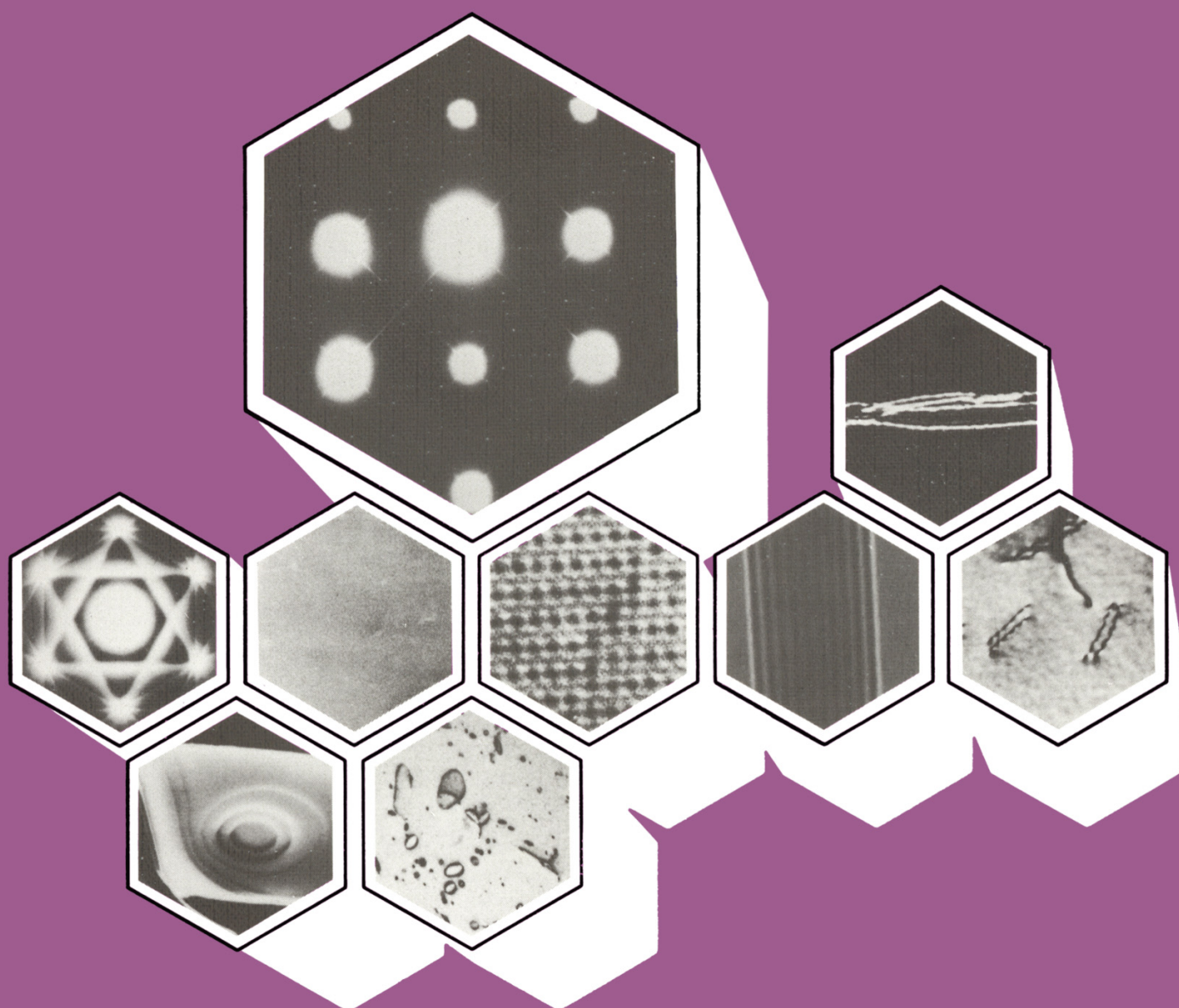


**PRACTICAL ELECTRON
MICROSCOPY
IN
MATERIALS SCIENCE**

2 Monograph Two **Electron Diffraction in the Electron Microscope**

J W Edington



Macmillan Education

ELECTRON DIFFRACTION IN THE ELECTRON MICROSCOPE

Already published in this series

1. The Operation and Calibration of the Electron Microscope

In preparation

3. Interpretation of Transmission Electron Micrographs
4. Typical Electron Microscope Investigations

Philips Technical Library

*Monographs in Practical Electron Microscopy
in Materials Science*

2

ELECTRON DIFFRACTION IN THE ELECTRON MICROSCOPE

J. W. EDINGTON

Department of Metallurgy and Materials Science, University of Cambridge, Cambridge, England

M

© N.V. Philips' Gloeilampenfabrieken, Eindhoven, 1975

All rights reserved. No part of this publication
may be reproduced or transmitted, in any form
or by any means, without permission

This book is sold subject to the standard
conditions of the Net Book Agreement

SBN 333 18292 8

ISBN 978-0-333-18292-5 ISBN 978-1-349-02595-4 (eBook)
DOI 10.1007/978-1-349-02595-4

First published 1975 by
THE MACMILLAN PRESS LTD
London and Basingstoke
Associated companies in New York, Dublin,
Melbourne, Johannesburg and Madras



PHILIPS

Trademarks of Philips' Gloeilampenfabrieken

Filmset at The Universities Press, Belfast, Northern Ireland

PREFACE

This is the second of a series of monographs on electron microscopy aimed at users of the equipment. They are written both as texts and sources of reference emphasising the applications of electron microscopy to the characterisation of materials.

In some places the author has referred the reader to material appearing in other monographs of the series. The following title has already been published:

1. *The Operation and Calibration of the Electron Microscope*

and the titles in preparation are:

3. *Interpretation of Transmission Electron Micrographs*

4. *Typical Electron Microscope Investigations*

ACKNOWLEDGEMENTS

It is a pleasure to acknowledge Drs L. M. Clareborough, C. P. Cutler, P. Humble, B. Kear, K. N. Melton, P. H. Pumphrey and M. N. Thompson for their useful comments on the manuscript. I would also like to acknowledge the many useful discussions over a number of years with all members of my research group in Cambridge. These formed the basis for many of the sections of this series of monographs. In addition, particular thanks are due to Messrs R. H. Bricknell, J. L. Henshall, D. A. Porter and Dr. D. B. Williams for their help in proof-reading the manuscript. I am also grateful to Professor R. W. K. Honeycombe for his continued advice and encouragement.

I would like to thank the large number of research workers who have provided me with illustrations and tables. They are acknowledged individually in the relevant figure and table captions. I am also grateful to the following journals and publishers for permission to reproduce a number of photographs, tables and figures: *Acta Metallurgica*, *Acta Crystallographica*, *Cambridge Review*, Adam Hilger Ltd, *Journal of Macromolecular Science*, *Journal of Materials Science*, *Journal of Microscopy*, *Journal of Physics F*, *Journal of Physics Radium*, *Journal of the Physical Society of Japan*, McGraw-Hill, *Metallurgical Reviews*, *Metallurgical Transactions*, *Metal Science Journal*, North-Holland Publishing Co., *Philosophical Magazine*, *Physica Status Solidi*, *Proceedings of the Royal Society*, *Transactions Quarterly of the American Society for Metals*, University of California Press.

This series of monographs has been published with the support of the Philips Company of The Netherlands. In particular I would like to thank Mr J. Fay of that organisation for his unfailing enthusiasm and encouragement.

CONTENTS

Preface

Acknowledgements

2. ELECTRON DIFFRACTION IN THE ELECTRON MICROSCOPE	1
PART I. INTRODUCTION TO ELECTRON DIFFRACTION	1
2.1 GENERAL INTRODUCTION	1
2.2 A GEOMETRICAL APPROACH TO ELECTRON DIFFRACTION FROM A CRYSTALLINE SPECIMEN	1
2.2.1 Scattering by an Individual Atom	1
2.2.2 Scattering by a Crystal	2
2.2.2.1 The Bragg law	3
2.2.2.2 The Laue conditions	3
2.2.3 The Reciprocal Lattice	4
2.2.4 The Reciprocal Lattice and Diffraction by a Single Crystal	5
2.3 A QUANTITATIVE APPROACH TO DIFFRACTION FROM A CRYSTALLINE SPECIMEN	6
2.3.1 The Structure Factor	6
2.3.2 The Intensity Distribution in Reciprocal Space	8
2.4 THE RECIPROCAL LATTICE AND TRANSMISSION ELECTRON DIFFRACTION IN THE ELECTRON MICROSCOPE	9
2.5 SUMMARY	10
PART II. ELECTRON DIFFRACTION PATTERNS IN THE ELECTRON MICROSCOPE	11
2.6 TYPES OF DIFFRACTION PATTERNS	11
2.6.1 Ring Patterns	11
2.6.2 Spot Patterns	11
2.6.3 Kikuchi Patterns	11
2.7 INDEXING DIFFRACTION PATTERNS	14
2.7.1 Ring Patterns	14
2.7.2 Spot Patterns	14
2.7.2.1 Simple patterns—spots are produced by planes in one zone	15
2.7.2.2 Complicated patterns—spots arising from different zones	18
2.7.2.3 Imperfect patterns	19
2.7.3 Kikuchi Patterns	20
2.7.3.1 Simple patterns—Kikuchi lines in the same zone	20
2.7.3.2 Complex patterns—Kikuchi lines from different zones	22
2.7.3.3 Kikuchi maps	25
2.7.3.4 Combinations of Kikuchi lines and spots	25
2.7.4 Summary of Procedures for Indexing Single-crystal Patterns	25
2.7.4.1 Spot patterns—identity of material known	25
2.7.4.2 Spot patterns—identity of material unknown	26
2.7.4.3 Kikuchi lines—identity of material known	26
2.7.4.4 Kikuchi lines—identity of material unknown	26
2.7.5 Accuracy of the Determination of B	26
2.7.6 Practical Comparison Between Methods of Analysing Diffraction Patterns	27
2.7.6.1 Consideration of further sources of error in determining B	27
2.7.7 Limitations in Usefulness of the Various Methods of Determination of B	27
2.8 UNIQUENESS IN INDEXING DIFFRACTION PATTERNS	27
2.8.1 The 180° Ambiguity	27
2.8.2 Coincidence Ambiguity	29

