of magnitude shorter than in the cited study [2]. For example, testing up to 10^8 loading cycles takes only 1.3 h. This time period is rather short for corrosive attack. Furthermore, the slopes of the two S–N curves obtained here are a further indication that corrosion did not occur, since in case of corrosion a more pronounced decrease of the fatigue strength with higher N can be expected [2]. The similarity to S–N curves of high strength steels tested in tension–compression mode [3] with respect to position and slope also renders corrosion improbable. SEM investigations of the crack origin, which will be presented later, have definitely shown that near-surface cracks started at large primary carbides or clusters, or nonmetallic inclusions and are frequently “near-surface” and not “surface”.

Thus, it could be shown here that surface-related damage mechanisms such as corrosion and cavitation are not responsible for the crack initiation observed at or close to the specimen surface.

3.2.5 Surface Zone Deformation During Fatigue Testing

As will be shown later, it was not possible to definitely identify the crack-initiating micro-constituents for all fractured specimens that failed due to fatigue cracks originated at or near the surface. In many cases of steel K110 fractures it seemed that the crack initiation zone was destroyed, probably during final fracture or during the fatigue process, which has been reported earlier by Marsoner et al. [4, 5]. Investigation of the fracture surface showed that only the region around the crack origin was affected (Fig. 3.12a). Other circumferential sites did not reveal any surface anomalies (Fig. 3.12b), which also supports the exclusion of stress corrosion of being responsible for near-surface crack nucleation. It is speculated that this damage of the initiation site occurs due to repeated shearing contact of the two free surfaces during stable crack growth before final failure. More specimens with longer lives tended to exhibit this anomaly; for those specimens that failed at shorter lives the crack origin could be identified with higher probability. Specimens that failed due to internal crack origins never exhibited such damage effects at the fracture surface, not even those who failed at comparable long lives (Fig. 3.12c).

Fracture surfaces of the other investigated steels did show such surface damages in the vicinity of the at/near-surface crack origins.

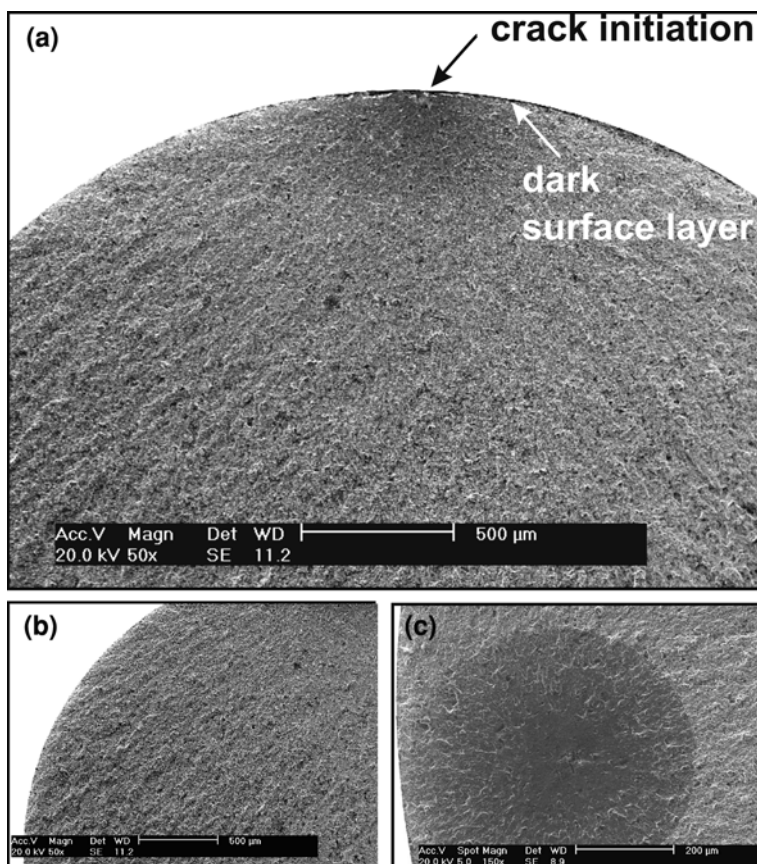


Fig. 3.12 Specimen surface after fatigue failure: **a** surface anomaly near the crack initiation site (K110L-64: failed at 650 MPa after 9.6×10^6 cycles), **b** no deformation at another site at the circumference of the same sample, **c** no surface anomaly of specimen that failed due to internal crack nucleation (K110L-40: failed at 800 MPa after 1.6×10^7)

3.3 Fatigue Behavior of Conventional Cold Work Tool Steel (Böhler K110)

3.3.1 Materials Characterization

3.3.1.1 Metallography, X-ray Diffraction and Electron Probe Microanalysis

Fatigue specimens were machined from two bars with different diameters (Bar “A”, diameter 15.5 mm and “B”, diameter 106.5 mm) as described in the

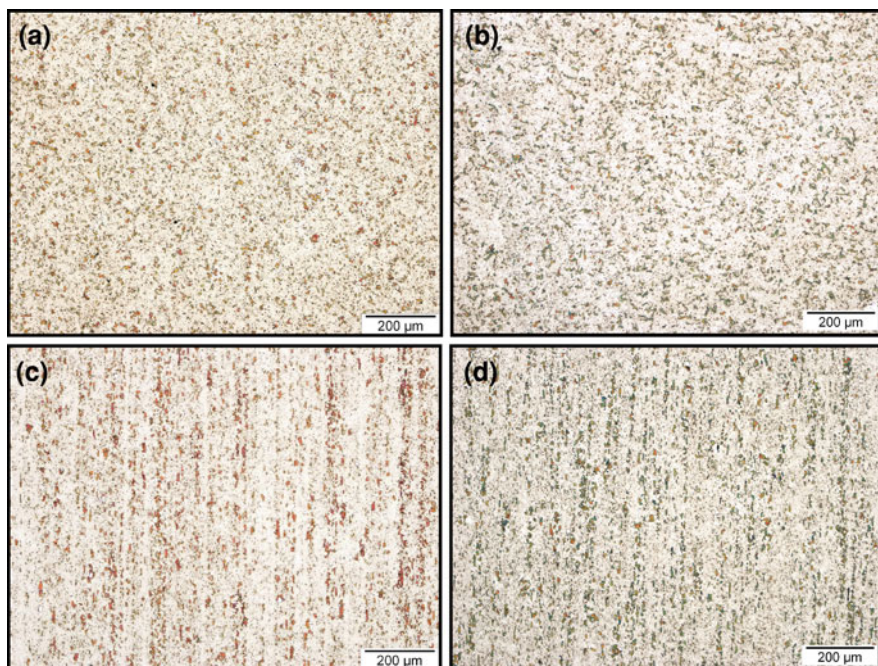


Fig. 3.13 Optical micrographs (100 $\times$, etched with Murakami reagent) of annealed (as-received) microstructure of steel K110: **a, b** samples bar A; **c, d** samples bar B, transverse and longitudinal sections, respectively

experimental part. The microstructure of the steel in the annealed condition is shown in Fig. 3.13 and 3.14 for both starting bars.

Uniform carbide distribution in the transverse section and typical parallel alignment of the primary chromium carbides of type M_7C_3 in the longitudinal direction was observed. The degree of deformation, which is lower for the bar with larger initial diameter, revealed a direct influence on the size of primary carbides, which were larger in bar B. Quantitative analysis of carbide sizes in quenched and tempered steel, presented later, definitely confirmed this impression. Beside large primary carbides, numerous finely dispersed cementite phases can be observed (Fig. 3.15) according Fig. 2.2. Closely-spaced large carbides can be found also, which will be referred to as “carbide clusters”. In general, larger carbide dimension were observed in the longitudinal direction, which is a direct result of the rolling process.

The steel was austenitized at 1,040 °C and oil quenched. Prior austenite grain boundaries can be detected in Fig. 3.16, confirming that undesired grain growth did not occur. Determination of the grain size was performed using the Snyder-Graff method. It turned out to be in the range of 10–15 μm regardless of the initial bar dimension. Comparison of the finely dispersed carbides before and

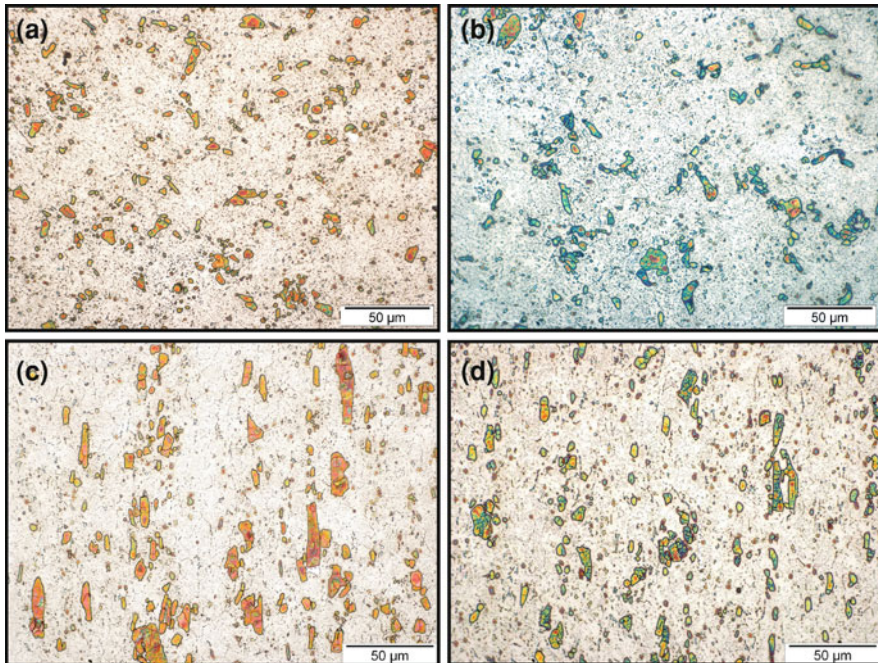


Fig. 3.14 Optical micrographs (500 $\times$, etched with Murakami reagent) of annealed (as-received) microstructure of steel K110: **a, c** samples bar A; **b, d** samples bar B, transverse and longitudinal sections, respectively

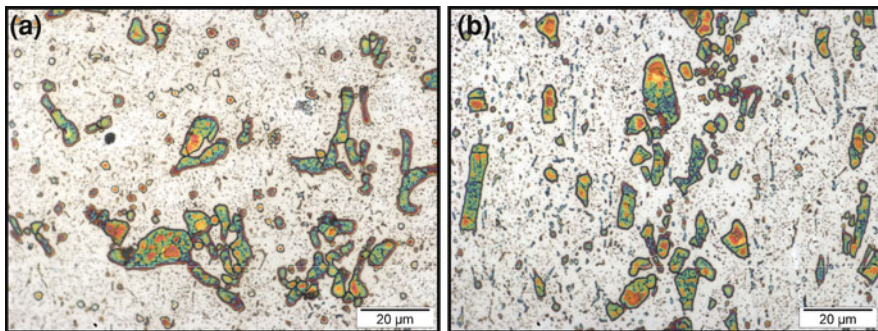
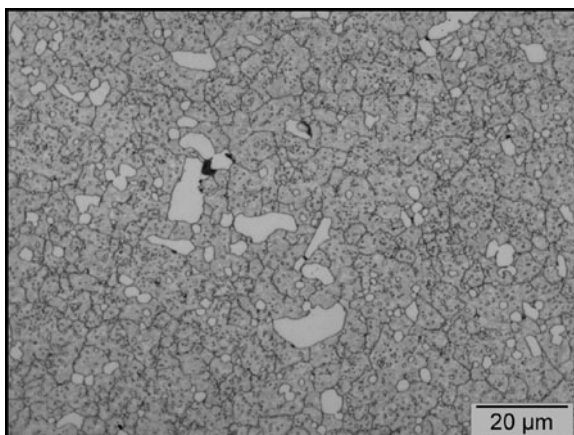


Fig. 3.15 Optical micrograph (1000 $\times$, etched with Murakami reagent) of annealed (as-received) microstructure of steel K110—bar B: **a** transverse section, **b** longitudinal section

after tempering (Fig. 3.17a, b) indicates that the amount of small carbides is higher after tempering.

Figure 3.18 shows the carbide distributions in steel K110 after the applied tempering for both samples of both bars. The carbides seem to be somewhat larger in the samples of bar B due to the lower degree of deformation. Furthermore, in the

Fig. 3.16 As-quenched microstructure of steel K110 revealing prior austenite grains and primary carbides appearing white (optical micrograph of transverse section of bar A at 1000 $\times$, etched with Pikral)



transverse section of bar B (Fig. 3.18c) more large carbide aggregates (carbide clusters) were observed compared to Fig. 3.18a). Figure 3.19 compares the microstructure of K110 in the as-received (a) and in heat treated = quenched and tempered (b, c) condition, both revealing very uniform structures. Fine-structured tempered martensite microstructure can definitely be detected in the as-heat treated steel (Fig. 3.19c). The etching behavior of the iron matrix (chemical composition—Fig. 3.20a) was rather uniform, indicating that apparently the amount of retained austenite is low—XRD proved it to be a few percent at maximum (Fig. 3.21). Dilatometric studies, which will be presented later, also showed that at the heat treatment conditions applied here the amount of retained austenite was very low, thus, its influence on fatigue behavior can be assumed to be negligible. The amount of retained austenite can be critical at this medium carbon content, since it is responsible for lower hardness. On the other hand, Ritchie et al. [6] claimed crack growth rates for HP9-4-20 steel in moist laboratory air to be reduced in the near-threshold region by the presence of 14% retained austenite, increasing the threshold stress intensity factor ΔK_0 by 20%.

Under the applied heat treatment conditions the existing carbides in the as-heat treated steel are known to be alloy carbides of type $(\text{Fe,Cr})_7\text{C}_3$, as discussed in Chaps. 1 and 2. This carbide type can take large amounts of iron (up to 70% [7]), which was confirmed by electron probe microanalysis (Fig. 3.20b) revealing a ratio of iron to chromium in the carbides of 5/4. Stene-Osen [8] performed TEM measurements on a similar steel and also identified these carbides as $(\text{Fe,Cr})_7\text{C}_3$, which show a hexagonal lattice structure [2]. Furthermore, XRD definitely confirmed those carbides to be of type M_7C_3 in the studied steel after the applied heat treatment (Fig. 3.21). The large primary carbide particles and clusters can act as stress-raising defects responsible for fatigue failure [4, 5, 9–12]. Thus, a rough classification of these potential defects after their size was performed for both initial bar diameters and for transverse and longitudinal direction, respectively. 11–13 light optical micrographs at magnification 200 $\times$ and at two specimens were investigated for every condition, respectively. A representative optical micrograph

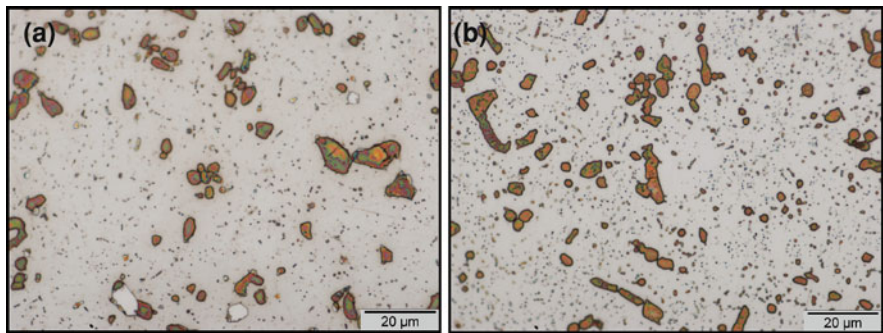


Fig. 3.17 Comparison of transversal as-quenched (a) and tempered microstructure (b) (optical micrographs at 1000 $\times$, etched with Murakami reagent) of steel bar A

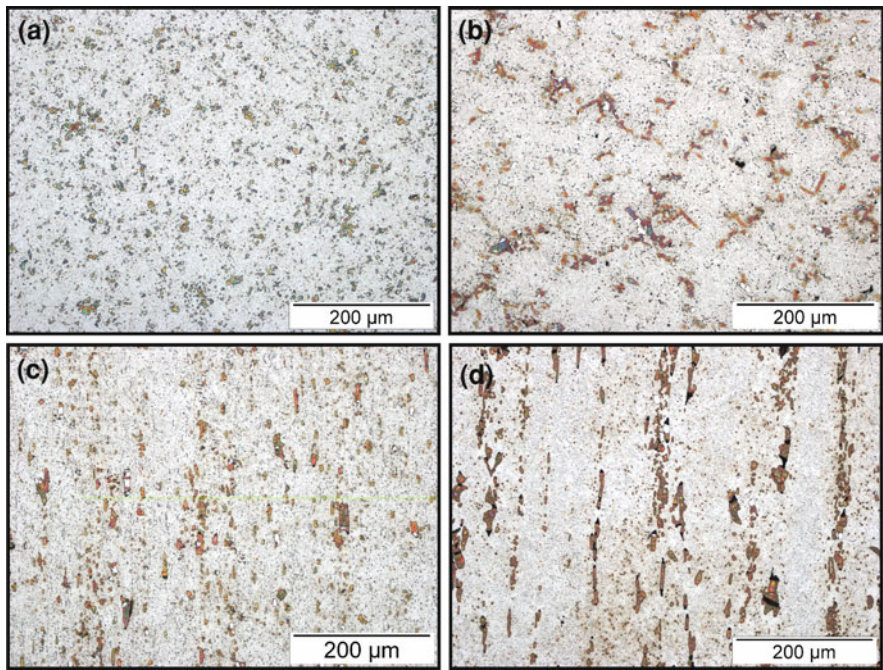


Fig. 3.18 Optical micrographs (1000 $\times$, etched with Murakami reagent) showing the carbide distribution in steel K110 after tempering: **a** and **b** sections of bar A, **c** and **d** sections of bar B, transversal and longitudinal, respectively

for which such a classification was performed using circles with defined diameters is presented in Fig. 3.22. The obtained numerical results are summarized in Table 3.3.

The overall largest carbide clusters of a diameter of about 100 µm were observed in bar B (lower degree of deformation). As a rule, the longitudinal

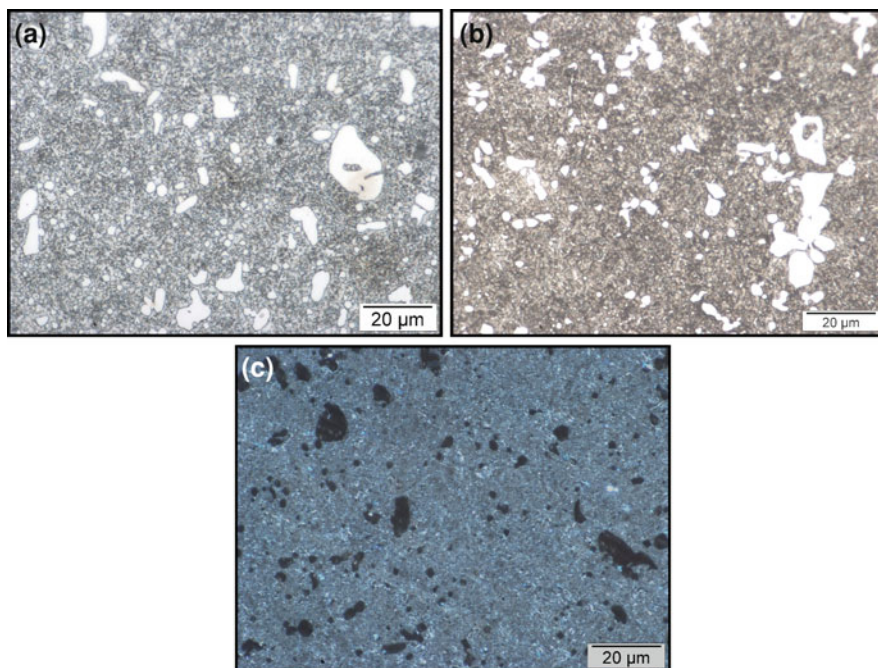


Fig. 3.19 Optical micrographs (1000 $\times$) of microstructure of steel K110 bar A: **a** annealed condition (etched with Adler reagent), **b** tempered condition (etched with Adler reagent), **c** tempered condition (etched with Picral—polarized light), dark phases correspond to coarse M_7C_3 carbides

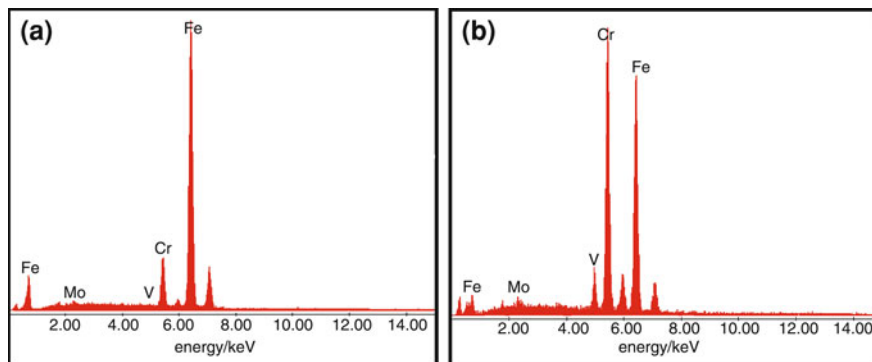


Fig. 3.20 Chemical composition (EDX spectra) of **a** matrix and **b** chromium carbides (M_7C_3) in steel K110 after d heat treatment

sections revealed significantly larger carbide dimensions than the transversal sections (Fig. 3.23). In the transversal section of bar A the largest carbide clusters had diameters of not more than 60 μm . Thus, if carbide (clusters) were responsible

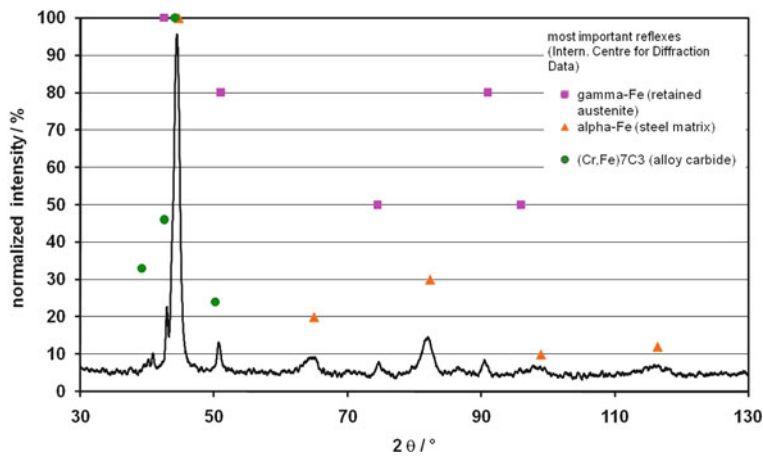


Fig. 3.21 XRD pattern of quenched and tempered K110 tool steel

Fig. 3.22 Carbide classification in transversal section of steel K110 (bar B, optical micrograph at 200×)

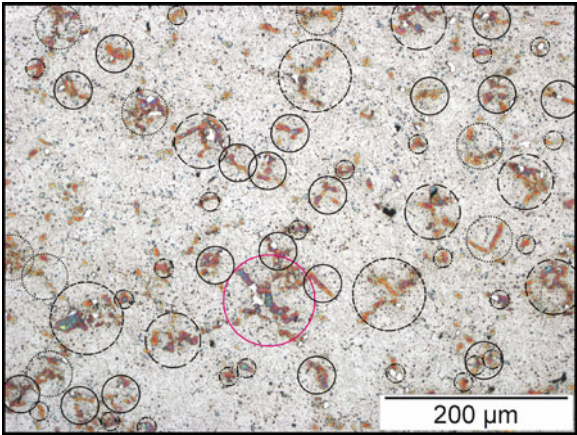
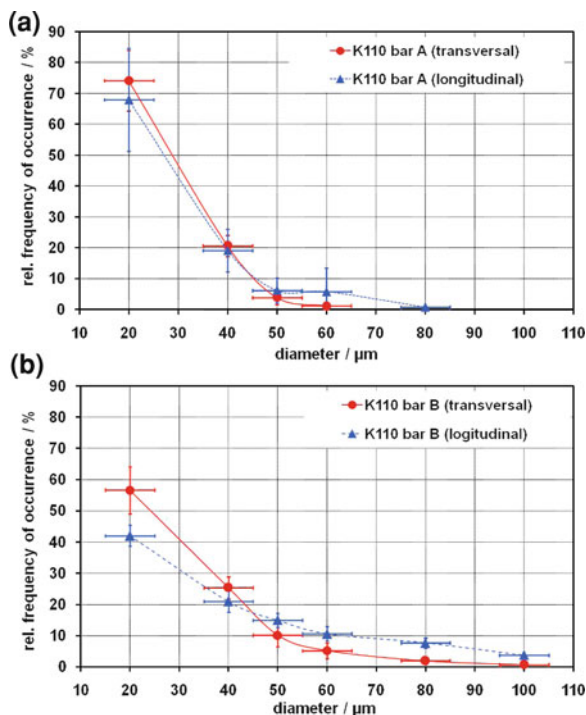


Table 3.3 Classification of carbides according to their sizes as found in metallographic sections of steel K110

Carbide (cluster) diameters (in μm)	Relative frequency (in %)			
	Bar A		Bar B	
	Transversal	Longitudinal	Transversal	Longitudinal
20	74	68	57	42
40	21	19	25	21
50	4	6	10	15
60	1	6	5	11
80	0	1	2	8
100	0	0	1	4

Fig. 3.23 Comparison of carbide sizes in longitudinal (filled triangle) and transversal (filled circle) sections for **a** bar A and **b** bar B



for fatigue failure, fatigue specimens K110L should show a higher fatigue strength than K110TT samples. Obviously, the degree of deformation, which is higher in bar A, affects the apparent dimensions of the primary carbides (Fig. 3.24). Higher deformation (bar A) means smaller carbides. The quantitative carbide size analysis confirmed the visual impression obtained from the optical micrographs.

The volume fraction of primary carbides in the steel was determined using image analyzing software, 25 optical micrographs at $500\times$ and $1000\times$ magnification each being evaluated, as for one case shown in Fig. 3.25. The carbide volume fraction turned out to be 12 ± 2 vol%, which was in good agreement with results found by Fukaura et al. [11]. Non-metallic inclusions have not been detected during metallographic investigations.

3.3.1.2 Mechanical Properties of Steel K110

The steel hardness, Young's Modulus, microhardness, and transverse rupture strength were measured, which are presented in Table 3.4. The hardness of the steel after heat treatment was similar for all K110 test series. In the as-received, annealed condition the steel had a hardness of about 22 HRC. The high hardness obtained after austenitizing ($1,040^\circ\text{C}$) and subsequent quenching in oil was decreased through tempering at 530°C to common working hardness of this steel

Fig. 3.24 Comparison of carbide sizes in **a** longitudinal section and **b** transversal section of bar A and B, respectively

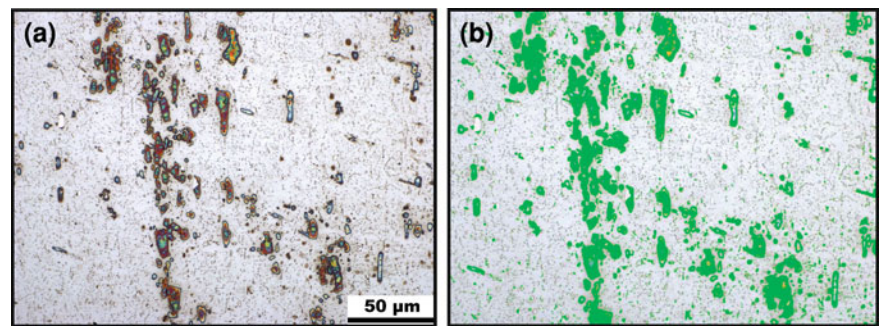
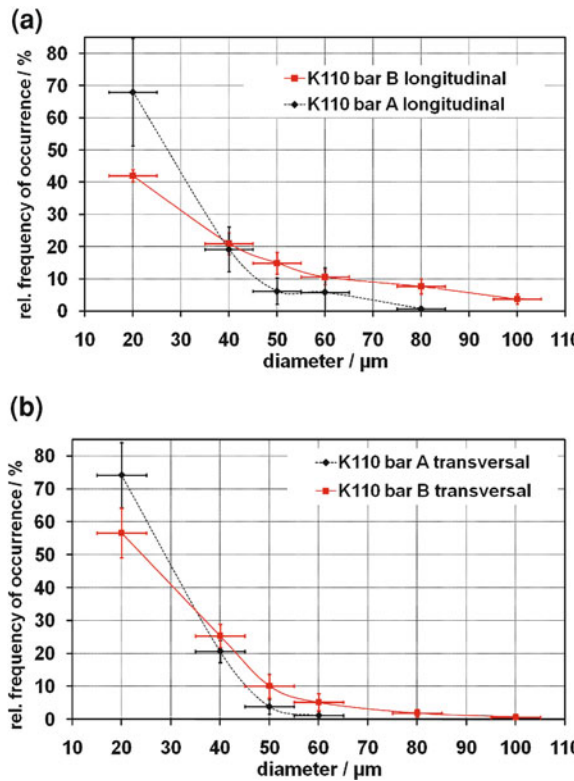


Fig. 3.25 Carbide volume fraction determined by optical micrograph (500×) analysis: **a** original micrograph, and **b** micrograph with carbides colored green as used for image analysis

of about 58 HRC. The Young's modulus was similar regardless of the test orientation to primary carbide bands.

The Young's Modulus of the chromium alloy carbide [13] is at least about 100 GPa higher than that of the steel, which can be decisive for the initiation of fatigue cracks at the carbide–matrix interface. The microhardness of the matrix and of the large carbide

Table 3.4 Mechanical properties of K110 steel

Steel “K110” AISI D2 DIN 1.2379	Rockwell hardness HRC 150 kg		Microhardness (HV 25 g)		T.R.S. (MPa)	Dynamic Young’s modulus (GPa)	
	As-quenched	Quenched and tempered	Matrix	Alloy carbides	Quenched and tempered	Quenched and tempered	Cr ₇ C ₃ [13]
K110L (parallel to RD)	64 ± 1	58 ± 2	755 ± 30	1540 ± 215	3600 ± 300	210 ± 7	>320
K110LL (sample axis parallel to RD)	64 ± 1	59 ± 1			3900 ± 300	206 ± 6	
K110TT (sample axis perpendicular to RD)	62 ± 2	58 ± 1			1900 ± 100 2000 ± 100	205 ± 6 (⊥) 211 ± 7 ()	

⊥ and ||: loading perpendicular and parallel to carbide alignment, respectively—see Fig. 3.26

species was also determined. The alloy carbides revealed twice the hardness of the steel matrix, which agrees with results in the work of Pernegger [14], who found a microhardness of 1500 HV10 for chromium carbides containing 70 mass% iron, and for plain chromium carbides Cr₇C₃ this author [14] reported a microhardness of 2200 HV10. Pernegger [14] claimed that the microhardness for this alloy carbide type decreases with the increasing incorporation of iron.

The bending strength (TRS) specimens with axis parallel to rolling direction (K110L and K110LL) were machined from both initial bars. From the larger bar, specimens with axis perpendicular to the rolling direction (K110TT) were manufactured also (Fig. 3.26). The K110L and K110LL samples showed similar strength, as presented in Table 3.4, while the K110TT revealed significantly lower transverse rupture strength, i.e. about one half of the strength of K110L,LL specimens. This can be attributed to the different orientation of primary carbide bands to the loading direction, very likely due to a reinforcement effect of these carbide bands within the iron matrix, similarly to fiber-reinforced materials.

The different transverse rupture strengths in the two orientations reveal the anisotropy of the material, which is also expressed in the obtained fracture surfaces (Fig. 3.27). Fracture surfaces of K110L and LL specimen (Fig. 3.27a, b) showed similar surface morphologies, with a large part of final fracture area exhibiting rather smooth brittle-like surface. In contrast, a major part of the fracture surface of K110TT specimens (Fig. 3.27c, d) revealed numerous cracks in loading direction and also in direction of primary carbide alignment (K110TT (||), (⊥)). All fractographs revealed lines and ridges that point back to the crack initiation sites (Fig. 3.28a, c, e). Figure 3.28b, d, f shows rather transgranular failure, with faceted surface texture. Cleavage of primary carbide particles was observed at the entire fracture surface. Cracks propagate parallel to the loading direction however, for K110TT specimen (⊥) those cracks frequently change their path parallel to primary carbide lines (Fig. 3.28f).

Fig. 3.26 Loading direction of TRS tests of K110TT samples: **a** orthogonal ($\perp$) and **b** parallel ($\parallel$) to carbide alignment in testing rods

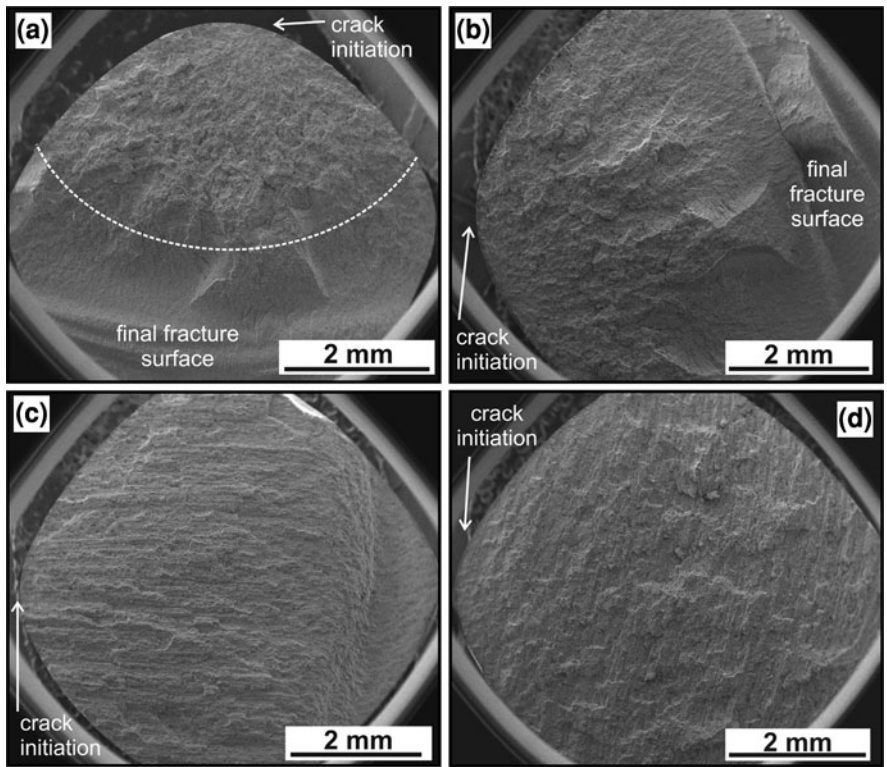
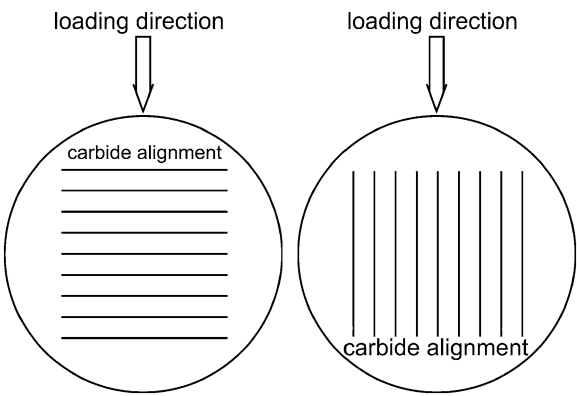


Fig. 3.27 Overview of fracture surfaces obtained in TRS tests of steel K110: **a** K110L, **b** K110LL, **c** K110TT ($\parallel$), **d** K110TT ($\perp$)

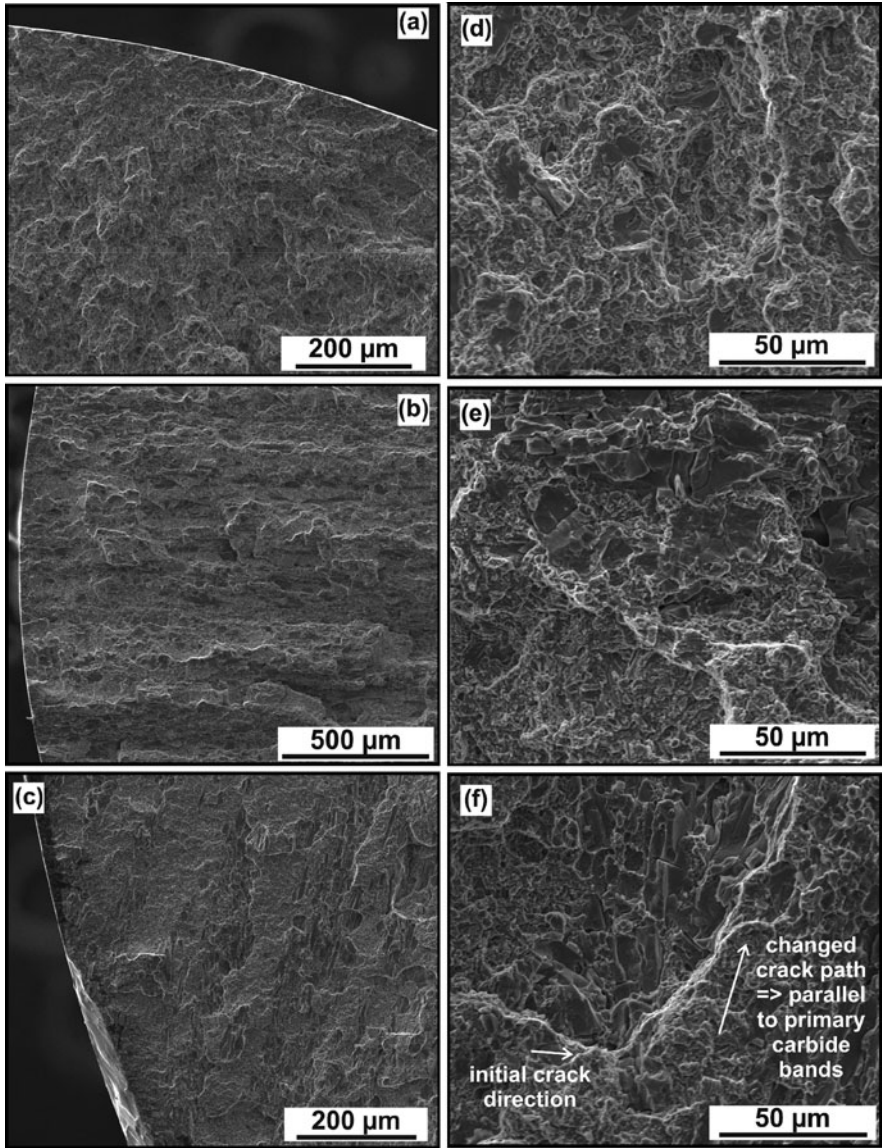


Fig. 3.28 Fracture surface details of fractures—K110L (a, d), K110 TT (||) (b, e), K110TT (⊥) (c, f), respectively—obtained in TRS tests: a–c zone of crack origin; d–f area of stable crack propagation

Fig. 3.29 Temperature–time profile for the two dilatometer test series (solid line: $T_{\max} \approx 750\text{ }^{\circ}\text{C}$; dashed line: $T_{\max} \approx 600\text{ }^{\circ}\text{C}$)

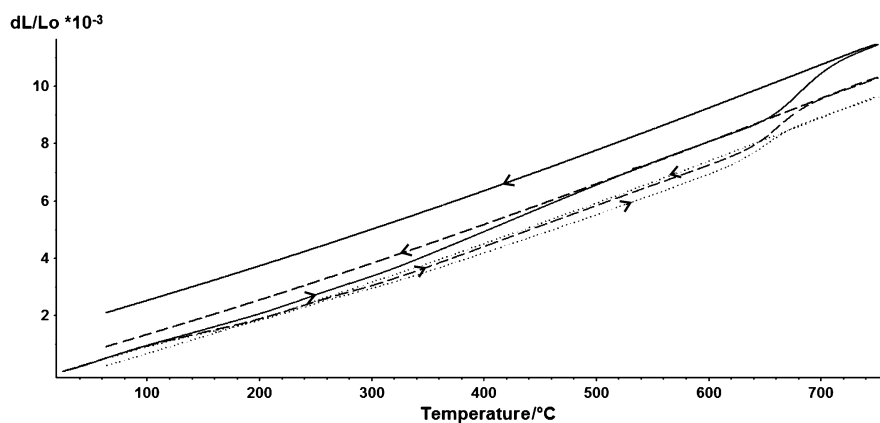
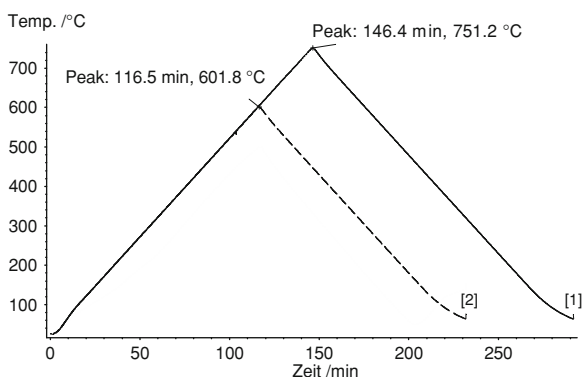


Fig. 3.30 Relative dimensional change during dilatometer test series up to maximum temperature of $750\text{ }^{\circ}\text{C}$ of three samples quenched from $1,110\text{ }^{\circ}\text{C}$ (solid line), $1,070\text{ }^{\circ}\text{C}$ (dashed line), and $1,040\text{ }^{\circ}\text{C}$ (dotted line)

3.3.2 Dilatometric Investigations of Cold Work Tool Steel K110

In order to investigate the extent of retained austenite after austenitizing at three different temperatures— 1040 , 1070 , and $1110\text{ }^{\circ}\text{C}$, and its subsequent transformation during tempering and cryogenic treatment, respectively, dilatometric experiments were performed on as-quenched samples, recording the dimensional changes during reheating of the specimens up to 600 or $750\text{ }^{\circ}\text{C}$, i.e. during tempering (Fig. 3.29). These two temperatures were chosen due to the fact that between 600 and $750\text{ }^{\circ}\text{C}$ a massive phase transformation was observed during the heating section (Fig. 3.30), characterized by a very significant length change. The system was calibrated against a Pt-standard sample, and the thermal expansion data measured for the K110 test specimens were corrected accordingly.

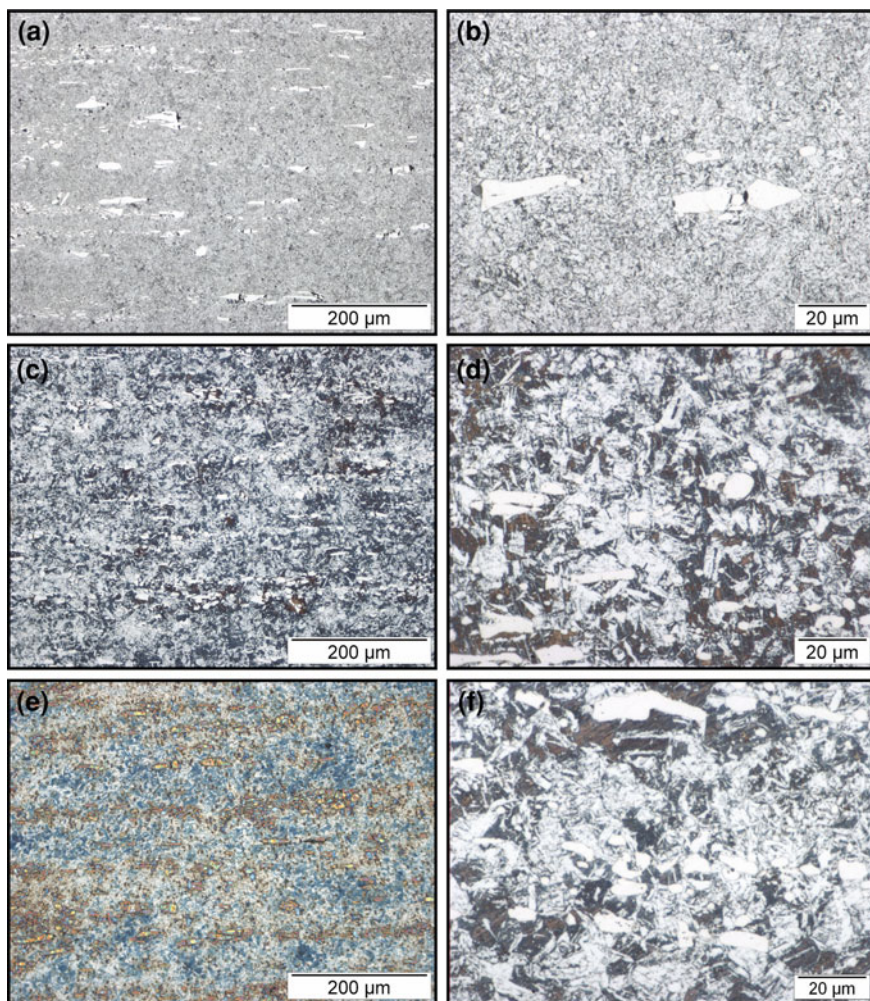


Fig. 3.31 Light optical micrographs of samples quenched from 1,040 °C (**a, b**), 1,070 °C (**c, d**), and 1,110 °C (**e, f**) at 200 $\times$ and 1000 $\times$ magnification, respectively, etched with diluted Adler reagent (logitudinal orientation)

Time periods between quenching and dilatometer experiments were about 20–30 min, i.e. this was the time after which the samples had attained approximately room temperature, except for those samples for which cryogenic treatment was applied.