PART III. USES OF DIFFRACTION PATTERNS FROM SINGLE CRYSTALS: THE BASIC DIFFRACTION PATTERN		30
2.9	GENERAL INTRODUCTION	30
2.10	SPECIMEN TILTING EXPERIMENTS	30
2.10.1	Tilting to Specific Orientations	30
2.10.1.1	A succession of two-beam conditions	30
2.10.1.2	Stereo microscopy	30
2.10.1.3	From one specific orientation to another	31
2.10.2	Control of the Sign and Value of s	33
2.10.3	Determination of the Sign of B	33
2.11	ORIENTATION RELATIONSHIP DETERMINATION	35
2.11.1	Parallel Directions and Planes	38
2.11.1.1	All precipitates have the same orientation relationship	38
2.11.1.2	Several variants of one orientation relationship or several phases present	40
2.11.2	Axis/angle Pairs	42
2.12	TWINNING	43
2.12.1	Origin of the Extra Spots	43
2.12.2	The Distribution of Twin Spots in the Diffraction Pattern	44
2.12.2.1	Cubic crystal structures	44
2.12.2.2	Hexagonal crystal structures	47
2.13	SECOND PHASES	51
2.13.1	Identification	51
2.13.2	Determination of Crystal System	54
2.13.3	Determination of Differences in Lattice Parameter Between Precipitate and Matrix	54
2.13.4	Detection of the Initial Stages of Phase Transformations	54
2.14	CRYSTALLOGRAPHIC INFORMATION	55
2.14.1	Habit Plane Determination	55
2.14.1.1	Defect intersects the foil surface	55
2.14.1.2	Defect contained within the foil	57
2.14.2	Line Direction u of a Dislocation	57
PART IV. USES OF DIFFRACTION PATTERNS FROM SINGLE CRYSTALS: THE FINE STRUCTURE IN THE PATTERN		59
2.15	GENERAL INTRODUCTION	59
2.16	EXTRA SPOTS	59
2.16.1	Double Diffraction	59
2.16.1.1	The distribution of spots during double diffraction	60
2.16.1.2	Detection of double diffraction in the electron microscope	61
2.16.2	Long-range Order	61
2.16.2.1	The distribution of spots in the electron diffraction pattern	61
2.16.2.2	The detection of long-range order in the electron microscope	61
2.16.2.3	Quantitative information available	61
2.17	SPOT SPLITTING AND SATELLITE SPOTS	61
2.17.1	Planar Crystal Defects	61
2.17.1.1	The general form of satellites	63
2.17.1.2	The detection of satellites	63
2.17.2	Periodic/modulated Structures	63
2.17.2.1	Spinodal decomposition	63
2.17.2.2	Regular arrays of antiphase domain boundaries	65
2.17.2.3	Regular dislocation array	66
2.17.2.4	Detection of satellites	67
2.17.3	Magnetic Domains	67

<i>Contents</i>		ix
2.18	STREAKS	67
2.18.1	Shape Effects	67
2.18.1.1	Precipitates	67
2.18.1.2	Stacking faults	70
2.18.1.3	Twins	70
2.18.1.4	Dislocations	70
2.18.1.5	Surface films	70
2.18.2	Elastic Strain Effects	71
2.18.3	Detection of Streaks	
2.19	DIFFUSE SCATTERING EFFECTS	72
2.19.1	Streaks in Reciprocal Space	72
2.19.2	Sheets in Reciprocal Space	73
2.19.2.1	Thermal diffuse scattering	74
2.19.2.2	β - ω phase mixtures	75
2.19.2.3	MC carbides	75
2:	<i>Recommended Reading</i>	75
2:	<i>References</i>	75
APPENDIX 1. BASIC CRYSTALLOGRAPHY		79
A1.1	INTRODUCTION	79
A1.2	INDEXING PLANES	80
A1.3	INDEXING LATTICE DIRECTIONS	80
A1.4	PLANE NORMALS	81
A1.5	ZONES AND THE ZONE LAW	81
A1.6	STEREOGRAPHIC PROJECTION	82
A1.7	USEFUL MANIPULATIONS WITH THE STEREOGRAPHIC PROJECTION AND WULFF NET	85
A1.8	USEFUL CRYSTALLOGRAPHIC FORMULAE FOR VARIOUS CRYSTAL STRUCTURES	87
	<i>Appendix 1: Recommended Reading</i>	87
	<i>Appendix 1: References</i>	87
APPENDIX 2. CRYSTALLOGRAPHIC TECHNIQUES FOR THE INTERPRETATION OF TRANSMISSION ELECTRON MICROGRAPHS OF MATERIALS WITH HEXAGONAL CRYSTAL STRUCTURE		90
A2.1	INTRODUCTION	90
A2.2	CRYSTALLOGRAPHIC RELATIONSHIPS FOR THE HEXAGONAL LATTICE	90
A2.2.1	Angles between Two Directions, θ	91
A2.2.2	Indices $[defg]$ of the Normal to the Plane $(hkil)$	91
A2.2.3	Directions $[wxyz]$ Lying in a Plane $(hkil)$	92
A2.2.4	Angle ϕ between Two Planes	92
A2.2.5	Direction of the Intersection of Two Planes	92
A2.3	STEREOGRAPHIC MANIPULATIONS IN THE HEXAGONAL LATTICE	92
A2.3.1	Indexing Diffraction Patterns	92
A2.3.2	Planes Containing a Given Direction	93
A2.3.3	Contrast Experiments	94
A2.3.4	Dislocation Geometry—Projection of Directions	94
A2.4	CRYSTALLOGRAPHIC DATA FOR THE HEXAGONAL LATTICE	94
	<i>Appendix 2: References</i>	94
APPENDIX 4. STANDARD SPOT PATTERNS		95
	<i>Appendix 4: Reference</i>	95
APPENDIX 5. KIKUCHI MAPS		105
APPENDIX 6. INTERPLANAR ANGLES AND SPACINGS OF SELECTED MATERIALS		109
	<i>Appendix 6: Reference</i>	109

APPENDIX 7. ELECTRON WAVELENGTH	112
APPENDIX 8. ATOMIC SCATTERING AMPLITUDES	
<i>Appendix 8: Reference</i>	113
APPENDIX 9. SUPERIMPOSED STEREOGRAMS FOR VARIOUS COMMON ORIENTATION RELATIONSHIPS	115
<i>Appendix 9: Reference</i>	116
APPENDIX 14. ILLUSTRATION OF THE INHERENT AMBIGUITY IN THE INTERPRETATION OF SELECTED AREA ELECTRON DIFFRACTION PATTERNS OF CEMENTITE	121

2. ELECTRON DIFFRACTION IN THE ELECTRON MICROSCOPE

Electron diffraction patterns are routinely obtained in the electron microscope and are used to gain quantitative information on the following.

- (1) The identity of phases and their orientation relationship to the matrix.
- (2) Habit planes of precipitates, slip planes in materials.
- (3) Exact crystallographic descriptions of crystal defects produced by deformation, irradiation, etc.
- (4) Order/disorder, spinodal decomposition, magnetic domains and similar phenomena.

The first part of this chapter contains those features of the kinematical diffraction theory necessary to interpret diffraction patterns obtained from the electron microscope. The second, third and fourth parts are devoted to indexing diffraction patterns and to their use in metallurgical investigations. In this monograph, all important stereograms are printed to fit the standard Institute of Physics 5 inch stereographic net so that it is possible to work through examples.

PART I. INTRODUCTION TO ELECTRON DIFFRACTION

2.1 General Introduction

Electrons may be regarded as particle waves with wavelength λ given by the de Broglie relation $mv = h/\lambda$. If the electron is accelerated to a voltage V_c , the relativistically corrected wavelength is

$$\lambda = \frac{h}{\{2mV_c e(1 + eV_c/2mc^2)\}^{1/2}} \quad (2.1)$$

where h is Planck's constant, m is the mass of the electron, e is its charge and c is the velocity of light. Values of λ for different accelerating voltages obtained from the above equation are tabulated in appendix 7. At 100 kV, the conventional accelerating voltage for transmission electron microscopy, the relativistically corrected wavelength is 3.7×10^{-3} nm.

In transmission electron microscopy a monochromatic beam of electrons is accelerated through a thin specimen which is usually a single crystal 0.1–0.5 μm thick. On the exit side of the specimen several diffracted beams are present in addition to the transmitted beam, and these are focussed by the objective lens to form a spot pattern in its back focal plane in the manner shown in figure 1.10(a). As explained in section 1.5, this diffraction pattern is magnified by the other lenses to produce a spot pattern, such as that shown in figure 2.1, on the viewing screen. In this case the incident beam direction B is $[100]$ in an aluminium (face-centred cubic, f.c.c.) single-crystal specimen. The transmitted beam is marked T and the arrangement of diffracted beams D around the transmitted beam is characteristic of the four-fold symmetry of the $[100]$ cube axis of aluminium. Here physical and mathematical descriptions of the diffraction process are given, to demonstrate both why diffraction

patterns such as that in figure 2.1 occur, and how they may be interpreted using simple geometrical concepts.

2.2 A Geometrical Approach to Electron Diffraction from a Crystalline Specimen

When a beam of electrons is incident on the top surface of a thin crystalline electron microscope specimen, specific diffracted beams arise at the bottom exit surface. Although each individual atom in the crystal scatters the incident beam, the scattered wavelets will only be in phase (that is reinforce) in particular crystallographic directions. Thus diffraction may be discussed in terms of the phase relationships between the scattered waves from each atom in the crystal.

2.2.1 Scattering by an Individual Atom

The scattering process at an atom is shown schematically in figure 2.2. Here a plane wave is incident on an atom A which acts as a source for a spherical wave propagating at an angle 2θ relative to the incident wave direction. The efficiency of the atom in scattering waves is described in terms of the atomic scattering factor f_θ which depends on both scattering angle θ and incident electron wavelength λ . The term f_θ is defined as

$$f_\theta = \frac{\text{amplitude scattered through angle } 2\theta \text{ by the atom}}{\text{amplitude scattered through angle } 2\theta \text{ by a single electron}}$$

and is given by

$$f_\theta = \frac{me^2}{2h^2} \left(\frac{\lambda}{\sin \theta} \right)^2 (Z - f_x) \quad (2.2)$$

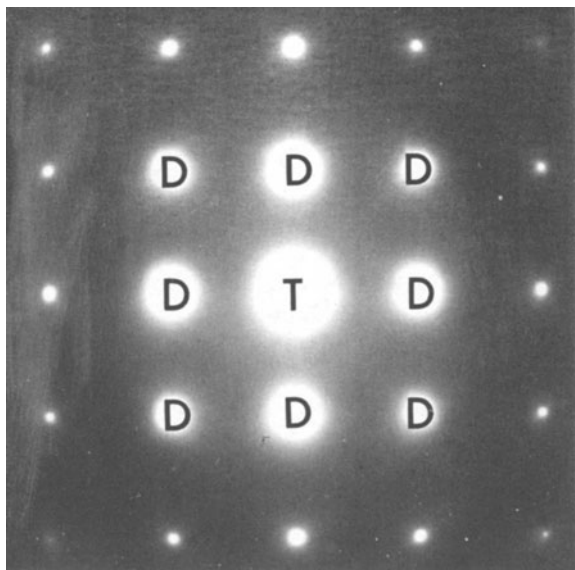


Figure 2.1 A typical spot pattern from an aluminium (f.c.c.) single crystal specimen, incident beam direction $B = [100]$. T, transmitted spot; D, diffracted spot

where Z is the atomic number, f_x is the atomic scattering factor for x-rays and the remaining symbols are those defined previously.

The atomic scattering factor increases with increasing atomic number and is normally expressed as a function of $(\sin \theta)/\lambda$ (see appendix 8). The general form of the relationship is shown schematically in figure 2.3.

2.2.2 Scattering by a Crystal

Before considering diffraction by a regular three-dimensional array of atoms, the principles involved are discussed by analogy with the diffraction of monochromatic light by a grating, see figure 2.4. In this diagram there is a set of slits consisting of plates that are infinitely long, perpendicular to the paper and have spacing a . A screen is placed at a large distance R from the grating and we consider the situation at a point X. If the waves scattered from openings 1 and 2 are in phase, that is their path difference PD is an integral multiple of their wavelength λ , X will be bright. Two in-phase

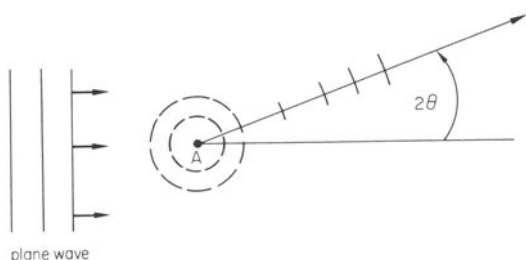


Figure 2.2 The scattering of a plane wave at an atom A through the formation of spherical wavelets travelling at an angle 2θ to the original direction of motion

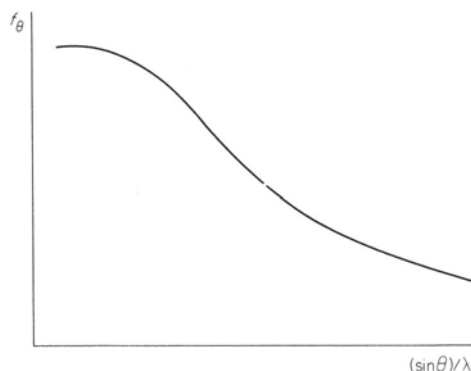


Figure 2.3 A schematic diagram showing the variation of the atomic scattering factor f_θ with $(\sin \theta)/\lambda$

waves are shown in figure 2.4 arriving at X. However, at other points on the screen, the path difference may be such that the waves are out of phase and the total intensity is very low or zero. When this interference process is considered over the whole screen, alternate bright and dark lines are obtained running perpendicular to the paper with the approximate intensity distribution shown in figure 2.4. As α increases the intensity of the fringes decreases because the efficiency for scattering through large angles is lower. In the case of a three-dimensional crystal, a similar path difference argument shows that the diffraction of the monochromatic electron beam by the regularly spaced three-dimensional array of atoms gives an interference pattern of beams, instead of lines.

The theoretical treatment of electron diffraction patterns generally relies on the kinematical theory of electron diffraction and the following assumptions.

- (1) The incident beam is monochromatic, that is the electrons all have the same energy and wavelength.
- (2) The crystal is free from distortion.
- (3) Only a negligible fraction of the incident beam is scattered by the crystal, that is every atom

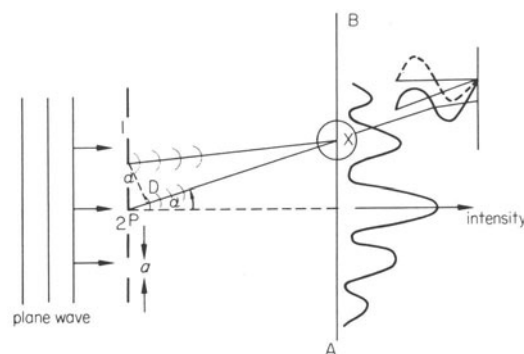


Figure 2.4 Diffraction of monochromatic light by a line grating. Inset shows waves arriving at X in phase

in the crystal receives an incident wave of the same amplitude.

(4) The incident and scattered waves may be treated as plane waves.

(5) There is no attenuation of the electron beam with increasing depth in the crystal, that is no absorption.

(6) There is no interaction between the incident beam and the scattered wavelets, that is the refractive index of the crystal is unity.

(7) There is no re-scattering of scattered waves.

In the electron microscope, the above assumptions are not in general true. Nevertheless, the kinematical approach is still satisfactory for a general description of *diffraction patterns*. In contrast, as we shall see in section 3, it is necessary to use the more realistic dynamical theory of electron diffraction to interpret the details of most *images* obtained in the electron microscope.

2.2.2.1 The Bragg law

The above treatment of diffraction introduced the important point that strong diffracted beams arise because scattered wavelets are in phase in particular directions in the crystal, that is the path difference is an integral number of wavelengths. This leads directly to a particularly simple method of visualising diffraction by a crystal, known as the Bragg law. Figure 2.5(a) depicts the situation describing diffraction in terms of the Bragg law for a transmitting thin electron microscope specimen $\sim 1000\text{--}3000 \text{ \AA}$ thick. Consider the particular case when the incident beam is made up of plane waves in phase and oriented at an angle θ relative to two (hkl) crystal planes I and II. Let the two waves be reflected by these crystal planes at an angle θ . At the plane wavefront CD two situations may occur.

(1) The two waves may be in phase, as shown in figure 2.5(a), in which case reinforcement will occur and a strong reflected beam will be present.

(2) The waves may be out of phase, that is they will interfere and there will be either zero or a very weak reflected beam.

Case (1), that is a strong beam, will occur if the path difference POD is an integral number of wavelengths $n\lambda$. Since $PD = OD = OL \sin \theta$, $2OL \sin \theta = n\lambda$ for a strong beam. However, OL is the interplanar spacing $d_{(hkl)}$. Thus, for a strong reflection, we must have

$$2d_{(hkl)} \sin \theta = n\lambda \quad (2.3)$$

which is known as the Bragg law. In effect we have shown that there will be a strong diffracted beam on the exit side of the crystal only if there is a set of crystal planes oriented at a critical angle θ relative to the incoming beam. For the present we may

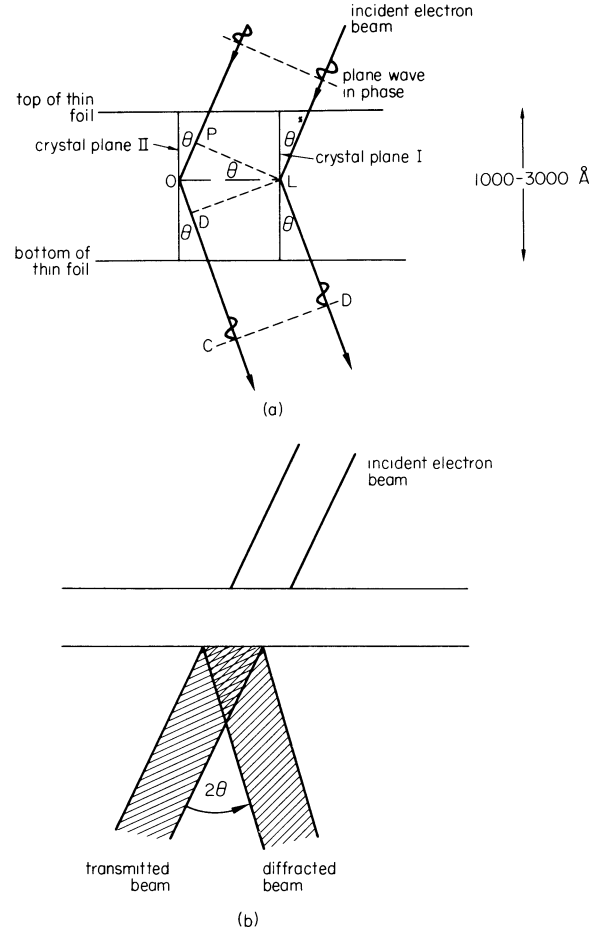


Figure 2.5 (a) Reflection at the Bragg angle θ from crystal planes in a thin foil electron microscope specimen. (b) The relationship between incident, transmitted and diffracted beams for a transmitting specimen

postulate that the reflective process is inefficient and that some of the electrons are not reflected but pass straight through the crystal. Thus there will be both a transmitted and a reflected beam at the bottom surface of the crystal with an angular relation 2θ (see figure 2.5(b)). In terms of the Bragg law the diffracted beams are referred to as reflected beams.