Before discussing the results of the dilatometer experiments in more detail, metallographic investigations on as-hardened samples quenched from 1040, 1070 and 1,110 °C are presented. It turned out that the diluted Adler reagent was capable of attacking the retained austenite in the as-quenched steel, thus the retained austenite appears as dark phase in the micrographs (Fig. 3.31). Microhardness

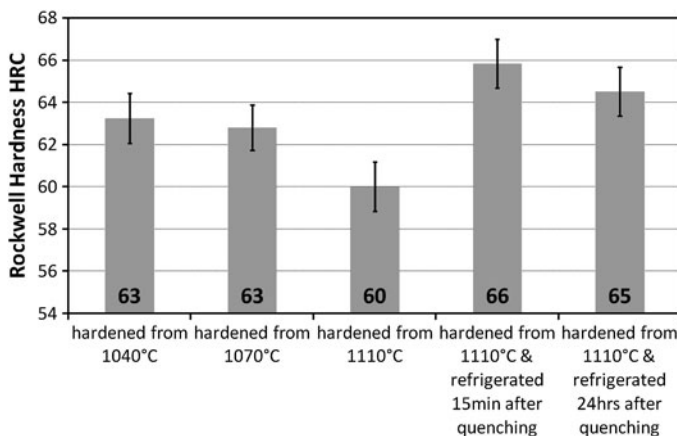


Fig. 3.32 Measured hardness (Rockwell C) of samples quenched from different austenitizing temperatures

measurements (HV0.01) confirmed that the dark phases definitely were retained austenite, since the hardness was 410 ± 60 , while the martensite—which was not attacked by Adler etch, thus remaining white—showed a higher microhardness of 570 ± 80 . It has to be considered that the only moderate difference of the microhardness values can be attributed to the fact that during the indentation, carbides located in the surroundings of the indented point affect the measurement considerably, i.e. increase the apparent hardness of the austenite areas. In addition, in Fig. 3.31e the primary carbides were colored by etching with Murakami reagent. Austenitizing at appropriate temperature such as 1,040 °C results in a very low amount of retained austenite (Fig. 3.31a, b), which is also confirmed by the relatively high hardness of 63 HRC (Fig. 3.32). Samples austenitized at higher temperatures (1,070 and 1,110 °C) showed significantly higher amounts of retained austenite (dark areas in Fig. 3.31). Despite that fact, the sample quenched from 1,070 °C revealed similar hardness as the appropriately austenitized steel (at 1,040 °C), so that it can be concluded that the existing amount of retained austenite in the sample quenched from 1,070 °C is too low to affect the steel hardness. In contrast, the sample hardened from 1,110 °C showed a lower hardness of 60 HRC.

In addition, the magnetic saturation of the as-quenched samples was also measured (Fig. 3.33), since this gives direct information on the amount of retained austenite present, i.e. the higher the amount of retained austenite, the lower the saturation. The results supported the observations described above: Lowest specific saturation was measured for the sample quenched from relatively high austenitizing temperature of 1,110 °C. The sample hardened from the correct (recommended) austenitizing temperature of 1,040 °C showed a magnetic saturation only slightly lower than the sample hardened from 1,040 °C and tempered at 530 °C for 2 h, for which XRD measurements showed that the amount of retained austenite is very low. Thus, it can be concluded that in the steel quenched

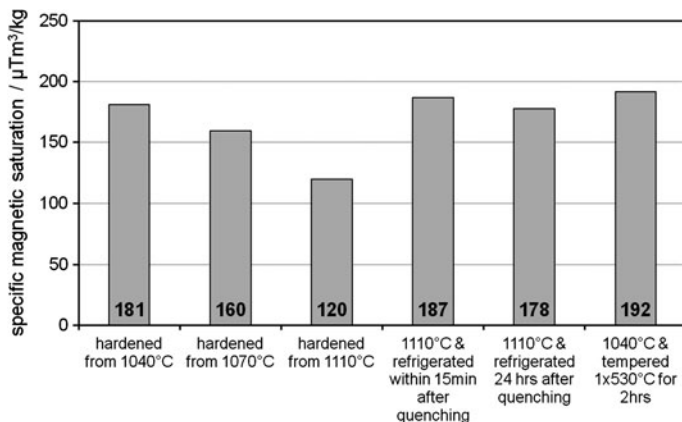


Fig. 3.33 Specific magnetic saturation $4\pi\sigma$ of steel K110 for different austenitizing temperatures

from 1,040 °C (non-tempered) the amount of retained austenite is also very low. The magnetic saturation measured for the steel quenched from 1,070 °C was in-between the values obtained for 1,040 and 1,110 °C, but closer to that for 1,040 °C. Metallographic investigations at samples quenched from 1,070 °C (Fig. 3.31c, d) showed somewhat less dark areas than the samples quenched from 1,110 °C but significantly more than samples quenched from 1,040 °C. However its hardness was similar to the sample quenched from 1,040 °C, as described above.

XRD measurements (Fig. 3.34) confirmed the findings obtained by the metallographic investigations and the magnetic saturation and hardness measurements, i.e. that the as-hardened samples quenched from 1,110 and 1,070 °C do contain some amounts of retained austenite that should at least partially transform upon tempering. However, generally the content of retained austenite in all samples was too low for showing strong peaks in the XRD patterns.

Tempering of the as-quenched samples was performed in the dilatometer, as described above. Figure 3.30 indicates that the retained austenite is transformed in the range between 600 and 750 °C. It is known that the retained austenite in the highly alloyed D-type steels is quite stable, at least until reaching the secondary hardening temperature range [15], i.e. for the investigated D2-type steel >530 °C for the different applied austenitizing conditions. Comparison of the change of the virtual “coefficient of thermal expansion” (“CTE”) with temperature, as presented in Fig. 3.35, shows that the only significant effect on the CTE above 530 °C was detected between 600 and 700 °C. Thus, it can be assumed that the transformation occurring there correspond to the decomposition of retained austenite into “bainitic mixtures of carbides and ferrite” [15]. Up to 600 °C the behavior of the three different samples was rather similar. However, between 600 and 750 °C a massive change of the CTE was observed for all three cases. The most significant change was obtained for the sample quenched from 1,110 °C, which showed a maximum of the virtual CTE of about $28 \times 10^{-6} \text{ K}^{-1}$. The specimen quenched

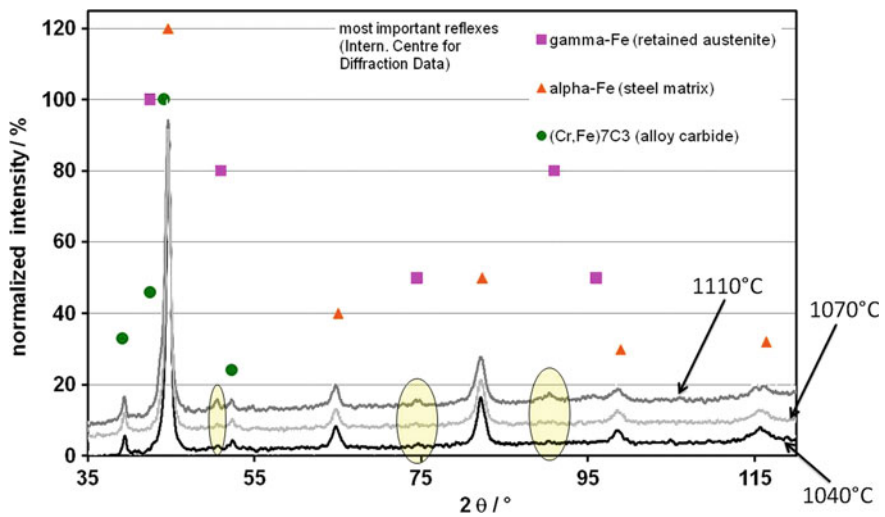


Fig. 3.34 XRD patterns of samples quenched from 1,040, 1,070, and 1,110 °C. Ellipses mark the significant peaks of the austenite phase

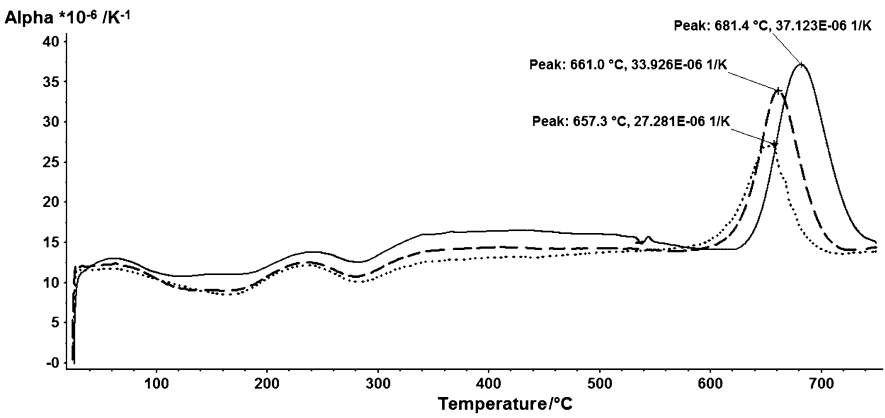


Fig. 3.35 The virtual “thermal expansion coefficient” versus temperature up to $T_{\max} = 750\text{ °C}$ for three as-hardened samples quenched from 1,110 °C (solid line), 1,070 °C (dashed line), and 1,040 °C (dotted line)

from the correct austenitizing temperature (1,040 °C) revealed the lowest effect. The curve for the sample quenched from 1,070 °C showed a behavior in-between. This effect was significantly more pronounced for the two samples quenched from too high austenitizing temperatures, supporting the results of the other investigations presented above.

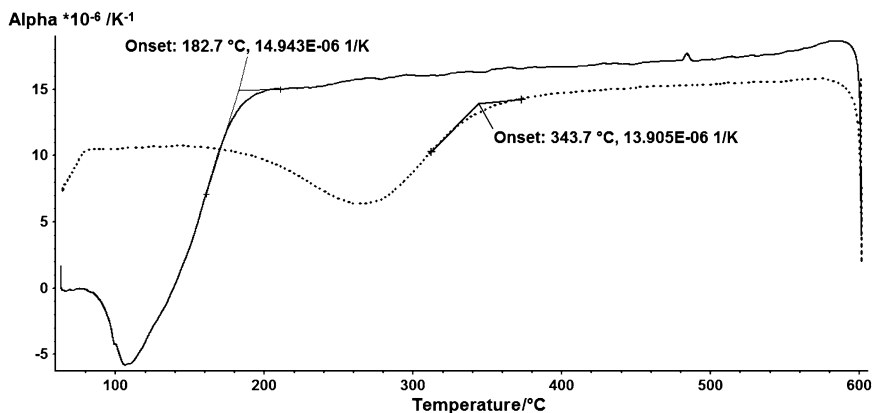


Fig. 3.36 Virtual CTE versus temperature upon cooling from 600 °C for samples quenched from 1,110 °C (*solid line*), and 1,040 °C (*dotted line*)

Furthermore, also the onset temperature of the retained austenite transformation was shifted to higher temperatures with increasing retained austenite content. However, at the usual tempering condition for the cold work tool steel studied here, during which the temperature reaches 550–600 °C maximum, retained austenite transformation does not occur during heating to and holding at the tempering temperature. Thus, it is supposed that this transformation occurs during subsequent cooling of the steel. In order to clarify whether retained austenite transformation really does occur during cooling from the tempering temperature, additional dilatometer experiments were performed, during which samples were heated up to $T_{\max}$ of 600 °C and then cooled down to room temperature in the dilatometer. This procedure was performed for samples quenched from 1,110 to 1,040 °C. The same samples then were heated up to $T_{\max} = 750$ °C again.

Figure 3.36 shows the dimensional behavior upon cooling from 600 °C for the two samples, which was very different to the cooling behavior of the specimens, which have been heated up to 750 °C. The measured “CTE” between 400 and 600° was somewhat higher for the samples quenched from 1,110 °C. The austenite-cubic martensite transformation of the small amount of retained austenite in the sample quenched from 1,040 °C started at about 343 °C, which was about 165 °C higher than for the sample austenitized at 1,110 °C. There, the phase transformation started below about 185 °C due to the large amount of retained austenite probably containing significant amounts of carbon and alloying elements, which are known to lower the M_s temperature.

The “virtual CTE” before and after the phase transformation differed only marginally in case of the sample quenched from 1,040 °C, which indicates that the amount of retained austenite present in the as-quenched steel was very low. In contrast, the other sample quenched from 1,110 °C showed a completely different image. Here, a massive change of the expansion coefficient was observed due to the considerable amount of retained austenite that has to be transformed.

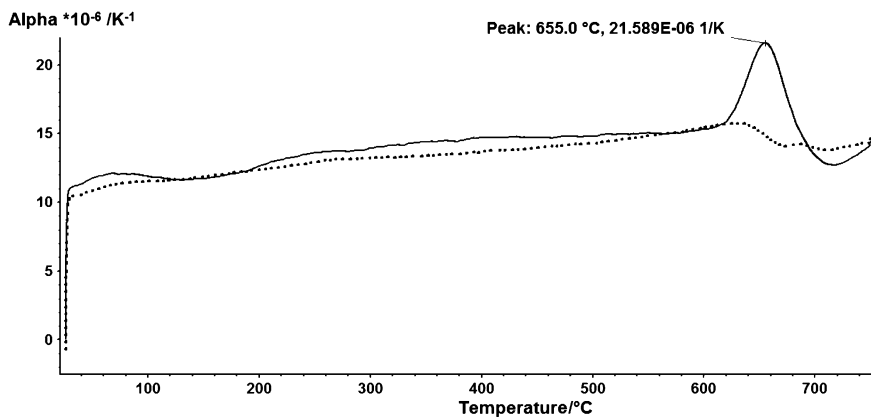


Fig. 3.37 Virtual CTE versus temperature for samples quenched from 1,110 °C (*solid line*), and 1,040 °C (*dotted line*) and both subsequently tempered to 600 °C before dilatometer experiments (heating up to 750 °C) was performed

It has to be mentioned here that the austenite-cubic martensite transformation can only occur upon cooling if the steel has been previously heated to temperatures above about 450 °C, which is commonly called the fourth stage of tempering. There, cementite is partly dissolved in the highly alloyed ferrite matrix, and simultaneously complex alloy carbides are precipitated [16]. During this period alloy carbides are also precipitated from the retained austenite; thus, the alloy and the carbon content within the retained austenite is reduced, which is usually referred as “conditioning” of the highly alloyed austenite. This phenomenon represents a prerequisite for the austenite–martensite transformation upon cooling.

In addition, a second tempering of the samples that were previously heated up to 600 °C was performed during which the samples now were heated up to 750 °C in order to evaluate if complete transformation upon cooling was attained. Figure 3.37 shows the observed dimensional behavior. Obviously, the sample initially quenched from 1,040 °C did not show any significant changes of the virtual CTE between 600 and 750 °C, which indicates that the retained austenite had been completely transformed during the cooling section of the first tempering. In contrast, the samples initially quenched from 1,110 °C revealed phase transformation in the mentioned temperature range. However, compared to Fig. 3.35 the peak maximum of the virtual CTE was low, indicating that most of retained austenite had been transformed in the cooling section of the first tempering cycle.

Thus, it can be concluded that the retained austenite is definitely almost completely transformed in the cooling section of the first tempering if the steel has been austenitized at the correct temperature, as done with the fatigue specimens of this thesis. This observation agrees with the aforementioned XRD measurements, which indicated the existence of only small amounts of retained austenite in the as-tempered steel quenched from the correct (=recommended) hardening temperature.

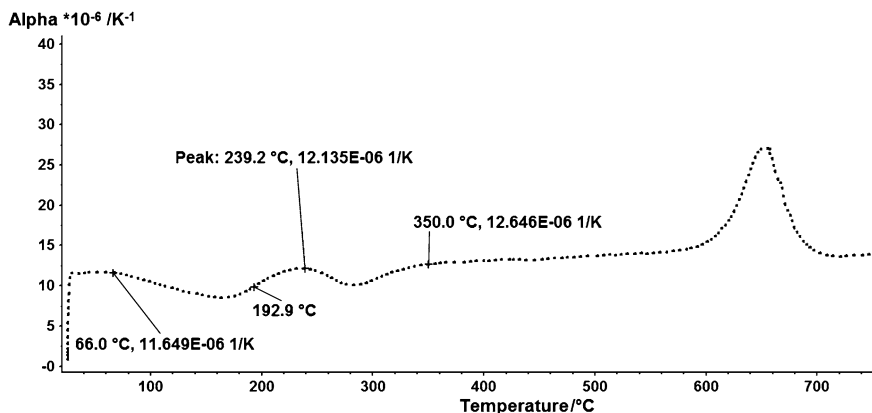


Fig. 3.38 Virtual CTE versus temperature for as-hardened sample quenched from appropriate austenitizing temperature of 1,040 °C. Temperatures relevant for the different stages of tempering are presented

In addition to the transformation of the retained austenite, the other microstructural changes that occur upon tempering are also of interest. Figure 3.38 shows the evolution of the thermal expansion coefficient during the heating section of tempering for as-hardened sample quenched from the correct austenitizing temperature of 1,040 °C. The temperatures relevant for the different transformation processes are presented. The first marked change of the virtual CTE was observed between 66 and 100 °C. Roberts et al. [15] reported that at temperatures between 100 and 200 °C the primary martensite begins to release the carbon, which is precipitated as so-called ϵ -carbides within the martensite grains accompanied by decrease in tetragonality of the martensite lattice. This process causes an overall contraction, as observed here (compare the virtual CTE at 66 and 234 °C). From about 230 to 350 °C another pronounced phase transformation was observed which corresponds to precipitation of cementite gradually replacing the ϵ -carbides. This precipitation of cementite, which can contain high amounts of alloying elements, is known to occur at the martensite laths or plates, thus, the martensitic crystals retain their lath and plate structure, even though the crystal structure of the martensite becomes cubic [15].

In addition, in the temperature range >425 °C [15] retained austenite can decompose into bainite and ϵ -carbides or cementite, which would increase the (real) CTE. However, a significant change of the CTE in that temperature region was not observed. Kulmburg and Swoboda [17] reported that carbide precipitation even below 1% can be detected from the differential change of sample length. Thus, it can be assumed that transformation of retained austenite did not occur very likely due to its high alloy content.

Considering the transformations described here, it is possible to interpret the observations presented in Fig. 3.35. The sample quenched from 1,110 °C showed less transformation in the temperature region of 100–200 °C compared to the other

two samples, which can be explained by the fact that the amount of martensite—which transforms in this temperature region—is lower due to the significantly higher content of retained austenite in the sample quenched from 1,110 °C—a retained austenite content of 85% for AISI D2 type steel quenched from 1,120 °C was reported in Ref. [18], which however is regarded far too high by the present author considering the magnetic saturation and hardness values obtained here. In contrast, the behavior from 200 to 350 °C was similar in all three cases, since here predominantly alloy cementite precipitation does occur.

Furthermore, it is also possible to describe the dimensional behavior observed for the samples that were first tempered up to 600 °C and then for a second time up to 750 °C, for which the second tempering up to 750 °C has been presented in Fig. 3.37. In the heating section of the second tempering cycle, the sample that had been quenched from the correct austenitizing temperature of 1,040 °C showed only negligible transformation in the temperature region of 66–200 °C, since after quenching only very limited retained austenite content was present, which transformed completely during cooling after the first tempering cycle up to 600 °C. Thus, untempered martensite—which would have been formed by transformation of retained austenite during cooling—was present in this sample only in a very limited amount, and therefore carbide precipitation from the martensite was not detected. In contrast, the sample quenched from 1,110 °C showed considerable transformation in the temperature region from 66 to 200 °C, corresponding to the carbon depletion of the—untempered—martensite that during cooling after the first tempering had been formed from the high amounts of retained austenite present after quenching. However, alloy cementite precipitation, which, as described above, can be detected in the temperature range of about 200–350 °C, was not detected here, since it already had occurred during the first tempering.

Another interesting question was whether the retained austenite obtained after quenching transforms during cryogenic treatment by liquid nitrogen or not. For that reason, samples quenched from 1,110 °C were refrigerated 15 min and 24 h after quenching, respectively. Figure 3.39 shows the obtained microstructure. Martensite needles can be seen quite clearly. The dark phases correspond to cubic martensite, which is attacked by Adler reagent in contrast to the tetragonal martensite formed from austenite–martensite transformation upon the cryogenic treatment, which remains unattacked (white needle-like structure). Hardness measurements revealed relatively high hardness of 66 HRC (refrigerated within 15 min after quenching) and 65 HRC (cryogenic treatment after 24 h), respectively, compared to as-quenched samples (Fig. 3.32), which indicated that the retained austenite had been fully transformed upon deep cooling. This was supported by the measured magnetic saturation (Fig. 3.33), which was relatively high, similar to the saturation obtained for a sample correctly austenitized at 1,040 °C and subsequently tempered at 530 °C for 2 h, i.e. in which retained austenite did not exist. However, the extent of transformation of retained austenite during cryogenic treatment after 24 h seems lower, since both magnetic saturation and hardness revealed slightly lower values compared to samples deep cooled

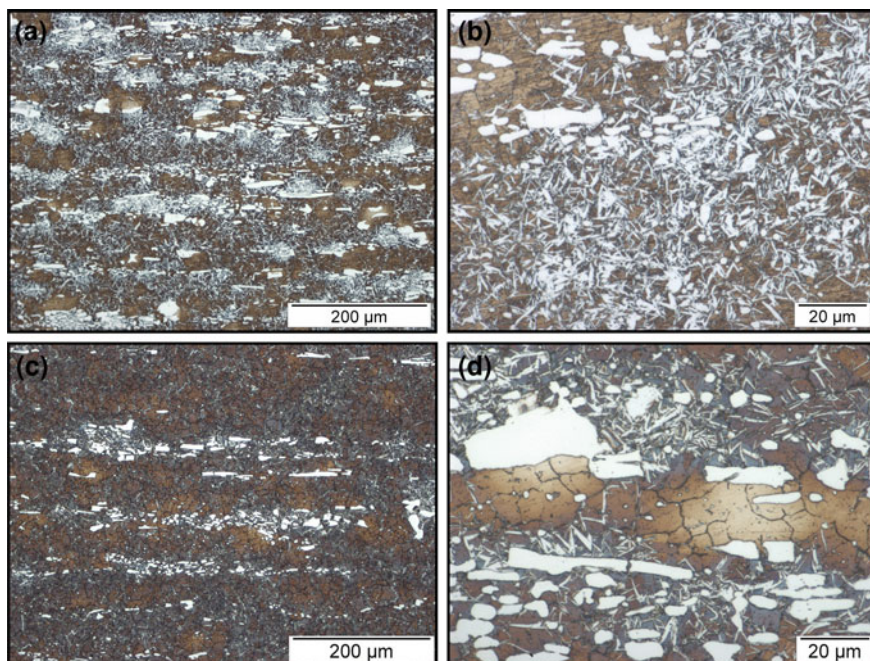


Fig. 3.39 Light optical micrographs (etched with Adler reagent) of as-hardened samples, quenched from 1,110 °C and refrigerated in liquid nitrogen: **a, b** within 15 min **c, d** after 24 h after quenching, respectively

within 15 min after quenching. This is also supported by the metallographic investigations, which showed less white, unattacked untempered martensite in the latter samples (Fig. 3.39c, d).

Dilatometer investigations on deep cooled as-quenched samples supported the metallographic observations and magnetic saturation results. A transformation between 600 and 750 °C was detected for these specimens (Fig. 3.40). However, the extent was rather small, the maximum measured “virtual CTE” was lower than that obtained for the correctly austenitized sample (1,040 °C—Fig. 3.35). Thus, retained austenite in the deep cooled sample can be assumed to be negligibly low. A slight stabilization of the retained austenite was obtained for the sample for which the cryogenic treatment was performed 24 h after quenching, which is expressed by a slightly higher austenite transformation peak (600–750 °C), and a shift to somewhat higher onset temperature.

The shrinkage caused by carbon depletion of the martensite and ϵ -carbide precipitation (66–200 °C) was very pronounced in the dilatometric graph for the deep cooled sample, simple due to the fact that the retained austenite had been transformed to martensite upon cryogenic treatment. Thus, this sample contained mainly untempered martensite, since also the alloy cementite precipitation was considerable (200–350 °C).

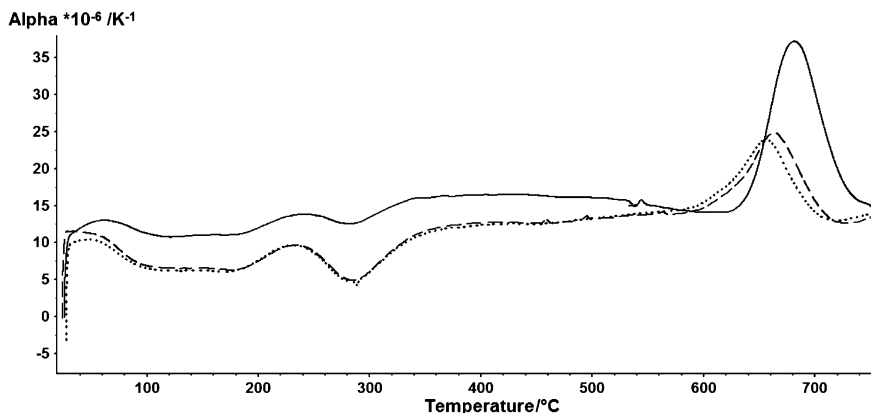


Fig. 3.40 Virtual CTE versus temperature during heating up to 750 °C for sample quenched from 1,110 °C: as-hardened (*solid line*), as-hardened and refrigerated in liquid nitrogen within 15 min after quenching (*dotted line*), and as-hardened and refrigerated in liquid nitrogen after 24 h (*dashed line*)

Concluding, retained austenite is transformed to martensite during the cooling section of the tempering cycle at usual conditions, which commonly employs 550–600 °C maximum. It is known that the prerequisite for the transformation is a process called conditioning, which simply describes the precipitation of alloy carbides in the temperature range from 200 up to 540 °C, during which the alloy content of the retained austenite is reduced. Since the high alloy content of the retained austenite is the reason that it does not transform in the heating section of tempering for the investigated type of steel, the alloy element depletion described above enables the transformation during cooling. It was further shown that the retained austenite is almost completely transformed during cryogenic treatment, even if there is considerable time between quenching and subzero treatment, and a coarse martensite needle structure is formed. At correct austenitizing conditions (1,040 °C) the content of retained austenite formed upon quenching is very low, and after single tempering it is negligible.

3.3.3 Fatigue Behavior of Cold Work Tool Steel (Böhler K110) in Longitudinal Direction

3.3.3.1 Fatigue Data and S–N Curve

Table 3.5 presents the obtained fatigue data for steel K110 with high compressive residual stresses (test series K110L-I, Table 3.5a) and with low residual stresses

Table 3.5 Fatigue data of (a) K110L-I (high compressive residual stresses at the surface) and (b) K110L-II (low residual stresses)

Internal fatigue sample number	Strain at gauge length (mm)	Stress amplitude (MPa)	Cycle number to failure	Crack origin location
<i>(a) Test series K110L-I</i>				
36	4.76E-03	1000	2.41E+05	Internal fish-eye
35	4.54E-03	950	4.25E+05	
39	4.42E-03	930	2.55E+06	
38	4.41E-03	930	2.40E+06	
56	4.41E-03	930	1.60E+05	
54	4.39E-03	930	2.80E+05	
37	4.35E-03	930	2.00E+05	
26	3.99E-03	830	1.45E+06	
24	3.90E-03	830	1.57E+06	
41	3.88E-03	830	1.17E+07	
44	3.88E-03	830	3.30E+07	
27	3.87E-03	800	6.00E+06	
28	3.82E-03	800	5.30E+06	
40	3.78E-03	800	1.57E+07	
25	3.76E-03	800	4.46E+06	
42	3.75E-03	800	8.48E+05	
30	3.71E-03	800	5.77E+06	
29	3.70E-03	800	3.60E+07	
12	3.50E-03	750	1.98E+08	
32	3.01E-03	640	2.11E+08	
47	2.75E-03	600	9.04E+08	At/near-surface
22	4.01E-03	830	6.47E+06	
23	3.91E-03	830	3.97E+07	
21	3.84E-03	800	3.60E+07	
3	3.71E-03	800	1.80E+07	
43	3.71E-03	800	1.07E+08	
13	3.61E-03	750	1.38E+08	
11	3.69E-03	750	1.91E+08	
14	3.67E-03	750	3.00E+07	
4	3.49E-03	750	1.83E+08	
8	3.46E-03	750	1.84E+08	
31	3.30E-03	690	1.30E+08	
10	3.18E-03	690	4.85E+08	
9	3.14E-03	640	8.48E+08	
34	3.06E-03	640	5.13E+09	
5	3.04E-03	640	4.23E+08	
33	3.01E-03	640	1.01E+09	
17	2.97E-03	640	2.96E+08	
15	2.90E-03	600	6.38E+08	
16	2.89E-03	600	1.44E+09	
46	2.87E-03	600	4.00E+08	
19	2.84E-03	600	4.50E+08	

(continued)

Table 3.5 (continued)

Internal fatigue sample number	Strain at gauge length (mm)	Stress amplitude (MPa)	Cycle number to failure	Crack origin location
48	2.81E−03	600	2.75E+08	Runout-specimen
20	2.79E−03	600	5.54E+08	
7	2.79E−03	585	1.00E+10	
18	2.73E−03	575	1.10E+10	
45	2.68E−03	560	1.10E+10	
<i>(b) Test series K110L-II</i>				
79	3.61E−03	750	5.85E+05	Internal fish-eye
78	3.47E−03	750	1.75E+06	
65	3.07E−03	650	1.00E+07	At/near-surface
60	2.88E−03	600	1.88E+07	
77	3.55E−03	750	2.00E+06	
62	3.31E−03	700	1.79E+06	
63	3.38E−03	700	2.65E+06	
73	3.31E−03	700	2.97E+06	
64	3.07E−03	650	9.55E+06	
76	3.10E−03	650	1.85E+07	
74	2.86E−03	600	1.57E+07	
52	2.77E−03	600	4.70E+07	
57	2.74E−03	550	3.70E+07	Runout-specimen
58	2.54E−03	550	3.90E+07	
72	2.55E−03	550	4.90E+07	
71	2.36E−03	500	3.69E+07	
67	2.36E−03	500	6.00E+07	
68	2.36E−03	500	1.87E+08	
59	2.36E−03	500	5.00E+09	
81	2.12E−03	450	3.97E+07	
83	2.12E−03	450	9.80E+07	
82	2.12E−03	450	1.41E+08	
80	2.13E−03	450	3.23E+08	
85	1.92E−03	400	4.54E+08	
87	1.98E−03	410	1.00E+10	
86	1.92E−03	400	1.03E+10	
84	1.94E−03	400	1.05E+10	

(test series K110L-II, Table 3.5b). The different extent of the occurring residual stresses has already been discussed comprehensively in Sect. 3.1.1. However, Fig. 3.41 shows the residual stress depth profiles for as-ground and polished specimens (test series K110L-I, ◆◆) and of samples for which the high compressive stresses had been eliminated through material removal using 15 μm diamond suspension (test series K110L-II, ▲).

Up to 6×10^6 cycles, the specimens of series I showed exclusively internal fish-eye failure (Fig. 3.42, triangles). With increasing cycle number the probability of near-surface failure (Fig. 3.42, circles) increases steadily; in the gigacycle

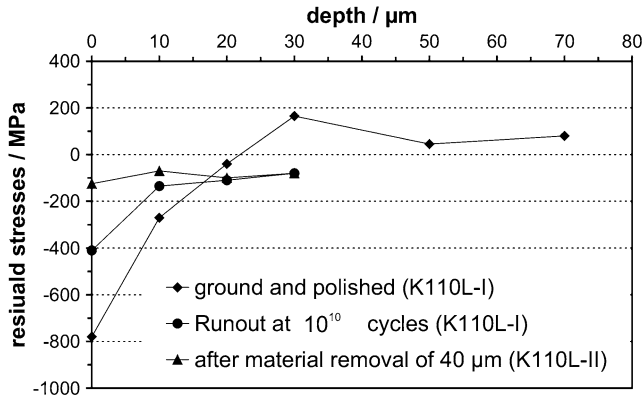


Fig. 3.41 Residual stress profiles for specimens with high compressive stresses (test series K110L-I) before (*filled diamond*) and after cyclic loading at 580 MPa for 10¹⁰ cycles without failure (*filled circle*). In addition the residual stress profile of specimens for which the high compressive stresses had been eliminated by material removal with 15 μm diamond suspension are presented (test series K110L-II, *filled triangle*)

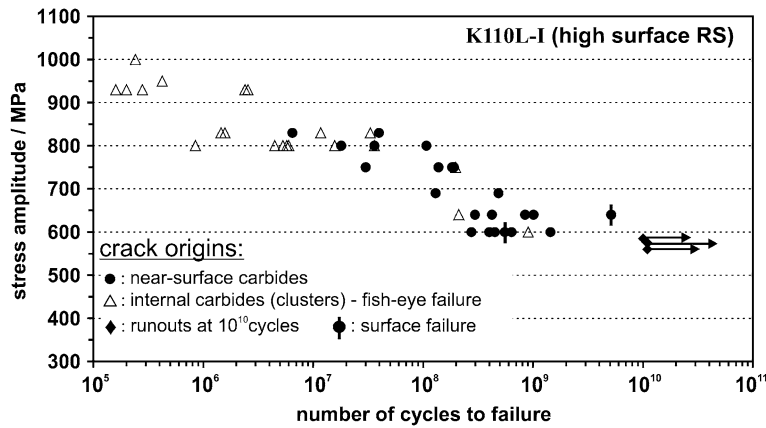


Fig. 3.42 S–N data of steel test series K110L-I (high compressive residual stresses (RS) at the surface), determined jointly with Dipl.-Ing. A.Betzwar-Kotas

regime internal crack nucleation was hardly obtained. The fatigue endurance strength of K110L-I test series at 10¹⁰ loading cycles was around 580 MPa, and thus, about 200 MPa higher than for the specimens with low residual stresses (K110L-II) which revealed a fatigue endurance strength at 10¹⁰ cycles of 400 MPa (Fig. 3.43). Both S–N curves are steadily decreasing and rather linear-shaped, and not a “duplex”- or “twofold”-S–N curve (see Fig. 1.12), which is in agreement with the literature [3], since a fully reversed tension–compression test was applied here (see also Fig. 1.13). However, the S–N curve of specimens with low residual

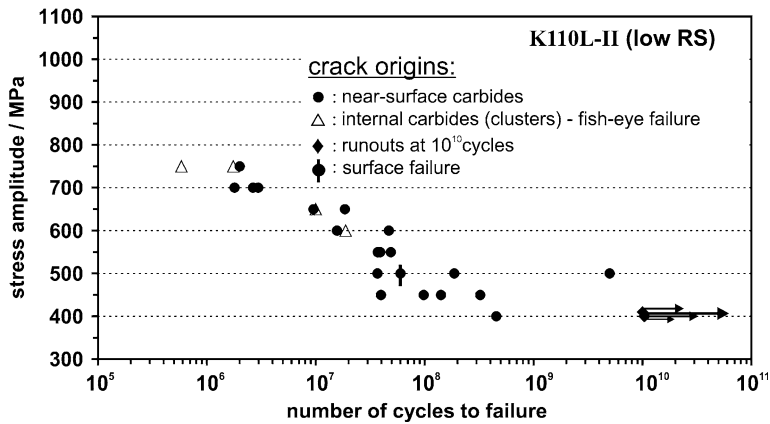


Fig. 3.43 S–N data of steel test series K110L-II (low residual stresses (RS) at the surface), determined jointly with Dipl.-Ing. A.Betzwar-Kotas

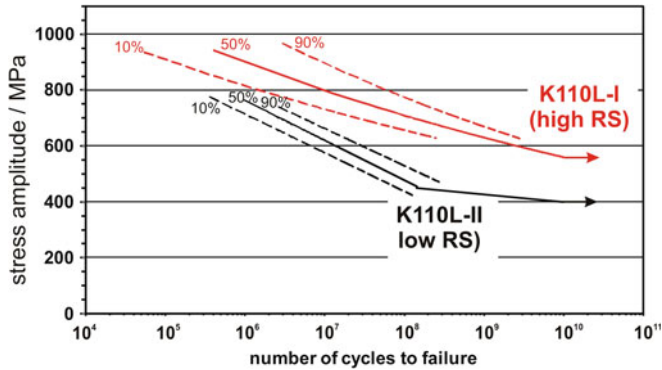


Fig. 3.44 S–N-curves of both K110L test series (with high and low compressive RS, respectively) showing 10, 50, and 90% fracture probability

stresses (K110L-II) showed a slightly steeper slope. Both S–N curves include both types of failure modes—internal and near-surface crack origins. The data scatter of both test series was low. The fatigue strength of K110L-I specimens was significantly higher than that of K110L-II samples with low residual stresses over the entire tested cycle number range (Fig. 3.44—fracture probabilities were calculated using Weibull distribution [19]).

Nearly all fractures of test series K110L-II originated near the surface, only a few internal fish-eye crack nucleation sites were obtained, which was in agreement with statistical estimations, which will be presented later. These suggest that surface crack origins dominate in absence of compressive residual stresses at the surface. Surprisingly, not a single internal failure in series K110L-II occurred below a

stress amplitude of 600 MPa, indicating that the internal defects present are not large enough for enabling the initiation of fatigue cracks at lower loadings, or—hypothetically—that crack growth rates of internal cracks are much lower than for near-surface ones. Crack nucleation sites were primary carbides and carbide clusters, both in the interior and in the surface region, as will be discussed later in more detail.

The occurrence of internally induced failures is most probably the consequence of high compressive residual stresses present at the specimen surface. The transition from internal to surface crack origins in series K110L-I can be attributed to the relaxation of these stresses, which was indicated by residual stress measurements at two runout specimens. Figure 3.41 shows a comparison of different residual stress depth profiles: The as-ground and polished specimen (◆, not yet cyclic loaded) revealed high compressive stresses at the surface, as comprehensively discussed in Sect. 3.1.1. The residual stress depth profile of a similarly as-ground and polished specimen—thus, initially having high compressive stresses at the surface—however, cyclically loaded to $N_{\max} = 10^{10}$ cycles without failure (runout specimen, ●), revealed significantly lower compressive stresses at the surface. A decrease of these stresses during cyclic loading to about one half was obtained.

This observation definitely proved that a relaxation of the initially high compressive residual stresses by cyclic loading took place, due to a “shake-off effect”. Such a residual stress relaxation has also been reported for shot-peened steels [20]. The relaxation of the initially high compressive stresses is thought to enable the crack initiation near/at the specimen surface, as it was obtained at cycle numbers $>6 \times 10^6$ cycles. This phenomenon will be discussed in more detail in the next sections.

3.3.3.2 Theoretical Consideration About the Effect of Residual Stresses on the Fatigue Response

Change of Applied Mean Stress

The influence of residual stresses on the fatigue response is well known in principle: By changing the effective mean stress (σ_m) the fatigue strength along the specimen cross section is directly affected. This was also presented earlier by Macherauch and Hauk [21]. Thus, it would be useful to calculate the location dependent cyclic strength ($\sigma_w(\sigma_m)$) as function of actual mean stress (σ_m) in order to quantify the residual stress influence, which was done using the Goodman equation:

$$\sigma_w(\sigma_m) = \sigma_{w0} * \left(1 - \frac{\sigma_m}{R_m}\right) \quad (3.1)$$

In this Eq. 3.1 σ_{w0} is the location dependent fatigue strength of residual stress-free specimens (which in fact should be “location-independent”, see below), and R_m is the maximum tensile strength of the steel. σ_{w0} is identical to the fatigue

strength of residual stress-free specimens of series II (in which the residual stresses have been removed by polishing with 15 μm diamond suspension), assumed to be constant over the entire cross section. This equation describes the dependence of the fatigue strength on the applied mean stress. Replacement of σ_m by $\sigma_{m,\text{true}}(x) = \sigma_m + \sigma_{\text{RS}}$ introduces the effect of residual stresses, which change the applied mean stress.

Since the residual stresses vary along the specimen cross section, their influence as a function of the distance from the surface (x) on the local fatigue strength can be defined as:

$$\sigma_w(\sigma_{m,\text{true}}(X)) = \sigma_{w0} * \left(1 - \frac{\sigma_{m,\text{true}}(X)}{R_m} \right) \quad (3.2)$$

Obviously, an increase of the compressive residual stresses, which per definition have a negative sign, results in a higher value of the bracket, i.e. in increased fatigue strength. In contrast, the presence of tensile residual stresses accordingly results in lower fatigue strength. Furthermore, it is possible to introduce the ratio r_{ind} , where the subscript “ind” means “indication”, between local fatigue strength and applied stress amplitude,

$$r_{\text{ind}} = \sigma_{w0}(\sigma_{m,\text{true}}(x)) / \sigma_a \quad (3.3)$$

This ratio enables a straightforward prediction where a fatigue crack would possibly start. It indicates the ability of crack nucleation, since for values $r_{\text{ind}} > 1$ the local fatigue strength is higher than the applied stress amplitude σ_a , thus crack initiation is not possible. This concept of local fatigue strength was applied for the present case, which will be presented in the discussion.

Relaxation of Compressive Residual Stresses

Residual stress measurements at runout specimens definitely proved that a decrease of the initial RS took place during cyclic loading (Fig. 3.41). Generally, the relaxation of residual stresses is known to occur as a consequence of thermally activated processes, or static or cyclic plastic deformation. Annealing of the steel at temperatures (T_{Degr}) of approx. 50% of the melting point of the steel matrix (T_m) in Kelvin or higher induce an exponential decrease of the residual stresses in the course of time [21]. The higher the applied annealing temperature, the faster the relaxation process takes place. However, in the present case the temperature was kept constantly low during fatigue testing by specimen cooling. Thus, only a relaxation process induced by micro-plastic or quasi-plastic deformation is conceivable. In the following section a simple model, earlier presented by Macherauch and Hauk [21], will be applied in order to estimate degradation behavior during cyclic loading, which is divided into the tension and the compression part, and it is assumed that the yield strength levels in tension and compression are identical.

Under tensile loading the core of the specimen at which slight tensile residual stresses ($+\sigma_{RS,Core}$) are effective would be deformed plastically first, since high compressive stresses near the surface ($-\sigma_{RS,sl}$) balance the applied tension, thus inhibiting deformation there. Relaxation starts when the loading stress amplitude ($\sigma_{tensile}$) reaches the yield strength ($\sigma_{y,m}$) of the matrix:

$$\boxed{\sigma_{tensile} = \sigma_{y,m} - (+\sigma_{RS,Core})} \quad (3.4)$$

In contrast, application of compressive loading causes deformation in the surface layer first as follows:

$$\boxed{\sigma_{compr} = \sigma_{y,sl} + (-\sigma_{RS,sl})} \quad (3.5)$$

where $\sigma_{y,sl}$ is the yield strength of the surface layer.

If these calculated stress amplitudes are reached during fatigue testing, relief of the residual stresses will mostly occur in the first cycle. If the applied stress amplitude is lower than the stresses required for monotonic yielding, continuous cyclic residual stress relaxation may occur with increasing cycle number if the stress amplitude exceeds the corresponding cyclic yield strengths. This residual stress relief is based on cyclic plastic deformation, dislocation rearrangement, and cyclic softening, which cause a decrease in hardness. The relaxation proceeds slower if stable obstacles inhibit dislocation movement, which might be performed by grain and phase boundaries, finely dispersed incoherent particles, and dissolved foreign atoms. In both above mentioned processes, the range of decrease of the residual stresses depends on several parameters such as the strength of the material, the temperature, the applied loading, extent of plastic strain, cycle number, and the occurrence of obstacles. Total relief of residual stresses can be inhibited by cyclic strain hardening in the material, leading to higher required stresses for residual stress relaxation, as described in the model above. In case of low cycle fatigue, where stress amplitudes are high, residual stress relaxation within the first loading cycle can be supposed. Thus, in this case the surface residual stresses are not as relevant as they are if the applied stress amplitudes are below the stress value where plastic deformation begins. Here, however, the latter was definitely the case, as will be shown in the following.

Assuming $\sigma_{y,m} = 1700$ MPa [11], $\sigma_{tensile} =$ cyclic stress amplitude σ_a and $+\sigma_{RS,Core} = +100$ MPa, the stress amplitude for which relaxation of residual stresses in the core of the specimen starts turns out to be 1,600 MPa during the tensile part of the loading. During the compressive loading, assuming $\sigma_{compr} =$ cyclic stress amplitude σ_a , $-\sigma_{RS,sl} = -800$ MPa, and $\sigma_{y,sl} = 1700$ MPa, the estimate for the stress amplitude at which relaxation of the residual stresses starts in the surface layer is 900 MPa. Since in this study the maximum applied stress amplitudes for most specimens were well below 1000 MPa, residual stress relief within the first loading cycle was not possible. The numerous small secondary carbides existing in the cold work tool steel studied here, which are obstacles to dislocation rearrangement as described above, combined with the high strength of the steel allowing only minimal plastic deformation, are assumed to inhibit

relaxation of the residual stresses. Another possibility for residual stress relaxation can be found in the formation of microcracks. The fatigue testing up to very high cycles renders this alternative highly improbable, since these microcracks would very likely result in fracture of the material. This was not detected here.

Nevertheless, the residual stress measurements at the runout specimen (Fig. 3.41, ●) exhibited a relaxation to about 50% of the initial value (from -800 to -400 MPa). The exact mechanisms how relief of the residual stresses occurs in this very hard material during gigacycle loading at low stress amplitudes still have to be identified.

3.3.3.3 Statistical Consideration of Potential Crack Origin Location

As mentioned in the introduction (Sects. 1.2.4 and 1.2.5), there is at least one fundamental difference between common high strength structural steels with martensitic microstructure, investigated by several cited researchers, and high alloy tool steels: The content of possible crack initiating “defects” is much lower in high strength steels. Quite generally, two prerequisites for a fatigue behavior similar to that of high strength structural steels in the high cycle fatigue regime, showing internal fish-eye crack origins, are (1) the existence of internal defects as possible nucleation sites of fatigue cracks, and (2) a low volume density of these defects so that their location at the specimen surface—where they would of course preferentially nucleate a crack—is highly improbable. The question that arises here for tool steels is at which critical defect concentration the prerequisite no. (2) is not fulfilled any more, since no. (1) is in any case satisfied by the numerous large primary carbides present in the cold work tool steel K110. Based on the investigations of the steel microstructure with respect to carbide sizes and their frequency of occurrence, in this chapter a statistical estimation of crack initiation location is presented in order to determine whether prerequisite no. (2) is fulfilled or not.

With increasing concentration of (supercritical) defects in the volume the probability that at least one of these defects may be found at or very near to the surface, thus causing surface-induced failure due to the higher stress intensity factor there, increases. Mughrabi [22] defined a critical value of volume density for inclusions below which prerequisite no. (2) is fulfilled. His concept will now be applied to carbides which are assumed to be potential crack nucleation sites, similar to the nonmetallic inclusions. The total number of carbides ($N_{\text{Car,V}}$) in the test volume (V_{test} ; specimen diameter d , gauge length l) of the specimen will be:

$$N_{\text{Car,V}} = n_{\text{Car}} * V_{\text{test}} \quad (3.6)$$

With

$$V_{\text{test}} = \frac{\pi * d^2 * l}{4} \quad (3.7)$$

where n_{Car} is the volume density of carbides. Furthermore, the number of carbides ($N_{\text{Car,sl}}$) located at the surface or in a surface layer volume (V_{sl}) of thickness d_{sl} is:

$$N_{Car,sl} = n_{Car} * V_{sl} \quad (3.8)$$

with

$$V_{sl} = \pi * d * l * d_{sl} \quad (3.9)$$

The carbide particles are assumed to have spherical shape. Mughrabi [22] defined the thickness of the surface layer to be equal to the defect particle diameter (here: $d_{sl} = d_{Car}$), since defects just touching the surface were found to exhibit the highest stress concentration [23]. The author of this thesis wants to highlight that this assumption ($d_{sl} = d_{Car}$) has to be critically reflected since larger defects automatically result in larger surface layer volume according to Eq. 3.9. Mughrabi [22] introduced the ratio between the number of carbides within the surface layer and the total number of carbides in the test volume as follows:

$$\frac{N_{car,sl}}{N_{car,v}} = 4 * \frac{d_{car}}{d} \quad (3.10)$$

This ratio represents the probability of occurrence of surface crack origins compared to internal origins, since it gives at least an idea of the probability for carbide particles being present at the specimen surface compared to the number of carbide particles within the specimen bulk material. Thus, a high ratio would favor surface crack initiation while a low one would definitely enhance the occurrence of failure with internal crack initiation from the statistical point of view. After Eq. 3.10 an increase of the specimen diameter results in a lower ratio, favoring internal crack origins, although a larger surface layer volume exists. The present author critically remarks that this implies the ratio being independent of the volume fraction of carbides in the material, and the author speculates if this ratio really allows prediction of crack origin location.

The question now is how to set a limit that allows a precise estimation of the location of the fracture origin. According to Mughrabi [22], the critical condition is given by the existence of at least one defect at the surface, thus if $N_{Car,sl} \geq 1$. Applying this definition of critical condition to Eq. 3.8 allows the calculation of the critical volume density of carbides in the steel as follows:

$$n_{car,crit} \geq \frac{1}{\pi * d * l * d_{car}} \quad (3.11)$$

If the volume density of carbides (n_{Car}) in the steel exceeds the critical value, the occurrence of surface induced fracture would be highly probable at least from the statistical viewpoint.

Based on the described model, the critical values were calculated. Corresponding results are shown in Table 3.6 together with the input parameters used. The specimen diameter d , gage length l and test volume V_{test} are determined by the specimen geometry and the testing system, which have already been described. A carbide diameter d_{Car} of 40 μm was assumed for the calculation. The total carbide volume fraction (TCVF) of the material was determined as 12% (see Sect. 3.3.1).

Table 3.6 List of input parameters and numerical results of statistical estimations for the location of crack initiation

List of input parameters		Numerical results	
Specimen diameter (d)	4 mm	Carbide volume fraction CVF ($d_{\text{Car}} \geq 0.04 \text{ mm}$)	3%
Gage length (l)	3.8 mm	$V_{\text{sl}} = \pi \times d \times l \times d_{\text{Car}}$	1.81 mm^3
Carbide diameter (d_{Car})	0.04 mm	$V_{\text{Car}} = 4/3 \times \pi (d_{\text{Car}}/2)^3$	$3.35 \times 10^{-5} \text{ mm}^3$
Test volume (V_{test})	45 mm^3	$n_{\text{Car}} = \text{CVF} \times V_{\text{test}}$	$900 \text{ carbides mm}^{-3}$
Total carbide volume fraction (TCVF)	12%	$n_{\text{Car,crit}}$ (using Eq. 3.11)	$1 \text{ carbide mm}^{-3}$
		$N_{\text{Car,V}} = n_{\text{Car}} \times V_{\text{test}}$	$4.0 \times 10^6 \text{ carbides}$
		$N_{\text{Car,sl}} = n_{\text{Car}} \times V_{\text{sl}}$	$1.6 \times 10^5 \text{ carbides}$
		$N_{\text{Car,crit,V(test)}} = n_{\text{Car,crit}} \times V_{\text{test}}$	25 carbides
		Ratio ($N_{\text{Car,sl}}/N_{\text{Car,V}}$)	4%
		Critical CVF = $n_{\text{Car,crit}} \times V_{\text{car}}/V_{\text{test}} \times 100$	$4 \times 10^{-5}\%$

The classification of carbides according to their size presented there reveals that for 25% of the total carbide volume fraction the particle—or particle cluster—diameter is larger than $40 \mu\text{m}$, thus, $\text{CVF} (d_{\text{Car}} \geq 0.04 \text{ mm}) = 12 \times 0.25 = 3\%$. Multiplying this value with the test volume gives the carbide volume density $n_{\text{Car}} (d_{\text{Car}} \geq 0.04 \text{ mm})$, which turned out to be $900 \text{ carbides mm}^{-3}$. The calculation of the critical carbide volume density using Eq. 3.11 shows that the actual carbide volume density significantly exceeds the critical value of $1 \text{ carbide mm}^{-3}$. The critical carbide volume fraction (critical CVF) is obtained by multiplying the critical carbide volume density ($1 \text{ carbide mm}^{-3}$) and the quotient of carbide and test volume (0.04). Definitely, the actual CVF is several orders of magnitude higher than the critical CVF. The number of carbides in the test volume and in the surface layer volume can be determined by multiplying the carbide volume density with the test volume and surface layer volume, respectively. The critical number of carbides within the test volume, i.e. the number of supercritical carbides above which failure from such a carbide located at the surface would occur, is given by multiplying critical carbide volume density and test volume. It turned out to be only 25 carbides. Actually, 4 million of supercritical carbides ($d_{\text{Car}} \geq 0.04 \text{ mm}$) exist in the test volume, thus the critical carbide number is by far exceeded.

All three actual material values—carbide volume density, carbide number in the test volume, and carbide volume fraction—significantly exceeded the corresponding critical values. This fact indicates that the occurrence of surface-induced failure is very plausible from the statistical point of view, since the probability that at least one supercritical carbide particle is located at the surface is very high. If such a supercritical defect is located at the surface, a fatal crack would be nucleated there and would cause failure, regardless of the number of defects located in the interior of the specimen. Furthermore, the stress intensity factors are higher for cracks starting at the surface compared to competing cracks originating

in the core material. This also supports crack initiation at the surface if defects are located there.

The ratio $N_{\text{Car,sl}}/N_{\text{Car,v}}$ would at first indicate that the probability of surface crack initiation is rather low, since it claims that only 4% of the number of the carbides within the test volume are located at the specimen surface. However, it is speculated that the ratio $N_{\text{Car,sl}}/N_{\text{Car,v}}$ is only applicable for discrimination of surface induced and interior failure type if the number of defects is rather diminutive, i.e. the defects are real “singularities”. However, it should be mentioned at this point that other factors have to be taken into account for predicting whether internal or surface induced failure would take place: These are the stress intensity factors, residual stresses, crack nucleation potential of the defect particles, and crack formation and propagation mechanism.

Summarizing, from the statistical point of view it is expected that the steel K110 would fail preferentially from the surface, at least if other factors such as compressive residual stresses can be excluded, which agrees with the experimental findings for test series K110L-II (Fig. 3.43). It is noted that the obtained results are qualitatively and quantitatively in good agreement with the concept of Mughrabi [22]. However, in addition to Mughrabi’s type I and II materials, the present authors propose to introduce a further material class—*type III materials*—which are characterized by a high defect concentration widely exceeding the critical volume density, resulting in a high probability of a defect being located at the surface, which thus triggers fatigue crack initiation there. The presented estimations will be further discussed in relation to the experimental results in Sect. 3.3.3.4.

3.3.3.4 Discussion of Observed Fatigue Crack Origin Locations

The occurrence of near-surface crack origins in fatigue loaded tool steels was reported earlier [5, 10–12]. However, those authors included this crack nucleation type in the “surface-related” failure mode. Here, it is tried to differentiate the type of surface failure observed here from a “usual” *surface* crack initiation failure, such as machining flaws or persistent slip bands, by referring to the former as “*at/near-surface*” crack initiation, since it is caused by primary carbides/clusters located in the surface layer, thus within the material structure. Other possible reasons for the observed at/near-surface failures could be stress corrosion and cavitation, which however could be excluded, as shown in Sect. 3.2.4.

In specimens with initially high compressive residual stresses (K110L-I), at N up to 6×10^6 cycles exclusively internally induced failure was observed. This is very likely the consequence of the existence of these compressive stresses at the specimen surface. Using the equation [21] $R_m = 4,02 \cdot \text{HV} - 374 \approx 2250 \text{ MPa}$ and the measured residual stresses at the various depth levels enables to calculate the local fatigue strength using Eq. 3.1, presented in Sect. 3.3.3.2. Furthermore, it is possible to calculate the ratio r_{ind} using Eq. 3.3 for the test series K110L-I

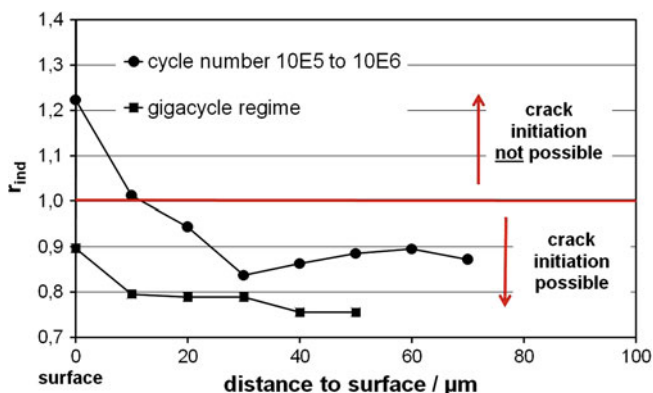


Fig. 3.45 Ratio r_{ind} of local fatigue strength and applied stress amplitude versus distance to surface for specimens of series I (K110L-I)

(high initial compressive RS) as a function of distance from the surface based on the model presented in Sect. 3.3.3.2.

Figure 3.45 (●) presents the obtained data of r_{ind} for specimens of test series K110L-I (high compressive RS) versus the distance from the specimen surface. Thereby, it is assumed that the residual stresses in the cycle number range of 10^5 to 10^6 are similar to the stresses measured at a ground specimen. This seems to be applicable since relaxation of the residual stresses does not occur up to very high cycle numbers (Fig. 3.1). For cycle numbers between 10^5 and 10^6 fatigue results had indicated that surface induced failures were inhibited supposedly by the high compressive residual stresses at the surface, so that only internal fish-eye failure was observed in this region (Fig. 3.42). As described before, compressive residual stresses change the applied mean stress in such a way that the loading is nearly exclusively compressive. However, since tensile loading would be required for crack growth, failure originating at the surface is not obtained, which is supported by the results in the fatigue regime of 10^5 to 10^6 (●) presented in Fig. 3.45, which revealed $r_{\text{ind}} > 1$ to a depth of more than $10 \mu\text{m}$. According to the definition of r_{ind} , this means that the local fatigue strength is higher than the applied stress amplitude (tensile part). It is noted here that, due to equilibrium of residual stresses within the part, slight tensile residual stresses are always found below the compressive layer that balance the compressive stresses. These tensile stresses can enhance the crack initiation and propagation in the affected zone, according to the above presented concept (Sect. 3.3.3.2).

The unusual transition from internal to surface crack origins with increasing cycle numbers, as observed with specimens with high initial surface residual stresses of series K110L-I, can be attributed to the relaxation of these residual stresses, as described above. This transition is in disagreement with published fatigue results for high strength steels, which always revealed a transition from surface to fish-eye type failure at increasing N , as discussed in the introduction (Sect. 1.2.4). However, in all of the cited studies, martensitic steels without

μm -sized carbides and containing a low amount of intrinsic defects, mostly non-metallic inclusions, have been under investigation. There, at $N > 10^7$ cycles the fatigue failure occurs due to these internal singularities as shown above, surface singularities are highly improbable, and residual stresses at the surface do not influence this crack initiation process. However, the surface crack initiation taking place at cycle numbers below 10^7 might be affected in the same way as presented here, but the level of applied stress amplitude has to be considered since at the high amplitudes in case of low N , relaxation of the residual stresses might occur within the first loading cycles, as discussed above. The low amount of defects in the fully martensitic steels is a major difference to the cold work tool steel K110, which contains numerous primary carbides (required for high abrasion resistance). According to the statistical consideration presented above, failure due to one of these large primary carbides located at the surface is most likely, unless other factors such as residual stresses play a role. The reason for the unusual transition from internal to near-surface crack initiation in test series K110L-I with increasing N /decreasing stress amplitude results from (a) different material being tested compared to the high strength bearing steels described in the literature and (b) the influence of initially high compressive residual stresses present at the specimen surface, which were then partially relieved during cyclic loading.

Naito et al. [24] observed fatigue failure at internal nonmetallic inclusions for electropolished samples of carburized steel beyond 10^6 loading cycles. If however surface anomalies were present (in the as-carburized, non-polished) specimens, crack initiation occurred at the surface within the entire cycle number range tested (10^4 to 10^8). This indicates that fish eye failure due to internal defects occurs only in absence of similarly sized surface defects—extrinsic or intrinsic—(or if any such defects are “shielded” e.g. by compressive residual stresses), which confirms the results for test series K110L-I and -II of the present study. The residual stresses in test series K110L-I suppress the crack formation at the surface as long as these stresses are not relieved. In the gigacycle regime, partial relaxation of the residual stresses takes place, so that in the entire cross section, not only in the core, the applied stress amplitude is higher than the local fatigue strength of the material (Fig. 3.45, ■), thus, enabling crack initiation also at/near the surface. However, in addition to influencing the location of crack origin, the residual stresses also considerably affect the level of the fatigue strength in particular in the gigacycle regime where the relative effect of the residual stresses on the mean stress—resulting in a shift to compressive mean stress—is most pronounced. It must be considered that a significant part of the applied tensile stress amplitude—which is required for crack nucleation—is consumed for balancing the compressive residual stresses near the surface where crack initiation takes place in this cycle number range. Therefore K110L-I yields a fatigue endurance strength that is about 30% higher than in K110L-II.

Considering the occurrence of both internal and near-surface failures, in the following the relevant literature is critically reflected in this respect: Fukaura et al. [11] investigated Japanese SKD-11 tool steel (AISI D2 equivalent) using a rotating bending testing system. For stress amplitudes above 1000 MPa,

those authors [11] obtained failures originating from the surface and from near the surface at one or more primary carbide particles. Below 1000 MPa the fatigue cracks initiated in the interior of the specimens, forming so-called fish-eyes. It is speculated here that these internally induced failures result from high residual stresses present at the surface, since the specimens have been polished using abrasive paper and 1 μm diamond paste. The present author assumes that this preparation procedure would induce high compressive residual stresses. Unfortunately, the authors in [11] did not measure these stresses and did not make any comment either. Marsoner et al. [5] found higher number of failures caused by surface carbides for specimens with low residual stresses—those authors definitely measured the surface residual stresses and performed removal of the affected surface layer. They also claimed that the residual stresses did not show any significant effect on the fatigue endurance strength at 10^6 cycles. This would at first disagree with the findings obtained in the present work; however, the S–N-curves of the two K110L test series (Fig. 3.44)—for high and low residual stresses—indicate that the gap between the two data scatter bands gets smaller with increasing stress amplitude, i.e. at lower N, and at $N = 10^6$ even overlaps, which would explain the observation by Marsoner et al. [5]. This can probably be attributed to the relatively lower influence of residual stresses on the mean stress at higher applied stress amplitudes. The same applies to findings of Meurling et al. [12], who also claimed that there is no influence of the residual stress on the fatigue limit at 2×10^6 cycles for the PM tool steel studied there.

3.3.3.5 Crack Origins and Fractography

Crack Initiating Microconstituents

Around the crack origin a (half) fish-eye was formed in case of near-surface and internal failure, respectively, appearing bright in the light microscope (Fig. 3.46). In the surrounding of the crack origin a dark area can be detected in the light optical micrograph, as described by Murakami et al. (see Sect. 1.2.4). However, in the SEM this area appears bright due to its high surface roughness. Macroscopically, the morphology of the fracture surfaces obtained for internal and at/near-surface failures looked quite similar, which can also be seen in Fig. 3.47, and showed very flat morphology. Secondary cracks and ridges point back to the crack origin.

Crack nucleation sites were primary alloy carbides and carbide clusters, located in the interior (Fig. 3.47a, b) and in the surface region (Fig. 3.47c, d), influenced by the existence of residual stresses at the surface, as described above. However, it was not possible to identify all of the near-surface origins since final fracture and the fatigue process seemed to damage or even destroy these near-surface areas, as described in Sect. 3.2.5.

Near-surface cracks originated at carbides/carbide clusters which just touch the surface or are located just below the surface, offering highest stress

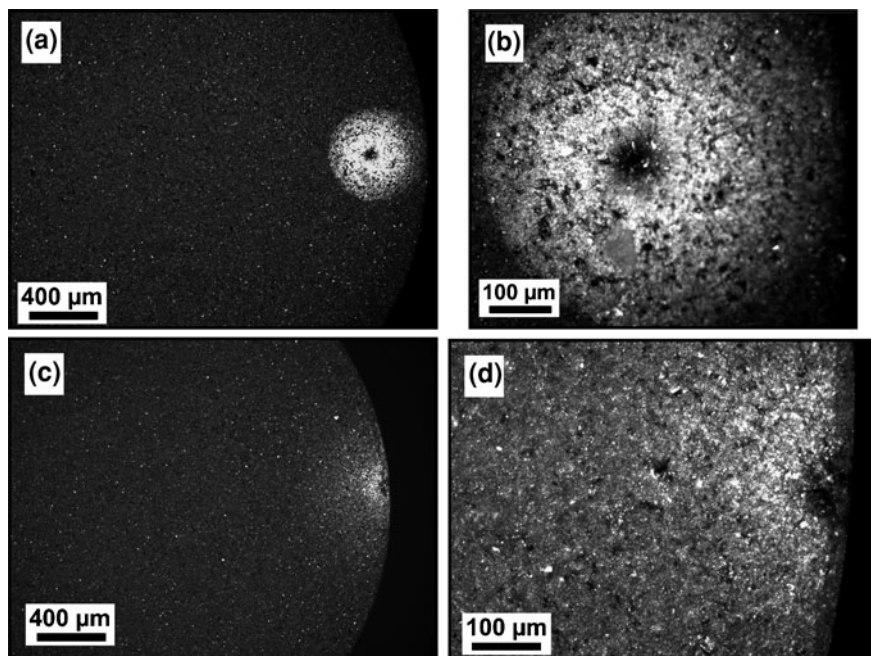


Fig. 3.46 Optical micrographs of K110L-I fracture surfaces: **a** and **b** internal fish-eye failure of sample K110L-12 broken at 750 MPa after 1.98×10^8 cycles, **c** and **d** at/near-surface failure of sample K110L-17 broken at 640 MPa after 2.96×10^8 cycles

concentration [23]. In most cases internal failures revealed carbide clusters in the center of the fish-eye, which involved several large single carbides closely adjacent to each other at distances of some microns. This constellation seems to be a prerequisite for internal crack formation here, since this arrangement might offer highest stress concentration due to superposition of the stress fields of the individual carbides. The obtained cluster diameter turned out to be in the range of 17–130 μm (95% confidence interval), the distances of the origins to the surface are between 340 and 800 μm and do not show any dependence on the stress amplitude and cycle number.

Only in a few cases one large single primary carbide with a size similar to the usually observed clusters initiated an internal fatigue crack. It should be noted that if a carbide cluster is located at the surface it may be assumed to act as a single defect with a size far larger than one single primary carbide particle. Of course the same holds in the interior Fig. 3.48a, b show the crack origin of a specimen of series K110L-II, which failed at a stress amplitude of 400 MPa after 4.5×10^8 loading cycles, at which three other specimens ran out at 10^{10} cycles without failure. The two images definitely reveal a carbide cluster with a diameter of about 30 μm just touching the surface, supporting the above described effect.

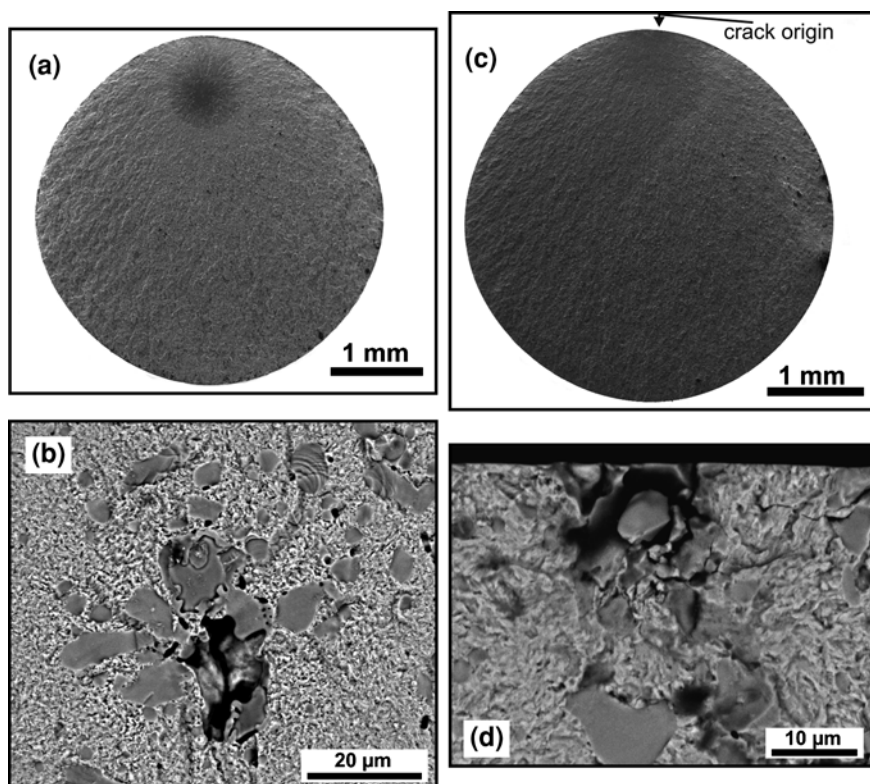


Fig. 3.47 Fractographs of **a, b** a specimen failed at 800 MPa after 6.00×10^6 cycles due to a large carbide cluster (K110L-27) and of **c, d** a specimen (K110L-17) failed at 640 MPa after 2.96×10^8 cycles due to primary carbides located just beyond the surface

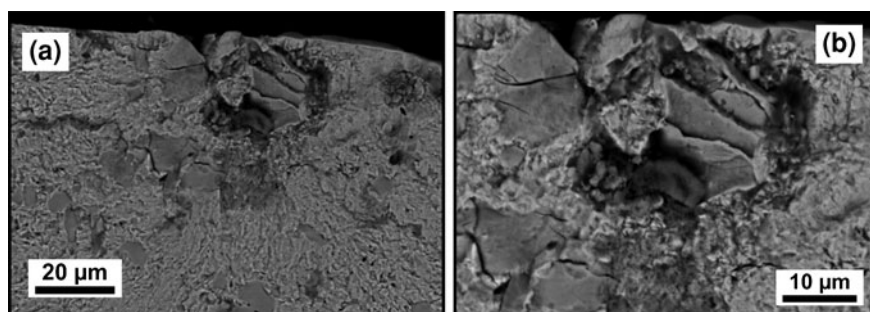


Fig. 3.48 Near-surface crack origin of specimen (K110L-II-85) failed at 400 MPa after 4.5×10^8 cycles due a large carbide cluster

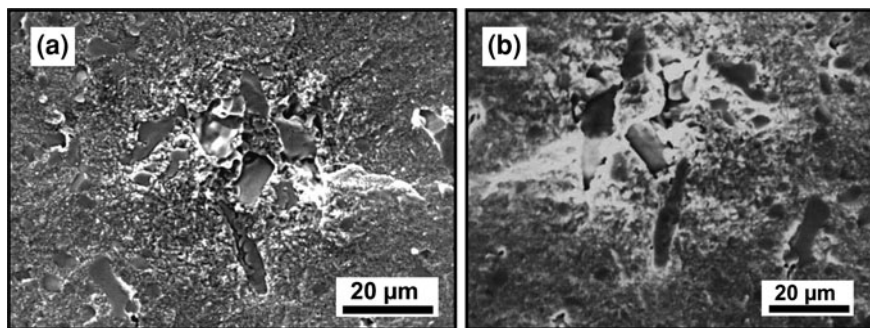


Fig. 3.49 Specimens (K110L-I-12) failed due to an internal carbide cluster. The carbide particles were found to be fractured at the crack origin: **a** and **b** show the mating fracture surfaces

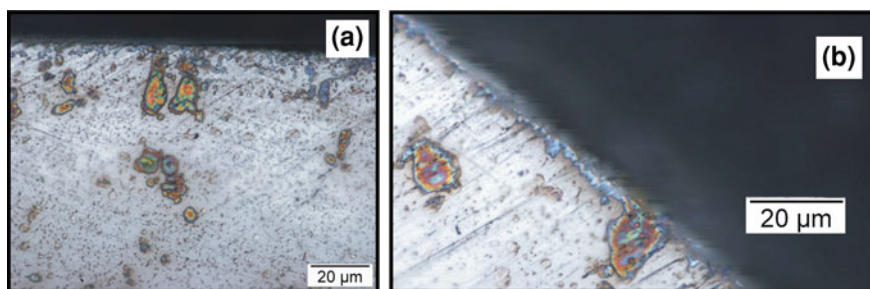


Fig. 3.50 Carbides located close **a** or **b** just at the surface of broken fatigue specimen

The hard carbide particles were fractured in transgranular mode at the entire fracture surface, including the crack origins, rather than decohering from the matrix, as shown in Fig. 3.49. In some cases the crack proceeded around the initiating carbide particles so that a hole was observed at the mating fracture surface. In case of near-surface failure, the question if carbide particle cracking either occurred during grinding/polishing [25] or due to cyclic loading might be of high importance for the crack initiation process. However, investigations of the surface after grinding and polishing and on fracture surfaces (see Fig. 3.50) did not reveal any damages of the surface or the carbides, which renders particle cracking during the grinding/polishing process highly improbable. Thus, carbide cracking can only occur during cyclic loading, or at final fracture due to excess energy.

It remains unclear during which stage internal carbide cracking occurs. Fukaura et al. [11] performed acoustic emission experiments revealing that carbide fracture occurs only above 1100 MPa under static tensile stressing. Mughrabi [22] did calculations for a high strength steel, estimating whether particle debonding or fracture act as crack initiation. It turned out that stress amplitudes of 1000 MPa and below are far too low to support either process. However, detailed SEM observations, which will be discussed later, indicated that particle debonding

might also be part of fatigue crack formation. In this respect, the particle–matrix interface is of high importance regarding also the fact that the two phases reveal considerable differences of hardness and Young’s modulus. Furuya et al. [26] claimed for hard nonmetallic inclusions that impurities such as TiN fracture during cyclic loading in contrast to Al_2O_3 that showed predominantly particle–matrix debonding. Berns et al. [9] claimed that the fracture of large primary carbides in AISI D2 type tool steel is the origin of fatigue cracks, which may indicate that plain energy consideration may not apply in case of cyclic loading, since progressive deformation of the particle and of the surrounding material occur even at low stresses, thus, representing a typical fatigue phenomenon. However, the author speculates that combination of particle fracture and matrix debonding due to stress superposition either with free surface or with closely arranged particles is involved in formation of fatigue cracks. Thus, it is unknown until now whether this particle fracture *initiates* the fatigue crack or if it is a *result* of fracture of the specimen.

Detailed Investigation of the Obtained Fracture Surfaces

Detailed investigation of the obtained fracture surfaces were performed, focusing on the life controlling defect size and the different stages of crack nucleation and propagation, in order to clarify the associated failure mechanisms.

The fish-eye appearance around the internal crack origin represents the propagation stages of the fatigue crack. It can be assumed that in this case, final failure occurs when the crack front reaches the specimen surface. In case of near-surface failure, half of fish-eye was obtained representing the crack spreading to the core of the sample. For both failure types the microscopic fracture surfaces revealed up to five zones, likely to be attributable to consecutive stages of fatigue crack growth.

Details of these zones as observed for internal failures are presented in Figs. 3.51 and 3.52. Four of those crack growth zones (including the granular area—see Fig. 3.51b—corresponding to crack formation) can be seen in Fig. 3.51a: “2a” represents a region of very low surface roughness. In some cases, short cracks perpendicular to the fracture surface were detected within this area, as shown in Fig. 3.53. This crack growth stage is followed by zones “2b” and “2c”, which both revealed a significantly rougher surface. There is however no clear boundary between these two zones but rather a gradual change. It seems that a continuous transition of the crack propagation process occurs there. In case of near-surface crack origins, the distinction between these two zones turned out to be still more difficult (Fig. 3.55). The area outside of the fish-eye (zone “3”) corresponds to the final fracture surface.

Primary carbide clusters were observed in the center of the internal fish-eyes, around these clusters a bright granular surface area being found (Figs. 3.51b, c and 3.53). This area, referred to as “granular bright area” (GBA), showed a specific surface morphology. High magnification SEM investigations revealed that there exist small cracks or holes in the material, probably resulting from decohesion of

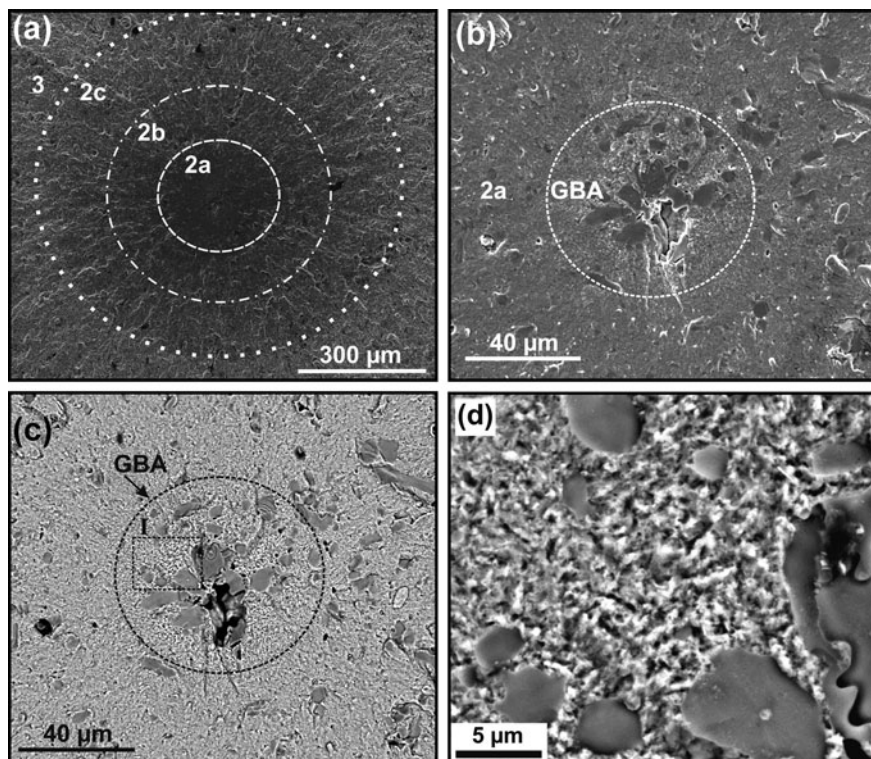


Fig. 3.51 Fracture surface of specimen of series K110L-I (K110L-27) failed after 6.00×10^6 cycles at 800 MPa: **a** SEM image of internal fatigue crack nucleation and propagation zones; **b** SEM image of granular bright area (GBA) in the vicinity of the carbide cluster; **c** and **d** BSE images revealing the granular surface morphology within the GBA

numerous finely dispersed secondary carbides from the matrix. Shiozawa et al. [27] proposed a model for the formation of such a granular area around the crack origin, called “dispersive decohesion of spherical carbides” (Fig. 3.54). There, multiple microcracks are formed by the aforementioned decohesion process which then grow and coalesce to short fatigue cracks. However, unless a sufficiently large short crack length (which corresponds to the GBA size) is reached, these short cracks are non-propagating. Thus, the described microcrack formation and coalescence within the granular area take place until a short propagating crack is formed. Shiozawa et al. [27] claimed that most of fatigue life is spent to form the granular area and that the fine spherical carbides are generated during the fatigue process although there are no informations given by which mechanism this should occur. This seems to be applicable for the material studied here as well. However, here the small carbides are more probably formed during the heat treatment process. In the as-quenched steel, large primary carbides and some small carbides existed. However, the number of small carbide precipitates

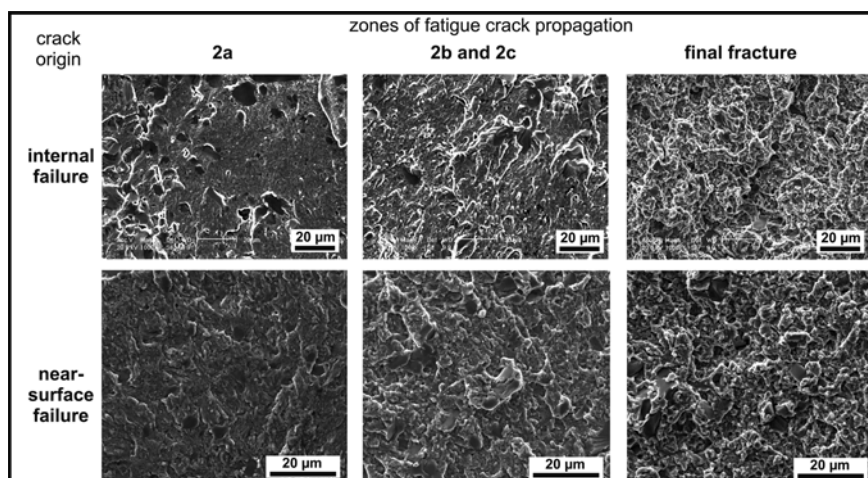


Fig. 3.52 Surface morphologies of the different crack growth zones for internal and near-surface failure

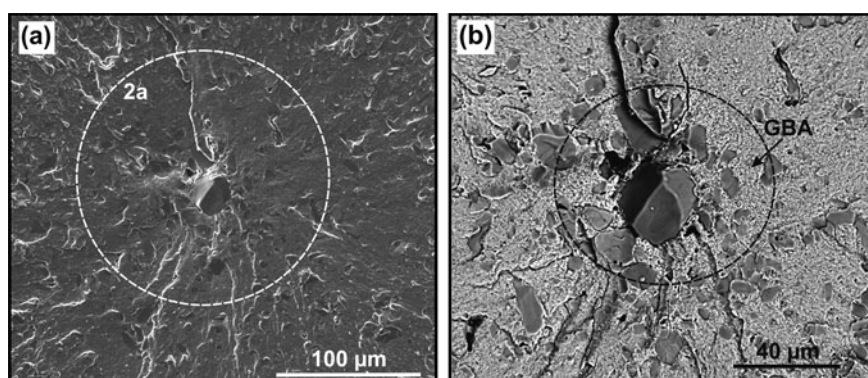


Fig. 3.53 Multiple propagating cracks observed in area “2a” for some of the failed specimens. **a** SEM and **b** BSE image of specimen of series K110L-I (sample nr. 24) failed after 1.57×10^6 cycles at 830 MPa

seemed to be significantly larger in the tempered steel. Furthermore, in place of the nonmetallic inclusion the large primary carbide particles of the carbide cluster offer the stress concentration in their vicinity that is assumed to be necessary to cause the decohesion process. Stickler and Weiss [28] reported that for pores in PM steels the field of stress concentration around an isolated pore can be assumed to be about twice of the diameter of the pore. However, here, in the case of large carbides instead of a pore, which show a significantly lower difference of Young's modulus, this field of stress concentration assumingly is smaller. Thus, the above

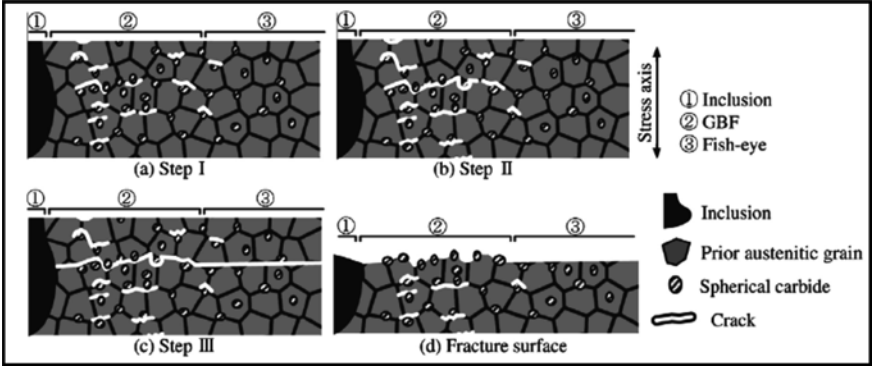


Fig. 3.54 Model for the formation of a granular area around the crack origin, called “dispersive decohesion of spherical carbides”, proposed by Shiozawa et al. (Reprinted from [27] with permission from Elsevier)

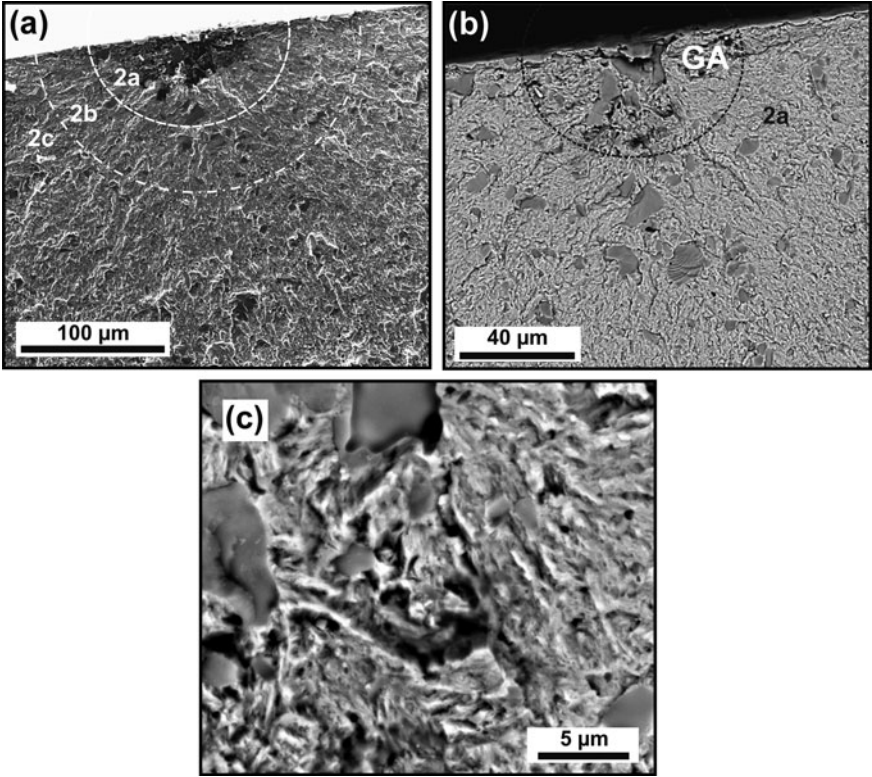


Fig. 3.55 Zones of near-surface fatigue crack nucleation and propagation of specimen (K110L-76) failed after 1.85×10^7 cycles at 650 MPa shown in **a** SEM and **b** BSE image; **c** granular area

mentioned process responsible for the occurrence of a granular area is essential for the formation of a propagating fatigue crack.

At/near-surface crack initiation revealed similar crack growth zones (Fig. 3.55) as observed for internal failures. The surface of the granular area (Fig. 3.55c) showed similar decohesion and microcrack formation around the crack-initiating primary carbide particles. The surface morphology of the subsequent crack growth stages of near-surface induced failures was comparable to the corresponding areas in case of internal crack origin (Fig. 3.52). However, for near-surface failures, stage 2a showed more intergranular facets. Generally, both surface morphologies of near-surface induced failure are similar to surface induced fractures obtained for AISI 4340 high strength steel in vacuum [29]. Shiina et al. [29] showed that surface morphology is similar for surface induced failure in vacuum and internal crack initiation, respectively, which is in agreement with the observations of the present study. However, here liquid cooling was performed. The similarity of fracture surfaces of near-surface originated failures to those obtained in vacuum environment [29] may result from the low compressibility, appreciable viscosity and high surface tension of the cooling medium and high frequency movements of the crack opening, preventing water from getting into the fatigue crack. It may be speculated that this might lead to de facto vacuum environment within the propagating crack.

Summarizing, fatigue fracture surfaces of K110L specimens revealed distinct zones of crack growth around the crack origin for both failure types—internal and near-surface. In both cases the morphologies of these zones are similar, indicating that fatigue crack initiation and propagation are fundamentally identical. The zone around the crack initiating carbide particles—GBA and GA—seems to correspond to microcrack growth, which might be explained by the model proposed by Shiozawa et al. [27].

3.3.3.6 Quantitative Evaluation on K110L Fractographs

The radii of the crack initiating defects and subsequent crack growth stages were determined and then correlated to applied stress amplitude and cycle number to failure. Evaluation of internal failures was performed exclusively for series K110L-I, since only four specimens of series K110L-II revealed internal fish-eye fracture. Thus, reasonable statistical evaluation was not possible there. But principally similar appearance of the fracture surfaces was obtained for internal failures of series II. Numerical results for internal failures are presented in Table 3.7. Evaluation for near-surface crack initiation type was performed for both series, however, it turned out that the high residual stresses in series K110L-I heavily influenced the results. Thus, here only the results for near-surface failures of series K110L-II with low residual stresses are presented (Table 3.8).

The radii of the largest single carbides in such clusters ranged from 5 to 22 μm . The distances of the crack origins to the specimen surface are in the range of 340–800 μm and did not show any relationship to the applied stress amplitude and

Table 3.7 Numerical results of quantitative evaluation of the crack nucleating and growth features observed on the fractographs of internal failures of series K110L-I (high compressive stresses at the surface)

Internal crack origins of series K110L-I (high compressive stresses at the surface)			Radii of						Distance of
Internal sample number	Cycle number to failure	Stress amplitude (MPa)	Largest single carbide of carbide cluster (μm)	Carbide cluster (μm)	GBA (μm)	Area 2a (μm)	Area 2b (μm)	Area 2c (μm)	Crack origin to surface (μm)
36	2.41E+05	1000	10	25	25	65	145	267	503
35	4.25E+05	950	7	15	24	84	146	255	365
39	2.55E+06	930	6	23	30	50	181	311	458
38	2.40E+06	930	7	13	24	64	200	350	780
37	2.00E+05	930	21	50	50	92	196	332	529
26	1.45E+06	830	10	29	48	79	212	350	499
24	1.57E+06	830	17	29	52	106		383	464
41	1.17E+07	830	5	18	30	93	252	355	551
44	3.30E+07	830	5	15	33	54	181	295	353
27	6.00E+06	800	7	15	43	83	235	451	632
28	5.30E+06	800	12	28	50	133	308	434	635
40	1.57E+07	800	14	38	38	99	175	282	336
25	4.46E+06	800	10	48	48	78		330	395
42	8.48E+05	800							
30	5.77E+06	800	19	45	45	135	246	370	461
29	3.60E+07	800	6	21	39	98	270	320	384
12	1.98E+08	750	5	21	33	113	195	288	344
32	2.11E+08	640	16	30	60	148	309	496	712
47	9.04E+08	600	10	18	43	170	380	504	623

cycle number to failure, which also holds for the largest single carbide sizes and carbide cluster sizes. In contrast, sizes of the GBA, the areas of stage 2a, 2b, and 2c showed a significant dependence on the applied stress amplitude (Fig. 3.56a). The lower the stress amplitude, the larger the obtained crack growth zones are. This observation can be explained considering the equation for the cyclic stress intensity factor:

$$\Delta K \sim \Delta \sigma_a \sqrt{a} \quad (3.12)$$

in which “a” corresponds to the defect size.

In order to reach the ΔK value required for subsequent crack propagation—which is constant for a given material—larger sizes of the distinct zones are required at lower applied stresses and vice versa. Note that, as mentioned before, the carbide clusters do not show such dependence. Thus, the carbide clusters seem to be too small to nucleate a short fatigue crack by themselves. Only through the

Table 3.8 Numerical results of quantitative evaluation of the crack nucleating and growth features observed on the fractographs of near-surface failures of series K110L-II (low residual stresses at the surface)

Near-surface crack origins of series K110L-II (low residual stresses at the surface)			Radii of					
Internal sample number	Cycle number to failure	Stress amplitude (MPa)	Largest single carbide of carbide cluster (μm)	Carbide cluster (μm)	GBA (μm)	Area 2a (μm)	Area 2b (μm)	Area 2c (μm)
52	4.70E+07	600						
57	3.70E+07	550			50	140	170	400
58	3.90E+07	550			43	140	150	405
59	5.00E+09	500			80	207	300	774
62	1.79E+06	700			35	95	200	336.5
63	2.65E+06	700	9		30	60	108	275.5
64	9.55E+06	650		12	30	75	130	330.5
67	6.00E+07	500			73	140	200	587.5
68	1.87E+08	500				171	189	592.5
71	3.69E+07	500						492.5
72	4.90E+07	550			65	131	175	481.5
73	2.97E+06	700	10	16	40	93		
74	1.57E+07	600	8.5	13	41	82	160	350
76	1.85E+07	650	9	18	45	72	320	340
77	2.00E+06	750	4	15.5	38	83	336	325
80	3.23E+08	450	6	13.5	85	230	264	582.5
81	3.97E+07	450	3	6.5	70	270	170	550
82	1.41E+08	450	10		83	240	280	782.5
83	9.80E+07	450	4.5	5	80	225	282	653.5
85	4.54E+08	400	8	23.5	124	265	221	779.5

mechanism of microcrack formation and coalescence taking place within the granular area, as described in the preceding section, the formation of a propagating short crack is possible. In some cases, for which the carbide cluster and the granular area revealed a similar size, short cracks evolved without formation of additional granular area since the carbide cluster was large enough for the initiation of a propagating short crack. The size of GBA, representing a short propagating crack, depends on the stress amplitude as indicated by the relationship (Fig. 3.56a) of the GBA size and the applied stress amplitude. The GBA size does not show a correlation with the cycle number to failure (Fig. 3.56b). However, the difference between GBA size and carbide cluster size shows such a relationship. Thus, the formation of an appropriate-sized GBA—the number of cycles required for GBA formation probably depends on the initial cluster size—represents a proportional part of total fatigue life since the area of GBA size minus carbide cluster size increases with increasing specimen life, which of course correlates with lower applied stress amplitudes. A similar cycle-number-to-failure relationship (Fig. 3.56c) was obtained for sizes of (stage 2a minus GBA) and (stage 2b

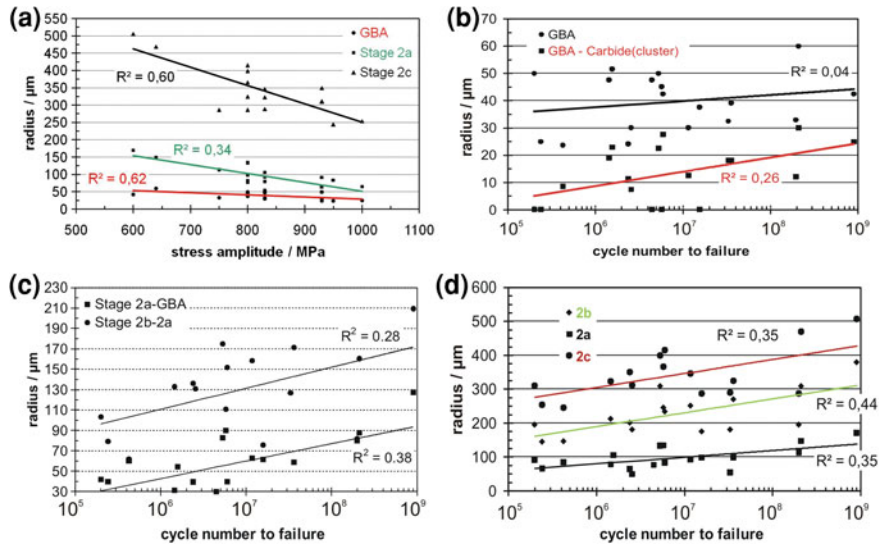


Fig. 3.56 Quantitative evaluation of the internal crack origins and crack growth zones of test series K110L-I (high compressive stresses at the surface)

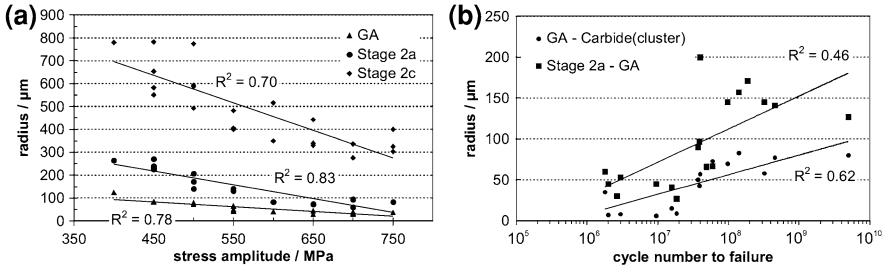
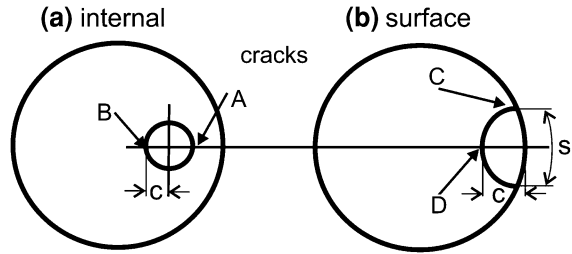


Fig. 3.57 Quantitative evaluation of the near-surface crack origins and crack growth zones of test series K110L-II (low residual stresses at the surface)

minus 2a) which seems plausible since the longer the fatigue life (the lower the stress amplitude) the more time of specimen life is spent during each stage of crack growth. However, for the areas of (stage 2c minus 2b) or (stage 2c minus 2a) such a relationship was not observed which indicates that crack growth stage 2c represents fast fatigue crack propagation just before final fracture regardless of applied stress amplitudes and specimen lives. Of course, the size of the area of stage 2c increased with increasing fatigue life, however, mainly due to larger sizes of areas corresponding to the preceding fatigue crack growth stages (Fig. 3.56d).