2.2.2.2 The Laue conditions

As an alternative to the above approach, diffraction may be considered in terms of scattering by individual atoms. This approach has the advantage that it may be used more quantitatively to describe diffraction in terms of the reciprocal lattice. Figure 2.6(a) shows this case for atoms O and L where the position of L relative to the origin O may be described in terms of a vector $\mathbf{r}$. The incident and scattered waves are now described in terms of the unit vectors $\mathbf{p}_0$ and $\mathbf{p}$, respectively. Again the scattered waves will be in phase if the path difference POD is an integral number of wavelengths. We may write the distance PO in

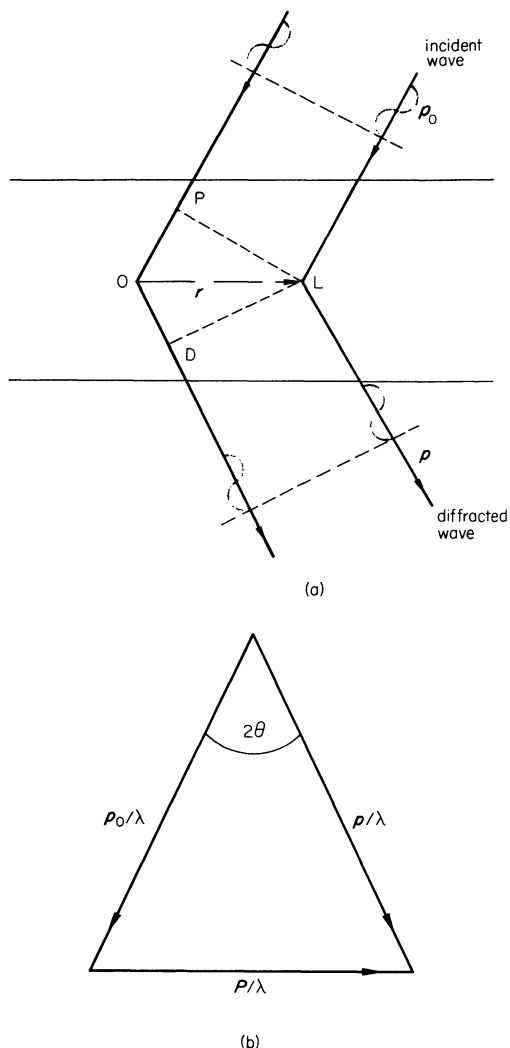


Figure 2.6 (a) The additive scattering from atoms situated at O and L in a thin foil specimen. (b) The vector diagram describing the scattering process

vector notation as $\mathbf{r} \cdot \mathbf{p}_0$ and OD as $\mathbf{r} \cdot \mathbf{p}$, that is $\text{POD} = \mathbf{r} \cdot \mathbf{p} - \mathbf{r} \cdot \mathbf{p}_0 = \mathbf{r} \cdot (\mathbf{p} - \mathbf{p}_0)$. Defining a scattering vector $\mathbf{P} = \mathbf{p} - \mathbf{p}_0$ we have

$$\text{POD} = \mathbf{r} \cdot \mathbf{P} = n\lambda \quad (2.4)$$

The relationship between $\mathbf{p}$, $\mathbf{p}_0$ and $\mathbf{P}$ may be described in terms of the vector triangle shown in figure 2.6(b) in which all vectors have been normalised by dividing by the wavelength λ .

It is convenient to describe the position of each atom relative to the x , y and z crystal axes instead of the simple vector $\mathbf{r}$. Thus we may resolve $\mathbf{r} \cdot \mathbf{P}$

into the components $\mathbf{P} \cdot \mathbf{a}$, $\mathbf{P} \cdot \mathbf{b}$ and $\mathbf{P} \cdot \mathbf{c}$ where $\mathbf{a}$, $\mathbf{b}$ and $\mathbf{c}$ are the repeat distances of the atoms along the crystal axes. Each of these resolved components must also be integral values of λ and we may express equation (2.4) as

$$\begin{aligned} \mathbf{P} \cdot \mathbf{a} &= h\lambda \\ \mathbf{P} \cdot \mathbf{b} &= k\lambda \\ \mathbf{P} \cdot \mathbf{c} &= l\lambda \end{aligned} \quad (2.5)$$

where h , k and l are integers. Equations (2.5) are known as the Laue conditions which must be satisfied for strong diffraction to occur and are equivalent to the Bragg law.

2.2.3 The Reciprocal Lattice

The reciprocal lattice is important because it may be used as a tool in conjunction with the Ewald sphere construction to simplify considerably the interpretation of electron diffraction patterns as described in section 2.2.4. The reciprocal lattice derives directly from the Laue conditions described in equation (2.5), because their solution is

$$\mathbf{P}/\lambda = h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^* \quad (2.6)$$

where $\mathbf{a}^*$, $\mathbf{b}^*$ and $\mathbf{c}^*$ are vectors defined such that $\mathbf{a} \cdot \mathbf{a}^* = \mathbf{b} \cdot \mathbf{b}^* = \mathbf{c} \cdot \mathbf{c}^* = 1$ and $\mathbf{a}^* \cdot \mathbf{b} = \mathbf{b}^* \cdot \mathbf{a}$, etc. = 0. Equation (2.6) may be shown to be the solution of equations (2.5) because forming the scalar product of (2.6) with $\mathbf{a}$ we have $\mathbf{P} \cdot \mathbf{a} = h\lambda$, the first Laue condition.

The conditions $\mathbf{a} \cdot \mathbf{a}^* = 1$ and $\mathbf{a} \cdot \mathbf{b}^* = 0$, etc., have a simple physical explanation. The relation $\mathbf{a}^* \cdot \mathbf{b} = \mathbf{a}^* \cdot \mathbf{c} = 0$ simply means that $\mathbf{a}^*$ is perpendicular to $\mathbf{b}$ and $\mathbf{c}$ and, by a similar argument, $\mathbf{b}^*$ is perpendicular to $\mathbf{a}$ and $\mathbf{c}$ while $\mathbf{c}^*$ is perpendicular to $\mathbf{a}$ and $\mathbf{b}$. This situation is depicted for non-orthogonal axes in figure 2.7. In practice, for crystal structures with orthogonal axes, that is cubic, tetragonal, orthorhombic, the axes of the reciprocal lattice coincide with the crystal lattice. The relations $\mathbf{a}^* \cdot \mathbf{a} = 1$, etc., define the magnitudes of the vectors as $|\mathbf{a}^*| = 1/|\mathbf{a}|$ which is the origin of the term reciprocal lattice.

The reciprocal lattice has the following two properties.

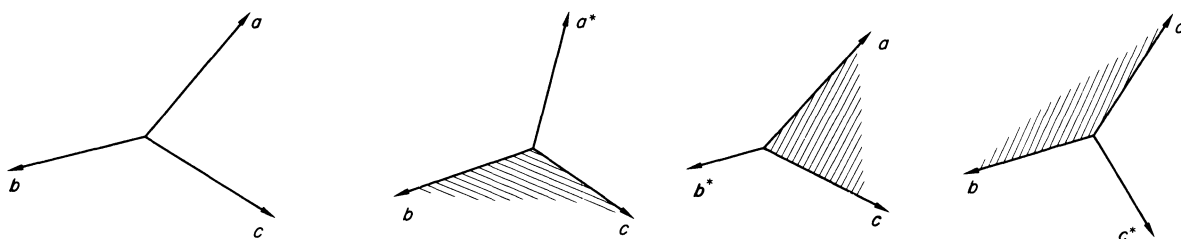


Figure 2.7 The geometrical relationships between the reciprocal lattice vectors $\mathbf{a}^*$, $\mathbf{b}^*$, $\mathbf{c}^*$ and the real lattice vectors $\mathbf{a}$, $\mathbf{b}$, $\mathbf{c}$

(1) The vector $\mathbf{g}_{(hkl)}$ to the point (hkl) of the reciprocal lattice is normal to the plane (hkl) of the crystal lattice.

(2) The magnitude of $\mathbf{g}_{(hkl)}$ is $1/d_{(hkl)}$ where $d_{(hkl)}$ is the interplanar spacing of the family of (hkl) planes (see appendix 1).

The first point may be proved with reference to figure 2.8 which shows the (hkl) plane in the crystal cutting the crystal axes at A, B, C. Then from the definition of Miller indices (appendix 1) the (hkl) plane intersects the axes at distances a/h , b/k and c/l . Consider the vector AB.

$$\frac{\mathbf{a}}{h} + \mathbf{AB} = \frac{\mathbf{b}}{k}, \quad \text{that is } \mathbf{AB} = \frac{\mathbf{b}}{k} - \frac{\mathbf{a}}{h}$$

The scalar product $\mathbf{g} \cdot \mathbf{AB}$ will be zero if $\mathbf{g}$ is perpendicular to AB.

$$\mathbf{g} \cdot \mathbf{AB} = (ha^* + kb^* + lc^*) \cdot \left(\frac{\mathbf{b}}{k} - \frac{\mathbf{a}}{h} \right)$$

Evaluating with the aid of $\mathbf{a} \cdot \mathbf{a}^* = 1$, $\mathbf{a}^* \cdot \mathbf{b} = 0$, etc., we find $\mathbf{g} \cdot \mathbf{AB} = 0$. Because this product is zero, $\mathbf{g}$ must be normal to AB. Similarly it may be shown that $\mathbf{g}$ is normal to AC. Consequently, because $\mathbf{g}$ is normal to two vectors in the plane (hkl) , it is normal to the plane itself.

To prove the reciprocal relation between $\mathbf{g}$ and d , let $\mathbf{n}$ be a unit vector in the direction of $\mathbf{g}$.

$$d = \text{ON} = \frac{\mathbf{a}}{h} \cdot \mathbf{n}$$

But

$$\mathbf{n} = \frac{\mathbf{g}}{|\mathbf{g}|}$$

$$d = \frac{\mathbf{a}}{h} \cdot \frac{\mathbf{g}}{|\mathbf{g}|} = \frac{\mathbf{a}}{h} \cdot \frac{(ha^* + kb^* + lc^*)}{|\mathbf{g}|} = \frac{1}{|\mathbf{g}|}$$

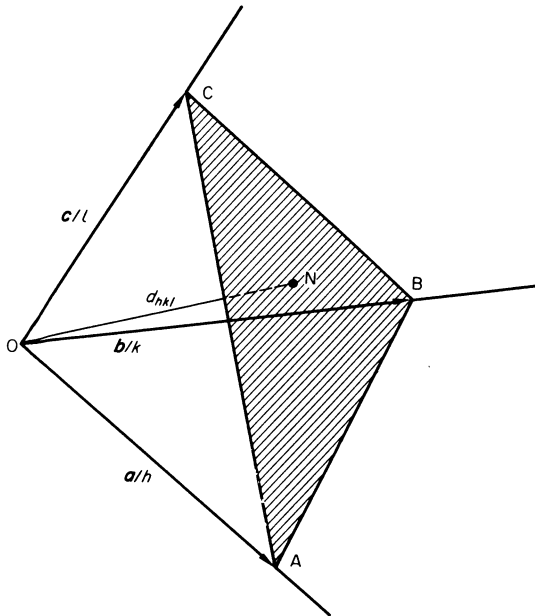


Figure 2.8 The geometrical relationship between the plane normal and $\mathbf{g}$

† From this point $\mathbf{g}_{(hkl)}$ is abbreviated as $\mathbf{g}$.