In case of at/near-surface induced failure, for which formation of half of fish-eye was observed, similar relationships with stress amplitude (Fig. 3.57a) and cycle number to failure (Fig. 3.57b) were obtained which indicates similar crack

Fig. 3.58 Possible fatigue crack starting points. The circle and half circle represent the crack fronts of internal and surface fatigue cracks, respectively



growth behavior. However, evaluation of crack propagation zones 2b and 2c was difficult to perform since it was not possible to exactly define the boundaries of these stages. Furthermore, in strong contrast to internally induced failure, for crack initiation at/near the surface, single primary carbides gave rise to fatigue crack in many cases, thus, a carbide cluster was often not present there.

For at/near-surface failures of specimens of test series K110L-I such relationships, as presented above, were not obtained, which can be attributed to the significant influence of the high compressive residual stresses at the surface on both, the crack formation and crack growth.

3.3.3.7 Fractographic Estimation of Stress Intensity Factors (Longitudinal Direction)

The fractographic data obtained as presented above were used for the calculation of threshold stress intensity factors for the different crack growth stages, assuming that the size of the area for each stage corresponds to a critical crack length. Theoretically, four crack starting points can be distinguished for the two failure modes, assuming the near-surface crack to be numerically similar to a semi-circular surface crack. The corresponding positions of those points are presented in Fig. 3.58. Table 3.9 gives the corresponding correction factors for calculation of the stress intensity factor. The following assumptions have been made in order to obtain the presented values of f_i : (1) Crack origins are regarded as spherical particles. (2) Near-surface cracks were approximated by surface crack equation. (3) Internal crack origins are situated well below the surface, thus, the ratio between the crack length c and the distance from the surface approaches zero. (4) Crack length c represents the radius of the defect—here, the size of the carbides or carbide clusters and of the areas for the different crack growth stages.

The stress intensity factor range ΔK for the crack heading to the surface from an internal crack origin can be calculated as follows [30]:

$$\Delta K = 1.13\sigma_a\sqrt{c} \quad (3.13)$$

where σ_a and c represent the stress amplitude and the crack radius, respectively.

The geometry factor $f_i \approx 1.13$ for internal cracks was obtained by including all constants of the original equation [30], i.e. $f_i \approx 2/\pi \sqrt{\pi} \approx 1.1284 \approx 1.13$.

Table 3.9 Equations used for calculation of ΔK

	Crack starting point (Fig. 3.58)	Geometry factor f_i and f_s
Internal [30] $\Delta K = f_i \sigma_a \sqrt{c}$	A	1.13 [30]
	B	
(Near-) surface [31] $\Delta K = f_s \sigma_a \sqrt{s}$	C	1.26 [31]
	D	1.13 [31]

Table 3.10 Calculated stress intensity factors (ranges represent the 95% confidence interval of the calculated mean value) of AISI D2 type tool steels for internal and near-surface crack origins of test series K110L-I and II

AISI D2	ΔK range/MPa $\sqrt{m}$		Fatigue process
	Internal CI	Near-surface CI	
Largest single carbide	2.4–3.4	1.3–2.5	Crack nucleation
Carbide cluster	3.9–5.5	1.7–3.5	
GBA	5.2–6.2	4.9–5.5	
Stage 2a	8.2–9.6	7.6–8.6	Start of Paris law regime
Stage 2b	12.8–14.6	n.a.	End of Paris law regime
Stage 2c	14.3–19.6	13.3–18.9	Fatigue fracture toughness

The tensile stress is assumed to be responsible for crack growth, thus, the stress *amplitude* is used in the equation instead of the *full stress range*. Residual stresses were neglected since only marginal or no tensile stresses are expected to be present in the interior of the material. The ΔK values for the near-surface failures were calculated in a similar way using the model [31] for a semi-elliptical surface crack (Fig. 3.58). ΔK was calculated for point C (since there the highest stress concentration takes place) according to the following equation:

$$(\Delta K = 1.26\sigma_a\sqrt{c}) \quad (3.14)$$

where σ_a and c represent the stress amplitude and the crack radius, respectively.

The geometry factor $f_s \approx 1.26$ in Eq. 3.14 was obtained by including all constants of the original equation [31] $\Delta K = 0.71 \sigma_a \sqrt{(\pi s)}$, i.e. $f_s \approx 0.71\sqrt{\pi} \approx 1.2584 \approx 1.26$. In the equation for near-surface cracks [31] “s” is replaced by “c”, which seems to be acceptable for small “c” and “s”. This substitution was performed since the evaluation of “c” in the obtained fracture surfaces turned out to be more accurate.

Table 3.10 shows the obtained numerical data for both types of crack origin and corresponding stages of fatigue crack growth, which were calculated for each specimen according to the aforementioned equations. For these calculations, fracture surfaces of specimens broken at stress amplitudes from 450 to 1000 MPa were evaluated. The presented range of ΔK corresponds to the 95% confidence interval of the calculated mean value. The obtained wide ranges for the ΔK values due to the uncertainty of the growth stage area measurements have to be considered during discussion of the results.

The obtained fatigue fracture toughness values are in good agreement with results found by Fukaura et al. [11] for similar steels tested in rotating bending. Interestingly, the ΔK value for the largest single carbides, the carbide clusters and the crack length of stage 2b and 2c (fracture toughness) revealed a direct linear relationship with the applied stress amplitude, in contrast to the stress intensity factors for the GBA and zone 2a, which did not show such a relationship. The stress intensity factors ΔK are significantly lower in case of near-surface cracks. However, the principal similarity of numerical ΔK data and the appearance of fracture surface morphologies for the two crack origin locations indicate that the mechanisms responsible for crack initiation and propagation are similar. The author speculates that the formation of the granular area plays a decisive role in the formation of short propagating fatigue cracks, as discussed above. During this process, numerous miniature cracks are assumed to be formed according to the model proposed by Shiozawa et al. [27]. Thus, the present author assumes that the threshold ΔK for crack growth of short fatigue cracks corresponds to the stress intensity factor ΔK calculated for the GBA ($4.9\text{--}5.2 \text{ MPa}\sqrt{\text{m}}$ —lower limits of obtained ΔK for near-surface and internal crack initiation, respectively), which turned out to be totally independent of the applied stress amplitude. There is also a good agreement with the experimentally determined fatigue crack growth behavior for AISI D2 steel performed in compact tension test at 6 Hz and $R = 0.1$ by Berns et al. [9] that showed increasing crack growth rates above $5 \text{ MPa}\sqrt{\text{m}}$. Furthermore, those results [9] also showed a narrow stable crack growth regime (Paris-law regime), which is supported by the data obtained here.

However, the nucleation of microcracks might start at even lower ΔK values, supposedly at the obtained ΔK values for single carbides or carbide clusters, which seems to be a prerequisite process for the subsequent formation of small fatigue cracks. Assuming that these ΔK levels are essential for initiation of fatigue failure, the corresponding ΔK values for single carbides and carbide clusters, respectively, were used for estimation of the fatigue endurance strength at 10^{10} loading cycles—a real fatigue limit does not seem to exist—of residual-stress-free specimens. It is calculated for internal and near-surface originating cracks according to the equations presented above in Table 3.9 ($\sigma_a = \Delta K / (f \sqrt{c})$, where c corresponds to the flaw size), using the largest sizes of carbides and carbide clusters observed in crack origins and metallographic investigations.

Table 3.11 shows the obtained results. Considering the fact that the fatigue endurance strength for residual stress-free specimens of series K110L-II at 10^{10} cycles was about 400 MPa, the calculation of the fatigue endurance strength based on near-surface cracks using the largest carbide and carbide cluster size obtained in near-surface crack origins—440 and 380 MPa, respectively—turned out to represent quite good estimates. Interestingly, the largest carbide cluster found in near-surface crack origin had the same size as the largest cluster observed in metallographic sections, thus indicating that the calculation of the fatigue endurance strength at 10^{10} loading cycles might be possible by using the cluster size observed in metallographic investigations.

Table 3.11 Estimates for fatigue endurance strength at 10^{10} cycles for AISI D2 type tool steel in longitudinal direction and low residual stresses at the surface

Defect type	Defect radius/ μm	Mean $\Delta K/\text{MPa}\sqrt{\text{m}}$	Estimated fatigue endurance strength/MPa	Failure origin location
<i>Largest single carbide</i> —observed in near-surface crack origin	12	1.9	440	Near-surface (low RS)
<i>Largest carbide cluster</i> —observed in near-surface crack origin; corresponds also to largest observed cluster size in metallographic investigation (see Table 3.3)	30	2.6	380	
<i>Largest single carbide</i> —obtained in internal crack origin	22	2.9	550	Internal
<i>Largest carbide cluster</i> —observed in internal crack origin	53	4.7	570	
<i>Largest single carbide</i> —observed in internal crack origin	22	1.9	320	Near-surface (low RS)
<i>Largest carbide cluster</i> —observed in internal crack origin	53	2.6	280	

Using the corresponding largest values of carbide (cluster) sizes obtained in internal crack origins for estimation of the fatigue endurance strength at 10^{10} loading cycles based on internal crack initiation revealed relatively high fatigue strength, which is in good agreement with experimentally obtained results, since internal fish-eye failure was not obtained at stress amplitudes below 600 MPa. Thus, the predominant occurrence of near-surface induced failure at stress amplitudes below 600 MPa in both test series might be explained in that way. If now those carbide (cluster) sizes that had been experimentally observed in internal crack origins are used for estimation of the fatigue endurance strength at 10^{10} loading cycles but based on near-surface cracks, significantly lower fatigue strength values resulted that are below those experimentally obtained, i.e. this case—as large carbides as were found in the core being present at the surface—has apparently not occurred. However, it is noted here that these large species of carbide (clusters) are in fact material singularities. The probability of occurrence of such a large cluster at the surface is much lower than in the interior of the

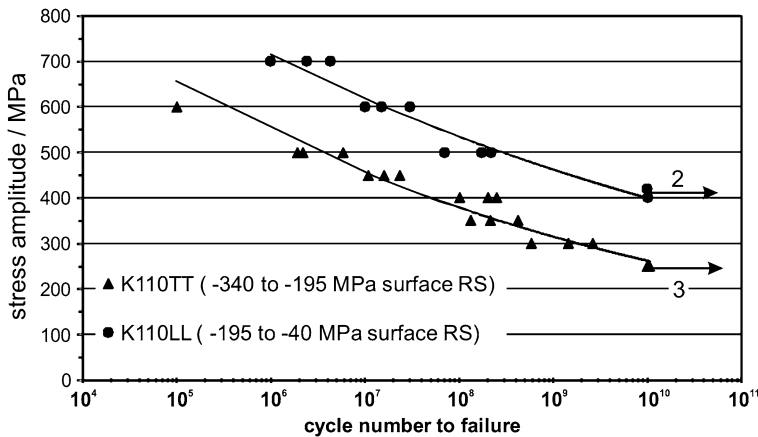


Fig. 3.59 S–N data of steel test series K110TT and LL (low residual stresses at the surface) determined jointly with Dipl.Ing.A.Betzwar-Kotas. Figures close to the *arrows* represent the number of runout specimens

specimen. Thus, also the probability of failure due to such large carbide clusters located at the specimen surface is low; however, it might be possible to take place. This has been already discussed in detail in [Sect. 3.3.3.3](#).

3.3.4 Fatigue Behavior of Cold Work Tool Steel K110 in Transverse Direction

In addition to fatigue investigations in longitudinal direction of steel K110, also the fatigue response transverse to the rolling direction of the steel bar was evaluated. This was done to identify possible anisotropy effects on the properties of conventionally produced steels. Thus, specimens were prepared with their axis parallel to the rolling direction (K110LL-M, K110LL-A) and orthogonal to the rolling direction (K110TT-M, K110TT-A) from the same starting bar. Specimens designated with “M”, machined from the inner core of the 106 mm bar and specimens “A” from “outside” were distinguished. Specimen preparation has been shown in detail in [Sect. 2.1.1.1](#).

3.3.4.1 Fatigue Data and S–N Curve

Figure 3.59 presents the S–N curves for the two test series K110LL and K110TT. Numerical data are presented in Table 3.12. The S–N data obviously revealed a strongly anisotropic gigacycle fatigue behavior of the steel. K110TT (▲) specimens showed significantly lower fatigue strength over the entire tested fatigue life range compared to K110LL specimens (●). It is noted that the somewhat higher

Table 3.12 Fatigue data of (a) K110TT (transverse samples) and (b) K110LL (longitudinal samples from same initial steel bar B2)

Internal fatigue sample number	Stress amplitude (MPa)	Cycle number to failure	Crack origin location	
<i>(a) Test series K110TT</i>				
Q15 L+R	600	1.00E+05	At/near-surface	
Q10 L+R	500	2.19E+06		
Q11 L+R	500	1.93E+06		
Q3 M	500	5.82E+06		
Q21 L+R	450	1.09E+07		
Q22 L+R	450	2.35E+07		
Q35 M	450	1.60E+07		
Q12 L+R	400	2.53E+08		
Q14 L+R	400	1.02E+08		
Q4 M	400	2.02E+08		
Q19 L+R	350	1.33E+08		
Q20 L+R	350	2.14E+08		
Q34 M	350	4.23E+08		
Q16 L+R	300	2.64E+09		
Q17 L+R	300	1.44E+09		
Q18 L+R	300	5.89E+08	Runout-specimen	
Q23 L+R	250	1.00E+10		
Q24 L+R	250	1.06E+10		
Q37 M	250	1.02E+10		
Internal fatigue sample number	Stress amplitude (MPa)	Cycle number to failure	Crack origin location	Crack origin location
<i>(b) Test series K110LL</i>				
L2-M	1.673	1.00E+06	700	At/near-surface
L10-A	1.673	2.41E+06	700	
L17-A	1.673	4.28E+06	700	
L13-A	1.444	1.00E+07	600	
L1-M	1.444	1.50E+07	600	
L9-A	1.444	3.00E+07	600	
L4-M	1.205	7.00E+07	500	
L11-A	1.205	1.74E+08	500	
L3-M	1.205	2.21E+08	500	
L5-M	0.986	1.00E+10	420	Runout-specimen
L12-A	0.966	1.01E+10	400	

compressive residual stresses—present at the surface of K110TT specimens—have to be considered in this respect: This means that theoretically, K110TT specimens with residual stresses similar to K110LL specimens are expected to show even lower fatigue strength than the experimental results obtained here. Crack initiation sites were closely-spaced primary carbides, referred to as carbide clusters, or, in a few cases, large primary carbides (Fig. 3.60). These defects were located at/near the surface. Interestingly, more near-surface crack origins were observed for

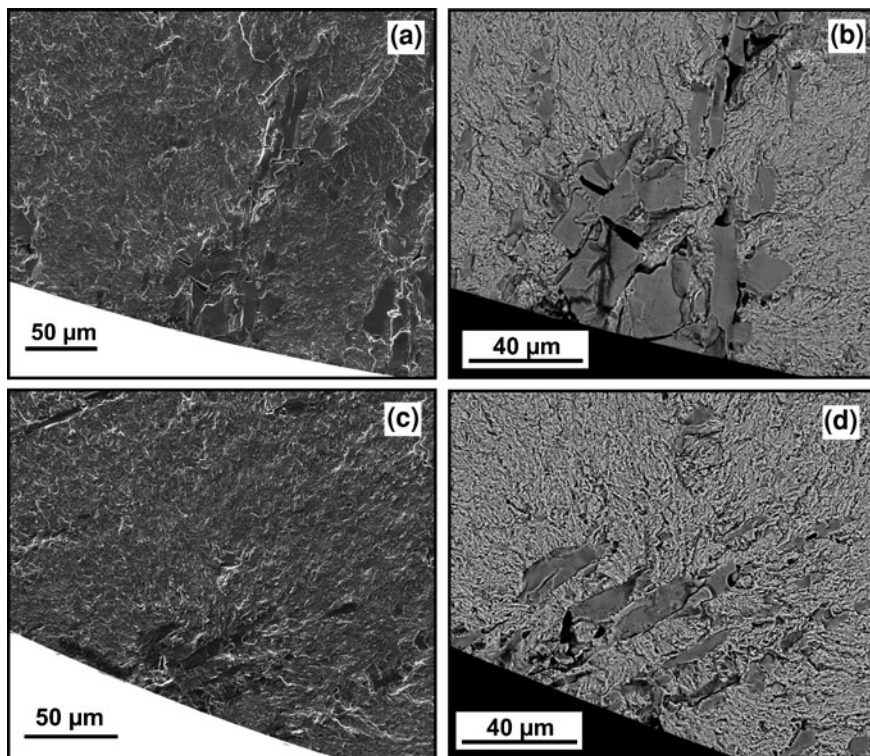


Fig. 3.60 Crack origins of K110TT specimens failed: **a, b** after 1.9×10^6 cycles at 500 MPa (carbide cluster touching the surface), and **c, d** after 1.3×10^8 cycles at 350 MPa (carbide cluster below the surface)

K110TT specimens. Carbides were always found to be cracked, i.e. initiation through cracking of larger carbides is dominant here compared to carbide-matrix decohesion. Some K110TT specimens failed due to surface cracks. The lower fatigue strength of K110TT specimens compared to the K110LL specimens can be attributed to the considerably higher frequency of occurrence of the large carbide clusters (80–100 μm diameter) in the longitudinal direction of the steel (bar B2)—which corresponds to the fracture area of K110TT samples (Table 3.3). The diameters of the carbide clusters that caused fatigue cracks in K110TT specimens ranged from 56 to 172 μm . In K110LL specimens, far smaller carbide clusters (10–48 μm in diameter) were found to be crack origins. This is reflected by the higher fatigue strength in the S–N data of the K110LL test series (Fig. 3.59, ●).

At this point it should be mentioned that no difference of the fatigue behavior was obtained between samples from the inner core (samples—“M”) and from the “outer part” (samples—“A”) of the bar B2. This means that the distribution of primary carbides seemed to be very homogeneous within the steel bar. Furthermore, the degree of deformation did not have an impact on the fatigue behavior of

the studied AISI D2 type tool steel either, at least with specimens oriented parallel to the rolling direction. Specimens of series K110LL (bar B2—larger initial bar diameter) failed within the same cycle number ranges as specimens of the more heavily deformed series K110L (bar B1), of which results have been presented in the preceding chapter. This finding was in agreement with results obtained for a spring steel by Furuya et al. [32], who claimed that differences of fatigue strength were small for specimens prepared from bars prepared with different degree of deformation, which was attributed to the fact that effective inclusion sizes were similarly small in both cases. This holds also for the steel studied here: The maximum carbide cluster size observed in transverse metallographic section (which corresponds to the fracture area of specimens K110L, and LL) of bar B2 was larger than in bar B1 (see Table 3.3). However, the relative frequency of these large species was rather low. Hence, the fatigue behavior of the two test series K110L and LL was similar. Some internal fish-eye type crack initiation was observed for K110L specimens in the cycle number range 10^5 – 10^7 , which is in agreement with the statistical considerations discussed in the preceding chapter. However, such internal failures were not obtained for K110TT samples, very likely due to the high frequency of occurrence of large carbides (clusters) which renders the existence of such a large species in the surface layer—where the highest stress concentration occurs [23]—much more probable.

3.3.4.2 Crack Origins and Fractography

Figure 3.61 presents representative fractographs of the two test series K110LL (Fig. 3.61a, b) and K110TT (Fig. 3.61c, d). Obviously, a strong difference of the macroscopic fracture surface between the two series was observed. While in K110LL fractographs numerous cracks, found at the major part of the fracture surface, point back to the fatigue crack origin, K110TT fractures looked completely different. Here, numerous cracks oriented parallel to the primary carbide bands were observed. It is noted here that K110L and K110LL specimens revealed very similar fracture surfaces, which is in accordance with the obtained S–N data that did not reveal any difference of fatigue lifetime (compare Fig. 3.59 to Fig. 3.44).

Around the crack origin a specific area was formed for all fractures of the three test series. This area appeared dark in SEM fractographs, indicating a rather flat surface morphology. The similarity of the appearance of the initial stages of the fatigue process for specimens of test series K110L, LL and TT and the fact that for both series K110LL and K110TT carbides (clusters) just touching the surface or being located just below the surface were nucleation point of fatigue cracks indicate that fatigue crack initiation and early propagation are quite similar for the two material orientations. In case of K110TT specimens, the crack initiating carbide clusters were far larger than those observed in K110LL samples, which directly corresponds to the larger carbide clusters sizes found in the longitudinal metallographic sections of bar B2 compared to the transverse direction

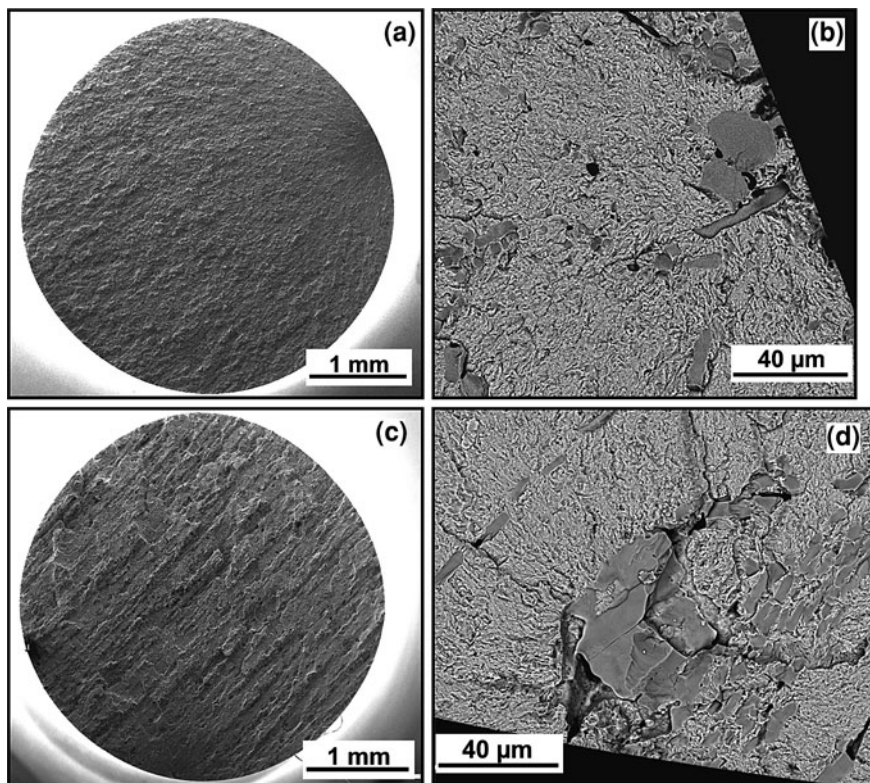


Fig. 3.61 Representative fractographs of specimens failed after **a, b** 1.50×10^7 cycles at 600 MPa (K110LL-1-M), and **c, d** after 1.60×10^7 cycles at 450 MPa (K110TT-Q35-M)

(Table 3.3—bar B2). Obviously, the larger carbides (clusters) are responsible for the significantly lower fatigue strength at a given sample life, as shown in the S–N data. This fact underlines the strong effect of the material anisotropy on the gigacycle fatigue behavior of this AISI D2 type tool steel.

An interesting observation was made with respect to the orientation of the primary carbide bands to the specimen surface at the crack origin. For about 60% of the failed K110TT specimens the carbide bands revealed an angle with the surface in the range between 20 and 50° (Fig. 3.60c, d). Only one specimen failed due to carbide bands parallel to the specimen surface (angle = 0°, see Fig. 3.62). About one-third of the failed specimen showed an angle between primary carbide bands and specimen surface of 60–90° (Figs. 3.60a, b and 3.61c, d). This observation is in strong contrast to K110LL specimens, for which all the fractures had more or less the same appearance.

Detailed investigations of the different zones on the obtained fracture surfaces of K110TT specimens were carried out in the same way as done for K110L-I and

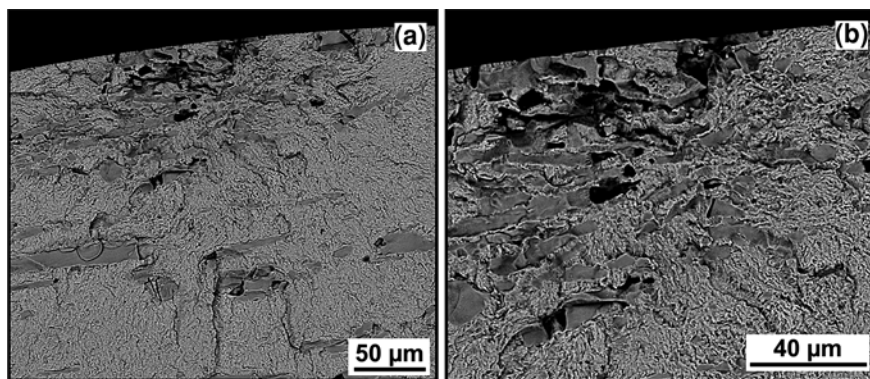


Fig. 3.62 Fracture surface of K110TT specimen failed after 2.6×10^9 cycles at 300 MPa; carbide bands oriented parallel to specimen surface

II specimens, shown above. K110LL and K110L fractographs showed similar appearance, which was described in Sect. 3.3.3 extensively.

Presently the focus is on the investigation of K110TT fracture surfaces. Figure 3.63 presents a typical zone around the crack origin of fractured K110TT specimens. Around the crack origins (carbide clusters) a granular area (GA) was observed, similar to that obtained for K110L specimens. This granular area (Fig. 3.63d) revealed a considerably roughened surface morphology compared to zone “2a” (Fig. 3.63c). It is speculated that the GA is formed according to the model introduced by Shiozawa et al. [27], described in the last chapter (see also Fig. 3.54). The process of microcrack formation and subsequent coalescence of these microcracks within the GA is rather slow. Shiozawa et al. [27] claimed that most of fatigue life is spent to form an appropriate size of GA which then triggers a short fatigue crack. The border of the granular area (GA) and the area “2a” probably marks the transition from a non-propagating to a propagating fatigue crack. More precisely, the short crack, formed through coalescence of numerous microcracks within the GA, reached the minimum length of a propagating crack. Before this crack length is attained, growth can only take place by microcrack coalescence, i.e. the crack does not “propagate” = become longer in the usual sense. The area “2a” (Fig. 3.63a), also observed for K110L and K110LL specimens and characterized by a rather flat surface morphology (Fig. 3.63c, see also Fig. 3.52), probably represents the second stage of the fatigue failure process, corresponding to the propagation of short fatigue cracks. At fracture surfaces of K110L specimens two more crack growth zones were observed, as was described in the last chapter. However, K110TT fractographs did not clearly exhibit such further stages of crack propagation. It seems that outside of area “2a”, a zone with higher surface roughness exists that passes into the final fracture area, where numerous cracks parallel to the primary carbide bands—possibly secondary cracks resulting from final failure—are visible.

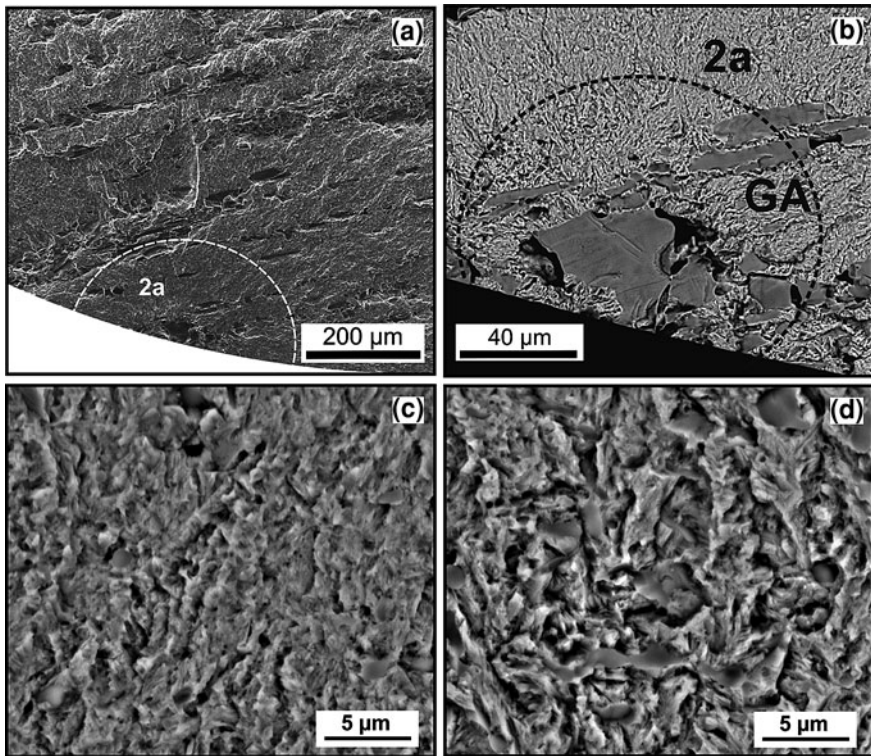


Fig. 3.63 Formation of granular area **a** and **b** subsequent area of short fatigue crack propagation (“2a”) in K110TT specimen failed after 2.4×10^7 cycles at 450 MPa; high magnification BSE image of surface morphology within **c** area “2a” and **d** the granular area (GA)

3.3.4.3 Quantitative Evaluation of K110TT Fracture Surfaces

The diameters of the carbide clusters, the granular area (GA) and of stage 2a were evaluated in order to obtain information about the fatigue process. However, it should be considered that exact determination of the size of area “2a” was possible only with difficulties, since the transition from stage “2a” to the subsequent fatigue stage was rather gradual. Numerical results are given in Table 3.13.

Figure 3.64a clearly shows that the lower the applied stress amplitude is, the larger is the observed size of the GA and stage 2a, respectively. This means the lower the applied stress amplitude is, the larger is the required GA size to trigger a short propagating crack. This observation supports the Shiozawa [27] model. The sizes of carbide clusters in the GA center did not reveal such a relationship, neither to the stress amplitude nor to the cycle number to failure. Furthermore, the GA formation can be described numerically by the difference of the granular area size minus carbide cluster size, which is plotted against cycle number to failure in Fig. 3.64b. There exists a strong correlation with the specimen life time, i.e. the

Table 3.13 Numerical results of quantitative evaluation of the crack nucleating and growth features observed on the fractographs of failed K110TT specimens

Steel K110			Test series K110TT		
Internal sample number	Cycle number to failure	Stress amplitude (Mpa)	Radii of		Angle between carbide bands to specimen surface at crack origin
			Carbide cluster (μm)	GBA (μm)	
Q10 L+R	2.19E+06	500	58	58	30
Q11 L+R	1.93E+06	500	61	50	90
Q21 L+R	1.09E+07	450	86	84	20
Q22 L+R	2.35E+07	450	68	88	40
Q35 M	1.60E+07	450	39	73	60
Q12 L+R	2.53E+08	400	40	53	90
Q14 L+R	1.02E+08	400	37	107	40
Q19 L+R	1.33E+08	350	46	92	45
Q20 L+R	2.14E+08	350	28	90	60
Q34 M	4.23E+08	350	33	102	80
Q16 L+R	2.64E+09	300		130	0
Q17 L+R	1.44E+09	300	64	100	85
Q18 L+R	5.89E+08	300	41	124	50

effective N to fracture is proportional to the distance GA has to grow to form a propagating crack. Longer fatigue life means larger granular area and larger difference of (GA minus carbide cluster size). Neither carbide cluster sizes nor sizes of the area “2a” showed any relationship to the fatigue life.

At cycle numbers below 10^7 —at high stress amplitudes—the observed GA size and carbide cluster size was rather equal, thus, the difference GA size minus carbide cluster size was zero. I.e. in this case the GA area is found only between the individual carbides forming the cluster but not around the cluster. Considering the fact that the GA size represents the critical crack length for a propagating short fatigue crack, this means that if this crack size can be reached by the size of the carbide cluster itself, no formation of GA is required. This seemed to be the case for the higher stress amplitudes. However, at lower stress amplitudes the carbide clusters by themselves are not large enough to nucleate a short fatigue crack. Thus, the microcrack formation and coalescence occur until appropriate GA size is reached according to the Shiozawa [27] model described above. It is speculated that the formation of the granular area takes a rather high number of loading cycles during the fatigue process, thus being decisive for fatigue life.

Interesting results were obtained combining the two datasets of K110TT and K110L specimens, for carbide clusters sizes and GA sizes, respectively. Figure 3.64c presents the observed relationship with the applied stress amplitude. Instead of showing one single correlation with the applied stress amplitude, the radii of carbide clusters rather revealed two data sets—one for transversal samples (Fig. 3.64c; K110TT(▲)) and one for the longitudinal samples (Fig. 3.64c, K110L(Δ)). Larger clusters (▲) were found in crack origins of K110TT specimens,

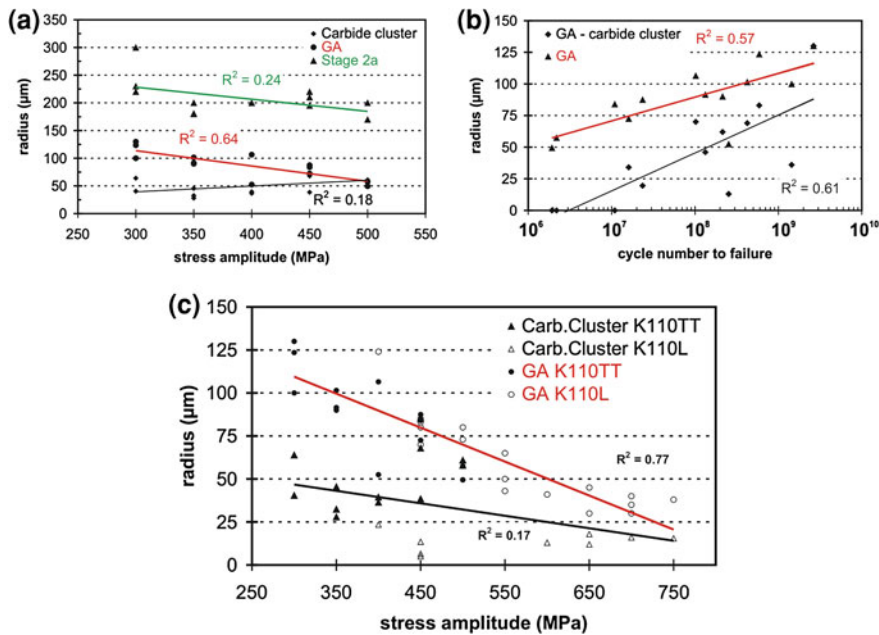


Fig. 3.64 Relationship of different zones of the fatigue process with: **a** the applied stress amplitude (K110TT specimens), **b** the cycle number to failure (K110TT), and **c** the applied stress amplitude for combined data set of K110TT and K110L specimens

as described above. In contrast, radii of carbide clusters observed in origins of K110L fractures (Δ) were not larger than $25\ \mu\text{m}$. This observation clearly underlines the material anisotropy, and corresponds also to the results of the metallographic investigations. In contrast to the carbide cluster size, the combined data of GA size of fractographs of K110TT and K110L specimens (Fig. 3.64c; $\bullet$, $\circ$) could be correlated linearly to the applied stress amplitude: The lower the applied stress amplitude was, the larger was the observed granular area, which corroborates the notion that the size of the GA represents the critical crack length for a propagating short fatigue crack. Interestingly, for K110L and LL specimens fractures did not occur below $400\ \text{MPa}$, which is attributable to the smaller carbide cluster dimensions into that direction, as discussed above. Considering the data of Fig. 3.64c, it seems that the carbide clusters in K110L were not large enough to form appropriately sized GA at stresses below $400\ \text{MPa}$ or that the GA formation might take longer than 10^{10} cycles to attain the critical GA size. This further supports the hypothesis that most of fatigue life to failure is spent for the GA formation and that *a real fatigue limit does not exist* for this steel. In contrast, carbide clusters in longitudinal direction of the rolled steel (K110TT specimens) were large enough to form a granular area also below $400\ \text{MPa}$, down to stress levels of $300\ \text{MPa}$. Thus, K110TT samples failed at these lower stress amplitudes. This further underlines the anisotropic fatigue behavior of the studied AISI D2 type tool steel.

Summarizing, all the above mentioned observations support the significance of the GA formation for the fatigue process at low stress amplitudes in the gigacycle fatigue regime.

Using the above presented value $\Delta K_{\text{carbide clusters}}$ (2.6 MPa $\sqrt{\text{m}}$) for carbide clusters, and the equation for surface cracks:

$$\boxed{\Delta K = 1.2584\sigma_a\sqrt{c}} \quad (\text{see Eq. 3.14})$$

where c represents half of the crack length—which is assumed to be equal to the radius of the defect (here: carbide cluster)—provides an estimation for probable fatigue endurance strength at 10^{10} cycles of the material. Using the radius of the largest primary carbide cluster (50 μm) observed in metallographic section (Table 3.3, bar B, longitudinal direction) give an estimate of the fatigue endurance strength of 290 MPa. If the largest cluster observed in fatigue failure origin of K110TT samples (90 μm) is used for the calculation, the fatigue endurance strength turned out to be 220 MPa. Both numerical results turned out to be reasonably good estimates for the fatigue endurance strength close to the experimentally obtained fatigue endurance strength at 10^{10} loading cycles which was about 250 MPa.

Summarizing the fatigue investigation of heat treated AISI D2 type tool steel, a considerable influence of surface residual stresses and material anisotropy was detected. It has been shown that primary carbides and carbide clusters located in the interior and at/just below the surface were origins of fatigue cracks. Singularities, especially nonmetallic inclusions did not play a role in fatigue failure initiation up to the gigacycle regime, highly probable due to the fact that these inclusions far smaller than the primary carbides in this chromium cold work tool steel. A granular area was observed around the crack initiating carbides, followed by a rather smooth zone. The granular area seems to play an important role in the fatigue process, which can probably be described by a model called “dispersive decohesion of spherical carbides” suggested by Shiozawa et al. [27]. A real fatigue limit was not attained in testing up to 10^{10} loading cycles.

3.4 Fatigue Behavior of Ingot Metallurgy High Speed Tool Steels (Böhler Steel S500 and S600)

3.4.1 Characterization of Mo-Based High Speed Steel (Böhler S500/M42/HS 2-10-1-8)

3.4.1.1 Metallography, X-ray Diffraction and Electron Probe Microanalysis

The as-received, annealed microstructure of steel S500 revealed uniform ferrite (Fig. 3.65), thus showing that forging, rolling, and subsequent soft annealing have

Fig. 3.65 Optical micrograph of as-received (annealed) microstructure of steel S500 (Nital etched)

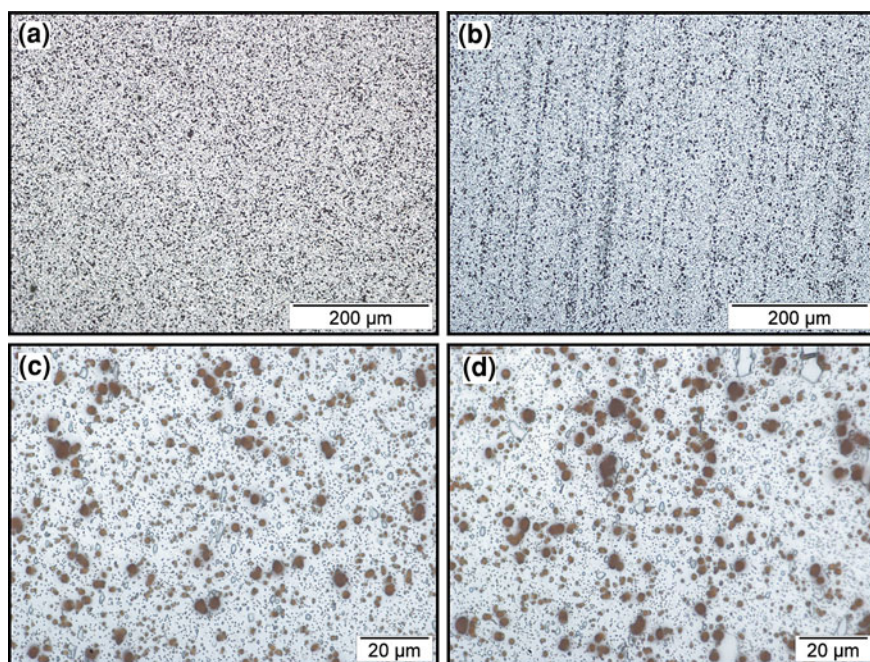
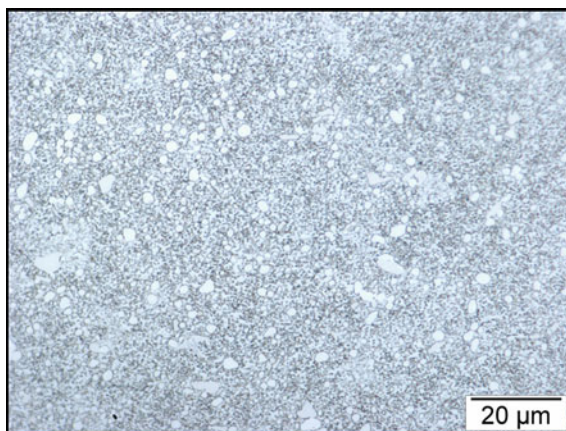


Fig. 3.66 Optical micrographs of as-received (annealed) microstructure of steel S500 (etched by Murakami reagent): **a, c** transverse direction, **b, d** longitudinal section, at two magnifications of 200 $\times$ and 1000 $\times$, respectively

been performed appropriately. Homogeneous carbide distribution was found in the transverse section (Fig. 3.66a), with primary carbides much smaller compared to those observed in cold work tool steel K110. Typical parallel alignment of the primary carbides was observed in longitudinal direction (Fig. 3.66b) resulting

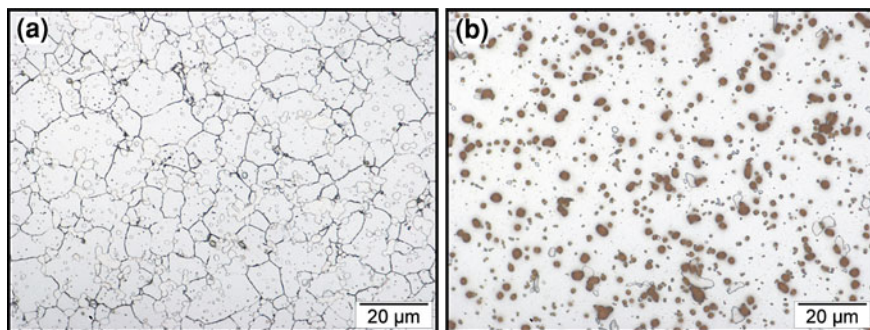


Fig. 3.67 Optical micrographs at 1000 $\times$ of S500 as-quenched microstructure: **a** Nitral etched and **b** Murakami etched

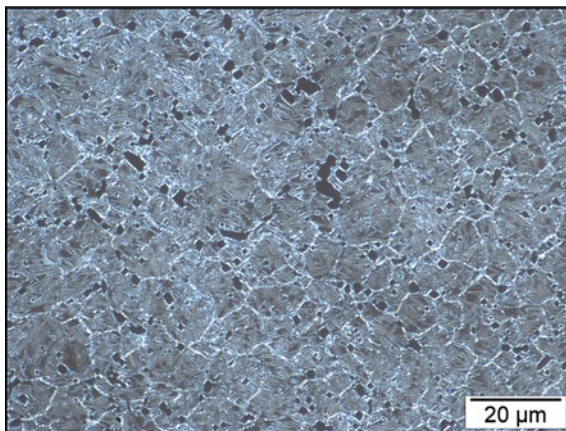
from casting and the rolling operation during the steel bar production process. The Murakami reagent colors the primary carbides of type M_6C , which is a multicomponent carbide. It contains between 40 and 70% iron, and the other main elements are Mo and W, but also V, which can be combined up to 60% in the M_6C carbide [7]. However, the etching does not clearly reveal the borders of the carbide particles as it does for M_7C_3 chromium carbides since for M_6C carbides Murakami etching results in a deposition effect, blurring the boundaries. Thus, the M_6C carbides tend to appear larger than they are. The M_6C type carbides have similar hardness as the Cr-rich M_7C_3 type carbides, described for steel K110, which was 1500 HV. The rather small carbide particles that were not colored by Murakami's reagent (Fig. 3.66c, d) represent MC type carbides, which are vanadium-rich species. These carbides can dissolve only limited amounts of iron (2–6%) and provide a high hardness of 2800–3000 HV [7].

The very small finely-dispersed carbides (Fig. 3.66c, d) are chromium carbides of type $M_{23}C_6$, which consist of 20–60% Cr and 40–70%Fe and some Mo or W [7]. These carbides can only be observed after annealing. The volume fraction in annealed AISI M42 steel is about 8% [33]. During austenitizing these Cr-rich carbides are completely dissolved (see Fig. 3.67b, providing most of the carbon content necessary for the matrix in hardening high speed steels [33]. Indeed, compared to the annealed condition (Fig. 3.66c, d), fewer very small carbides were observed in the as-quenched material (Fig. 3.67b).

Cobalt, about 8% are contained in steel S500, is predominantly dissolved in the matrix. It has an important effect on the transformation behavior during hardening: Co-containing steels show significantly faster austenite–bainite, martensite, or perlite transformation than Co-free high speed steels [34].

As described in detail in the experimental chapter (Chap. 2), the fatigue specimens were hardened by quenching from 1,190 °C in oil. Figure 3.67 shows the microstructure of the as-quenched steel in transversal section. Prior austenite grain boundaries are visible (Fig. 3.67a). Undesired grain growth did not occur. The grain size was determined using the Snyder–Graff method. It turned out to be

Fig. 3.68 S500 as-quenched microstructure (Pikral etched—dark field optical micrograph at 1000 $\times$)



10 $\pm$ 1 μm for the applied austenitizing conditions. Figure 3.67b exhibits the primary carbides of type M_6C (Mo-rich, colored) and MC (V-rich, not colored). Fine martensitic needle structure was observed within the grains (Fig. 3.68) and the primary carbides were predominantly found at the prior austenite grain boundaries (carbides are the black phases in Fig. 3.68).

The tempered martensitic structure and parts of prior austenite grain boundaries are clearly visible in the as-heat treated (quenched and tempered) microstructure of steel S500 (Fig. 3.69). BSE investigations (Fig. 3.70) revealed the fine needle structure of tempered martensite, and, as mentioned before, primary carbides are located predominantly at the grain boundaries. Electron probe microanalysis of the matrix (Fig. 3.71a) revealed that some Mo, Cr, and more of Co was dissolved in the iron matrix of the S500 steel. The primary carbides contained mainly Mo, but also some W, V, and Cr (Fig. 3.71b). XRD measurements (Fig. 3.72) proved that retained austenite had been totally transformed by three times tempering.

The carbide distribution in as-heat treated steel S500 steel is shown in Fig. 3.73 in transversal (a, c, e) and longitudinal (b, d, f) section. In the transverse direction uniform carbide distribution was observed (Fig. 3.73a, c) whereas in longitudinal sections the typical vertical alignment of the primary carbides, forming carbide bands, was found (Fig. 3.73b, d), characteristic for cast and rolled tool steels, although the alignment was less pronounced than in K110, in particular the carbides themselves are equiaxed. Two types of carbides can be identified in the metallographic sections at high magnification (optical micrographs at 1000 $\times$ —Fig. 3.73e, f) due to different etching behavior. M_6C carbides are colored orange by Murakami etching, as described above, whereas MC carbides remain white. However, the MC boundaries are attacked by the etchant, so that the MC carbides can clearly be distinguished. Compared to the as-received, annealed steel, fewer finely dispersed small carbides exist in the as-heat treated condition due to (1) the dissolution of the chromium M_{23}C_6 carbides during austenitizing and (2) the re-dissolution of cementite precipitated during the first tempering stage (room temperature up to about 270 $^{\circ}\text{C}$) during the second stage of tempering

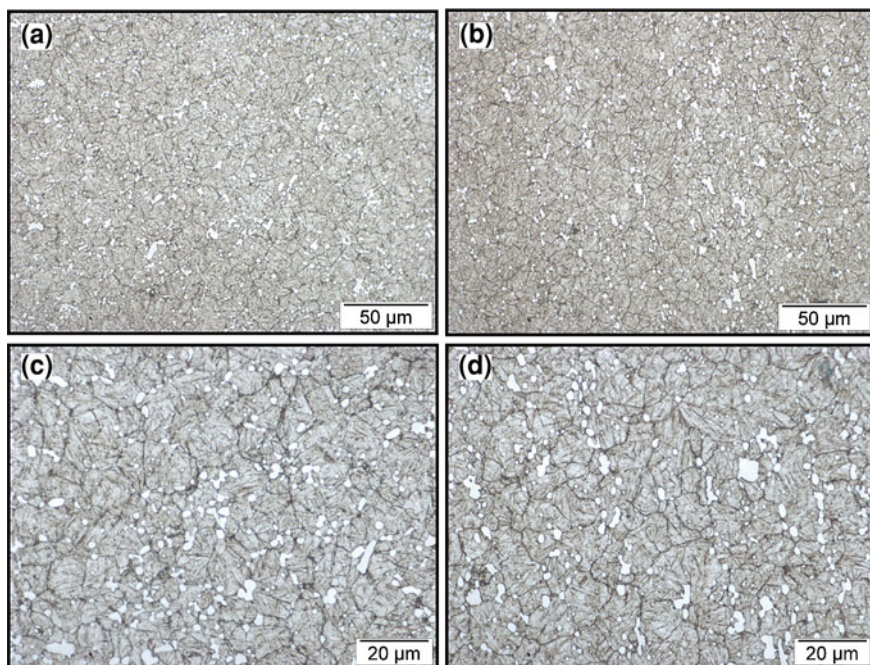


Fig. 3.69 Optical micrographs of quenched and 3× tempered microstructure of steel S500 (Nital etched): **a, c** transverse direction, and **b, d** longitudinal section at magnifications of 500× and 1000×, respectively

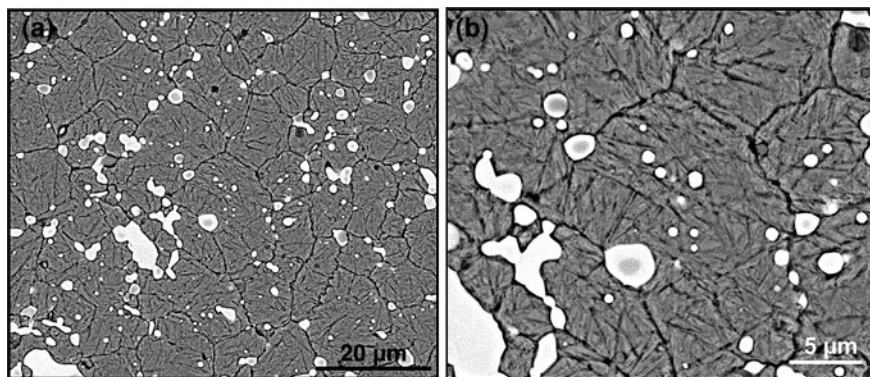


Fig. 3.70 Transverse microstructure of steel S500 after heat treatment, i.e. quenched and 3× tempered (BSE images, Nital etched)

(400–570 °C). The occurrence of the two different types of carbides, indicated by the different coloration, was confirmed by EDX analysis: M_6C type carbides (Fig. 3.71b) contain predominantly Mo. Some W, V, Cr, and significant amount of

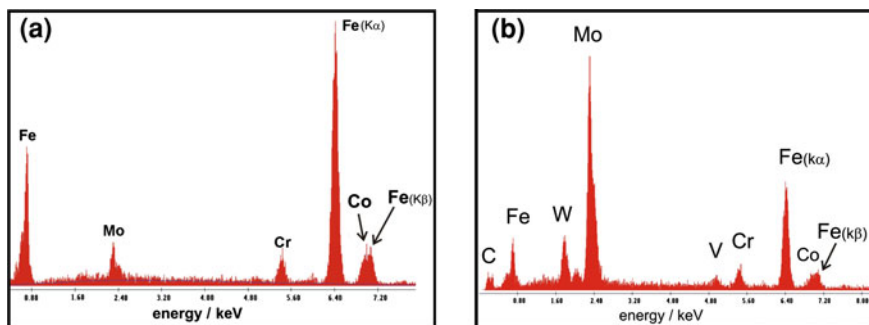


Fig. 3.71 Chemical composition (EDX spectra) of **a** the matrix, and **b** of the M_6C type primary carbides (Mo-rich) in as-heat treated (quenched and $3\times$ tempered) steel S500

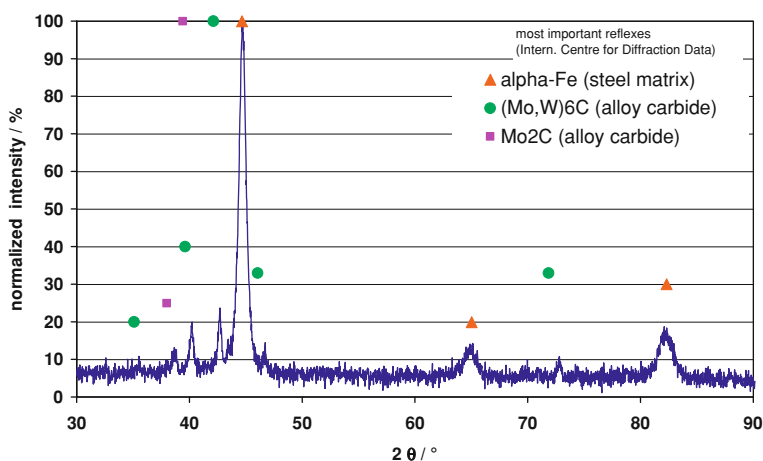


Fig. 3.72 XRD pattern of quenched and $3\times$ tempered S500 tool steel

Fe is also dissolved in this carbide type. MC type carbides, which were not colored by Murakami etching, definitely revealed high V content. Furthermore, XRD (Fig. 3.72) at least proved the existence of the M_6C carbides. The content of MC carbides was too low here to be detected by XRD.

Furthermore, XRD indicated small amounts of Mo_2C carbides. These carbides come into existence during tempering at temperatures $>500^\circ C$, i.e. the precipitation in form of very fine (3–10 nm sized) M_2C carbides from the martensite and residual austenite [7]. This process is responsible for the secondary hardening effect, especially of Mo-based high speed steels, due to the very fine, homogeneously dispersed M_2C carbides with a high hardness of 1800 HV [4].

Beside the carbides, very few nonmetallic inclusions (Fig. 3.74a) were detected in the metallographic sections of steel S500 containing predominantly Ca, Mg, Al and Si—probably as oxides or sulfides (Fig. 3.74b). These cannot be distinguished

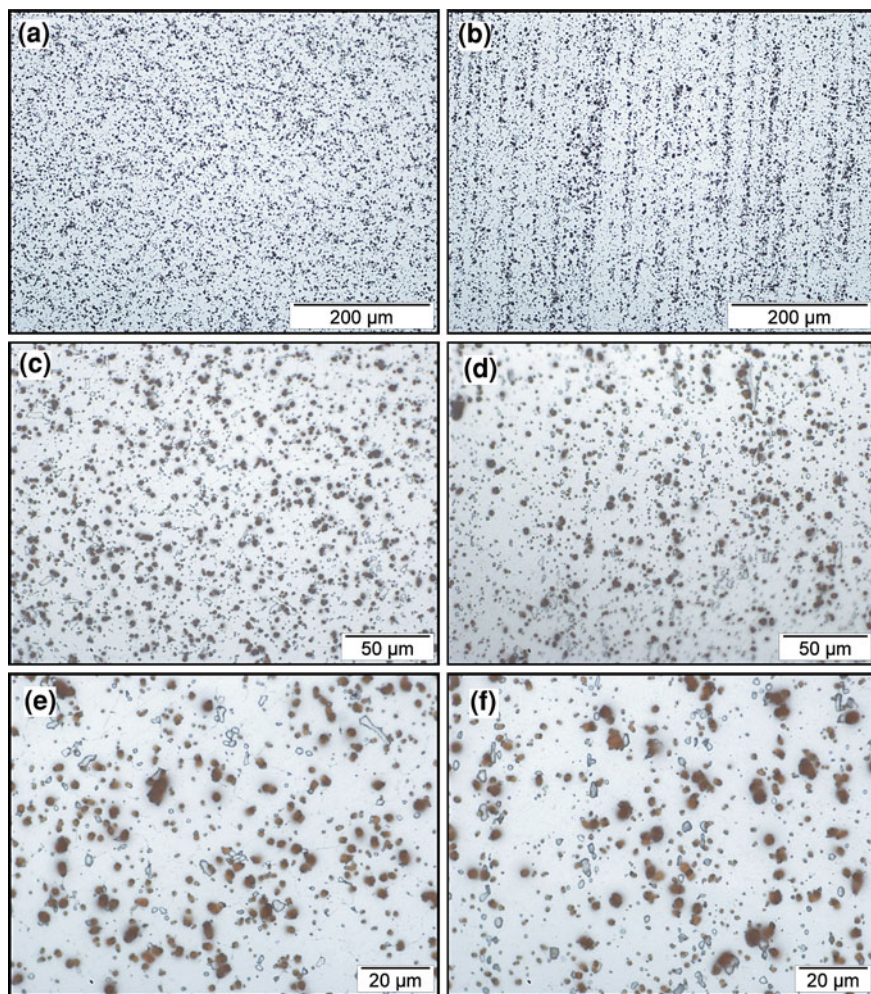


Fig. 3.73 Carbide distribution in as-heat treated steel S500 (optical micrographs, etched by Murakami reagent): **a, c, e** transverse section, and **b, d, f** longitudinal section at magnifications of 200 $\times$, 500 $\times$, and 1000 $\times$, respectively

by EDX due to the interference between Mo L_{α} and S K_{α} . However, the presence of oxides is more probable here.

Quantitative evaluation of the primary carbide size in as-heat treated steel was performed using light optical micrographs at 1000 $\times$ magnification in transverse and longitudinal section, six parallel images each. The classification according to carbide Feret diameter is presented in Fig. 3.75. Obviously, the largest species observed had a diameter of 20 μm , which is rather small compared to the chromium carbides of steel K110. Only a few carbide clusters have been detected that exhibited a maximum size of 20 μm .

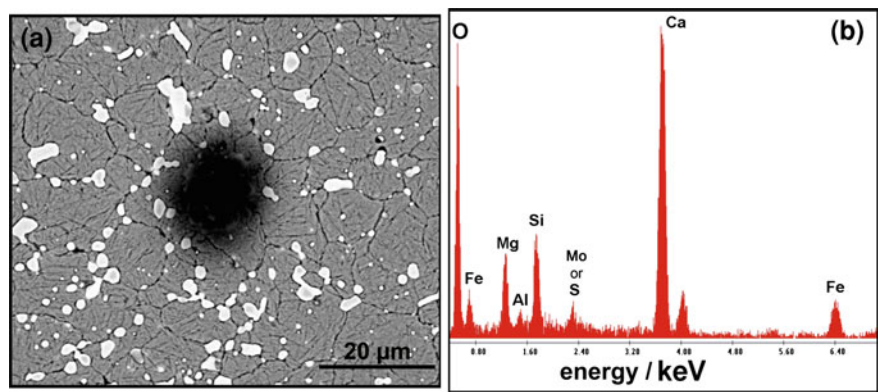


Fig. 3.74 Nonmetallic inclusion in metallographic section of S500 tool steel: **a** BSE image, **b** chemical composition (EDX spectrum)

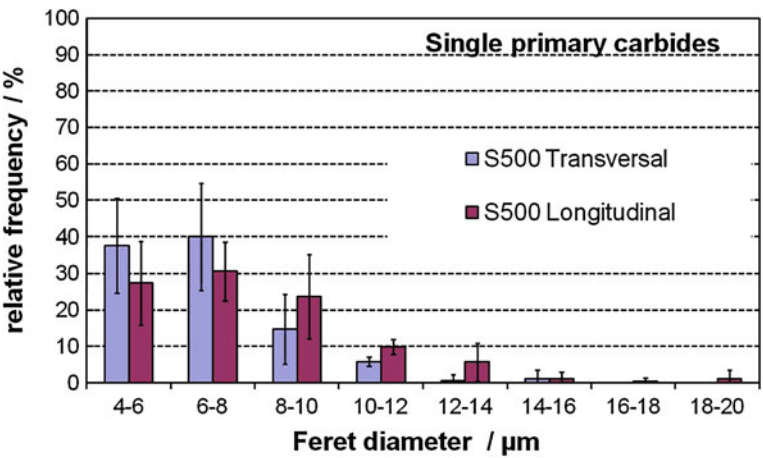


Fig. 3.75 Classification of primary carbides in steel S500 according to their size

Furthermore, the volume fraction of primary carbides in the steel was determined using image analyzing software applied on 18 optical micrographs at 500× and 1000× magnification (Fig. 3.76). Figure 3.76b reveals the corresponding image in which the carbide phases are colored green. The volume fraction of primary carbides turned out to be 7 ± 1 vol%.

3.4.1.2 Mechanical Properties of Böhler Steel S500

Steel hardness, Young's Modulus, and transverse rupture strength (TRS) have been determined (Table 3.14). In the as-received, annealed condition the steel

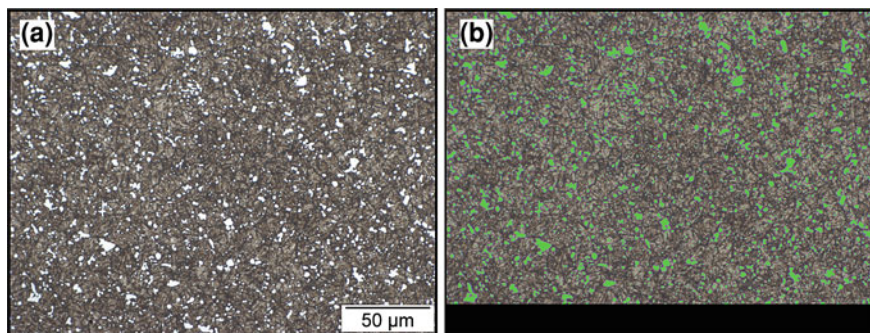


Fig. 3.76 Determination of carbide volume fraction in optical micrograph (500×-Nital etched)

Table 3.14 Mechanical properties of S500 steel

Steel	Rockwell hardness HRC 150 kg		Microhardness (HV 25 g)	T.R.S. (MPa)	Dynamic Young's modulus (GPa)
	As-quenched	Quenched and tempered	Quenched and tempered	Quenched and tempered	Quenched and tempered
“S500” AISI M42 HS 2-10-1-8 DIN Nr. 1.3247 (parallel to RD)	58 ± 2	66 ± 2	1037 ± 25	3400 ± 250	207 ± 7

S500 had a hardness of about 22 HRC, which was quite similar to the wrought cold work tool steels in the same condition. Increase of hardness from 58 HRC after hardening to about 66 HRC in as-tempered steel is a consequence of the secondary hardening effect in high speed steels and transformation of retained austenite. This precipitation hardening takes place in the second stage of tempering between 400 and 570 °C, during which precipitation of 3–10 nm-size alloy carbides (VC, Mo₂C, W₂C [7]) from the tempered martensite occurs. M₂C type carbides are metastable and transform into M₆C species at temperatures >900 °C.

Similarly, in the third stage of tempering (cooling from the tempering temperature), retained austenite decomposes accompanied by the precipitation of alloy carbides and martensite, which gives a significant rise of hardness at both room and elevated temperature.

Although exhibiting higher hardness, which has to be considered when comparing to the wrought cold work steel K110, the bending strength of S500 high speed steel was similar to that of K110, indicating that at the same strength level the HSS S500 offers significantly higher material hardness.

Representative fractographs obtained by TRS tests are presented in Fig. 3.77. Cracks emanated from the specimen surface (Fig. 3.77b, c).

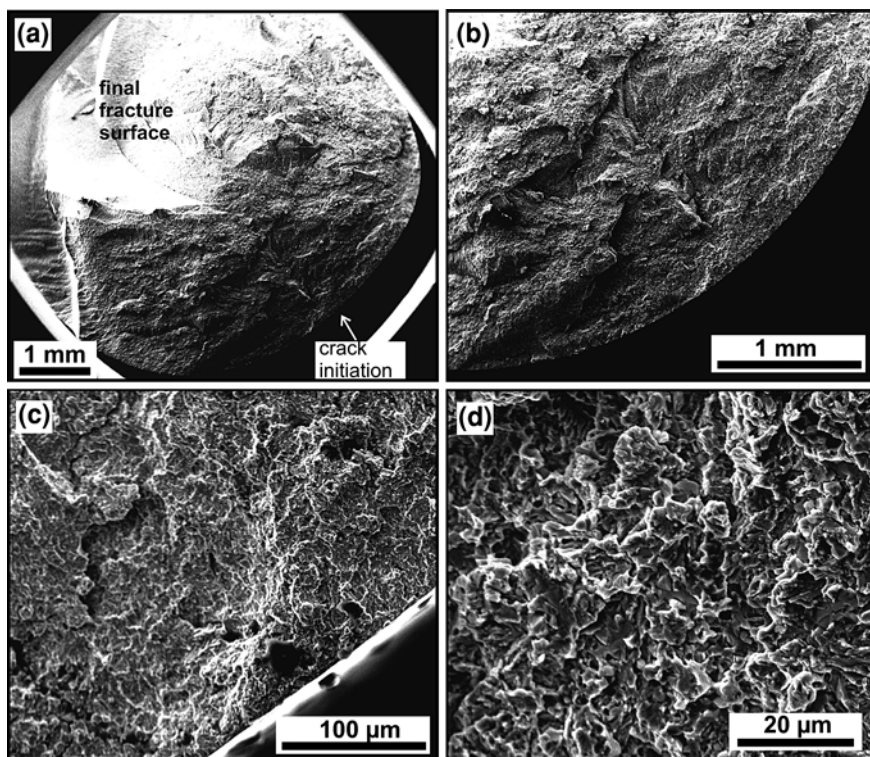


Fig. 3.77 Bending test fractographs of high speed steel S500: **a** overview, **b** area of crack initiation, **c** crack initiation site, **d** area of slow crack propagation near the crack origin

3.4.2 Characterization of Mo-W-Based High Speed Steel (Böhler S600/M2/HS 6-5-2)

3.4.2.1 Metallography, X-ray Diffraction and Electron Probe Microanalysis

The as-received annealed microstructure was uniformly ferritic in the matrix (Fig. 3.78c, d). Beside the numerous primary carbides, several nonmetallic inclusions have been detected. The primary carbides revealed homogeneous distribution in the transverse section (Fig. 3.78a), whereas in the longitudinal section (Fig. 3.78b) typical parallel alignment of primary carbides—forming carbide bands—was observed, resulting from casting and rolling of the steel ingot. The Murakami reagent attacked the primary carbides of type M_6C , which also here is a multicomponent carbide containing Mo, W and Fe, as described above for steel S500. The carbide particles showed similar size as in steel S500. The small finely-dispersed carbides are likely chromium carbides of type $M_{23}C_6$, which can only be

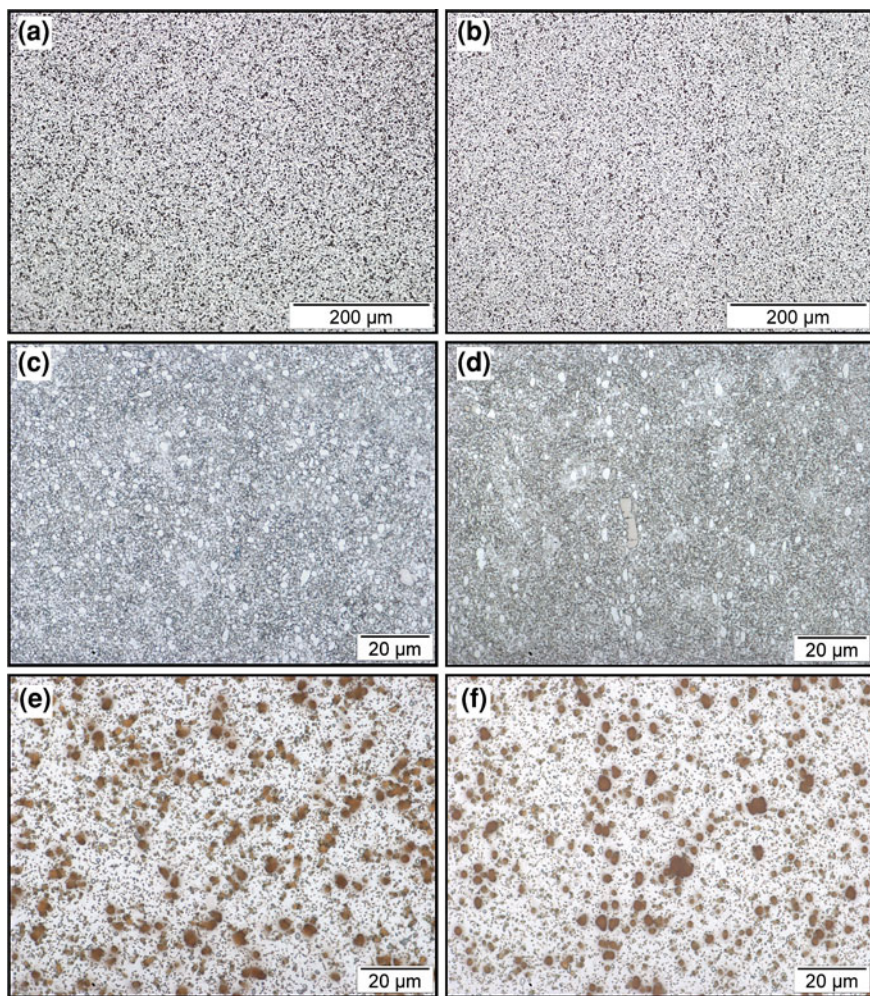


Fig. 3.78 Light optical micrographs of annealed microstructure of steel S600: **a–c** transverse **d–f** longitudinal section; **c, d** etched with Nital, **a, b, e, f** etched with Murakami reagent

observed in annealed steel, as discussed for steel S500. Some carbides of type MC (V-rich) were observed in the annealed condition, hardly visible in the micrographs. Kulmburg reported not more than 2% MC carbides in annealed AISI M2 high speed steel [4]. The fatigue specimens were hardened from 1,200 °C in oil. In the as-quenched microstructure, primary carbides (white particles in Fig. 3.79a) are located predominantly at the prior austenite grain boundaries. Undesired grain growth did not occur. The grain size, evaluated using the Snyder–Graff method, turned out to be $11 \pm 1 \mu\text{m}$ for the austenitizing procedure applied here. Figure 3.79b (Murakami etching) exhibits the primary carbides of type M_6C

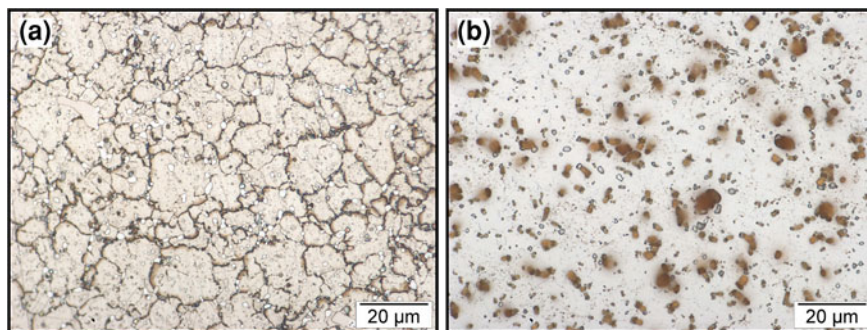
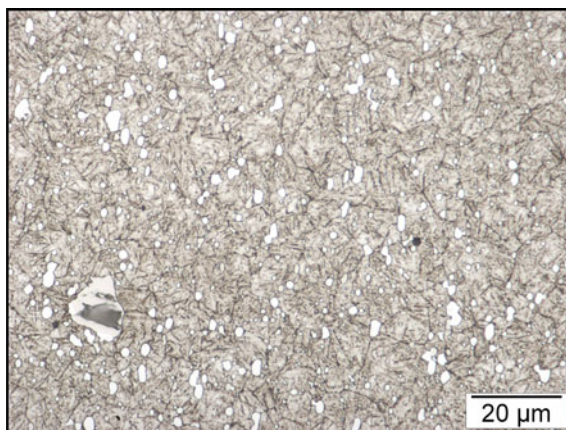


Fig. 3.79 As-quenched microstructure of S600 in the transverse section at 1000 $\times$ magnification: **a** Nital etched, **b** Murakami etched

Fig. 3.80 As-tempered microstructure in longitudinal section of steel S600 (etched with diluted Adler reagent)



(Mo-rich, colored) and some MC type carbides (V-rich, not colored). Fewer very small carbides were observed in the as-quenched steel S600 compared to the annealed condition, since the $M_{23}C_6$ Cr-rich carbides have been dissolved during austenitizing and remained dissolved upon quenching.

The as-tempered microstructure resembled that of tempered steel S500. Short etching with Adler etch (Fig. 3.80) revealed the tempered martensitic structure and parts of prior austenite grain boundaries. XRD proved that retained austenite had been transformed after three times tempering. Figure 3.81 shows the fine-structured martensite more clearly. Electron probe microanalysis (Fig. 3.82) revealed that the as-heat treated iron matrix contained some W, Mo, and Cr. The M_6C type carbide contained predominantly Mo and W, some Cr and V seemed also to be dissolved in this carbide type. The MC type carbides revealed high amounts of V.

The carbide distribution in as-heat treated steel in the transverse section (Fig. 3.83a) was very homogeneous. In the longitudinal section (Fig. 3.83b),

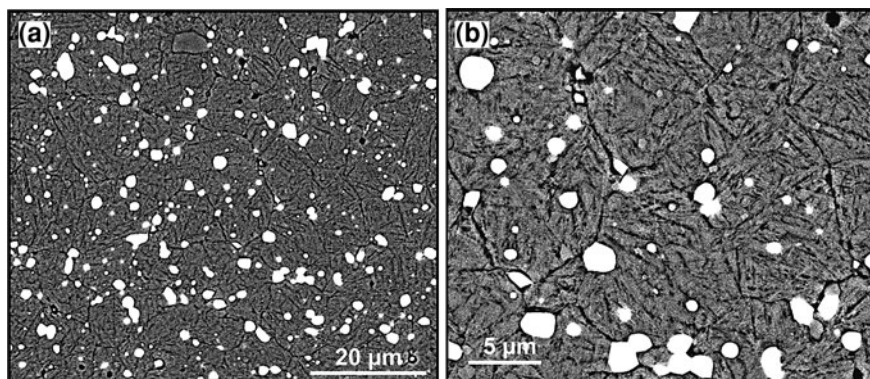


Fig. 3.81 Transverse as-tempered microstructure of steel S600 (BSE images, etched with diluted Adler reagent)

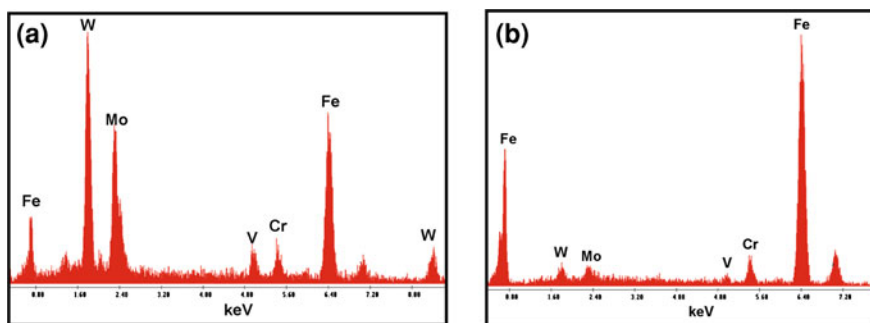


Fig. 3.82 Elemental analysis of **a** M_6C type primary carbides (Mo-W-rich) and **b** the matrix of as-heat treated steel S600

characteristic carbide bands were observed, which in this steel were somewhat more pronounced than in S500. Two types of carbides— M_6C and MC type—can be identified in the metallographic sections at high magnification (Fig. 3.83e, f) due to their different etching behavior, as already described. Only very few MC carbides were detected, most of which were smaller than $5\ \mu\text{m}$. XRD proved the existence of M_6C carbides (Fig. 3.84). In contrast, MC carbides were not detected by XRD due to the low content of these carbide species in the steel. In addition to the numerous primary carbides, a few nonmetallic inclusions were found in the metallographic sections of steel S600, similar to those observed in steel S500.

Quantitative evaluation of the primary carbide size in as-heat treated steel was performed using light optical micrographs at magnification of $1000\times$ in transverse and longitudinal section, six images each (etched with Nital or diluted Adler reagent; the effect was the same for both agents) The obtained classification

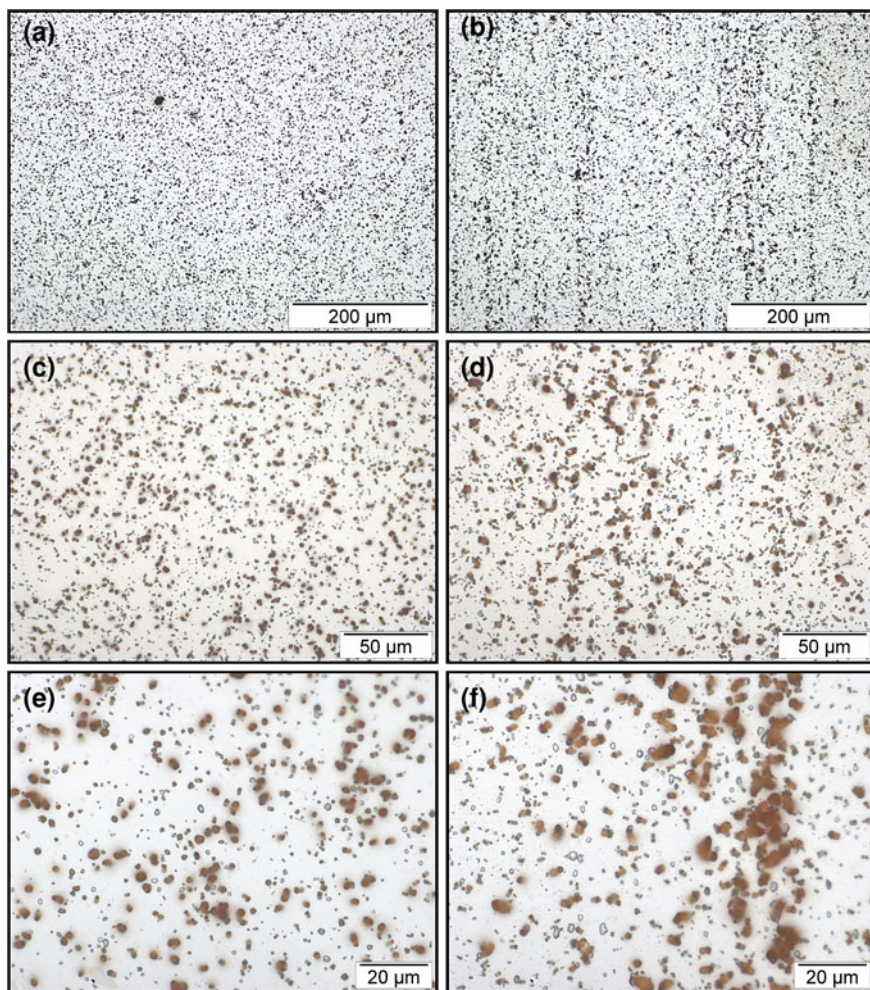


Fig. 3.83 Carbide distribution in steel S600 after heat treatment (quenched and $3\times$ tempered): **a, c, e** transverse sections, **b, d, f** longitudinal sections at $200\times$, $500\times$ and $1000\times$ magnification, respectively (etched with Murakami reagent)

according to carbide Feret diameter is presented in Fig. 3.85. Sizes of the primary carbides were similar in both sections. Furthermore, the volume fraction of primary carbides in the steel was determined using image analyzing software applied on ten optical micrographs at $500\times$ and $1000\times$ magnification (etched with Nital or diluted Adler reagent). The carbide volume fraction for steel S600 after the applied heat treatment, determined as shown for steel K110 and S500, turned out to be 8 ± 2 vol%, which agrees with the total carbide volume fraction of 9% reported by Kulmburg [4] for hardened AISI M2 type tool steel.

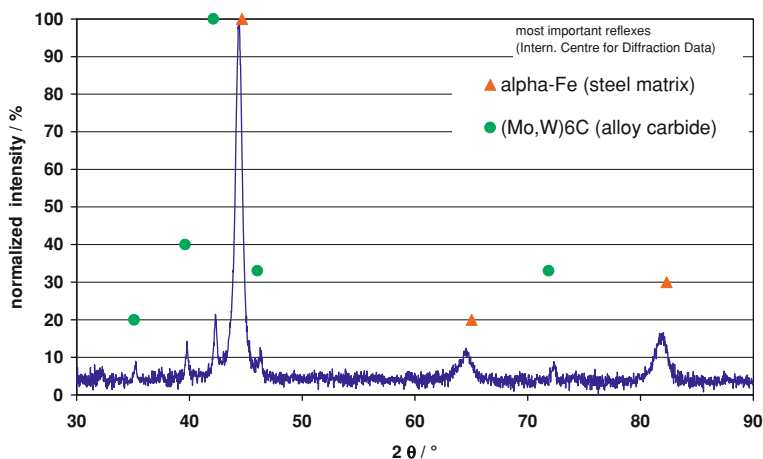


Fig. 3.84 XRD pattern of quenched and $3\times$ tempered steel S600

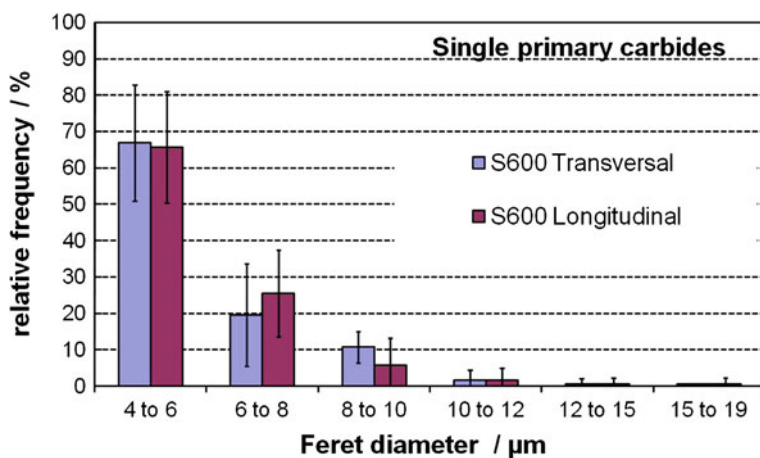


Fig. 3.85 Classification of primary carbides in steel S600 according to their size

3.4.2.2 Mechanical Properties of Böhler Steel S600

The mechanical properties are presented in Table 3.15. In the as-received, i.e. annealed, condition the steel S500 had a hardness of about 26 HRC. As-quenched hardness was about 6 HRC higher than for steel S500, which can be attributed to the very hard $(\text{Fe,W})_6\text{C}$ carbides that are not dissolved during austenitizing at 1,200 °C. A hardness increase was not observed during tempering. The TRS of S600 was similar to that of the other two wrought tool steels S500 and K110

Table 3.15 Mechanical properties of S600 steel

Steel	Rockwell hardness HRC 150 kg		Microhardness (HV 25 g)	T.R.S. (MPa)	Dynamic Young's modulus (GPa)
	As- quenched	Quenched and tempered	Quenched and tempered	Quenched and tempered	Quenched and tempered
“S600” AISI M2 HS 6-5-2 DIN Nr. 1.3554	64 ± 2	64 ± 2	965 ± 10	3600 ± 400	208 ± 6

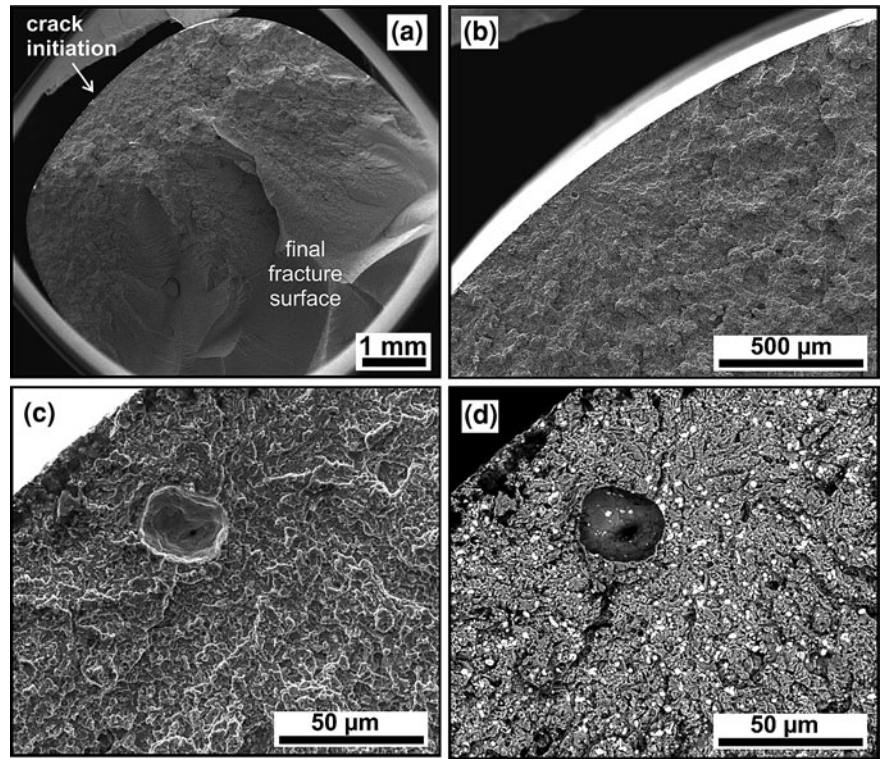


Fig. 3.86 Bending test fractographs (SE images) of high speed steel S600: **a** overview, **b** area of crack initiation, **c** crack nucleation site, **d** BSE image of crack nucleation site

studied here. SEM images of the TRS fracture surfaces are presented in Fig. 3.86. Here, the crack originated at a subsurface nonmetallic inclusion (Fig. 3.86c, d). Other specimens failed from the surface.

3.4.3 Fatigue Behavior of High Speed Steels S500 and S600

3.4.3.1 Fatigue Data and S–N Curves

Figure 3.87 shows the obtained S–N data for steel S500 with low residual stresses, tested in longitudinal direction (Table 3.2). Numerical results are presented in Table 3.16. The types of crack origin are specially marked, and in addition the graphs for 10, 50, and 90% fracture probability are also presented. The fatigue strength decreases by half from 850 MPa at 10^5 loading cycles (the minimum N reasonably attainable with ultrasonic testing) to 450 MPa at 10^{10} loading cycles, where three runout specimens ($\blacktriangle$, $N_{\max} = 10^{10}$ cycles) were obtained. In addition, at 500 MPa one further runout sample ($\blacktriangle$) was obtained, however, at this stress amplitude three other samples failed after about 10^8 cycles. Obviously, the observed scatter of the data was rather small. Two specimens used for preliminary samples had failed at surprisingly long lives at 900 MPa; these are assumed to have had significantly higher initial surface residual stresses compared to the rest of the specimens, since residual stress measurement at specimen ground and polished in a similar way as these two specimens revealed compressive stresses at the surface in the range of -400 MPa, which probably inhibited or delayed the crack initiation at the surface, as described in Sect. 3.3.3.4.

Five different types of crack origins were observed: At higher amplitudes, internal failures started at internal carbides ($\blacklozenge$) and, in some cases, at a nonmetallic inclusion ($\blacklozenge$) forming so-called fish-eye patterns at the fracture surfaces. Furthermore, two subsurface failures due to large nonmetallic inclusions (Δ) with diameters about 73 and 33 μm , respectively, were found, which confirms Furuya's statement that by gigacycle fatigue testing, nonmetallic inclusions are found that are hardly detectable in metallography. The major part of the failed specimens showed fatigue crack initiation at primary carbides and carbide clusters located at or close to the surface ($\bullet$). For a few samples ($\circ$) it was impossible to identify the crack origins; however, the fatigue cracks definitely started within the surface region.

Fatigue behavior of high speed steel S600 (longitudinally oriented, low residual stresses, see Table 3.2) was similar to steel S500, as can be clearly seen in Fig. 3.88. Numerical results are presented in Table 3.17. Fatigue tests of steel S600 were done for a few specimens at stress amplitudes of 700, 600, 500 and 450 MPa, two samples each, and sample failures occurred within the confidence band obtained for steel S500. Crack initiation was found to take place at primary carbides or carbide clusters located at/near the surface and at subsurface nonmetallic inclusions, as described above for steel S500. However, internal failures were not observed here, probably due to the limited number of specimens tested. Furthermore, two runouts were obtained at 450 MPa after 10^{10} cycles; thus, the fatigue strength at 10^{10} loading cycles was similar to that obtained for steel S500.