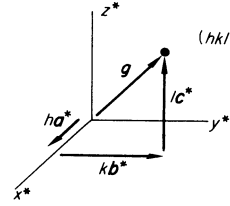
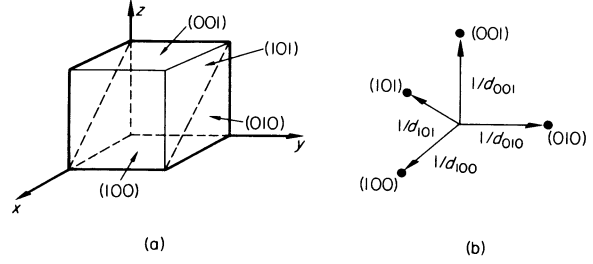


Figure 2.9 The relationship between (a) crystal planes, (b) equivalent reciprocal lattice points and (c) the geometric description of equation (2.7)

Thus we have defined the reciprocal lattice as an array of points, each point corresponding to a particular (hkl) plane and defined by a vector described by points (1) and (2) above. Figure 2.9 shows this relationship between planes in the real lattice and points in the reciprocal lattice for a cubic crystal structure. Each point is labelled with the particular (hkl) indices of the corresponding reflecting plane. Note that a point (hkl) in reciprocal space (figure 2.9(c)) is defined by the steps ha^* along the x axis, kb^* along the y axis and lc^* along the z axis. Thus, as shown in figure 2.9(c),†

$$\mathbf{g}_{(hkl)} = ha^* + kb^* + lc^* \quad (2.7)$$

2.2.4 The Reciprocal Lattice and Diffraction by a Single Crystal

The process of diffraction using the Bragg law may be readily visualised in terms of the reciprocal lattice and the Ewald sphere construction. Referring to figure 2.6(b) we have described the Bragg law in terms of a vector triangle. Equation (2.6) describes the base of this triangle in terms of the reciprocal lattice vectors $\mathbf{a}^*$, $\mathbf{b}^*$, $\mathbf{c}^*$, that is $\mathbf{P}/\lambda$, and equation (2.7) defines the position of an (hkl) reciprocal lattice point in the same terms. Thus $\mathbf{P}/\lambda = \mathbf{g}$ defines the reciprocal lattice point.

The significance of this fact may be made clear with reference to figure 2.10 in which a thin single-crystal electron microscope specimen is oriented in the electron microscope to produce reflection from only one set of (hkl) planes. Assuming a unit incident wave vector, that is

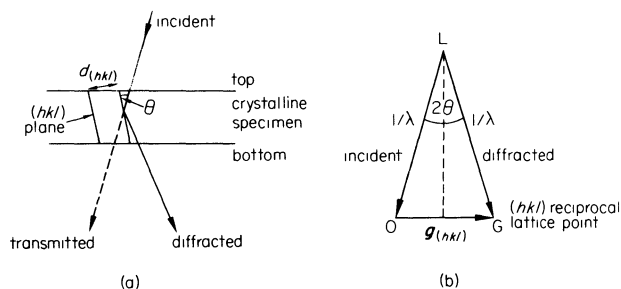


Figure 2.10 (a) Reflection by a set of (hkl) crystal planes, and (b) the vector diagram in reciprocal space describing the same process

$p_0 = p = 1$, the direction of the diffracted beam may be obtained by constructing the following.

(1) A line in the direction of the incident beam and with magnitude $1/\lambda$ running from a point L in the reciprocal lattice to the origin of the reciprocal lattice.

(2) A line LG of the same magnitude $1/\lambda$ from L to the reciprocal lattice point G described by the vector $g_{(hkl)}$ for the particular (hkl) reflecting plane.

In this way, we have reproduced the vector diagram in figure 2.6(b) which describes the Bragg law and have defined the direction of the diffracted beam. Because the reciprocal lattice is three-dimensional and LO and LG are both equal to $1/\lambda$, the construction in figure 2.10(b) represents a small part of a sphere radius $1/\lambda$ in reciprocal space, known as the Ewald or reflecting sphere with centre defined by (1) above. The Ewald sphere construction in the reciprocal lattice is extremely important because it immediately and simply describes the form of the diffraction pattern for a given incident beam direction in the crystal, see section 2.4 for further discussion.

2.3 A Quantitative Approach to Diffraction from a Crystalline Specimen

2.3.1 The Structure Factor

The structure factor describes the contribution of the entire unit cell to the diffracted intensity. Up to this point diffraction has been considered in geometric terms and both the position of atoms in the reflecting plane and atomic identity have been ignored. The structure factor enables both of these factors to be included in the description of the diffraction process, and leads to either systematic absence of reflections or differences in intensity from one (hkl) reflection to another.

A physical picture describing the importance of both atomic position and identity is presented in figure 2.11 in terms of the Bragg law. The first-order reflection which might be expected from a cubic crystal structure is $\{001\}$. For such a first-order

reflection $n = 1$ and a path difference of λ would occur for waves reflected from successive (001) planes A in figure 2.11(a). However, waves will also be reflected from the layer of atoms (B plane) situated halfway between the (001) planes. Because these are $\lambda/2$ out of phase with those from the A planes destructive interference will occur and the overall reflected intensity will be zero. Consequently for a body-centred cubic (b.c.c.) crystal structure $\{001\}$ reflections are absent. Figure 2.11(b) shows that second-order reflections from (002) planes will be present because the path difference between the A planes will be 2λ and there is complete reinforcement with wave's reflection from B planes which have a path difference of λ . Similar reasoning shows that the first-order (111) reflection is absent for the b.c.c. crystal structure whereas the (222) reflection is present. Consideration of systematic absences from other reflecting planes enables a rule to be formulated which states that, for this crystal structure, if $h + k + l$ is odd, then the reflection is absent.

Scattering from a unit cell may be expressed more rigorously in terms of atomic scattering factors and a path difference argument applied to scattering by each atom within it. This enables the structure factor F defined as

$$F = \frac{\text{amplitude of the wave scattered by all the atoms of a unit cell}}{\text{amplitude of wave scattered by an electron}}$$

to be calculated.

Consider the position of the n th atom in the unit cell in figure 2.12. The vector r_n defines the

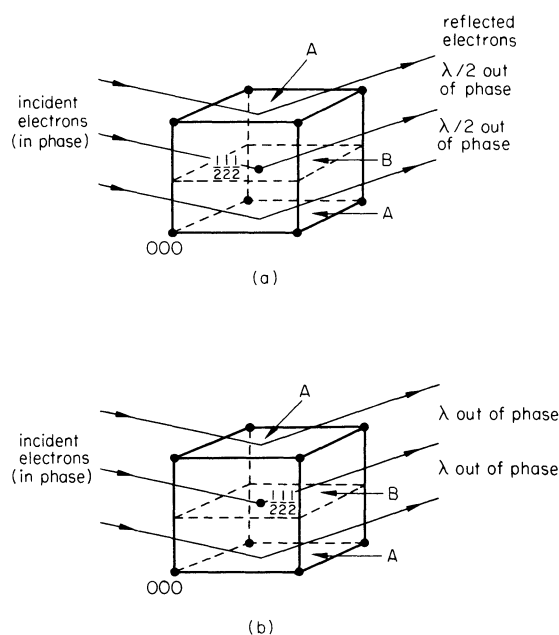
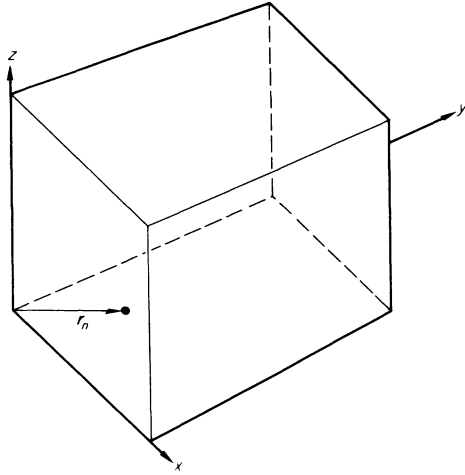


Figure 2.11 The phase relationships for reflection by the layers of atoms shown for (a) (001) and (b) (002) reflections in a b.c.c. crystal structure

Figure 2.12 The n th atom in the unit cell

position of the atom in terms of the fractions x_1 , y_1 , z_1 of the unit vectors a , b , c along the x , y , z axes as

$$\mathbf{r}_n = x_1 \mathbf{a} + y_1 \mathbf{b} + z_1 \mathbf{c} \quad (2.8)$$

The path difference between an atom at the origin of the unit cell and the n th atom is $(\mathbf{r}_n \cdot \mathbf{P})$ and the resultant phase difference $\phi = 2\pi/\lambda \times$ path difference, that is

$$\phi = k \mathbf{r}_n \cdot \mathbf{P}$$

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

The structure factor F is the sum of the scattered amplitudes of the individual atoms f_n and all the phase differences arising from all path differences, that is

$$F = \sum_n f_n \exp(i\phi_n) = \sum_n f_n \exp(ik \mathbf{r}_n \cdot \mathbf{P}) \quad (2.9)$$

Substituting equations (2.6), (2.8) for $\mathbf{r}_n$ and $\mathbf{P}$ we have

$$\mathbf{r}_n \cdot \mathbf{P} = \lambda(hx_1 + ky_1 + lz_1)$$

that is

$$F_{hkl} = \sum_n f_n \exp\{2\pi i(hx_1 + ky_1 + lz_1)\} \quad (2.10)$$

The presence or absence of reflections in the b.c.c. crystal structure can be obtained mathematically from the above structure factor equation as follows.

Intensity of diffracted beam is proportional to

$$\begin{aligned} |F|^2 &= [f_1 \cos\{2\pi(hx_1 + ky_1 + lz_1)\} \\ &\quad + f_2 \cos\{2\pi(hx_2 + ky_2 + lz_2)\} + \dots]^2 \\ &\quad + [f_1 \sin\{2\pi(hx_1 + ky_1 + lz_1)\} \\ &\quad + f_2 \sin\{2\pi(hx_2 + ky_2 + lz_2)\} + \dots]^2 \end{aligned}$$

that is

$$\begin{aligned} |F|^2 &= \sum_i f_i \cos\{2\pi(hx_1 + ky_1 + lz_1)\} \\ &\quad + \sum_i f_i \sin\{2\pi(hx_1 + ky_1 + lz_1)\} \end{aligned} \quad (2.11)$$

For b.c.c. metals there are identical atoms at coordinates 000 and $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ in the unit cell as shown in figure 2.11. Thus the intensity

$$\begin{aligned} I &\propto f^2 \left[\cos(2\pi \cdot 0) + \cos\left\{2\pi\left(\frac{h}{2} + \frac{k}{2} + \frac{l}{2}\right)\right\} \right]^2 \\ &\quad + f^2 \left[\sin(2\pi \cdot 0) + \sin\left\{2\pi\left(\frac{h}{2} + \frac{k}{2} + \frac{l}{2}\right)\right\} \right]^2 \end{aligned} \quad (2.12)$$

$$\begin{aligned} I &\propto f^2 [1 + \cos\{\pi(h + k + l)\}]^2 \\ &\quad + f^2 [\sin\{2\pi(h + k + l)\}]^2 \end{aligned} \quad (2.13)$$

that is $I = 0$ if $h + k + l$ is odd, as pointed out earlier in this section.