Comparison of the fatigue data of these two high speed steels S500 and S600 to the data obtained for AISI D2 type wrought cold work tool steel (Böhler grade K110), which has been presented in the previous chapter, showed surprisingly

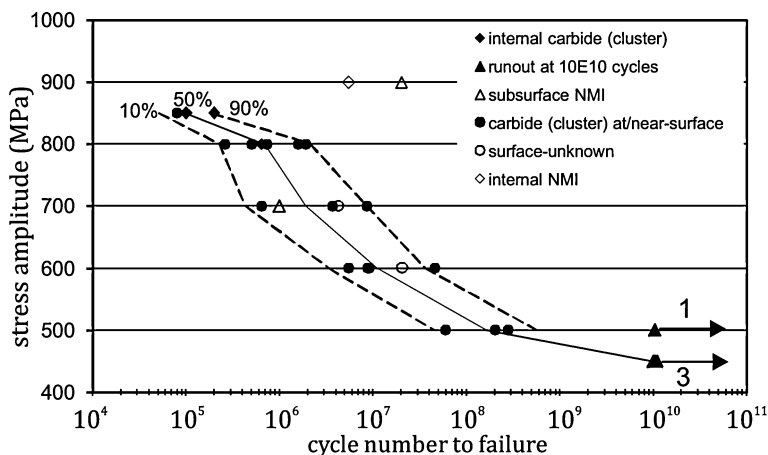


Fig. 3.87 S–N data of steel S500 (longitudinal direction, low residual stresses) determined jointly with Dipl.-Ing.A.Betzwar-Kotas (University of Vienna). The *solid* and the *two dashed lines* represent 50, 10 and 90% fracture probability, respectively. Figures close to the *arrows* indicate the number of runout specimens

that the gigacycle fatigue behavior of these steels (K110, S500 and S600) is similar (in case of low residual stresses at the specimen surface for all materials), despite the fact that the hardness of the high speed steels is about 8 HRC higher than that of the cold work steel. Also the microhardness of the two high speed steels was significantly higher than that of the cold work tool steel K110. However, it has to be considered that the TRS and Young's moduli are similar, too. Consequently, it seems that the elastic behavior and the strength of the matrix have a more pronounced influence on the fatigue behavior than simply the hardness, which is predominantly determined by the very hard primary W and Mo carbides. For both steel types, primary carbides and carbide clusters at/near the surface represent the most important group of fatigue crack origins, if only low residual stresses exist at the specimen surface as was the case here. If, as has already been shown in this thesis, high compressive surface residual stresses were present, the S–N curve for steel K110 was shifted significantly towards longer specimen lives, and specimens failed due to internal carbide clusters in the cycle number range of 10^5 to 10^7 . As described above, two (preliminary) specimens of steel S500 failed at 900 MPa at significantly longer lives, very likely due to high compressive stresses at the specimen surface, which is corroborated by the fact that K110L-I specimens (high compressive stresses) revealed similar fatigue live and showed also internally induced failure.

3.4.3.2 Crack Origins and Fractography

Macroscopically, the obtained fracture surfaces of steel S500 and S600 looked very much the same as those obtained for wrought cold work tool steel K110.

Table 3.16 Fatigue data of high speed steel S500 (longitudinal direction, low residual stresses at the surface)

Steel S500				
Internal fatigue sample number	Strain at gauge length (mm)	Stress amplitude (MPa)	Cycle number to failure	Crack origin location
S500-S4		900	5.45E+06	Internal nonmetallic inclusion
S500-S5		900	2.00E+07	Subsurface nonmetallic inclusion
S500-S32		850	8.00E+04	Surface carbide
S500-S11	4.14E−03	850	1.00E+05	Internal carbide cluster
S500-S16	4.09E−03	850	2.00E+05	Internal carbide
S500-S30		800	2.60E+05	Surface carbide
S500-S26		800	6.00E+05	Internal carbide
S500-S20	3.83E−03	800	7.30E+05	Surface carbide
S500-S21	3.83E−03	800	1.60E+06	
S500-S24		800	1.93E+06	
S500-S1		800	5.00E+05	
S500-S28		700	6.40E+05	
S500-S13	3.34E−03	700	1.00E+06	Subsurface nonmetallic inclusion
S500-S19	3.42E−03	700	3.70E+06	
S500-S25		700	4.27E+06	Surface-unknown
S500-S18	3.41E−03	700	8.60E+06	Surface carbide
S500-S6	2.90E−03	600	5.50E+06	
S500-S10	2.89E−03	600	8.80E+06	
S500-S9	2.90E−03	600	9.16E+06	
S500-S12	3.13E−03	600	2.07E+07	Surface-unknown
S500-S27		600	4.60E+07	Surface carbide
S500-S7	2.45E−03	500	2.07E+07	
S500-S22	2.40E−03	500	2.02E+08	
S500-S14	2.45E−03	500	2.82E+08	
S500-S29		500	5.22E+08	
S500-S8	2.44E−03	500	1.04E+10	Runout specimen
S500-S17	2.21E−03	450	1.00E+10	
S500-S31		450	1.01E+10	
S500-S15	2.17E−03	450	1.09E+10	

Around the crack origin a dark area was detected under the light microscope (Fig. 3.89). Ridges and cracks point back to the crack origin, as can be also seen in Fig. 3.90. Especially at high stress amplitudes, part of the fracture surfaces exhibited areas with high edges and ridges (Fig. 3.90a), probably deriving from a heavy overload during final fracture of the specimen. The area of this part of the final fracture surface decreased considerably with increasing sample life and totally disappeared at lower stress amplitudes (Fig. 3.90b).

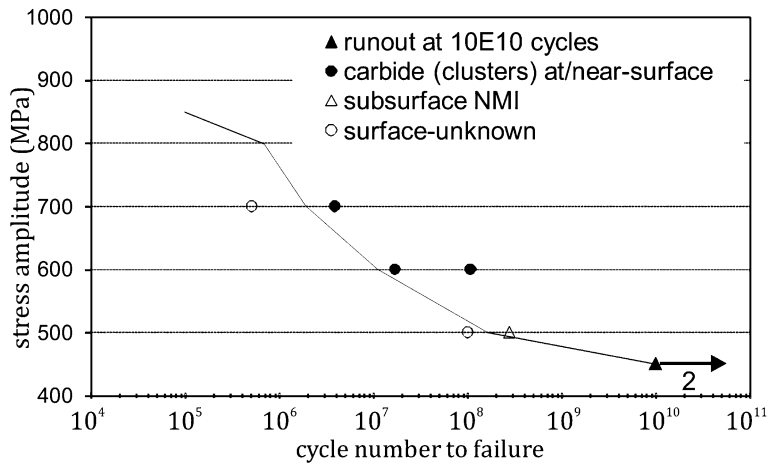


Fig. 3.88 S–N data of steel S600 (longitudinally oriented, low residual stresses at the surface) determined jointly with Dipl.-Ing.A.Betzwar-Kotas (University of Vienna). The solid line represent 50% fracture probability of steel S500 (figures close to the arrows indicate the number of runout specimens)

Table 3.17 Fatigue data of high speed steel S600

Steel S600				
Internal fatigue sample number	Strain at gauge length (mm)	Stress amplitude (MPa)	Cycle number to failure	Crack origin location
S600-S55	3.19E–03	700	5.11E+06	Surface-unknown
S600-S54	3.19E–03	700	3.89E+06	Surface
S600-S57	2.74E–03	600	1.70E+07	Carbide
S600-S56	2.76E–03	600	1.07E+08	(cluster)
S600-S52	2.32E–03	500	1.04E+08	Surface-unknown
S600-S53	2.32E–03	500	2.79E+08	Subsurface nonmetallic inclusion
S600-S58	2.13E–03	450	1.00E+10	Runout
S600-S59	2.11E–03	450	1.18E+10	Specimen

Fracture surfaces on the microscopic level revealed two zones within the (half) fish-eye pattern, depending on the type of crack origin and applied stress amplitude. In contrast, fracture surfaces of cold work tool steel K110 showed five different zones of crack growth in case of internal failure. Here, for the two high speed steels fish-eye- or half-fish-eye-like pattern (see Figs. 3.91 and 3.93) was formed around the internal and at/near-surface crack origin, respectively, of which however, exact definition of the borderline to final fracture surface was difficult since a rather gradual change of the structure was observed.

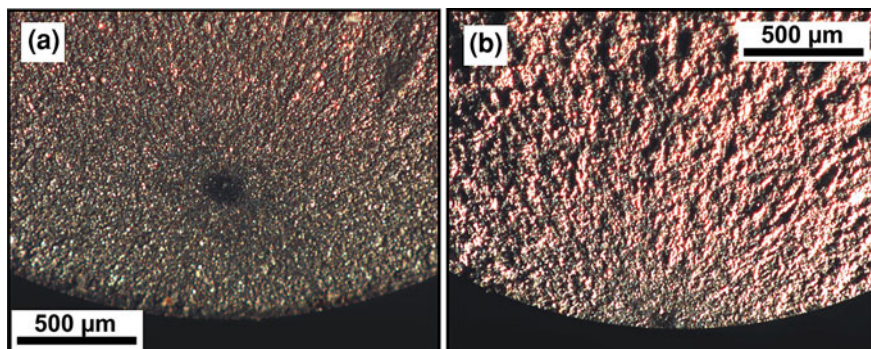


Fig. 3.89 Light optical fractographs of S500 specimens failed from **a** an internal nonmetallic inclusion (S500-S4) at 900 MPa after 5.45×10^6 cycles and **b** from a subsurface nonmetallic inclusion (S500-S5) at 900 MPa after 2.00×10^7 cycles

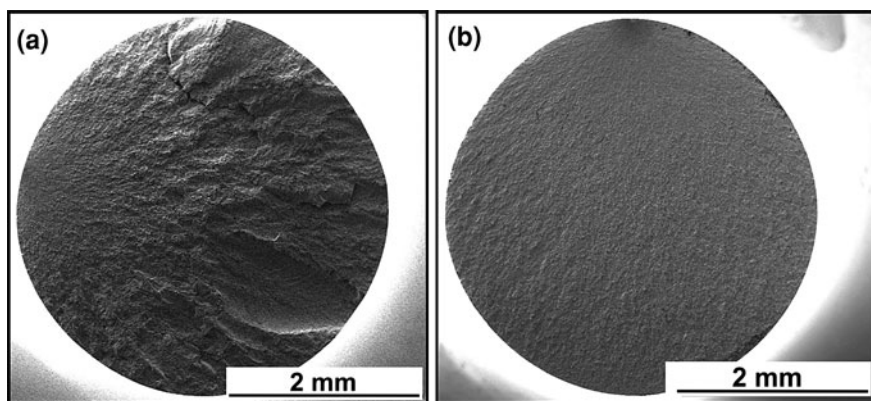


Fig. 3.90 Macroscopic fracture surface of **a** S500 specimen failed after 5.00×10^5 cycles at 800 MPa (S500-S1) and **b** S600 specimen failed after 3.89×10^6 cycles at 700 MPa

Four types of crack origins (CI) have been identified (except for a few specimens for which the determination of the crack initiation site was not possible). The four CI types can be categorized into internal and surface/subsurface failures. Figure 3.91 shows characteristic internal failures starting from a carbide cluster (Fig. 3.91a–c) and a nonmetallic inclusion (Fig. 3.91d–f). The primary carbides appear bright in the BSE image due to the high average atomic number. In contrast, the nonmetallic inclusions appear dark due to their lower average atomic mass. While the nonmetallic inclusion is rather isolated, the large primary carbide is surrounded by numerous somewhat smaller carbides closely arranged to each other. This constellation seems to be a prerequisite for internal crack formation from primary carbides, since this arrangement might offer highest stress concentration due to superposition of the stress fields of the individual carbides, probably

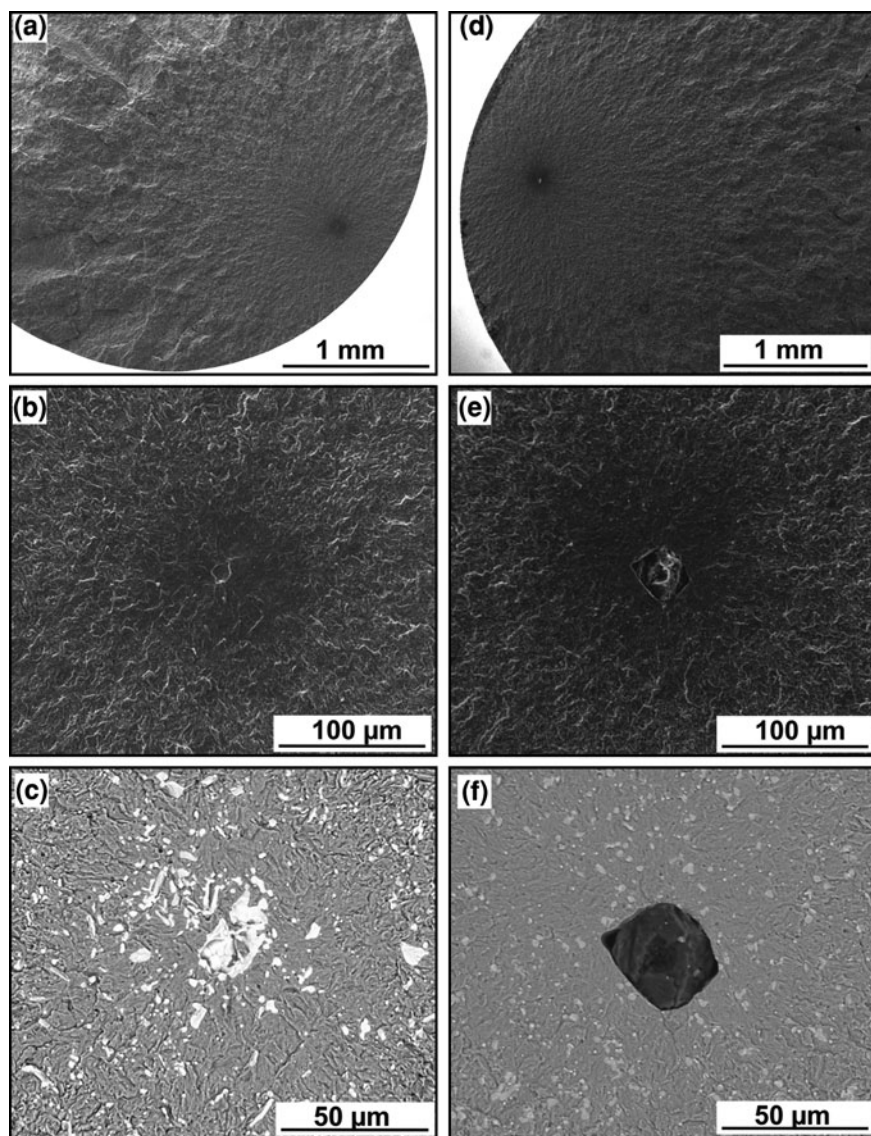


Fig. 3.91 Representative fractographs of internal failures of steel S500: **a–c** Specimen (S500-S16) failed at 850 MPa after 1.0×10^5 cycles from primary carbide cluster; **d–f** Specimen (S500-S4) failed at 900 MPa after 5.5×10^6 cycles from a nonmetallic inclusion

causing cracking of the carbides. In most cases the hard primary carbides fractured rather than decohering from the matrix, thus, they were observed on both mating fracture surfaces (compare Fig. 3.91c to Fig. 3.92a). EDX analysis (Fig. 3.92b) of the crack nucleating carbides revealed high amounts of molybdenum, iron and

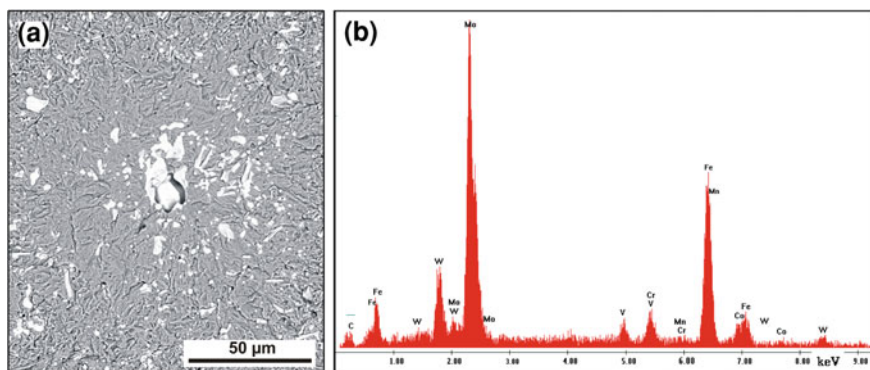


Fig. 3.92 a Mating fracture surface of fractograph of specimen S500-S16—shown in Fig. 3.91; b EDX spectrum of the crack initiating carbide particle

tungsten, similar to the spectra shown in the experimental section (Chap. 2). Thus, these crack initiating carbides were η -carbides (M_6C type). Only in a few cases a hole was observed at one fracture surface while on the mating surface a carbide particle turned out to be the crack origin. The latter indicates that the crack proceeded around the initiating carbide particles, which were M_6C type carbides, Mo-rich and W-Mo-rich in steel S500 and S600, respectively. The diameters of the carbide clusters that caused internal failure ranged from 30 to 40 μm (see Table 3.18).

According to electron probe microanalysis (Fig. 3.94b), the crack-nucleating nonmetallic inclusions contained large amounts of Ca, Al, Mg and oxygen and were somewhat larger, i.e. 30–75 μm , than the internal carbide clusters. Thus, it can be assumed that the inclusions were typical slag impurities such as CaO, MgO, and Al_2O_3 . In contrast to the carbides, at these nonmetallic inclusions decohesion from the matrix occurred in place of transgranular fracture, as can be seen in Figs. 3.91f and 3.94a.

Distances of the internal crack origins to the specimen surface, which ranged from 380 to 1800 μm , did not reveal any relationship to the applied stress amplitude nor to the cycle number to failure. Thus, it can be concluded that these internal defects represented singularities within the tested material volume. Since the number of specimens that failed from internal and subsurface defects was too low, correlating with applied stress amplitude and cycle number to failure would not be meaningful.

Figure 3.93 shows characteristic crack origins of fatigue failure from primary carbides located at or close to the specimen surface (Fig. 3.93a–c) and from subsurface nonmetallic inclusions (Fig. 3.93d–f). In case of at/near surface failure, half of fish-eye was formed, showing a similar dark surface area in the vicinity of the carbide aggregates as described before. The determination of crack nucleating micro-constituents at/near the surface was difficult in many cases. It seemed that during final fracture or during compressive cycles the surface region in the vicinity

Table 3.18 Numerical results of quantitative evaluation of the crack nucleating and growth features observed on the fractographs of failed S500 specimens

Steel S500			Diameter		Distance of
Internal sample number	Cycle number to failure	Stress amplitude (MPa)	Carbide cluster (μm)	Area 2a (μm)	Crack origin to surface (μm)
S500-S13	1.00E+06	700	73	217	136
S500-S5	2.00E+07	900	33	73	52
S500-S1	5.00E+05	800	17	98	
S500-S4	5.45E+06	900	36	97	536
S500-S11	1.00E+05	850	37	88	382
S500-S16	2.00E+05	850	37	101	985
S500-S26	6.50E+05	800	33	110	1800
S500_1	5.00E+05	800	17	98	
S500_32	8.00E+04	850	29	134	
S500_30	2.60E+05	800	23	134	
S500_20	7.30E+05	800	15	130	
S500_21	1.60E+06	800	12	106	
S500_24	1.93E+06	800			
S500_28	6.40E+05	700	59	174	
S500_19	3.70E+06	700	10	114	
S500_18	8.60E+06	700		130	
S500_6	5.50E+06	600	32	192	
S500_10	8.80E+06	600	28	140	
S500_9	9.16E+06	600	25	190	
S500_27	4.60E+07	600	12	216	
S500_7	6.00E+07	500		222	
S500_22	2.02E+08	500		214	
S500_14	2.82E+08	500			
S500_29	5.22E+08	500	19	220	
S500_25	4.27E+06	700		130	

of the crack origin is damaged or even destroyed, which has been discussed earlier. However, identification of the crack initiation site was really impossible only for a few samples. Other possible reasons for the observed surface failures, such as corrosion and cavitation, have been discussed and excluded in [Sect. 3.2.4](#).

3.4.3.3 Quantitative Evaluation on S500 and S600 Fractographs

The diameters of these at/near-surface crack-initiating carbides and carbide clusters ranged from 10 to 60 μm, thus they were in part far larger than the maximum carbide sizes found in the metallographic investigations. Nevertheless, these large carbide clusters seem to exist in numbers sufficiently high that the probability of occurrence at/near the specimen surface was high, thus, they do not represent

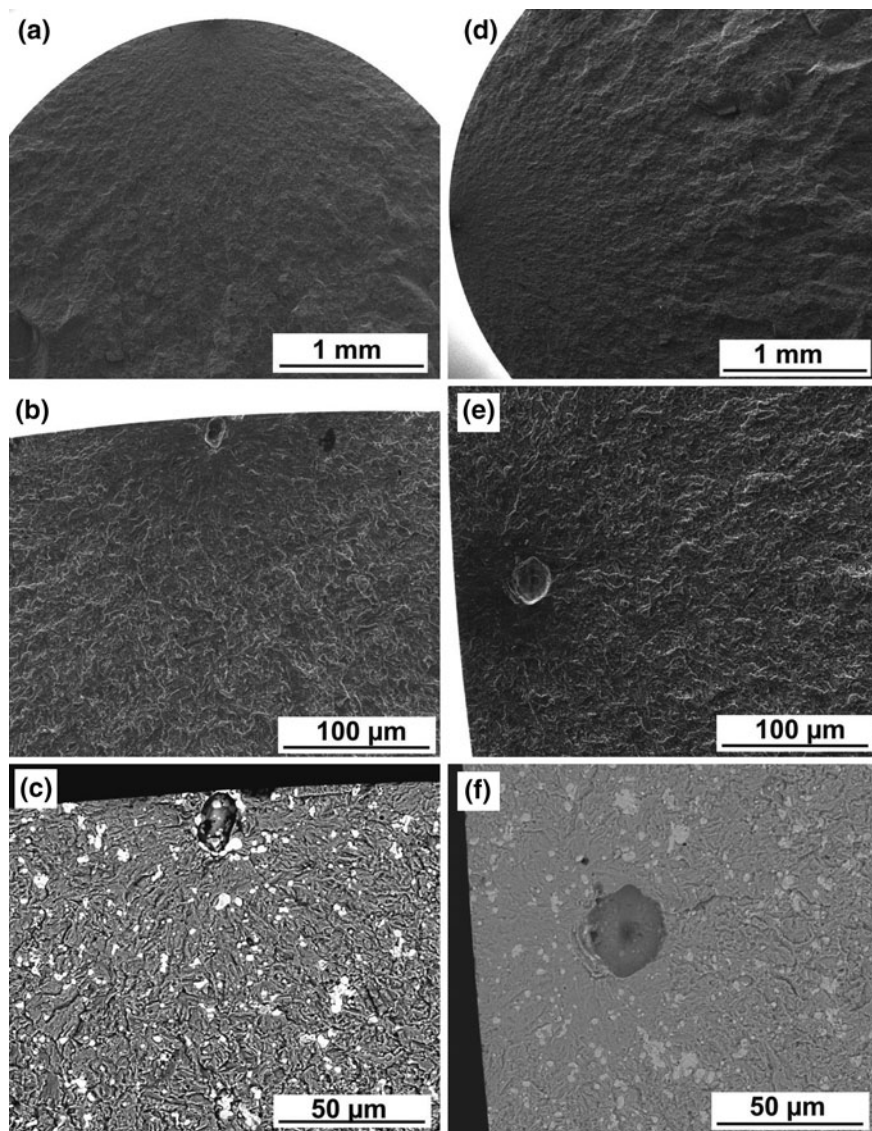
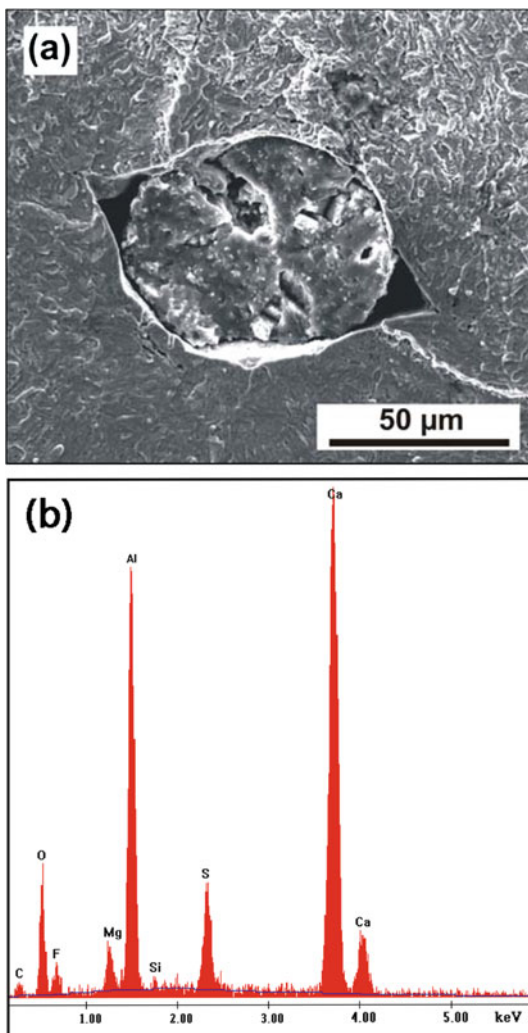


Fig. 3.93 Representative fractographs of at/near-surface (a–c) and subsurface failures (d–f) of steel S500: **a–c** Specimen failed due to large primary carbide at 800 MPa after 2.6×10^5 cycles; **d–f** Specimen failed at 900 MPa after 2×10^7 cycles due to a subsurface non-metallic inclusion

material singularities. An estimation of the location of the crack origin—internal or at/near-surface—can be performed according to the statistical concept presented in Sect. 3.3.3.3. With increasing “defect” volume content, which here means volume content of potentially crack initiating carbides, the probability that at least

Fig. 3.94 **a** Fractograph of specimen S500-S13 failed from a subsurface nonmetallic inclusion; **b** EDX spectrum of the inclusion



one of them is located at/near the surface increases. Consequently, it is essential to calculate a critical carbide volume content, above which surface induced failure will be dominant. Assuming a carbide diameter of 10 μm —which was found to be the smallest crack initiating carbide—and considering the total carbide volume fraction for the two high speed steels, i.e. about 7%, the critical carbide volume fraction is $3 \times 10^{-6}\%$, which is by far exceeded by the volume fraction of carbides having a diameter larger than 10 μm (0.56%) as observed in the metallographic investigations. Thus, the probability of finding such a potential crack initiator at the specimen surface is high and consequently at/near-surface failures are dominant for the two high speed steels, which was confirmed by the results of the fatigue experiments.

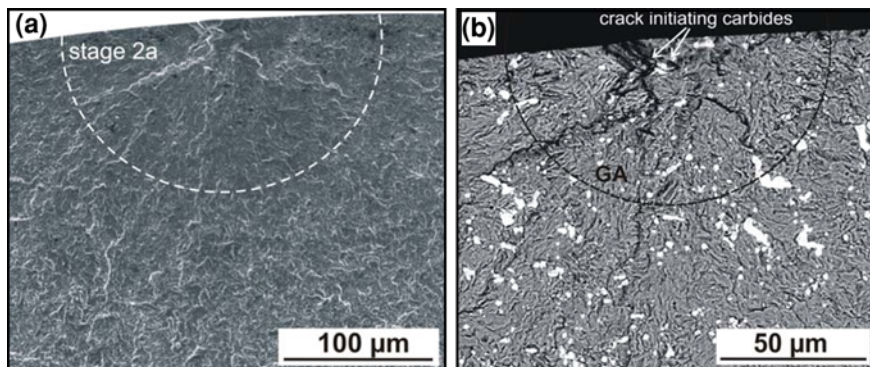


Fig. 3.95 Fracture surface of S500 specimen (S500-S29) failed due to closely arranged primary carbides at 500 MPa after 5.22×10^8 loading cycles: **a** SEM image showing zone with rather low surface roughness (stage 2a), **b** BSE image revealing the crack origin and the surrounding granular area (GA)

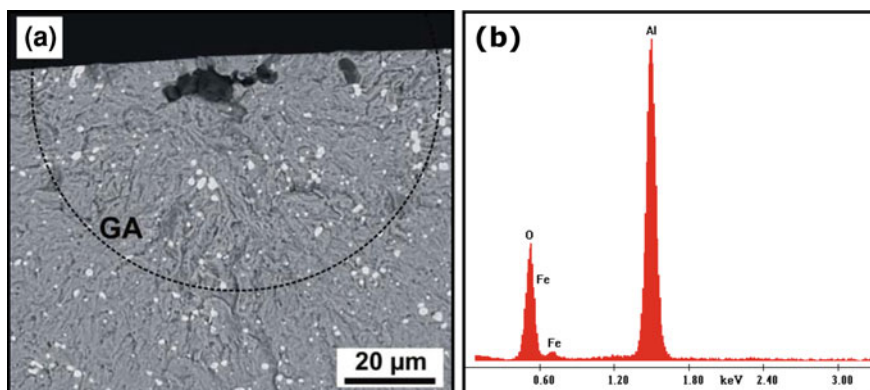


Fig. 3.96 **a** Fractograph of S600 (S600-S52) specimen failed at 500 MPa after 1.04×10^8 cycles from a surface aluminium oxide inclusion (EDX analysis (**b**)) showing a granular area around the inclusion

In the vicinity of the crack-nucleating carbides an area exhibiting a granular surface morphology (GA in Fig. 3.95b) was observed, especially at longer fatigue lives and lower amplitudes <700 MPa. In cases in which large nonmetallic inclusions initiated the fatigue crack, such a surface structure was not obtained, which can be attributed to the large dimension of these inclusions. Around smaller inclusions this granular area, however, was also obtained (Fig. 3.96). Similar granular surface morphology was observed around the crack-nucleating carbides in wrought cold work tool steel K110. However, in contrast to steel K110, for the two high speed steels studied here the granular surface morphology was by far not as pronounced, and an exact determination of the size of this granular area (GA)

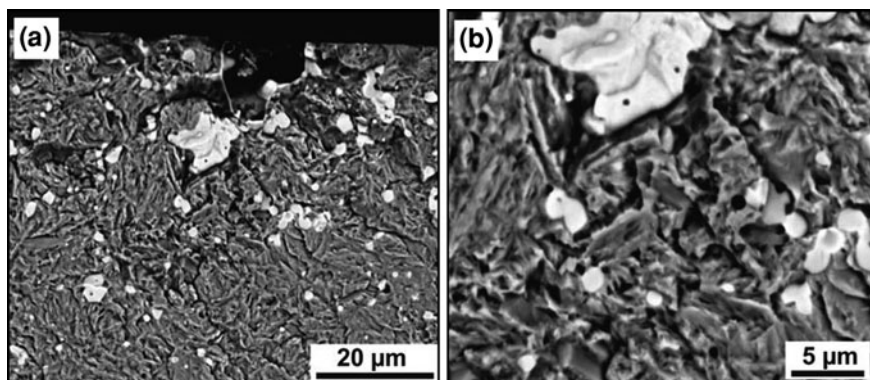


Fig. 3.97 Fractographs of specimen S500-S53 failed at 500 MPa after 2.79×10^8 cycles from a surface carbide cluster: **a** granular area around the carbide cluster particles, **b** surface morphology within the granular area

was not possible. However, the radius of the GA was in the range of 30–100 μm depending on the applied stress amplitude.

The carbide/carbide cluster size did not reveal any relationship to the applied stress amplitude and cycle number to failure, which agrees with findings for the cold work tool steel K110. There, it was argued that the formation of a granular area is a prerequisite for the formation of a short fatigue crack, i.e. when the granular area reaches a certain size. At stress amplitudes >700 MPa the crack initiating carbides were large enough to trigger a propagating fatigue crack, and thus, it seemed that a granular area was not formed.

The formation of this granular can be explained by the model of Shiozawa et al. discussed in Sect. 3.3.3.5. The formation and coalescence of microcracks in the vicinity of the larger particles, whether primary carbides or nonmetallic inclusions, seemed to occur also for these two high speed steels, as can be seen in Fig. 3.97.

The multiple microcracks are formed by the decohesion of the matrix from small spherical carbides, which however is enhanced by stress concentration as in the vicinity of larger primary carbides. These decohesion minicracks then grow and coalesce to form propagating fatigue cracks. However, unless an appropriate crack length—which corresponds to the size of the granular area—is reached, these short cracks are non-propagating cracks. Thus, the described decohesion minicrack formation and coalescence within the granular area continues until a short propagating crack is formed. The fact that for amplitudes <700 MPa a correlation to applied stress amplitude or cycle number to failure did not exist for the carbide size (but existed for the GA) was observed supports the hypothesis that the formation of the granular area is essential for the development of a propagating fatigue crack at low amplitudes and gigacycle fatigue life. Furthermore, the occurrence of fatigue failures up to 10 billion cycles is a direct result of this subcritical fatigue mechanism.

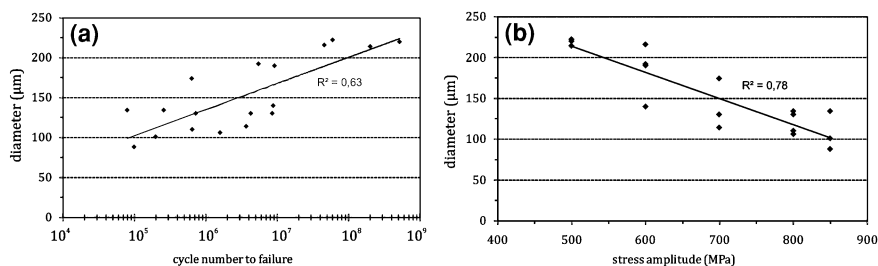


Fig. 3.98 Relationship between radius of crack growth stage 2a and **a** cycle number to failure, and **b** applied stress amplitude

The change from the granular area to the subsequent crack growth stage “2a” was gradual, which also made the assessment of the granular area size difficult. This subsequent area “2a” is characterized by a rather low surface roughness and thus appears dark in the SEM (Fig. 3.95a). It is speculated that the low surface roughness might be due to the very slow crack growth here, which can be supposed to result in crack growth exactly perpendicular to the stress orientation. Since it can be assumed that crack growth in this region is rather slow, a large proportion of fatigue life is spent there. The transition from stage 2a to the subsequent crack propagation process seems to be fairly continuous. Outside of area 2a, crack growth is assumed to be relatively fast, consequently the surface roughness is fairly high. The size of the stage 2a area showed a direct relationship to the cycle number to failure (Fig. 3.98a), which was also found for steel K110. Consequently, the longer the specimen lives, the larger is the stage 2a area, which means that a significant part of the specimen life is spent within this crack growth zone, which presumably corresponds to short crack growth. Furthermore, it turned out that the lower the applied stress amplitude is, the larger is the observed size of stage 2a (Fig. 3.98b).

3.4.3.4 Fractographic Estimation of Stress Intensity Factors

For the carbide cluster and the zone 2a, stress intensity factor thresholds for at/near-surface failure were calculated according to the equation for a surface crack presented in Sect. 3.3.3.7. For these calculations, fracture surfaces of specimens broken at stress amplitudes from 500 to 800 MPa were evaluated. The presented range of ΔK corresponds to the 95% confidence interval of the calculated mean value. The obtained wide ranges for the ΔK values due to the uncertainty of the growth stage size measurements have to be considered. The $\Delta K_{\text{carbide cluster}}$ value obtained for at/near-surface failure of steel S500 turned out to be in the range of 2.2–3.6 $\text{MPa}\sqrt{\text{m}}$, which was very similar to the values obtained for near-surface cracks for steel K110. However, the ΔK values for at/near-surface failure of zone 2a were in the range of 6.8–7.8, which was

slightly lower than those obtained for steel K110, indicating that the Paris-Law crack growth regime starts at lower ΔK , i.e. at smaller crack length, which can be attributed to the significantly higher hardness of the high speed steels. A mean ΔK value of $2.9 \text{ MPa}\sqrt{\text{m}}$ and the largest carbide cluster size observed as crack nucleation point, i.e. $30 \text{ }\mu\text{m}$ radius, were used for the calculation of an endurance fatigue limit for steel S500. It turned out to be about 420 MPa, which was a quite good estimate compared to the experimentally obtained fatigue endurance strength of 450 MPa. Using the largest cluster size obtained in metallographic investigations, which had a radius of $10 \text{ }\mu\text{m}$, gave a fatigue endurance strength of 730 MPa, which is by far too high compared to the experimentally determined value.

This fact clearly shows that fatigue testing at low amplitudes/high N can detect the largest defects in the material, which cannot be found by metallographic investigations, simply due to the fact that these large carbide clusters represent singularities within the studied high speed steels. This is a major difference to the studied cold work tool steel K110, in which large carbides/carbide clusters exist in very high numbers.

Concluding the fatigue investigation on the ingot metallurgical high speed steels S500 and S600, the two steels failed at $N > 10^6$ loading cycles at stress amplitudes lower than 900 MPa, showing similar fatigue behavior. The observed S–N curves, which revealed fairly low scatter, resembled the data obtained for wrought cold work tool steel K110. Crack initiation sites were internal carbides/carbide clusters, internal and subsurface nonmetallic inclusions, and primary carbides/carbide clusters located at/near the surface, which represented the dominant group of fatigue crack origins. In the vicinity of the crack-initiating carbides a granular area was detected for amplitudes $< 700 \text{ MPa}$, similar to that observed for steel K110, that also here plays an important role in the fatigue process at low amplitudes and long fatigue lives.

3.5 Fatigue Behavior of Powder Metallurgy Cold Work Tool Steel (Böhler K390)

3.5.1 Material Characterization

3.5.1.1 Metallography, X-ray Diffraction and Electron Probe Microanalysis

The as-received, annealed microstructure revealed very homogeneous distribution of the very small ($< 5 \text{ }\mu\text{m}$) primary alloy carbides (white spots in Fig. 3.99). These carbides are vanadium MC type carbides. Transverse and longitudinal metallographic sections showed completely identical distribution of the carbides, i.e. fully isotropic microstructure, as resulting from the PM production route.

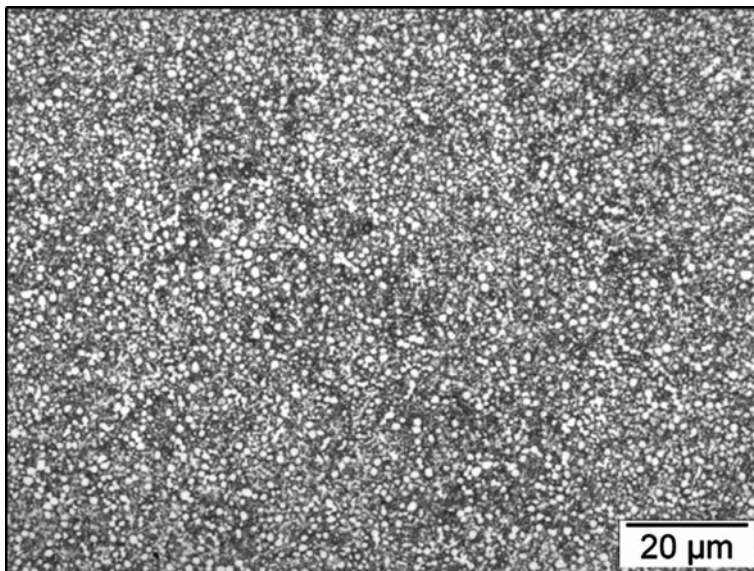


Fig. 3.99 As-received (annealed) microstructure of steel K390 (1000 $\times$, etched with Adler reagent)

The fatigue specimens were hardened from 1,040 °C in oil. Due to the sluggish etching behavior of this highly-alloyed vanadium steel, prior austenite grain boundaries are only partially visible. However, at least it was possible to estimate the grain sizes, which were between 10 and 15 μm . Thus, undesired grain coarsening did not take place, and the austenite grain size was similar to that observed in the already described ingot metallurgical steels. Figure 3.100 shows the as-tempered microstructure. Tempering was performed three times at 560 °C, as described in Sect. 2.1.2. Vanadium-rich MC type carbides and matrix can be distinguished clearly; carbides appear darker than the matrix in the BSE image due to the lower atomic number of vanadium compared to iron. Obviously, the carbides are quite small—a few μm maximum.

EDX analysis of the as-heat treated steel matrix (Fig. 3.101a) showed predominantly Fe, but also some Cr, Mo and V, the detection of which however might result from very small carbides embedded in the matrix.

The VC carbides contain also some Mo and Cr, and maybe some Fe (Fig. 3.101b). However, the fairly high amount of Fe detected in the carbides definitely is a measurement artifact, since the signal generation, i.e. the emission of X-rays, occurs within a lateral area that is larger than the carbides. Thus, it is obvious that due to the small carbide sizes also part of surrounding matrix contributes to the generated signal. Kulmburg [7] reported the iron content being in the range of 2–6% in VC carbides, which contain usually about 40% V, and additionally W or Mo. XRD proved the presence of MC type, V-rich carbides (Fig. 3.102). Retained austenite was not observed after the applied heat treatment.

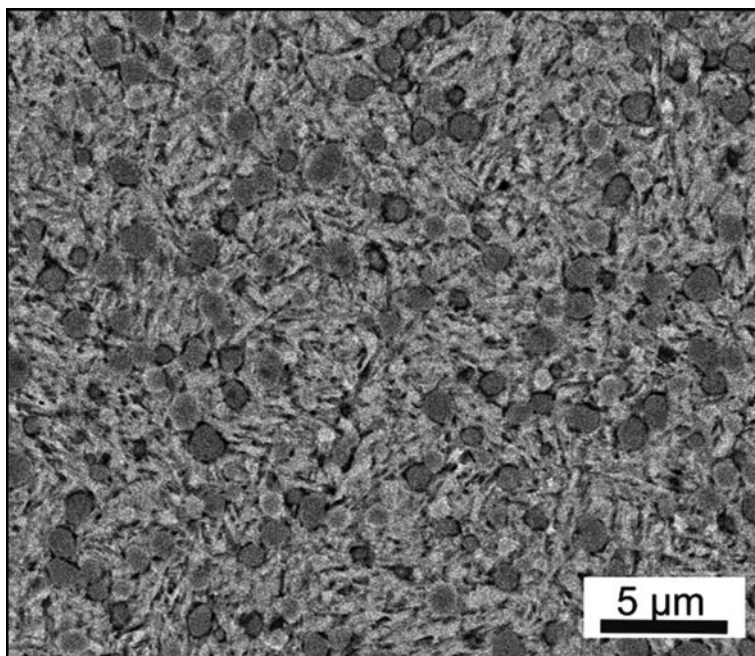


Fig. 3.100 As-tempered microstructure of PM cold work tool steel K390 (Nital etch)

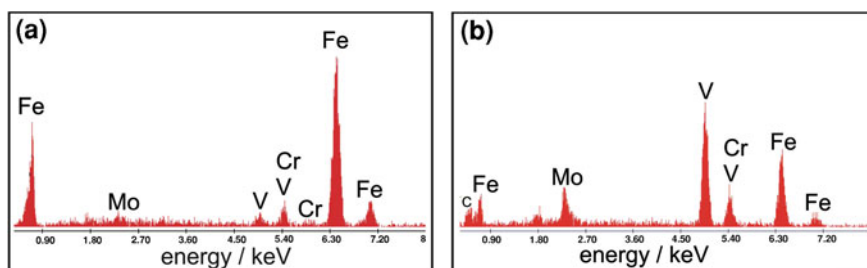


Fig. 3.101 Elemental analysis (EDX) of **a** the matrix, **b** of MC type primary carbides (V-rich) of as-heat treated (quenched and tempered) steel K390

Quantitative evaluation of the primary carbide size in as-heat treated steel—as done for the conventionally produced (ingot metallurgy) steels—was not reasonable here since the carbides were too small ($<5\ \mu\text{m}$ diameter). Obviously, there existed closely arranged carbides, i.e. carbide clusters. However, it was not possible to define a reliable criterion for the decision whether carbides belong to a “carbide cluster” or not. Furthermore, nonmetallic inclusions were not observed in metallographic sections of steel K390. The volume fraction of the primary carbides in the steel K390 was determined using image analyzing software applied

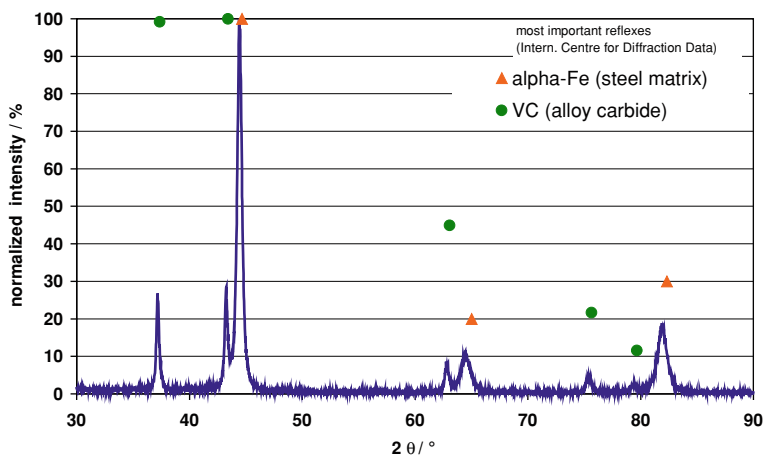


Fig. 3.102 XRD pattern of quenched and tempered K390 PM cold work tool steel

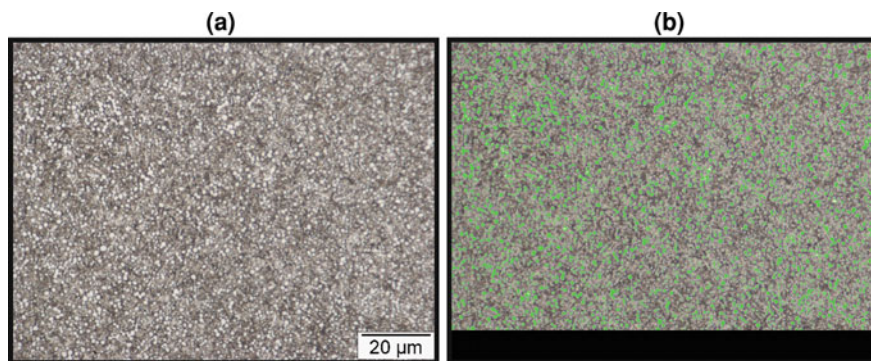


Fig. 3.103 Determination of carbide volume fraction by quantitative metallography (1000 $\times$ -Picral etch)

to five optical micrographs at 1000 $\times$ magnification, as shown in Fig. 3.103. The carbide volume fraction turned out to be 14 ± 3 vol%.

3.5.1.2 Mechanical Properties

The mechanical properties determined are presented in Table 3.19. In the as-received, annealed condition the steel K390 had a hardness of about 24 HRC, which was quite similar to the cold work tool steels K110. The as-tempered hardness was adjusted through the heat treatment in such a way that the same hardness as for the steel K110, i.e. 59 ± 1 HRC, was attained. The dynamic Young's modulus was significantly higher for steel K390 compared to K110,

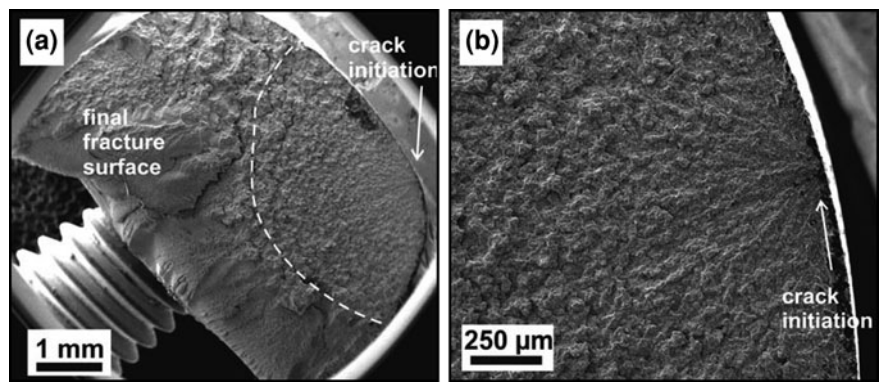


Fig. 3.104 TRS fractographs of PM cold work tool steel K390: **a** overview, **b** area of crack initiation

Table 3.19 Mechanical properties of K390 tool steel

Steel	Rockwell hardness HRC 150 kg		T.R.S. (MPa)	Dynamic Young's modulus (GPa)
	As-quenched	Quenched and tempered	Quenched and tempered	Quenched and tempered
Böhler “K390”	64 ± 1	59 ± 2	6800 ± 650	228 ± 8

probably due to the higher amount of alloying elements and higher carbide volume fraction. Impressive bending strength of the steel was obtained, which was nearly twice as high as for the ingot metallurgy cold work tool steel K110. The bending test bar burst into many fragments during fracture. Obtained fracture surface are shown in Fig. 3.104. Cracks initiated from the surface area.