If the above argument is applied to an ordered intermetallic compound with the B_2 structure such as NiAl, the atom at 000 will be Ni and that at $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ will be Al. Consequently, since the atomic scattering factors are *not* the same, the diffracted intensity is

$$\begin{aligned} I &\propto [f_{\text{Ni}} + f_{\text{Al}} \cos\{\pi(h + k + l)\}]^2 \\ &\quad + [f_{\text{Al}} \sin\{\pi(h + k + l)\}]^2 \end{aligned} \quad (2.14)$$

that is

$$I \propto (f_{\text{Ni}} + f_{\text{Al}})^2 \text{ when } h + k + l \text{ is even}$$

and

$$I \propto (f_{\text{Ni}} - f_{\text{Al}})^2 \text{ when } h + k + l \text{ is odd}$$

Thus $\{001\}$ reflections will occur with an intensity proportional to the *difference* in scattering factors of the atoms in the material and are generally less intense than the fundamental reflections. Such reflections are known as superlattice reflections and may or may not be present for the same superlattice depending upon the difference in atomic scattering factor (that is atomic number) of the constituent atoms. Table 2.1 shows structure factor information in relation to the absence of reflections in specific crystal structures. A detailed description of structure factors for all crystal structures will be found in the *International Tables for X-ray Crystallography* (1962).

A physical picture describing the occurrence of superlattice reflections may be obtained from figure 2.11(a). The atoms in the B plane would be

Table 2.1 Structure factor effects

Structure	Reflections absent if
simple cubic	all present
f.c.c. (Al, Cu, etc.)	h, k, l , mixed odd and even
b.c.c. (V, W, α -Fe)	$h + k + l$ odd
c.p.h. (α -Ti, Zr, Mg)	$h + 2k = 3n$ and l is odd
b.c.t. (martensite α -Fe)	$h + k + l$ odd
zinc blende (complex cubic) ZnS	h, k, l , mixed odd and even
sodium chloride NaCl	h, k, l , mixed odd and even
diamond (Si, Ge)	h, k, l all even and $h + k + l$ not divisible by four, or h, k, l mixed odd and even

f.c.c., face-centred cubic; b.c.c., body-centred cubic; c.p.h., close-packed hexagonal; b.c.t., body-centred tetragonal.

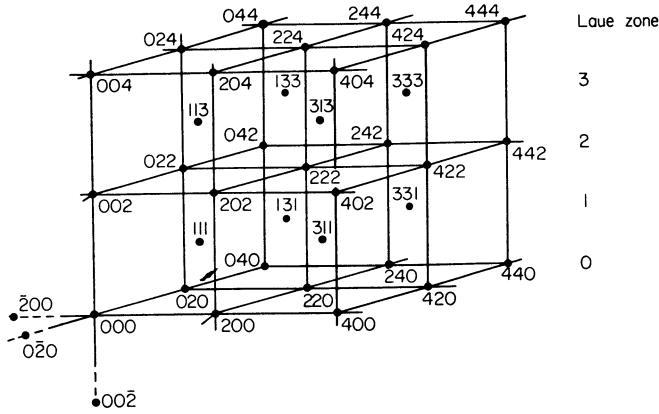


Figure 2.13 The reciprocal lattice for f.c.c. crystal structures

different from those in the A planes and, although the waves interfere as shown before, their intensities are not equal. Consequently a weak (001) superlattice reflection will occur with an intensity depending upon the difference in atomic scattering factors of the constituent atoms, as pointed out before.

It is important to relate the results of these structure factor calculations to the reciprocal lattice. In effect, if the structure factor is zero, the reciprocal lattice point is removed because no reflection will be present in any diffraction pattern. Thus, using the selection rules in table 2.1 for all f.c.c. crystals, the reciprocal lattice is b.c.c. and may be indexed as shown in figure 2.13. For a b.c.c. crystal the reciprocal lattice is f.c.c.

2.3.2 The Intensity Distribution in Reciprocal Space

After having considered the influence on diffracted intensity of atomic position and identity within the unit cell, it is necessary to consider the diffracted intensity from the large array of unit cells that go to make up the electron microscope specimen. Figure 2.14(a) shows a thin electron microscope specimen made up of $N_x N_y N_z$ unit cells along the x , y and z axes. The position of the n th unit cell relative to the origin may be defined by the vector $\mathbf{r} = n_x \mathbf{a} + n_y \mathbf{b} + n_z \mathbf{c}$ where $\mathbf{a}$, $\mathbf{b}$, $\mathbf{c}$ are unit vectors along x , y , z respectively.

Thus, if F is the structure factor of each (identical) unit cell the total scattered amplitude A is the sum of all the phase differences $\phi = \mathbf{k} \cdot \mathbf{a} \cdot \mathbf{P}$ along the x , y and z axes for N_x , N_y and N_z unit cells, that is

$$A = F \sum_{n_x=0}^{N_x-1} \exp(ikn_x \cdot \mathbf{a} \cdot \mathbf{P}) \times \sum_{n_y=0}^{N_y-1} \exp(ikn_y \cdot \mathbf{b} \cdot \mathbf{P}) \times \sum_{n_z=0}^{N_z-1} \exp(ikn_z \cdot \mathbf{c} \cdot \mathbf{P}) \quad (2.15)$$

Each of these terms is a geometric progression of the form

$$\sum_{m=0}^{N-1} X^m = X^0 + X^1 + X^2 \dots X^{N-1} = \frac{1 - X^N}{1 - X} \quad (2.16)$$

Thus the first part of equation (2.15) may be written

$$\sum_{n_x=0}^{N_x-1} \frac{1 - \exp(ikN_x \cdot \mathbf{a} \cdot \mathbf{P})}{1 - \exp(ik \cdot \mathbf{a} \cdot \mathbf{P})} \quad (2.17)$$

Multiplying equation (2.17) by its complex conjugate

$$|\sum|^2 = \frac{1 - \cos(kN_x \cdot \mathbf{a} \cdot \mathbf{P})}{1 - \cos(\mathbf{k} \cdot \mathbf{a} \cdot \mathbf{P})} = \frac{\sin^2(\frac{1}{2}N_x \mathbf{k} \cdot \mathbf{a} \cdot \mathbf{P})}{\sin^2(\frac{1}{2}\mathbf{k} \cdot \mathbf{a} \cdot \mathbf{P})} \quad (2.18)$$

Thus the expression for the total diffracted intensity is

$$|A|^2 = |F|^2 \times \frac{\sin^2(\frac{1}{2}N_x \mathbf{k} \cdot \mathbf{a} \cdot \mathbf{P})}{\sin^2(\frac{1}{2}\mathbf{k} \cdot \mathbf{a} \cdot \mathbf{P})} \quad \text{I} \\ \times \frac{\sin^2(\frac{1}{2}N_y \mathbf{k} \cdot \mathbf{b} \cdot \mathbf{P})}{\sin^2(\frac{1}{2}\mathbf{k} \cdot \mathbf{b} \cdot \mathbf{P})} \quad \text{II} \\ \times \frac{\sin^2(\frac{1}{2}N_z \mathbf{k} \cdot \mathbf{c} \cdot \mathbf{P})}{\sin^2(\frac{1}{2}\mathbf{k} \cdot \mathbf{c} \cdot \mathbf{P})} \quad \text{III} \quad (2.19)$$

Strong diffraction by the crystal will occur when $|A|^2$ is a maximum, that is when each term I–III is a maximum. It has been shown previously that

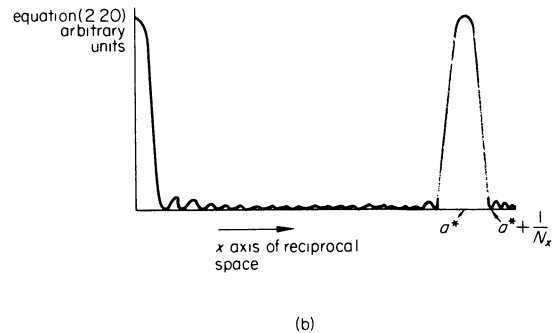
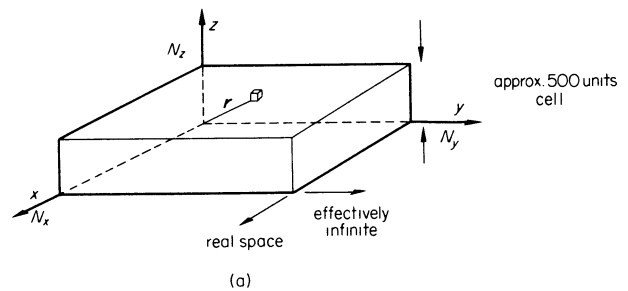


Figure 2.14 (a) The position of the n th unit cell in an electron microscope specimen consisting of N_x , N_y and N_z unit cells in the x , y , z directions respectively. (b) The variation of equation (2.20) along the x axis of reciprocal space

the diffracted wave vector $\mathbf{P}$ is a maximum when $\mathbf{P}/\lambda = \mathbf{g}$. Substituting equation (2.6) into term I of equation (2.19) we have

$$\frac{\sin^2 \left[\frac{1}{2} N_x k a \cdot \{ \lambda(ha^* + kb^* + lc^*) \} \right]}{\sin^2 \left[\frac{1}{2} k a \cdot \{ \lambda(ha^* + kb^* + lc^*) \} \right]} \quad (2.20)$$

is a maximum if $a \cdot \{ \lambda(ha^* + kb^* + lc^*) \} = h\lambda$ and zero if it is $h\lambda/N_x$. Thus, in effect this term maps the diffracted intensity as a function of position along the x axis of reciprocal space as shown in figure 2.14(b). The fact that the diffracted intensity falls very rapidly to zero on moving a small distance $1/N_x$ from the reciprocal lattice point for a large crystal shows that the reciprocal lattice does indeed consist of an array of points.

2.4 The Reciprocal Lattice and Transmission Electron Diffraction in the Electron Microscope

In most cases electron microscope diffraction patterns are obtained from individual grains and therefore are single-crystal diffraction patterns. They are most easily visualised in terms of the Ewald sphere construction in the reciprocal lattice, but first the reciprocal lattice must be modified to take account of the thin sheet shape of the electron microscope specimen using the results of equation (2.19). It was shown in section 2.3.2 and figure 2.14(b) that the width of the reciprocal lattice point is $2/N_x$, $2/N_y$, and $2/N_z$ in the x , y , z direction. However, the typical electron microscope specimen shown schematically in figure 2.14(a) is a sheet, effectively infinite in its xy plane but finite along the z direction, that is ~ 500 unit cells thick. Consequently the reciprocal lattice points are very narrow in the z and y directions with intensity distributions of the form shown schematically in figure 2.15(b). In contrast the intensity distribution around the reciprocal lattice points in the z direction is much broader than in the x and y

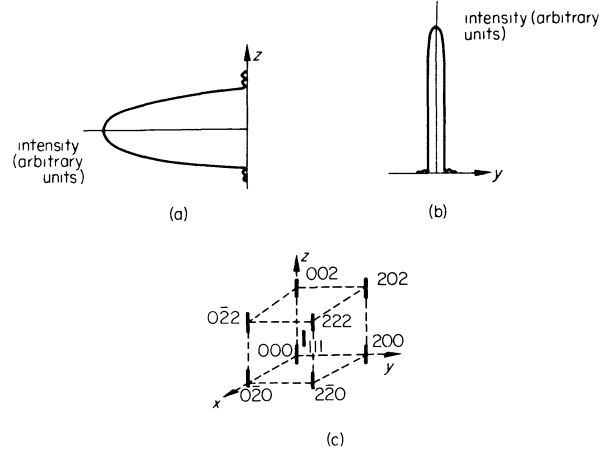


Figure 2.15 (a) and (b) The resulting approximate intensity distribution in reciprocal space parallel to z and y , respectively. The effective streaking of all points in reciprocal space normal to the specimen surface is shown in (c)

directions owing to the thinness of the sheet, see figure 2.15(a). Consequently the reciprocal lattice points must in fact be treated as streaks parallel to z , the foil normal, see figure 2.15(c). This is equivalent to stating that the Laue condition in the direction z is relaxed and thus a significant diffracted intensity will be obtained even when the Bragg condition is not *exactly* satisfied. The modified Ewald sphere construction which takes account of this is shown in figure 2.16. A vector $\mathbf{s}$ is defined describing the deviation from the exact Bragg position when the Ewald sphere cuts the streak. Clearly, as s increases, the diffracted intensity will decrease, see figure 2.16 and if $s \neq 0$ the reciprocal lattice vector is $\mathbf{g}' = \mathbf{g} + \mathbf{s}$.

The above discussion has important implications for electron diffraction in the electron microscope that can be readily seen with the aid of the Ewald sphere construction in figure 2.17. Here the position of the thin foil is indicated, together with the direction of the incident beam. Although it is only a device to aid interpretation of diffraction

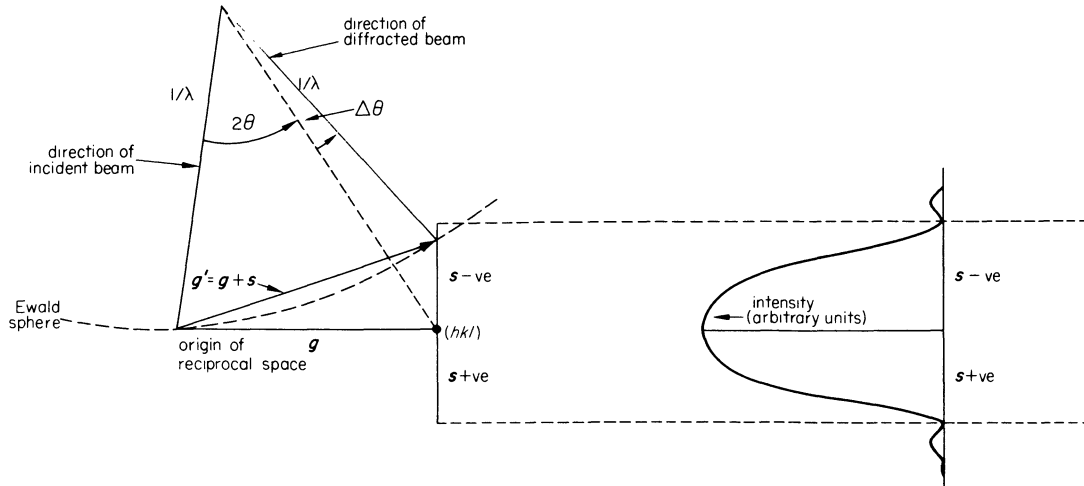


Figure 2.16 The definition of vectors $\mathbf{g}$, $\mathbf{s}$, $\mathbf{g} + \mathbf{s}$ in terms of the Ewald sphere construction in reciprocal space

patterns, the Ewald sphere is drawn schematically as if it were centred at the specimen where the diffraction event occurs and the layer of the reciprocal lattice points containing the origin is also shown with the points streaked to allow for specimen thinness. For an incident 100 kV electron beam $1/\lambda \approx 27 \text{ \AA}^{-1}$ but a typical value for $1/d$ is $0.5 \text{ \AA}^{-1}$, that is the radius of the Ewald sphere is ~ 50 times that of the spacing between reciprocal lattice points. Thus, because the Ewald sphere is almost flat and the reciprocal lattice points are streaked parallel to the foil normal, several reciprocal lattice points may be cut simultaneously and therefore several diffracted beams may occur. The distribution of the reciprocal lattice streaks on the surface of the Ewald sphere corresponds to that obtained by inserting a plate below the specimen, see figure 2.17. Since the action of the lens imaging system is simply to produce a magnified image of the spot distribution in the plate (see section 1.5.1) the diffraction pattern is effectively a *magnified* image of the spot array on the surface of the Ewald sphere, that is a planar section in reciprocal space perpendicular to the incident beam direction B . Thus, if an electron beam is incident on a single-crystal f.c.c. material parallel to $[001]$, the diffraction pattern will consist of an array of spots with four-fold symmetry corresponding to a magnified view of the (001) of the reciprocal lattice in figure 2.13. Compare this section of the reciprocal lattice with the diffraction pattern from aluminium in figure 2.1.

The above section through the origin of reciprocal space is called the zero-order Laue zone and is shown labelled 0 in figure 2.13(a) for $B [001]$. The first- and second-order Laue zones are parallel to the zero-order zone and are labelled 1, 2 in figure 2.13(a), that is $l = 1$, $l = 2$. Laue zones are discussed in more detail in section 2.7.2.2.

2.5 Summary

(1) If an electron beam is incident on a crystal, several beams in addition to the transmitted beam may be observed on its exit side. The extra beams arise because the incident electrons are scattered by the atoms of the crystal and the scattered electrons are in phase (that is reinforce) in certain directions in the crystal.

(2) The diffraction process may be conveniently visualised as reflection by (hkl) crystal planes spacing $d_{(hkl)}$. The diffracted beam corresponds to reflection for a particular angle θ —between the incident beam and the crystal plane for which $n\lambda = 2d \sin \theta$, where d is the spacing of the (hkl) planes and λ is the wavelength. This law is known as the Bragg law and corresponds to satisfaction of the Laue conditions.

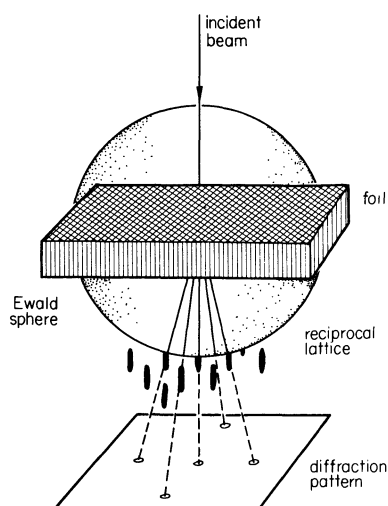


Figure 2.17 The specimen, transmitted and diffracted beams and the diffraction pattern. Superimposed is the Ewald sphere construction in reciprocal space that describes the diffraction pattern

(3) The reciprocal lattice provides a very useful method of describing single-crystal diffraction patterns. It consists of points, each corresponding to a particular (hkl) plane, distance $|g| = 1/d_{(hkl)}$ from the origin in a direction perpendicular to the individual (hkl) plane. In effect the reciprocal lattice is a map of the diffracted intensity distribution in reciprocal space.

(4) For a given incident beam direction B the number of diffracted beams occurring in the diffraction pattern and the crystal planes responsible may be determined rapidly by using the Ewald sphere construction within the reciprocal lattice. A diffracted beam occurs when the Ewald sphere cuts a reciprocal lattice point specifying the indices of the reflecting plane, the Bragg angle and the direction of the diffracted beam.

(5) For diffraction in the electron microscope the specimen is normally a single crystal. Because of the relaxation of the Laue conditions reciprocal lattice points are streaked perpendicular to the specimen surface. Furthermore, the radius of the Ewald sphere $\gg$ the reciprocal lattice vectors g_{hkl} . Thus the single-crystal electron diffraction pattern is a series of spots equivalent to a magnified view of a planar section through the reciprocal lattice normal to the incident beam direction.

(6) The intensity of a particular beam depends on (a) the position and identity of the atoms in the unit cell through the structure factor F , (b) the size and shape of the crystal through the Laue condition, (c) the deviation s from the exact Bragg reflection position.

PART II. ELECTRON DIFFRACTION PATTERNS IN THE ELECTRON MICROSCOPE

2.6 Types of Diffraction Patterns

Electron diffraction patterns produced in transmission in the electron microscope can be of three different types.

- | | |
|--------------------------|--|
| (a) Ring pattern | } Polycrystalline specimen
Single-crystal region
of the specimen |
| (b) Spot pattern | |
| (c) Kikuchi line pattern | |

(b) and (c) often occur on the same diffraction pattern. In general, spot and Kikuchi patterns will be taken from a specific area of the specimen and are known 'selected area' diffraction patterns (SADPs).