3.5.2 Fatigue Behavior of PM Cold Work Tool Steel K390

3.5.2.1 Fatigue Data and S–N Curve

The S–N data obtained for PM cold work tool steel K390 with low residual stresses at the surface (–80 to –240 MPa) is presented in Fig. 3.105. Numerical data is shown in Table 3.20. Testing was done in longitudinal direction of the bar, which is however irrelevant in this case since PM tool steels are microstructurally isotropic.

The curve shows a continuously decreasing shape with increasing cycle number to failure and that a real fatigue limit does not exist for this material, too. The level of stress amplitudes required for material failure within the tested cycle numbers was in the range of 700–1400 MPa, thus very high. At 10¹⁰ loading cycle a fatigue

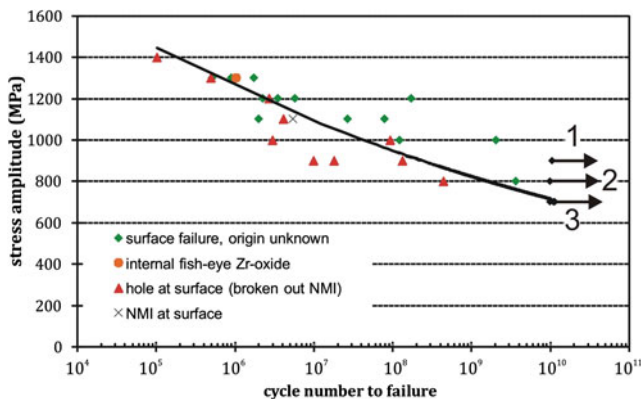


Fig. 3.105 S–N data of PM cold work tool steel K390 with low residual stresses at the surface (figures close to the *arrows* indicate the number of runout specimens) determined jointly with Dipl.-Ing.A.Betzwar-Kotas (University of Vienna)

endurance strength of about 700–800 MPa was observed, i.e. significantly higher than with K110.

Crack initiation took place at “holes” at the surface (Fig. 3a, ▲), which can be caused by broken-out nonmetallic inclusions, at a nonmetallic inclusion located close to the surface (Fig. 3a, ×), and one sample failed due to an internal defect, i.e. a Zr-oxide inclusion (Fig. 3a, ○). However, in many cases the specimen failed from the surface, but the exact crack nucleating microstructural constituent was not evident (Fig. 3a, ◆), which was also reported by Marsoner et al. [4] for 12% Cr PM cold work tool steel (Böhler grade K190), and discussed earlier in this thesis.

Figure 3.106 shows a comparison to the S–N curve obtained for steel K110 (also with low residual stresses), which clearly reveals that the PM cold work tool steel offer a fatigue strength about *twice as high* as the fatigue strength obtained for conventional wrought cold work tool steel K110. This can be attributed to the significantly larger size of the primary carbides (clusters) that initiated fatigue cracks in the K110 grade and the significant higher strength and toughness of PM steel K390 (TRS of nearly 7000 MPa). A further interesting fact is that the scatter for steel K390 is markedly more pronounced than for steel K110, which is in accordance with findings of Marsoner et al. [5]. The fact that K390 specimens broke from singularities, i.e. nonmetallic inclusions, located at the surface can be the reason for the significantly larger scatter of the S–N data. In contrast, in steel K110 the crack-nucleating large primary carbides exist in a very high number in the steel. Thus, as it was shown in the statistical consideration in Sect. 3.3.3.3, the probability of occurrence of large carbides/carbide clusters at the surface is very high. Therefore the fatigue stress amplitudes are lower for steel K110 but the scatter band of S–N data is quite narrow.

Table 3.20 Fatigue data of PM cold work tool steel K390

Steel K390						
Internal fatigue sample number	Strain at gauge length (mm)	Stress amplitude (MPa)	Cycle number to failure	Crack origin location	Crack nucleating microconstituent	Diameter of crack nucleating feature (μm)
K390-KP10	6.18E-03	1400	1.03E+05	Surface	Broken out	5
K390-KP21	5.66E-03	1300	5.00E+05		nonmetallic inclusion	7
K390-KP13	5.75E-03	1300	5.20E+05		Unknown	
K390-KP17	5.67E-03	1300	9.10E+05		Unknown	
K390-KP22	5.73E-03	1300	1.04E+06	Innen	Zr-Oxide inclusion	12
K390-KP12	5.74E-03	1300	1.75E+06	Surface	Unknown	
K390-KP19	4.87E-03	1200	2.26E+06		Unknown	
K390-KP30	5.28E-03	1200	2.70E+06		Broken out nonmetallic inclusion	17
K390-KP18	5.29E-03	1200	3.50E+06		Unknown	
K390-KP28	5.25E-03	1200	5.74E+06	Subsurface	Unknown	
K390-KP20	5.28E-03	1200	1.72E+08	Surface	Unknown	
K390-KP23	4.87E-03	1100	2.00E+06		Unknown	
K390-KP14	4.88E-03	1100	4.15E+06		Broken out nonmetallic inclusion/MC carbide	4
K390-KP15	4.87E-03	1100	5.42E+06		Nonmetallic inclusion	9
K390-KP16	4.86E-03	1100	2.67E+07		Unknown	
K390-KP31	4.85E-03	1100	7.90E+07		Unknown	
K390-KP27	4.40E-03	1000	3.00E+06		Broken out	10
K390-KP29	4.39E-03	1000	9.21E+07		nonmetallic inclusion	8
K390-KP26	4.41E-03	1000	1.22E+08		Unknown	
K390-KP9	4.50E-03	1000	2.04E+09		Unknown	
K390-KP40	3.97E-03	900	1.00E+07		Broken out	18
K390-KP41	3.99E-03	900	1.80E+07		nonmetallic inclusion	48
K390-KP33	3.96E-03	900	1.33E+08			12
K390-KP39	3.97E-03	900	1.05E+10	Runout specimen		
K390-KP42	3.55E-03	800	4.41E+08	Surface	Broken out nonmetallic inclusion	24
K390-KP34	3.50E-03	800	3.68E+09		Unknown	
K390-KP37	3.54E-03	800	1.01E+10	Runout specimen		
K390-KP38	3.56E-03	800	1.04E+10			
K390-KP35	3.09E-03	700	1.00E+10			
K390-KP36	3.09E-03	700	1.10E+10			
K390-KP43	3.11E-03	700	1.15E+10			

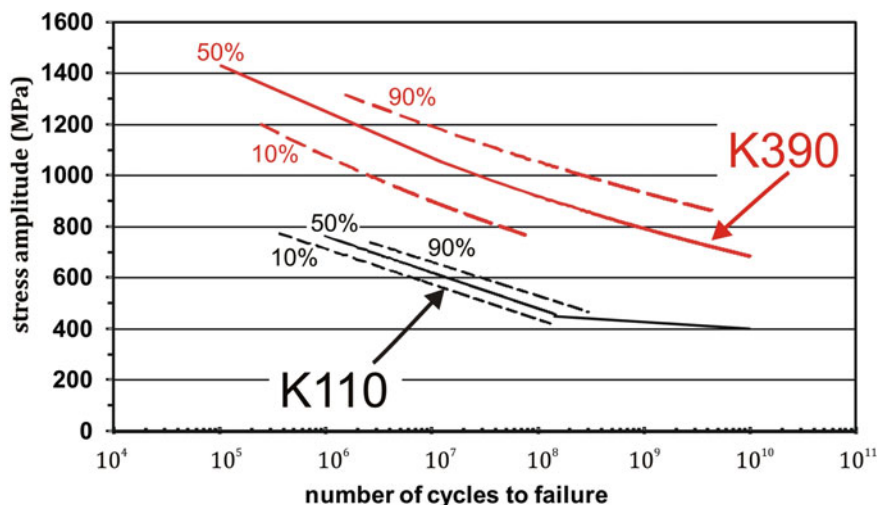


Fig. 3.106 Comparison of the S-N curves of PM cold work tool steel K390 to wrought cold work tool steel K110 (low residual stresses at the surface); Bold and dashed lines represent the 50, 10 and 90% fracture probability, respectively

3.5.2.2 Crack Origins and Fractography

The macroscopic appearance of failures from surface defects (inclusions, holes and unidentified) located at or just below the surface—thus, referred to as at/near-surface failures—and from an internal defect is presented in Fig. 3.107a, b, respectively. The two fractographs reveal rather flat, fine-structured surface morphology despite the fact that both samples failed at quite high stress amplitudes, which can be attributed to the relatively high strength and toughness of steel K390 (TRS nearly 7000 MPa). Cracks and ridges point back to the crack origin. A fish-eye pattern was formed around the internal crack origin. In case of at/near-surface failures a half fish-eye was obtained.

In contrast to wrought cold work tool steel K110, crack growth zones within the fish-eye patterns are hardly distinguishable. Even the border of the fish-eye cannot be determined exactly (Fig. 3.108). It seems that transition occurs rather gradually, similar to the behavior observed for the ingot metallurgical high speed steels. Around the crack origin an area with low surface roughness was detected (zone 2a). A granular area in the vicinity of the crack origin was observed only in some cases, for which the crack initiating micro-constituents were very small (see Fig. 3.108d). In most cases when nonmetallic inclusions were responsible for fatigue failure, these inclusions were found to be broken out very likely during the fatigue test, as shown in Fig. 3.108. EDX analysis showed in most of these cases small amounts of slag elements such as Si, Al, Ca, Mg, K and O, as will be shown later. The question that arises is whether the holes were formed during polishing or

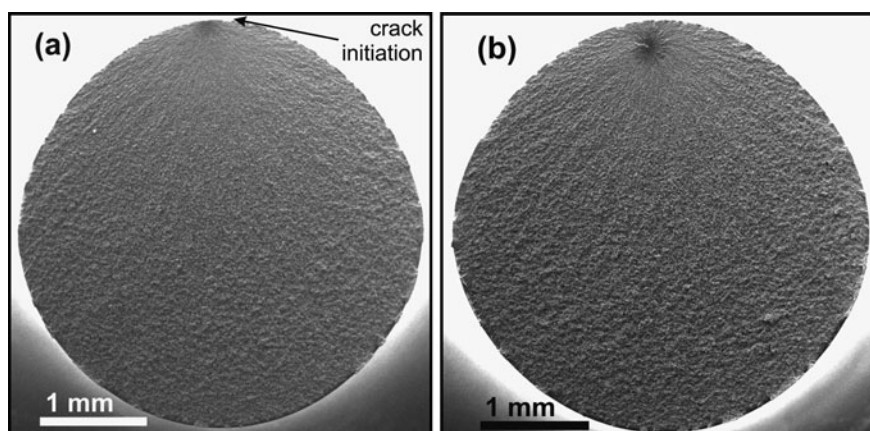


Fig. 3.107 Macroscopic appearance of fractures failed from **a** a nonmetallic inclusions (K390-KP30) at 1200 MPa after 2.70×10^6 cycles and **b** failure due to an internal inclusion (K390-KP22) at 1300 MPa after 1.04×10^6

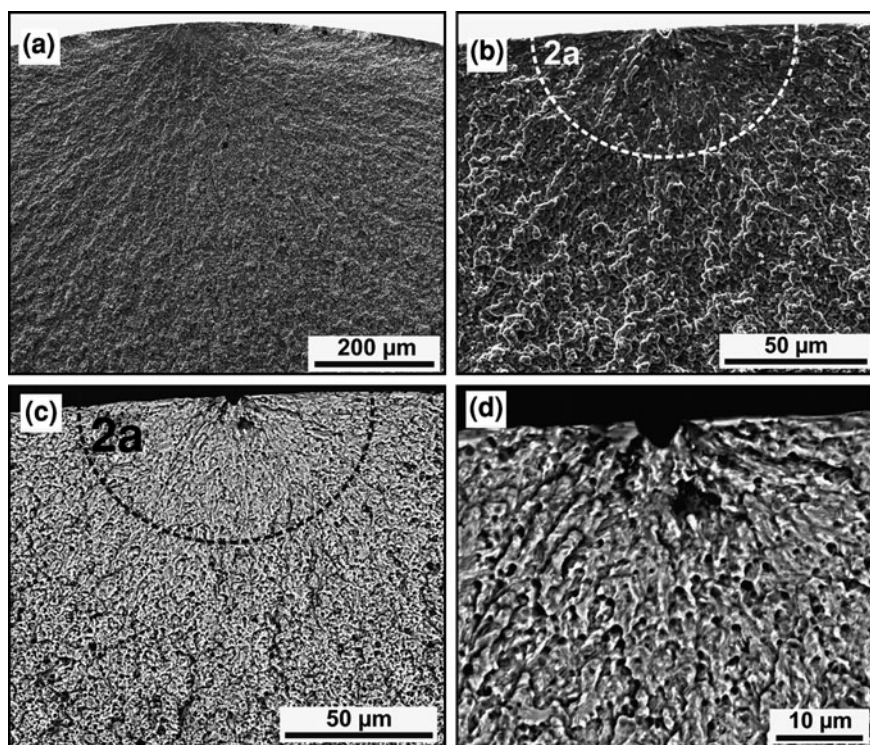


Fig. 3.108 Specimen K390-KP10 failed from a nonmetallic inclusion at 1400 MPa after 1.03×10^5 cycles: **a** half-fish-eye around the crack origin, **b** and **c** zone with lower surface roughness (area 2a) around the crack origin, **d** crack origin

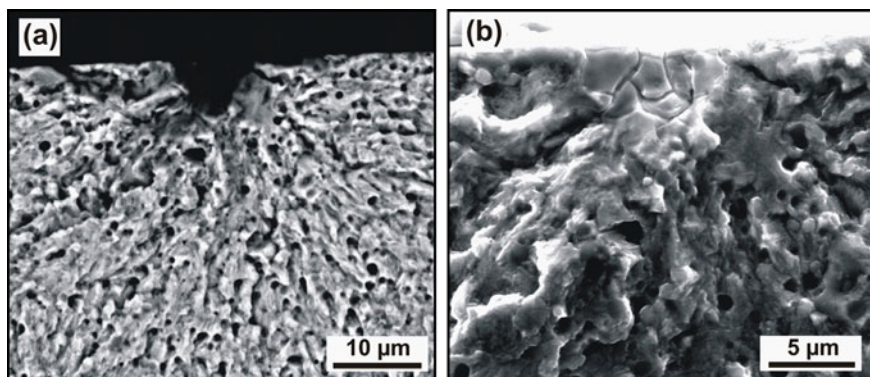


Fig. 3.109 K390 specimen failed at 1100 MPa after 5.42×10^6 cycles: **a** BSE image of the crack origin (nonmetallic inclusion appears dark due to the low average atomic number), **b** SEM image of the crack nucleating inclusion that seems to be fractured

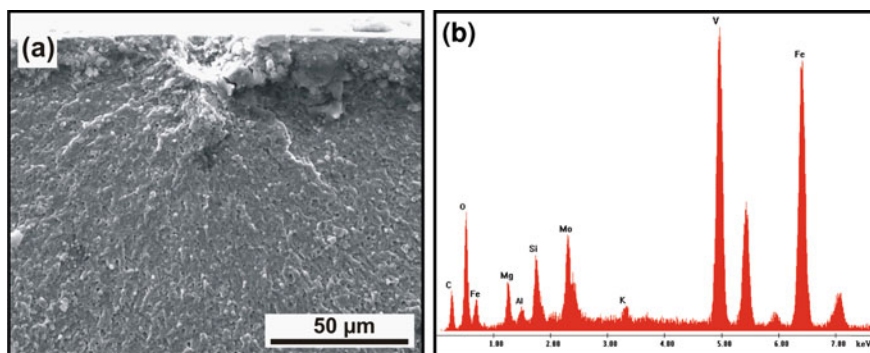


Fig. 3.110 Specimen K390-KP42 that failed at 800 MPa after 4.41×10^8 cycles from an surface inclusion: **a** SEM image of the crack origin and surrounding area, **b** EDX spectrum revealing high amounts of oxygen, Mg, Si, and some Al and K

during fatigue testing of the specimen. The latter argument seems more probable since surface inspection before fatigue testing did not reveal any such holes.

However, there was one specimen, for which definitely a nonmetallic inclusions was detected in the crack origin (Fig. 3.109). EDX analysis indicated that it was a silicon oxide inclusion. Comparing Fig. 3.109a to Fig. 3.108d, an obvious similarity of the two fractographs can be stated, which supports the statement that for specimens for which a hole was detected in the crack origin (Fig. 3.108d) a nonmetallic inclusion caused the fatigue failure and broke out during fatigue testing, i.e. probably at the shock during final fracture.

Figure 3.110 shows another example for a specimen that failed from a non-metallic inclusion, which very likely broke out during the fatigue process. The EDX spectrum revealed high amounts of oxygen, Mg, Si, and some Al and K

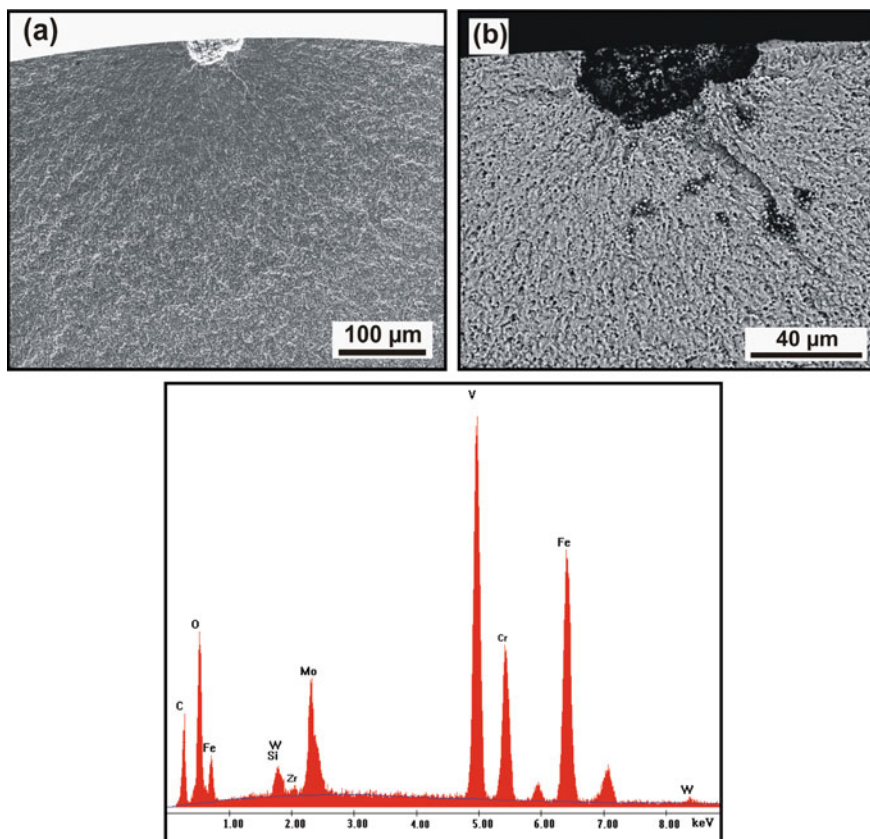


Fig. 3.111 Specimen K390-KP41 failed from a large nonmetallic inclusion at 900 MPa after 1.8×10^7 cycles: **a** SEM image, **b** BSE image, **c** EDX spectrum

indicating that a slag particle (oxide) was located at the site where the hole was observed after the specimen fracture.

One specimen (K390-KP41) failed from a very large nonmetallic inclusion—probably silicon oxide and zirconium oxide as the EDX analysis in Fig. 3.111c indicates. The observed fractographs are presented in Fig. 3.111a, b.

A Zr-oxide inclusion was also responsible for the only internal failure that was observed for steel K390 (specimen K390-KP22). The corresponding macroscopic appearance of the fracture surface is presented in Fig. 3.107b. Figure 3.112 shows the fracture surface on microscopic level, the crack origin and electron probe microanalysis (EDX) that definitely proved that this inclusion contained predominantly zirconium and oxygen and some calcium. It might probably derive from the nozzle through which the metal melt is atomized. The ceramic nozzle material is known to contain Zr-oxide.

It seems that in the vicinity of the inclusion a granular area, similar to those obtained for wrought cold work tool steel K110, was formed (see Fig. 3.112b, d).

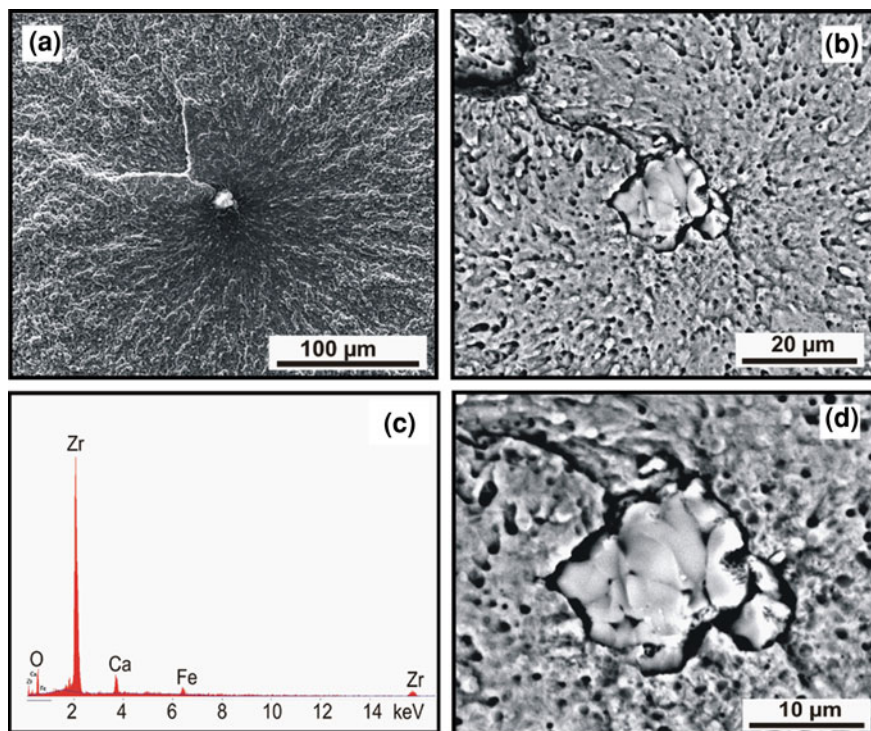


Fig. 3.112 Fractographs of specimen (K390-KP22) failed from an internal Zr-oxide inclusion

However, it is rather small. Furthermore, decohesion of the matrix from the inclusion surface took place, probably during fatigue testing.

As mentioned above, for a significant number of specimens the exact determination of the crack nucleating micro-constituent was not possible. Figure 3.113 shows representative fractographs. Fatigue failure started somewhere at the surface or just below, where the two detectable cracks point to. However, externally introduced surface defects such as machining flaws were not detected. Thus, some microstructural feature has to be responsible for the fatigue crack formation, which seems to be too small to be detectable by SEM. TEM measurements might be helpful in this respect, for which however sample preparation might be very tedious due to the high hardness of the material.

3.5.2.3 Quantitative Evaluation on K390 Fractographs

A quantitative evaluation of different crack growth zones, as performed for the ingot metallurgical steels, was not possible for this PM cold work tool steel. Discrete distinction could not be made, as can be seen from the presented fractographs. However, for the cases in which the crack nucleating micro-constituent

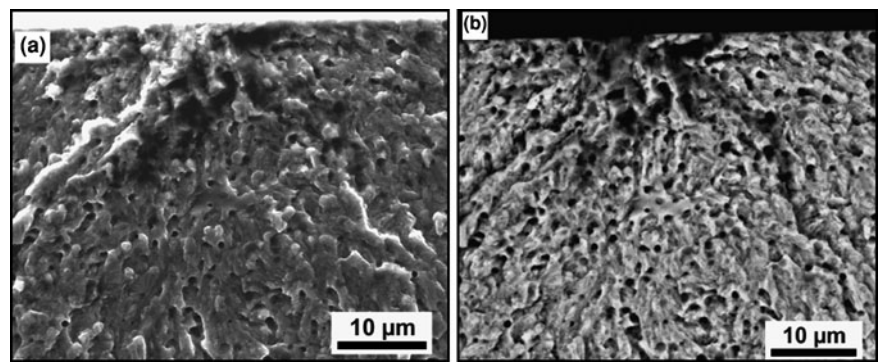


Fig. 3.113 Representative fractographs for specimens for which exact determination of the crack nucleating micro-constituent was not possible **a** SEM image, **b** BSE image of same sample

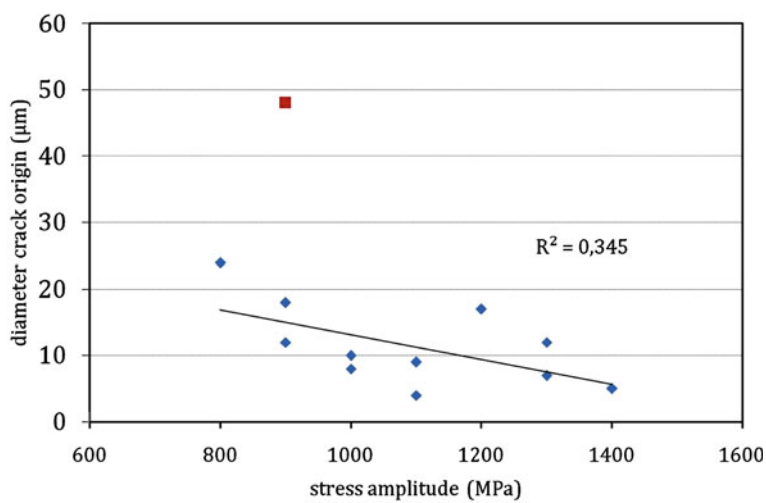


Fig. 3.114 Relationship between crack nucleating micro-constituents (nonmetallic inclusions) at/near-surface in steel K390 and applied stress amplitude

could be determined, also the size of this feature was measured. Corresponding data are presented in Table 3.20. Interestingly, a relationship between the inclusion size and the applied stress amplitude was observed, as is shown in Fig. 3.114, which is in strong disagreement with the results obtained for the ingot metallurgical steels. Together with the absence of the granular area this indicates that the inclusions were—though rather small—large enough to trigger a small fatigue crack without the requirement of the decohesion microcrack formation and coalescence, which turned out to be a prerequisite for fatigue failure of the ingot metallurgical tool steels, *at least at lower amplitudes*. This might be the decisive

reason for the absence of the granular area in case of the PM cold work tool steel, since the stresses were relatively high (>700 MPa). The fact that for steel S500 at amplitudes above 700 MPa a granular area was not detected, either, support this explanation.

3.5.2.4 Fractographic Estimation of Stress Intensity Factors

Calculation of the threshold stress intensity factor for the crack initiating inclusions ΔK_{incl} was performed using the equation and principle for a surface crack as presented in Sect. 3.3.3.7. It turned out to be in the range of $2.4\text{--}3.6$ MPa $\sqrt{\text{m}}$ (95% confidence interval of the mean value). Similar to the ΔK_{GA} values of the granular area for steel K110, the ΔK_{incl} values did show a relationship to the applied stress amplitude which indicates that the obtained mean ΔK_{incl} represents a material constant. Using the mean value for ΔK_{incl} , i.e. 3.0 MPa $\sqrt{\text{m}}$ and the radius of the largest inclusion observed as a crack origin, i.e. 14 μm (apart from the one large inclusion that had a radius of 24 μm , which represented a single event), a fatigue endurance strength of about 690 MPa results, which is a quite good estimate for the experimentally obtained fatigue strength after 10^{10} cycles (700 MPa). However, considering the largest inclusion, which had, as just mentioned above, a diameter of 24 μm , a fatigue endurance strength of 490 MPa would be obtained. Thus, fatigue failure down to this stress cannot be excluded.

Summarizing, the investigations of the fatigue behavior of PM cold work tool steel K390 showed significantly higher fatigue strength compared to the ingot metallurgical steel K110, which can be mainly attributed to the fact that the primary carbides are too small here to trigger fatigue cracks in steel K390. In contrast, nonmetallic inclusions are responsible for fatigue failure, which however, compared to the numerous crack-nucleating carbides in steel K110, are singularities in steel K390.

3.6 Fatigue Behavior of Powder Metallurgy W-Mo Based High Speed Tool Steel (Böhler S590)

3.6.1 Material Characterization

3.6.1.1 Metallography, X-ray Diffraction and Electron Probe Microanalysis

The as-received, annealed microstructure of the PM high speed steel S590 (Fig. 3.115) is very similar to that of the PM cold work tool steel K390: It exhibits a very homogenous distribution of primary carbides, in this case M_6C type tungsten-molybdenum-rich alloy carbides, which are quite small (<5 μm).

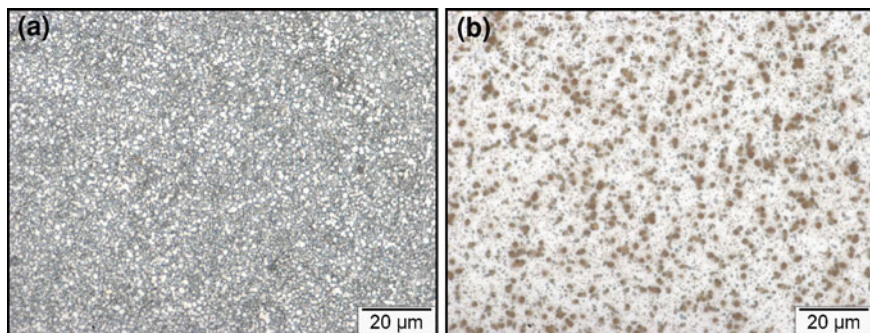


Fig. 3.115 As-received (annealed) microstructure of PM high speed steel S590 at 1000 $\times$ magnification: **a** etched with Adler reagent, carbides remained unattacked, **b** etched with Murakami reagent

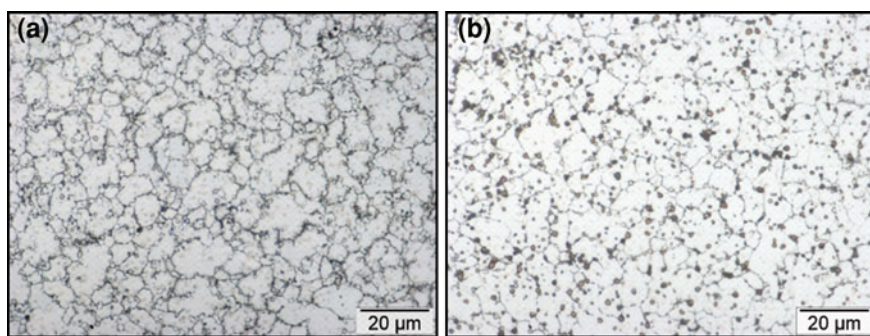


Fig. 3.116 As-quenched microstructure of steel S590 at 1000 $\times$ magnification: **a** etched with Nital, **b** etched with Murakami reagent

In addition to the M_6C carbides also some chromium carbides ($M_{23}C_6$) can exist, which however are dissolved completely during austenitizing, as described in [Sect. 3.4](#) for the two ingot metallurgy high speed steels. Transverse and longitudinal metallographic sections showed uniform and isotropic carbide distribution, as also observed for the PM cold work tool steel K390, characteristic for tool steels produced by the PM route.

As described in detail in the experimental [Chap. 2](#), the fatigue specimens were hardened from 1,180 °C in oil. The microstructure of the as-quenched S590 steel is shown in [Fig. 3.116](#). Prior austenite grain boundaries are visible in metallographic section etched with Nital reagent ([Fig. 3.116a](#) and [3.117a](#)). Undesired grain growth did definitely not occur. The grain size was determined using the Snyder–Graff method, and it turned out to be $11 \pm 1 \mu m$ for the applied austenitizing conditions. [Figure 3.117b](#) exhibits the primary carbides of type M_6C (W,Mo-rich, colored). Compared to the annealed steel ([Fig. 3.115b](#)), fewer carbides were

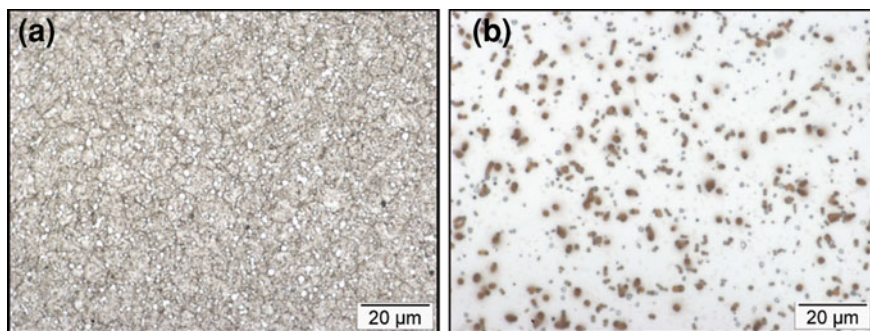


Fig. 3.117 As-heat treated microstructure of steel S590: Etched with **a** Nital and **b** Murakami reagent

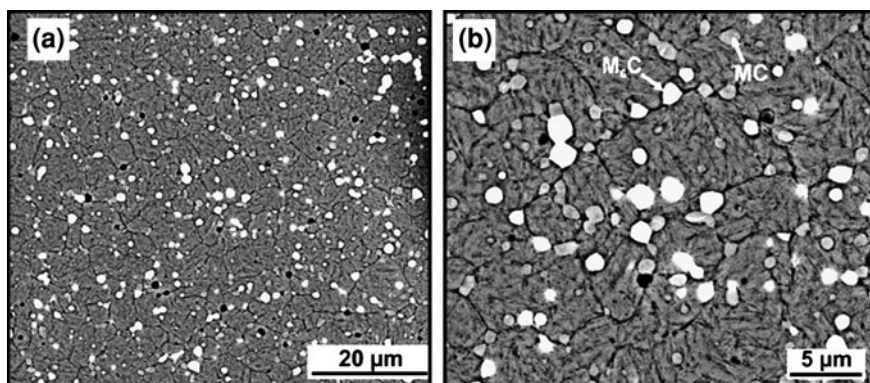


Fig. 3.118 As-heat treated microstructure of steel S590 (SEM-BSE image, etched with Nital) at **a** 2000 $\times$, **b** 5000 $\times$ magnification

observed in the as-quenched material, which probably can be attributed to the dissolution of the chromium carbides during austenitizing. The primary carbides were predominantly found at the prior-austenite grain boundaries (Fig. 3.117).

The fully heat treated microstructure revealed uniform, tempered martensite with primary carbides predominantly located at the prior-austenite grain boundaries (Fig. 3.118). The uniform etching behavior indicated that retained austenite has been completely transformed by three times tempering, which was confirmed by XRD measurements (Fig. 3.119). EDX analysis of the matrix of the as-heat treated S590 steel (Fig. 3.120a) revealed a large amount of cobalt dissolved in the tempered martensite, and also some W, Mo, V, and Cr was detected which, however, might originate from primary carbides located in the analyzed volume just beneath the surface.

Two alloy carbide types, Mo,W-rich M_6C carbide and V-rich MC type carbide, were detected by XRD (Fig. 3.119), which can also be distinguished in the

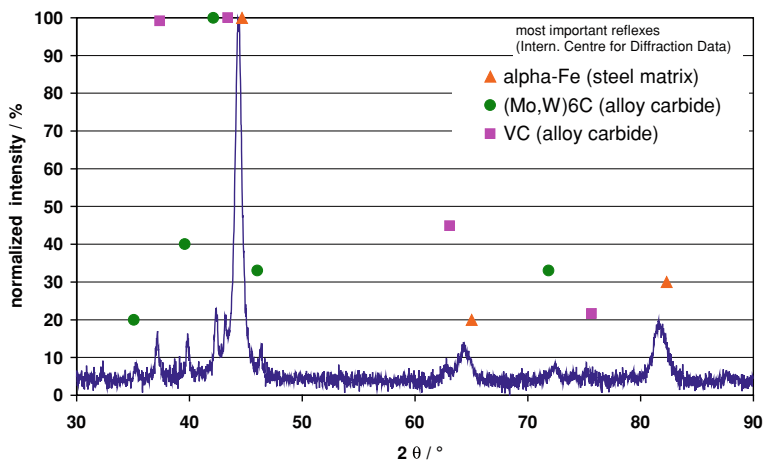


Fig. 3.119 XRD diffraction pattern of quenched and tempered S590 PM high speed steel

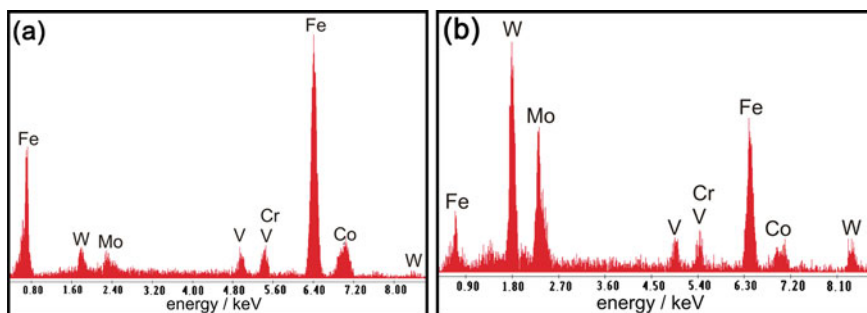


Fig. 3.120 Elemental analysis (EDX-spectra) of **a** the matrix, **b** of M_6C type primary carbides (W, Mo-rich) in as-heat treated steel S590

micrograph presented in Fig. 3.118b: the bright white spots correspond to the M_6C , which was also confirmed by EDX analysis (Fig. 3.120b). The grayish carbides are the vanadium carbides, which reveal a high content of V in the EDX spectrum.

Quantitative evaluation of the primary carbide particle size in the as-heat treated steel was not performed since the size of the single carbides was too small ($<5 \mu\text{m}$ diameter), and thus, the carbides most likely do not play a role in fatigue crack initiation. Similar to steel K390, closely arranged carbides can be observed in the metallographic sections. However, the definition of a reliable criterion for the decision whether carbides belong to a “carbide cluster” or not was not possible. Nonmetallic inclusions were not found in metallographic sections of steel S590. The volume fraction of the primary carbides in the steel S590 was

Table 3.21 Mechanical properties of S590 high speed steel

Steel	Rockwell hardness HRC 150 kg		T.R.S. (MPa)	Dynamic Young's modulus (GPa)
	As- quenched	Quenched and tempered	Quenched and tempered	Quenched and tempered
Böhler "S590"	64 ± 2	67 ± 2	4900 ± 500	236 ± 10

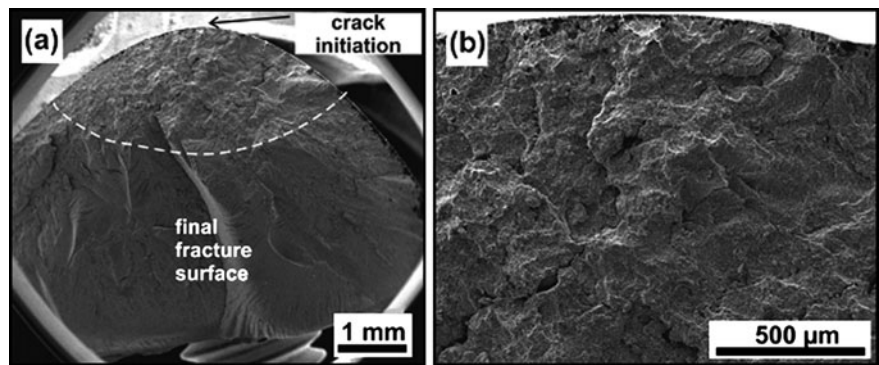


Fig. 3.121 TRS fractographs of S590 high speed steels

determined using image analyzing software; data were collected on five optical micrographs at 1000× magnification. The carbide volume fraction turned out to be 14 ± 1 vol%.

3.6.1.2 Mechanical Properties

The mechanical properties determined for steel S590 are presented in Table 3.21. The as-received, i.e. annealed, steel had a hardness of about 25 HRC, which was quite similar to the other tool steels investigated here. The as-quenched steel showed a hardness similar to as-quenched steel S600, which can be attributed to the hard primary η -carbides ($\text{Fe}_3(\text{Mo,W})_3\text{C}$) that remain undissolved during austenitization. After three times tempering a hardness increase of three HRC was achieved due to the precipitation hardening of Mo_2C carbides as described for steel S500. The dynamic young's modulus of steel S590 was significantly higher than for the conventional (wrought) high speed steels S500 and 600. The bending strength of the steel was more than 1000 MPa higher than for the wrought high speed steels. The TRS tested bar burst into many fragments during final fracture, as observed for the PM cold work tool steel, apparently as a consequence of the very high elastic energy stored during loading. Cracks initiated from the surface at several starting points (Fig. 3.121). Precise determination which micro-constituents actually initiated the cracks was not possible.

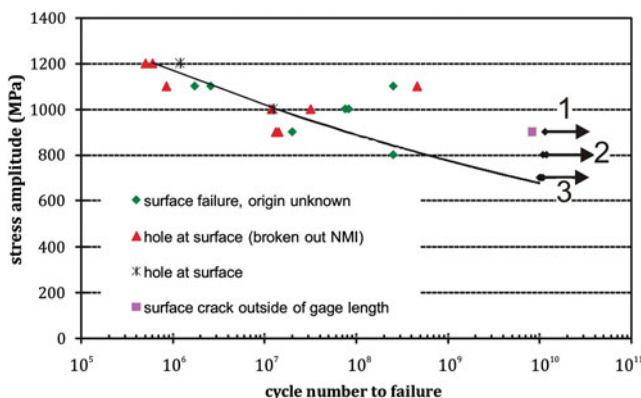


Fig. 3.122 S–N data of PM high speed steel S590 with low residual stresses at the surface (figures close to the arrows indicate the number of runout specimens) determined jointly with Dipl.-Ing.A.Betzwar-Kotas (University of Vienna)

3.6.2 Fatigue Behavior of Böhler S590 Steel

3.6.2.1 Fatigue Data and S–N Curve

The S–N curve obtained for the steel S590 (Fig. 3.122) showed a continuous slightly decreasing shape, with rather broad data scatter. Specimens failed at relatively high stress amplitudes from 800 to 1200 MPa in a range of 10^5 to 10^9 loading cycles. For numerical data see Table 3.22.

Runout specimens ($N_{\max} = 10^{10}$ cycles) were obtained at three different stress amplitudes. The fatigue endurance strength at 10 billion cycles was about 700 MPa. Specimens failed predominantly due to non-metallic inclusions located at/near the surface. For some specimens, exact determination of the crack-nucleating micro-constituent was not possible, similarly to PM cold work tool steel K390. One sample failed due to a large inclusion located at the surface, however, outside of the gauge length, where the stress amplitude is significantly lower than in the center. Therefore, this specimen had a relatively high lifetime of nearly 10 billion cycles.

The two S–N curves for PM high speed steel S590 and PM cold work tool steel K390 are nearly identical (Fig. 3.123), which was rather surprising since the PM high speed steel had a significant higher hardness (about 8 HRC harder) and considerable lower TRS (2000 MPa lower). Thus, it seems that, similar to the results of the ingot metallurgical high speed steel and cold work tool steel, the ductility and hardness of the matrix is quite irrelevant. The existing crack-nucleating constituents and their size, which affects the fatigue more severely, are more decisive. For this respect it is noted that fatigue experiments with S590 samples heat treated to lower hardness of 62 HRC were performed, which however failed

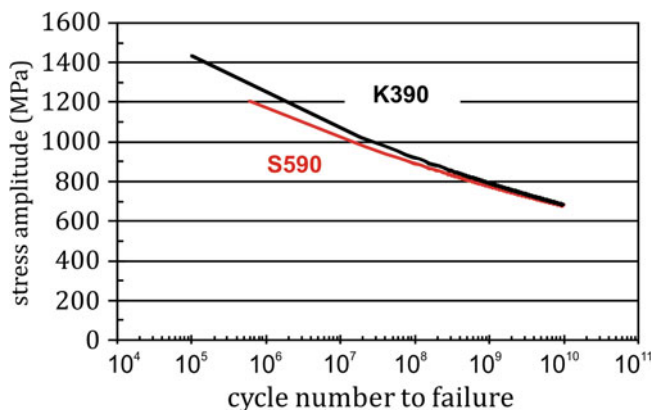


Fig. 3.123 Comparison of S–N curves of PM high speed steel S590 and PM cold work tool steel K390 (low residual stresses at surface; 50% failure probability)

in the same range as the samples with hardness of 67 HRC, which corroborates the statement made just above.

A comparison of the S–N curves of PM high speed steel S590 and the studied ingot metallurgical high speed steel S500 (Fig. 3.124) showed a similar result as obtained for PM and wrought cold work tool steels K390 and K110, respectively. The fatigue strength of the PM steel was nearly twice as high as for the conventional wrought high speed steel, which can be attributed to the presence of larger primary carbides in steels S500 and also S600 that caused fatigue failure.

In PM tool steels K390 and S590, nonmetallic inclusions in the material caused fatigue failure. These inclusions were far smaller than the crack-initiating carbides in the ingot metallurgical tool steels. The considerable data scatter for the PM steel results from the fact that singularities, i.e. the (rare) nonmetallic inclusions, and not (numerous) regular microstructural constituents such as the primary carbides were responsible for fatigue failure.

3.6.2.2 Crack Origins and Fractography

Macroscopically, fracture surfaces of “short”-life specimens revealed a large area with ridges and edges (Fig. 3.125a) and a small area with more flat surface morphology that partly corresponds to the stable fatigue crack growth process (in part it also results from final fracture). In contrast, for long-life specimens a quite flat surface morphology was obtained at the entire cross section (Fig. 3.125b), i.e. flat areas not necessarily indicate stable crack growth; they may also be caused by final fracture. In both cases cracks and ridges point back to the crack origin, which was always at or near the surface.

Table 3.22 Fatigue data of PM high speed steel S590 (low residual stresses at the surface)

Steel S590						
Internal fatigue sample number	Strain at gauge length (mm)	Stress amplitude (MPa)	Cycle number to failure	Crack origin location	Crack nucleating micro-constituent	Diameter of crack origin (μm)
S590-PS4	5.07E-03	1200	5.00E+05	Surface	Nonmetallic inclusion	8
S590-PS36	5.03E-03	1200	6.00E+05		Hole	7
S590-PS37	5.10E-03	1200	1.20E+06		Nonmetallic inclusion	12
S590-PS12	4.69E-03	1100	8.51E+05			26
S590-PS19	4.69E-03	1100	1.75E+06		Unknown	
S590-PS20	4.68E-03	1100	2.59E+06			
S590-PS17	4.69E-03	1100	2.55E+08			
S590-PS8	4.68E-03	1100	4.60E+08		Nonmetallic inclusion	23
S590-PS15	4.23E-03	1000	1.20E+07			
S590-PS30	4.25E-03	1000	1.25E+07		Unknown	
S590-PS14	4.28E-03	1000	3.17E+07		Nonmetallic inclusion	7
S590-PS18	4.26E-03	1000	7.60E+07		Unknown	
S590-PS29	4.27E-03	1000	8.39E+07			
S590-PS16	3.88E-03	900	1.33E+07		Nonmetallic inclusion	8
S590-PS23	3.82E-03	900	1.43E+07			18
S590-PS25	3.82E-03	900	2.01 E+07		Unknown	
S590-PS11	3.82E-03	900	8.25E+09	Surface/ outside of gauge length	Surface crack	
S590-PS7	3.84E-03	900	1.16E+10	Runout specimen		
S590-PS21	3.39E-03	800	2.55E+08	Surface	Nonmetallic inclusion	21
S590-PS31	3.40E-03	800	1.10E+10	Runout specimen		
S590-PS27	3.40E-03	800	1.20E+10			
S590-PS6	2.97E-03	700	1.00E+10			
S590-PS38	2.98E-03	700	1.03E+10			
S590-PS32	2.96E-03	700	1.10E+10			

In most cases, crack origins were nonmetallic inclusions located at the surface or just below (see Figs. 3.126b, 3.128b). Around the surface crack origin a half fish-eye was formed, the border of which cannot be determined exactly. It seems that in the vicinity of the crack nucleating inclusion a granular area (Fig. 3.126b) exists, as it was found for the ingot metallurgical steels. However, the granularity was by far not as pronounced as in fractures of cold work tool steel K110. The EDX spectrum presented in Fig. 3.127 shows a high oxygen content and some Na, Mg, Al, K, Ca, and Zr indicating that it was a typical slag impurity that caused the fatigue failure of this sample. Again Zr was detected, which was also found in some crack origins of steel K390.

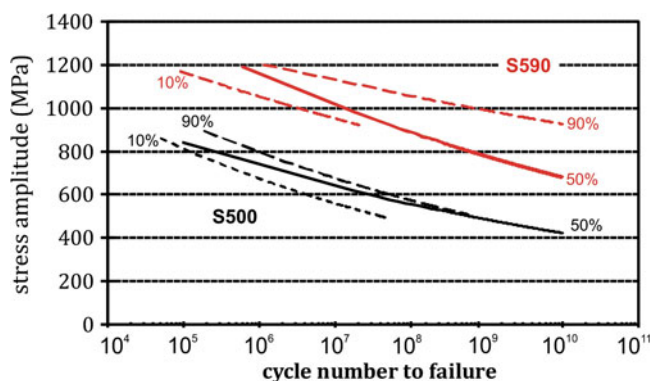


Fig. 3.124 Comparison of S–N curves of PM high speed steel S590 and ingot metallurgical high speed steel S500 (in both cases with low residual stresses at the surface, lines represent 10, 50 and 90% fracture probability, respectively)

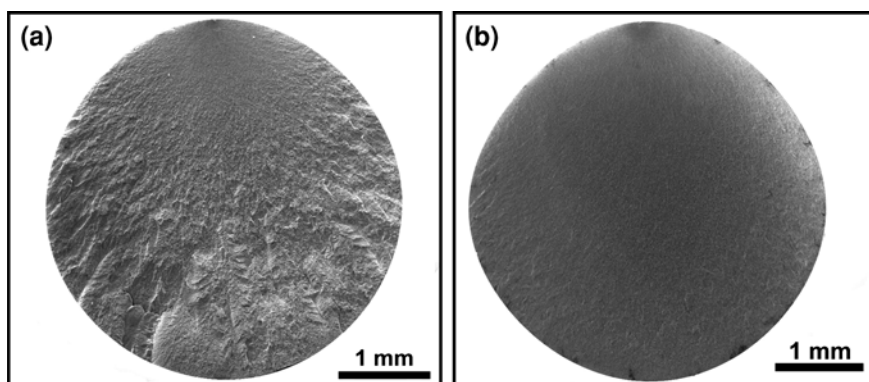


Fig. 3.125 Macroscopic appearance of fractures failed from a nonmetallic inclusion: **a** short life specimen failed at 1100 MPa after 8.51×10^5 cycles (S590-PS12), and **b** long-life specimen failed at 800 MPa after 2.55×10^8 cycles

The granular area is followed by a rather smooth area (half circle in Fig. 3.126a, 3.128a). The size of this zone with low surface roughness decreased with increasing stress amplitude. It probably marks a zone of rather slow crack propagation perpendicular to the loading direction, as intensively discussed in Sect. 3.3.3.5 for steel K110.

Figure 3.128 shows another example of a specimen failed from a surface inclusion, which seemed to be a Ca, K, Mg-oxide or oxides from Cr and V according to the EDX spectra (Fig. 3.129). Here, for this short-run sample a granular area around the crack origin does exist only very closely to the inclusion. Again, a smooth surface around the origin is visible (Fig. 3.128a).

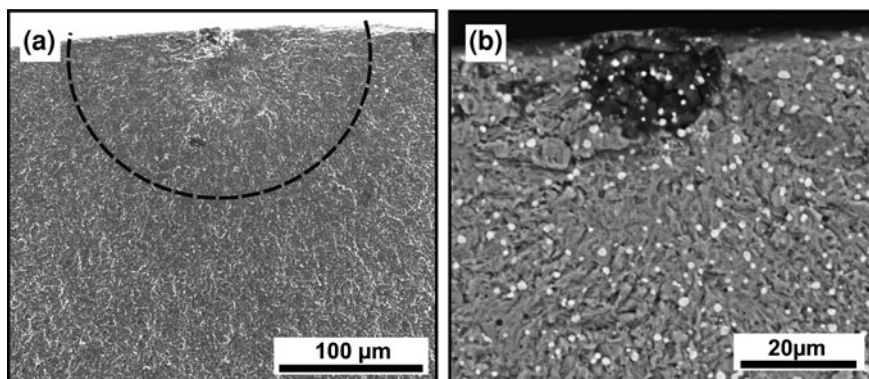


Fig. 3.126 Fractographs of specimen S590-PS21: **a** smooth zone around the crack origin, **b** nonmetallic inclusion (hole that was left behind) and granular area in the vicinity of the inclusion

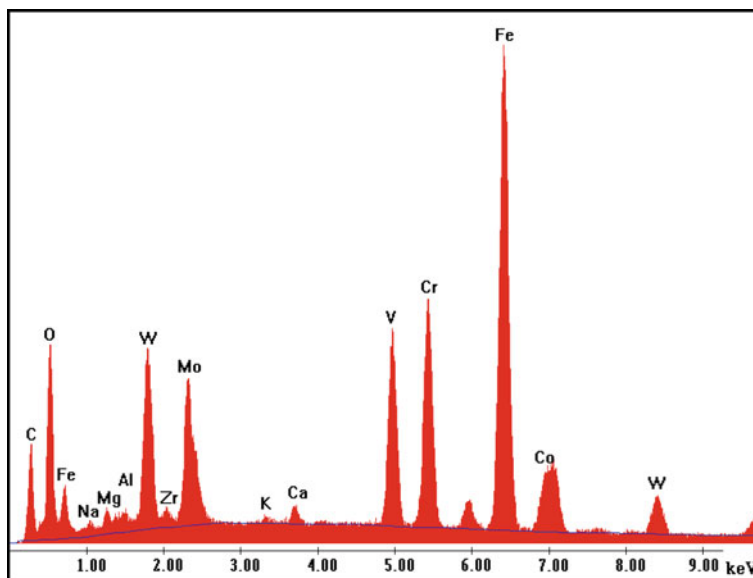


Fig. 3.127 EDX spectrum of nonmetallic inclusion that cause failure of specimen S590-PS21

Figure 3.130 shows fractographs of a “short”-run specimen broken at 1200 MPa after 5.00×10^5 loading cycles. In short-life specimens, characteristic large area with high ridges and edges pointing back to the crack origin can be seen in Fig. 3.130a. The smooth area around the crack origin is quite small due to the short specimen life. However, in Fig. 3.130c a granular area can definitely be seen despite the fact that it is a short-run specimen. The occurrence of the granular area indicates that the inclusion at which the fatigue failure started was too small to

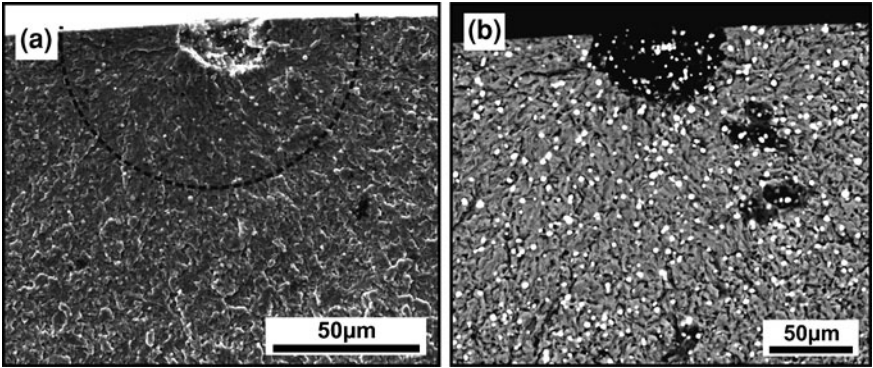


Fig. 3.128 Fractographs of specimen S590-PS12: **a** smooth zone around the crack origin, **b** hole that was left behind from nonmetallic inclusion

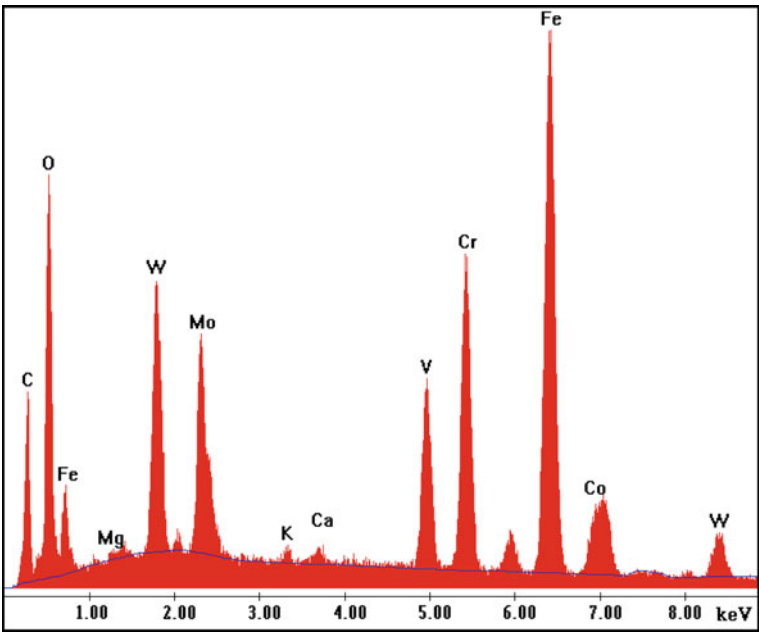


Fig. 3.129 EDX spectrum of the crack nucleating nonmetallic inclusion in specimen S590-PS12

trigger a propagating small crack. Thus, through the formation this granular area the critical crack length was attained, as described for steel K110. EDX analysis (Fig. 3.131) revealed that the inclusion most likely was an aluminum oxide impurity.

The sizes of the crack nucleating nonmetallic inclusions did not show any relationship to the applied stress amplitude, which was in contrast to PM steel

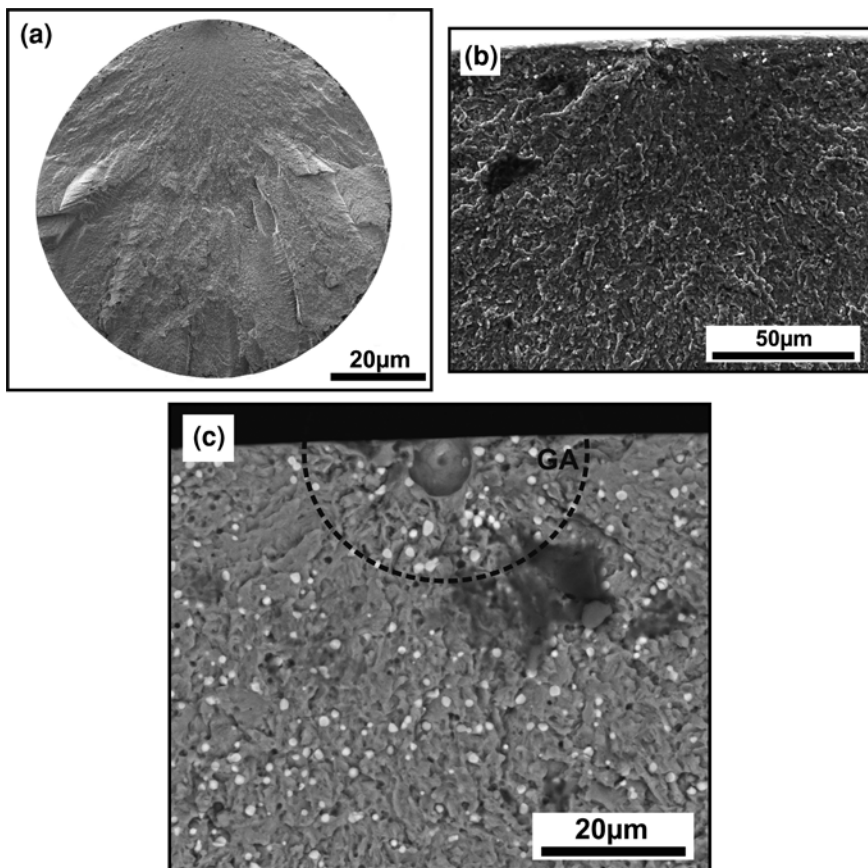


Fig. 3.130 Fractographs of specimen S590-PS4 that failed from an aluminium oxide inclusion at 1200 MPa after 5.00×10^6 cycles: **a** macroscopic appearance of the fracture surface, **b** zone with low surface roughness around the crack origin, **c** Granular area in the vicinity of the crack origin

K390. This fact and the occurrence of granular areas around the smaller crack nucleating micro-constituents indicate that at least for specimens in which the nonmetallic inclusions were too small to trigger a propagating fatigue crack themselves, the process of microcrack formation and coalescence in the vicinity of the higher stress field around the inclusion plays an important role in the fatigue process.

Unfortunately, the exact determination of the size of the granular area was not possible since the borderline to the subsequent crack growth zone was hardly distinguishable. However, the diameters of the crack nucleating inclusions were determined and the stress intensity threshold ΔK_{incl} calculated according to the principles presented in Sect. 3.3.3.7. It turned out to be in the range of 2.4–4.5 MPa $\sqrt{\text{m}}$, which was quite similar to values obtained for near-surface carbide clusters in steel S500 and K110, and also for nonmetallic inclusions in

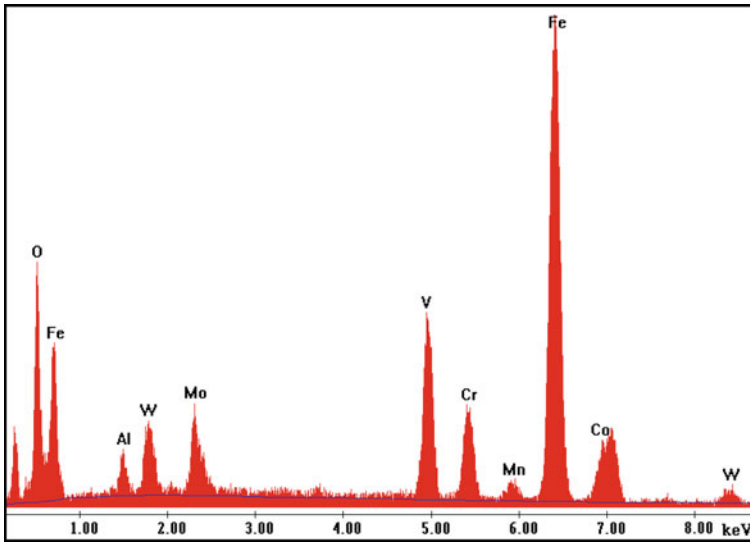


Fig. 3.131 EDX spectrum at the crack origin of specimen S590-PS4

steel K390. The mean ΔK_{incl} value ($3.4 \text{ MPa}\sqrt{\text{m}}$) combined with the largest inclusion size observed as crack origin, i.e. $31 \mu\text{m}$ in diameter, was used for estimation of fatigue endurance strength. A strength of 700 MPa was obtained through this calculation, which was a very good estimate compared to the experimentally obtained fatigue strength at 10^{10} cycles (700 MPa).

3.6.2.3 Fatigue Testing of Specimens with Artificially Introduced Surface Defect

In order to shed more light on the fatigue process, fatigue tests on samples with artificially introduced surface defects were performed. For this, at the surface within the gauge length of two specimens two defects with given size were produced by means of fast ion bombardment with Ga ions (Ga-FIB).

Figure 3.132 shows the point at the fatigue specimen surface where the defect was introduced and the structure of the hole that resulted from the ion bombardment. The defect had a diameter of about $10\text{--}12 \mu\text{m}$, thus, corresponding to nonmetallic inclusion sizes observed as crack origins in steel S590.

Fatigue testing of the two samples revealed that one of these specimens failed from the introduced defect within the scatter range of the S–N curve of steel S590 (Fig. 3.133). In the other case a large nonmetallic inclusion caused fatigue failure, simply because it was larger than the introduced defect. Figure 3.134 shows the observed fractographs for the sample failed from the artificial defect, which can be recognized very clearly. Note that the hole does not appear dark as it was the case for holes left behind from decohesion of nonmetallic inclusions, which has been

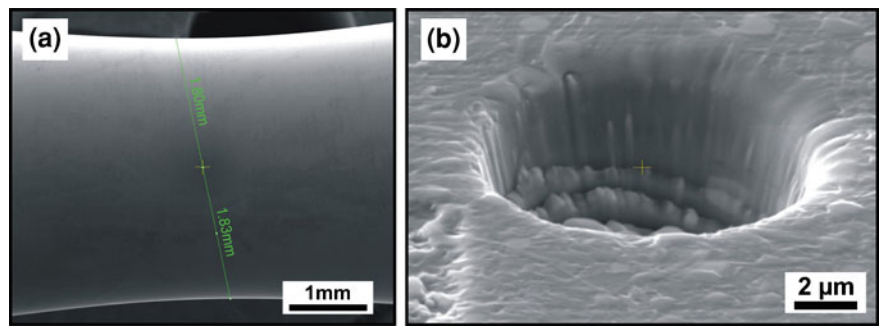


Fig. 3.132 **a** Point at the gauge length where the artificial defect was introduced by FIB and **b** structure of the produced hole

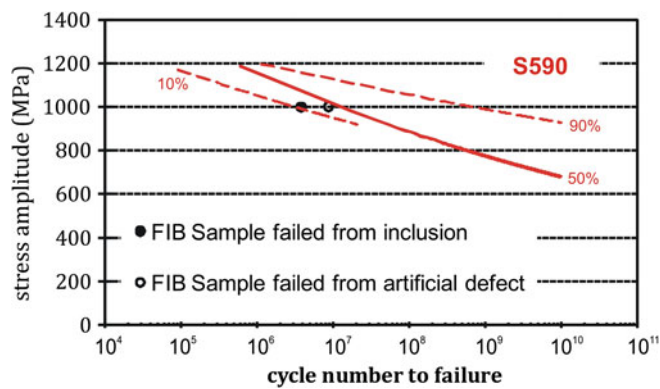


Fig. 3.133 S-N curve of PM high speed steel S590 and FIB samples with an artificial defect (solid and the two dashed lines indicate 50%, 10 and 90% fracture probability, respectively)

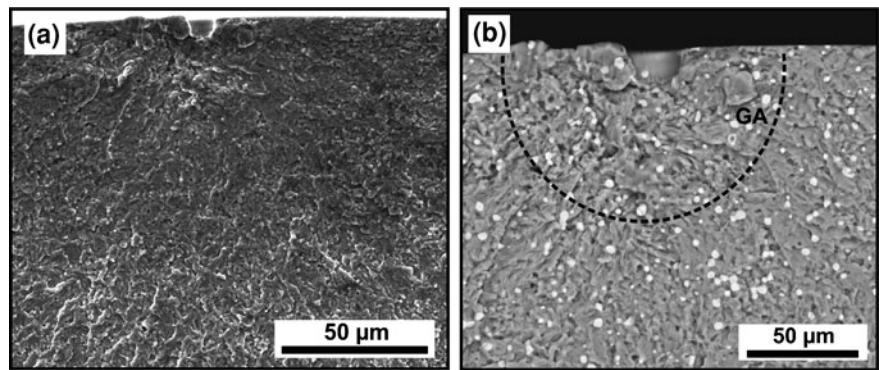


Fig. 3.134 Fractographs of specimen with artificial defect introduced by FIB: **a** zone with low surface roughness around the crack origin, **b** granular area in the vicinity of the crack origin, i.e. the introduced defect

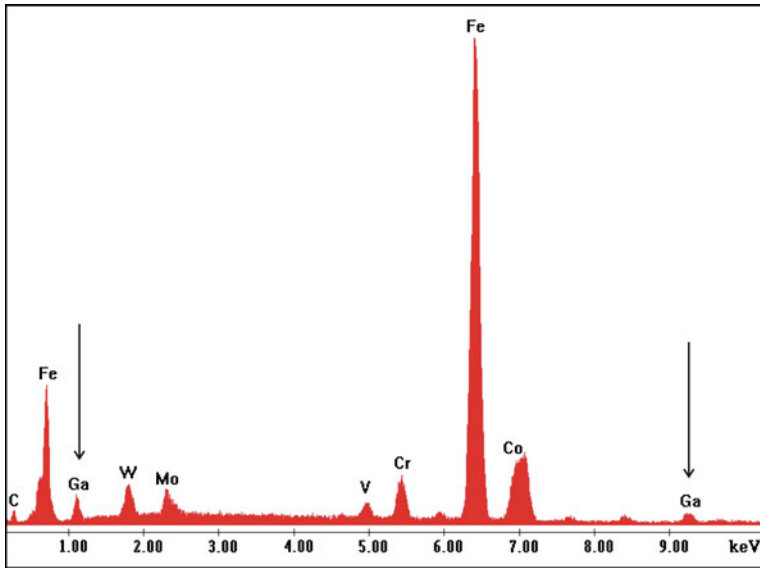


Fig. 3.135 EDX spectrum at the *hole* showing implemented Ga (marked with *arrows*)

presented above. EDX analysis of the hole proved that this hole definitely resulted from the FIB treatment since Ga was detected there from implementation during the bombardment (Fig. 3.135).

Furthermore, in Fig. 3.134b a granular area can be detected around the introduced defect, which indicates that the defect itself was too small to trigger a propagating fatigue crack. Thus, microcrack formation and coalescence within the granular seem to have occurred. The fact that the specimen failed due to this rather small defect indicates further that another defect such as a nonmetallic inclusions was not located at the surface. This underlines that the nonmetallic inclusions responsible for fatigue failure of S590 and very likely also for the PM cold work tool steel K390 represent in fact material singularities, i.e. very rare phenomena.

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Chapter 4

Summary and Outlook

4.1 Summary

This work describes investigations into the gigacycle fatigue behavior of tool steels, which were accomplished in the framework of a joint research project (FWF project P17650-N02) of Vienna University of Technology (Institute of Chemical Technologies and Analytics; Prof. Dr. H. Danninger) and University of Vienna (Faculty of Physics; Prof. Dr. B. Weiss). The principal researchers of this project were Dipl.-Ing. A. Betzwar-Kotas (University of Vienna) and the author of this thesis (Vienna University of Technology).

Tool steels, which are very hard steels with relatively low ductility, are difficult to study by conventional fatigue testing routines, and especially testing to loading cycle numbers $>10^6$ is time consuming. The ultrasonic resonance fatigue test method used here enabled testing of steel specimens with hardness levels up to 68 HRC and maximum loading cycle numbers of typically 10^{10} , which is virtually impossible with standard fatigue test procedures. At such high N, effects can be observed that are not, or at least not as clearly, revealed by static or standard fatigue testing.

The first part of the project comprised the installation of an optimized and fully computerized ultrasonic frequency fatigue testing system. A new generator was acquired and adapted for the ultrasonic fatigue testing. Alike the acoustic setup was developed and adjusted to the needs given by the fact that materials with very high hardness and strength are aimed to be tested. This included the appropriate design of the acoustic horn, offering the required displacement wave amplification, but also the design of fatigue specimens for successful testing results. Especially the latter was a time consuming part of the work since the specimens have to withstand the high loads that have to be applied to cause failure of these materials, i.e. fracture should occur within the gauge length and not at other locations of the specimen, as e.g. at the screw. Another important issue was the specimen cooling system, since it turned out that considerable heat was generated through damping effects within the samples during application of the relatively high loading

amplitudes of up to 1,400 MPa. Thus, the samples are cooled by a liquid non-corrosive coolant that is splashed onto the specimens, from which it drops down into a collecting vessel below. From this vessel the coolant is recovered and re-circulated. Appropriate adjustment of the way the coolant is applied on the specimen prevents erosion of the specimen through cavitation. Usage of the cooling system applied provided a rather constant sample temperature of below 30 °C.

One major aim of the project was to identify the role of potential crack-initiating microstructural constituents, i.e. singularities, in the tool steels. Here, the role of slag inclusions is intensely discussed in the literature. However, it turned out that for tool steels produced by ingot metallurgy the primary carbides and carbide clusters are the lifetime-limiting constituents. e.g. in wrought medium-carbon high chromium cold work tool steel primary alloy carbides or alloy carbide clusters of type $(\text{Cr,Fe})_7\text{C}_3$ gave rise to fatigue cracks (Fig. 4.1a), cracking of the carbides themselves rather than decohesion being the initial effect. The same holds for wrought high speed steels for which fatigue failure occurred predominantly from to primary alloy carbides of type $(\text{Fe,Mo,W})_6\text{C}$ and alloy carbide clusters (Fig. 4.1b). Here, however, in some cases crack initiation at non-metallic inclusions was also observed (Fig. 4.2).

In contrast, the powder metallurgical grades showed predominantly fatigue failure from (slag) impurities, thus, definitely failed due to material singularities, since the primary carbides were too small to nucleate fatigue cracks even in the very high cycle regime. Figure 4.3 shows a typical fractograph obtained for PM high speed steel, for which a non-metallic inclusion caused fatigue failure. Here, the inclusion broke out during cyclic loading, leaving a hole behind.

Thus, by this it was also shown that fatigue testing at low stress amplitudes (however “low” only for these high strength materials!) and extremely long lives is well suited for detection of material micro-constituents potentially causing failure of the tool steel material and in particular for detecting the last few critical inclusions in steels.

The powder metallurgical (PM) grades proved their superior fatigue behavior compared to the ingot metallurgical (IM) tool steel variants (Fig. 4.4). At 10^{10} loading cycles the fatigue endurance strength of nearly residual stress-free wrought tool steels was about 400 MPa, while the PM tool steels revealed a fatigue endurance strength of about 700 MPa. Thus, PM tool steels attained nearly twice the fatigue strength of the conventional, i.e. cast and wrought, tool steels. With higher stress amplitudes this gap increased even more, since the slope of the S–N curve for the PM steels is steeper than for the IM steels. Surprisingly, it showed that the composition of the steels is much less relevant for the fatigue behavior than the manufacturing route: for ingot metallurgy Cr alloyed cold work tool steel and two grades of high speed steels virtually the same S–N curve was obtained; equally, for V-alloyed PM cold work tool steel and PM HSS the same S–N curve was recorded, but at significantly higher stress level than for the IM steels (see Fig. 3.1).

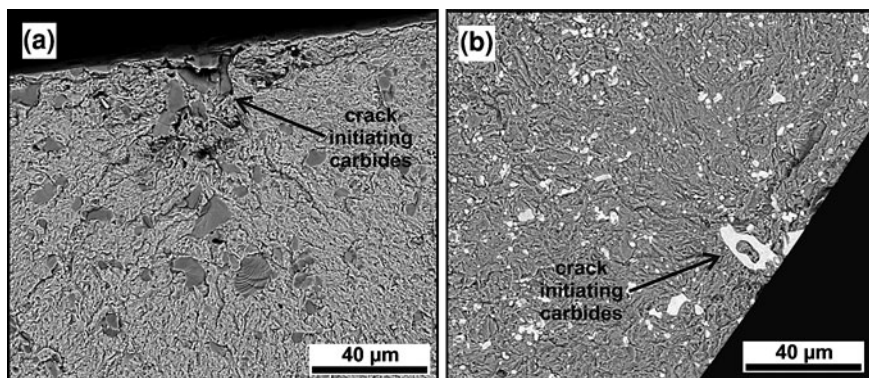


Fig. 4.1 Representative fatigue crack origins for **a** medium-carbon high-chromium cold work tool steels (AISI D2): crack nucleating chromium carbides $(\text{Fe,Cr})_7\text{C}_3$; **b** Mo-based AISI M42 high speed steel: crack nucleating eta-carbides $(\text{Fe,Mo,W})_6\text{C}$

Fig. 4.2 Crack nucleating non-metallic inclusion causing fatigue failure in the very high cycle regime in AISI M42 high speed steel

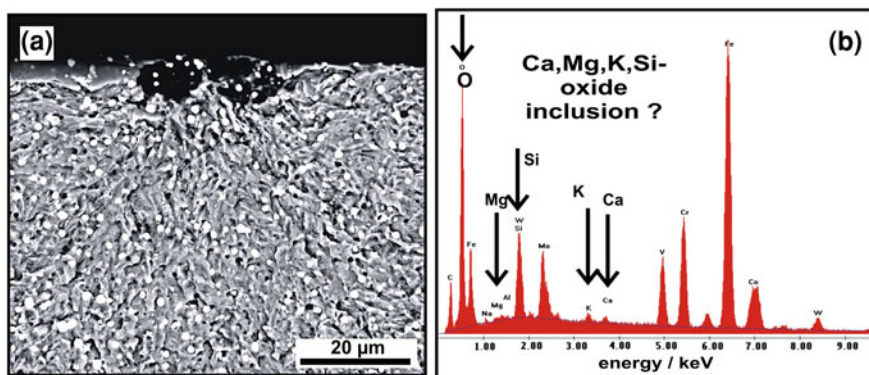
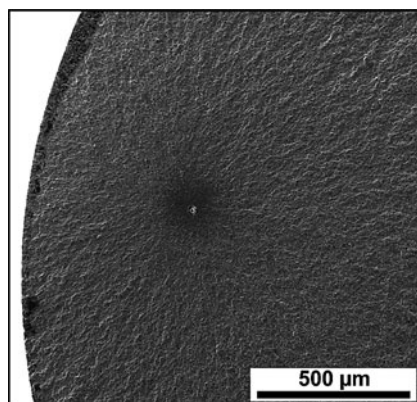
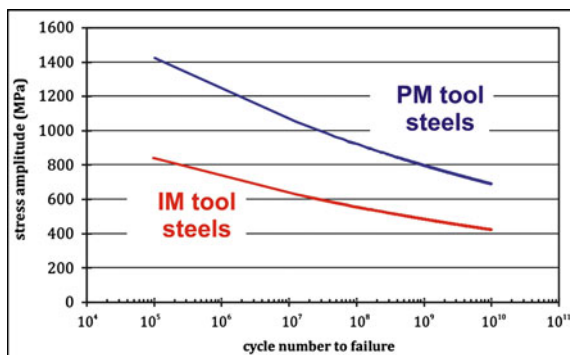


Fig. 4.3 Nonmetallic inclusion causes fatigue failure of PM high speed steel (Böhler grade S590): **a** hole left behind from inclusion that broke out during cyclic loading, **b** EDX spectrum at crack origin indicating an oxide inclusion was located there

Fig. 4.4 Schematic comparison of S–N curves obtained for powder metallurgical (PM) and ingot metallurgical (IM) tool steels



Another aim of the project was to produce comprehensive fatigue data up to the very high cycle regime in order to supply input to the current scientific discussion about the shape of the S–N curves of tool steels and to the very fundamental question whether a real fatigue limit does exist for such high strength steels or not. The obtained S–N curves showed continuously decreasing fatigue strength with increasing number of cycles. A multistage shape such as “twofold” or “stepwise” curve, which can be found in the literature for high strength bearing and spring steels, was not obtained. This observation agrees with findings by Murakami et al. who noted for the fatigue behavior of bearing steels that under tension–compression testing condition such multistage curves cannot be expected to be observed. Furthermore, the performed experiments also showed quite clearly that a real fatigue limit was not attained up to 10 billion cycles, strongly indicating that a real fatigue limit *does not* exist for the investigated tool steels.

Furthermore, the influence of compressive surface residual stresses—which was investigated in detail on the wrought medium-carbon high-chromium cold work tool steel grade—was found to be very significant and affected both the observed fatigue strength and the location of the crack origin. The fatigue strength of specimens with high compressive residual stresses at the surface was more than 200 MPa higher than of those with low residual stresses (Fig. 4.5). When high compressive residual stresses (about -800 MPa) existed at the specimen surface, fatigue cracks originated in the interior at large primary carbide clusters forming so-called fish-eye patterns at the fracture surface (Fig. 4.5a, b). If these residual stresses were lower, cracks started from carbides or carbide clusters located at or just below the specimen surface, which was referred to as “at/near-surface failure” in order to differentiate from the usual surface failures resulting from machining defects, persistent slip bands, corrosion pits, etc.

Furthermore, a very surprising relaxation of the residual stresses through gigacycle loading was observed. The initially high compressive stresses at the surfaces of fatigue test specimens—introduced by surface preparation—were lowered to about one half of the initial value during testing to 10^{10} cycles (Fig. 4.6), likely due to the cycling loading. However, the mechanisms responsible for this relaxation are currently unknown, since the common processes causing relaxation of residual stresses do not apply here.

Fig. 4.5 Effect of high compressive residual stresses at the surface on the fatigue response of medium-carbon high-chromium cold work tool steel (AISI D2):

a Internal fish-eye failure from carbide cluster, **b** crack nucleating carbide cluster

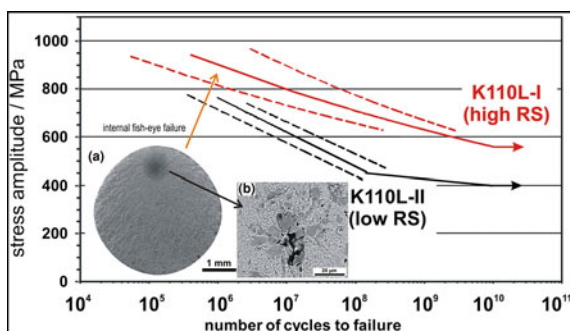
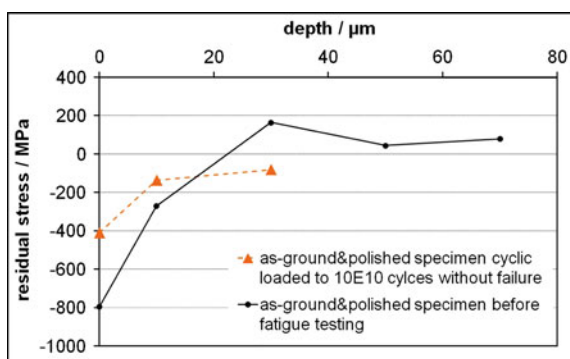


Fig. 4.6 Residual stress profiles at specimens before and after cyclic loading to $N_{\max} = 10^{10}$ cycles without failure



The observed residual stress relaxation resulted in a transition from internal (see Fig. 4.5a, b) to at/near-surface crack origins (primary carbides or carbide clusters located at or just below the surface, see Fig. 4.1a) with increasing cycle number to failure, which contradicts the usual opinion that internal crack initiation becomes more probable at higher N .

A theoretical estimation of the crack origin location was made by a simple model calculating the local fatigue strength along the specimen cross section, which supported the experimental findings.

Statistical considerations concerning the location of the crack origins based on a concept proposed by [1] were made for wrought cold work tool steel, which showed that for this steel, fatigue failure should occur from one of the numerous carbides and carbide clusters located at or just below the surface, at least in absence of high residual stresses, which prediction was definitely verified by the experimental results. This statement holds also for wrought high speed steels, in contrast to PM tool steels for which singularities—e.g. very rare (slag) inclusions—caused fatigue failure, however, also they were generally located at the specimen surface.

A further point of investigation was whether the anisotropic arrangement of the primary carbides in the ingot metallurgical tool steels does have an influence on the fatigue behavior in the two directions, perpendicular and parallel to the rolling direction. This was investigated on the wrought medium-carbon high-chromium

Fig. 4.7 Influence of anisotropic arrangement of primary carbides in ingot metallurgy tool steel (AISI D2 cold work tool steel) on the fatigue response

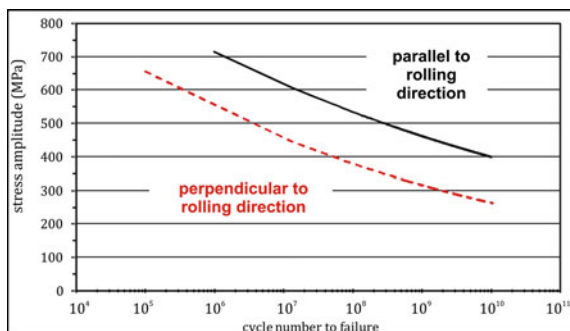
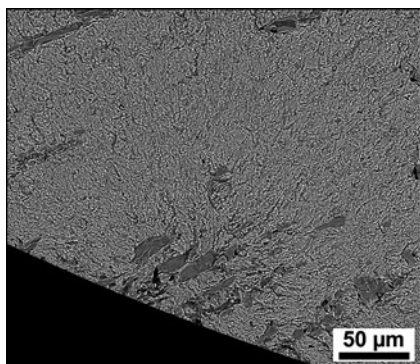


Fig. 4.8 Fatigue crack nucleation at large primary carbides in AISI D2 cold work tool steel samples with axis perpendicular to the rolling direction



cold work tool steel grade, since in this steel the carbide bands were particularly pronounced, and also the carbides themselves were both elongated and oriented (unlike in HSS in which there were also carbide bands but the carbides themselves were more or less equiaxed). Indeed, a significant difference of the fatigue response was observed for the two orientations: Specimens with axis parallel to the rolling directions showed a fatigue strength that was about 150 MPa higher over the entire tested cycle number range compared to the specimens with axis perpendicular to the rolling direction (Fig. 4.7). Thus, the larger dimensions of the primary carbides in the longitudinal directions of the rolled bar (i.e. the plane of fracture surface in the samples with axis transverse to the rolling direction) caused the significantly inferior fatigue response of the transverse specimens. Figure 4.8 shows such a large carbide cluster arrangement comprising several large elongated carbides that caused fatigue failure of the sample.

Furthermore, the macroscopic appearance of the obtained fracture surfaces showed to be completely different for the two directions. While for the specimens with axis parallel to the rolling direction a rather flat surface morphology with ridges and cracks pointing back to the crack origin was observed (Fig. 4.9a), the transverse samples revealed numerous cracks oriented parallel to the primary carbide bands on a rough fracture surface (Fig. 4.9b).

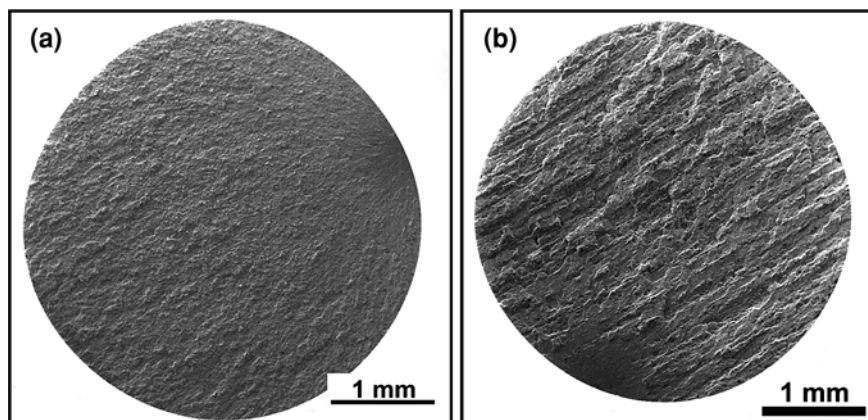


Fig. 4.9 Macroscopic fracture surface of AISI D2 cold work tool steel: **a** axis parallel and **b** axis perpendicular to the rolling direction of the initial steel bar

Detailed investigation of the obtained fracture surfaces was performed by means of light microscopy and scanning electron microscopy. Fractographic analysis of crack growth zones close to the origin revealed fish-eye or half fish-eye pattern around the origin for internal and at-near-surface failures, respectively. Depending on the steel type, several zones of growth were evaluated within this fish-eye pattern. Most important was the detection of a granular area in the vicinity of the crack origins (see Fig. 4.5b), which was observed at least at lower stress amplitudes. It was shown that the formation of this zone is a prerequisite for the formation of a short propagating fatigue crack, which could be explained by a model introduced by the Japanese researchers [2]. They explained the microcrack formation in the enhanced stress-field of large non-metallic inclusions, or in the present case primary carbides, by decohesion of small carbides from the matrix, which forms miniature cracks which then subsequently coalesce and thus grow until the critical crack length for a propagating crack is attained. It seems that this model was also well applicable here for the tool steels studied.

The areas of the different crack growth stages observed were evaluated quantitatively, in order to obtain further information about the fatigue process. Threshold stress intensity factors were estimated from the size data for the individual fatigue crack growth stages obtained through the quantitative fractographic evaluation at the fractographs. The fatigue thresholds were then used for estimation of fatigue endurance strength. The obtained values turned out to be quite good estimates for the experimentally determined strengths at 10 billion cycles.

Concluding, it was shown by this work that tool steels do not exhibit a real fatigue limit and reveal continuously decreasing S–N curve in tension–compression mode. Furthermore, this study showed the significant effect of compressive residual stresses at the surface on the fatigue response, i.e. residual stresses of about –800 MPa improved the fatigue endurance strength in the tested cycle number range (10^5 – 10^{10} loading cycles) by about 200 MPa. However, surprisingly a partial

Table 4.1 Summary of the obtained fatigue endurance strength levels for the investigated tool steels at 10 billion cycles under fully reversed tension–compression loading

Tool steel				Fatigue endurance strength at 10^{10} cycles (MPa)
Type	Grade	Production pathway		
Cold work tool steels	AISI D2 DIN 1.2379 (Böhler K110) (high Cr–medium C)	Ingot metallurgy	High compressive residual stresses at the surface (parallel to rolling direction of the initial steel bar)	580
			Low residual stresses at the surface (parallel to rolling direction of the initial steel bar)	400
			Transverse to rolling direction of the initial steel bar (low residual stresses)	250
High speed steels	Böhler K390 (high V–high C)	Powder metallurgy	Low residual stresses at the surface	700
	AISI M42 DIN HS 2-10-1-8 (Böhler S500) (Mo-rich, high Co)	Ingot metallurgy		450
	AISI M2 DIN HS 6-5-2 (Böhler S600) (Mo-W-rich)			450
	Böhler S590 (W-Mo based, high Co)	Powder metallurgy		700

relaxation of the residual stresses through cyclic loading was observed influencing the location of the crack origin. The relaxation mechanism is currently unknown.

Fatigue testing up to a very high number of cycles ($N_{\max} = 10^{10}$) at low stresses employing the ultrasonic frequency resonance testing method turned out to be capable for identifying crack nucleating micro-constituents in the material, whether singularities such as nonmetallic inclusions or large carbide clusters, or important microstructural features such as primary carbides. Comparison between ingot metallurgy and powder metallurgy steels showed that the latter are definitely superior in gigacycle fatigue, regardless if cold work or high speed steels are studied. A summary of the obtained fatigue endurance strength data at 10^{10} loading cycles are presented for the investigated steels in Table 4.1.

4.2 Outlook

For future work, three main topics might be of high interest:

- First, ultrasonic resonance fatigue testing offers the chance to find also very rare defects within a material, especially in tool steels. This might be the basis for implementing this method as standard procedure in the development process of tool steels, in order to find and analyze any potentially detrimental defects in the steels at reasonable time and costs, since ultrasonic fatigue testing is a very fast method and enables investigation of reasonable volumes. An appropriate strategy in that respect could be the testing of a larger number of samples at e.g. two selected stress amplitudes in order to achieve reasonable statistical results about the crack initiating constituents responsible for failure.
- A more detailed investigation of the process through which the granular area is formed, which seems to be essential for the nucleation of a propagating fatigue crack, might be of interest in order to enable explaining the occurrence of fatigue failure in the gigacycle fatigue regime. Comprehensive systematic evaluation of crack initiation could be helpful in this respect, to be performed on samples with artificial defects introduced by means of FIB or by electric discharge machining, as it was briefly described in this thesis.
- A further point of interest might be the effect of residual stresses on the fatigue behavior, which was shown in this work to be considerable. However, comprehensive investigations are required, especially concerning the relaxation process that seems to occur during cyclic loading up to very high cycle numbers at low loading amplitudes. This is of high importance since in practice, residual stresses always exists in a tool, and especially for tools which operate at lower loads and high cycle numbers, the cyclic degradation of these residual stresses might be a decisive factor for tool life. However, also for tool steels applied as engine parts or other automotive components the fatigue behavior up to very high cycles combined with the influence of residual stresses can be assumed to be a very important issue.
- At last, fatigue testing up to the gigacycle regime of even harder tool materials such as hardmetals might be an interesting research field, using the knowledge about fatigue behavior of hard tool steels acquired in this work and also the experimental routines developed. In this respect, the effect of the fine microstructure and the cemented carbides (geometries, types) on the fatigue failure are interesting, and the possibility of the ultrasonic fatigue testing method to support the material development, i.e. identifying potential defects such as undesired carbide geometries and sizes. Also here, the effect of residual stresses could be of high relevance.

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