2.6.1 Ring Patterns

The major use of ring patterns is in the identification of phases using extraction replicas. These diffraction patterns also arise from very fine grain size polycrystalline material such as physically or chemically vapour deposited or electrodeposited thin foils. A typical ring pattern from a thin (~ 50 Å) carbon film covered in randomly oriented small precipitate particles shown in figure 2.51 is shown in figure 2.18(a). The situation which produces this pattern is shown geometrically for one ring in figure 2.18(b). For a given beam direction a number of precipitate particles within the area illuminated by the beam will be oriented to satisfy the Bragg law for all allowed reflecting planes. However, one individual precipitate will produce a specific beam reflected from a particular (hkl) plane, such that the angle between it and the incident beam is 2θ , satisfying the Bragg law. For randomly oriented particles and a specific reflecting plane (hkl) these beams will lie in a cone with apex angle $4\theta_{(hkl)}$ centred on the incident beam direction to produce a ring in the diffraction pattern as shown in figure 2.18(b). Since a number of $\{hkl\}$ planes will reflect depending on the structure factor (section 2.3.1) a series of concentric rings will be produced as shown in figure 2.18(a), each one corresponding to a particular set of (hkl) reflections, as indexed.

2.6.2 Spot Patterns

It was shown in section 2.4 that spot patterns correspond to magnified images of planar sections through the reciprocal lattice taken normal to the incident beam direction B . Using spot patterns the following are possible.

- (1) The foil orientation in the microscope can be determined (often by inspection because the

various simple low-index beam directions are easily recognised, see appendix 4).

- (2) The crystal can be quickly surveyed and the accessibility of certain orientations and hence diffracting vectors determined.

- (3) A well-defined tilt axis by tilting about a strong Bragg reflection can be established, that is bright spot, see section 2.10.1.

- (4) Precipitates, twins, martensite plates, etc., can be identified, see sections 2.12, 2.13.

- (5) The orientation relationship between phases can be determined, see section 2.11.

- (6) Details of the fine structure of the pattern can be used to obtain information on the defect structure of the specimen, see sections 2.16–2.19.

These investigations are described in more detail in parts III and IV.

2.6.3 Kikuchi Patterns

These occur if a SADP is taken from a single-crystal region of the specimen provided the specimen is reasonably thick ($\sim \frac{1}{2}$ maximum usable penetration) and has a low defect density. Kikuchi patterns consist of pairs of parallel bright and dark lines $B'B'$ and $D'D'$, figure 2.22. Kikuchi patterns are very important for the following reasons.

- (1) They enable the tasks (1)–(3) and (5) outlined above to be performed more accurately than with spot patterns.

- (2) They constitute a 'Kikuchi map' which describes the distribution of Kikuchi lines within a unit triangle of the stereogram. By comparison with this map (see section 2.7.3.3 and appendix 5) it is possible to identify Kikuchi patterns rapidly and to determine B . Furthermore, the specimen may be tilted in a self-consistent manner while working in the microscope (see section 2.8). An example of such a tilting sequence is shown in figure 2.35.

- (3) They determine the sign and magnitude of the deviation s from the exact Bragg position as defined in figure 2.16. The relationship between the bright Kikuchi line and its associated diffraction spot is used, see section 2.10.2.

- (4) They define the sense of tilt because the Kikuchi pattern behaves as if it is fixed to the bottom of the crystal and consequently it moves in the same sense as the crystal, that is the lines move in a direction perpendicular to the tilt axis and towards the edge of the specimen tilted *up*, see later in this section.*

* This is also true for the image, provided the 180° rotation between image and diffraction pattern, section 1.11.3, is correctly allowed for.

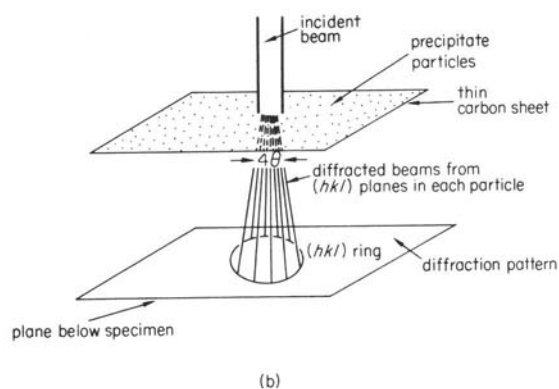
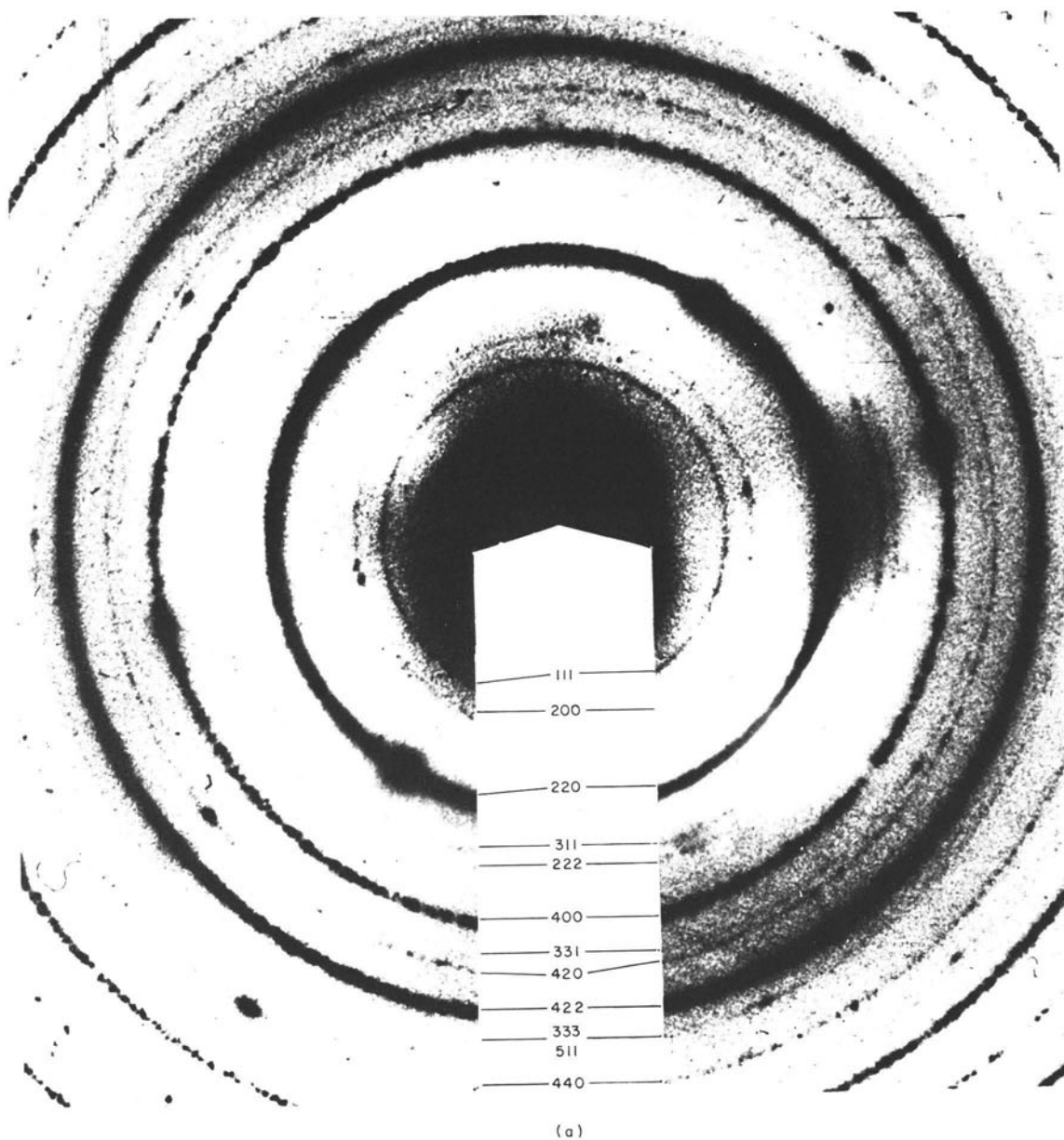


Figure 2.18 (a) A ring diffraction pattern from a polycrystalline gold specimen. The faint rings are from the support film.
(b) The geometry of formation of a single (hkl) ring by accumulation of (hkl) beams from different grains

(5) Small angular tilts, δ , can be estimated, where $\delta = m/L$ and m is the linear displacement of the Kikuchi line pattern, normal to the tilt axis; L is the camera length, see section 1.

(6) Large angular tilts can be measured by identifying specific B accurately, see section 2.7.3.

(7) They determine crystal symmetry because, unlike spot patterns, Kikuchi patterns exhibit real crystal symmetry, see figure 2.25.

The main features of the geometry of Kikuchi patterns can be understood by making use of the simplified treatment first proposed by Kikuchi (1928) in which the lines are considered to arise from the diffraction of electrons that have been previously *inelastically scattered* by the specimen. These inelastically scattered electrons are present in most spot electron diffraction patterns such as that shown in figure 2.1 both as a diffuse halo around the transmitted and strong diffracted spot and as an overall faint background intensity. Since many more electrons suffer small energy changes than large ones the diffuse halo loses intensity with increasing distance from the transmitted beam, and the intensity distribution may be described in terms of the polar diagram in figure 2.19(a).

Figure 2.19(b) illustrates a geometrical description of the occurrence of Kikuchi lines. A thin single-crystal specimen is oriented such that the Bragg law is not satisfied for the incident beam relative to the pair of (hkl) planes shown. However, an inelastic scattering event occurs at O , the electron loses a small amount of energy, $\lesssim 50$ eV, and it changes direction slightly so that it now satisfies the Bragg law for plane XY . Thus instead of contributing to the intense halo around the transmitted beam, see figures 2.1, 2.19(a), it is diffracted away towards the low background intensity region well removed from the transmitted beam down the path OBC in figure 2.19(b). The result is a local reduction in background intensity near the transmitted beam and an increase in a region far removed from it. Very many fewer electrons will be inelastically scattered through the large angle necessary to be diffracted back into the region near the transmitted beam following the path OAD because only a few are scattered into OA in the first place, see figure 2.19(a). When all possible incident directions into the (hkl) planes are considered, and the three-dimensional distribution of the inelastic scattering events is included, the diffraction process can be shown to produce cones of radiation centred on the specimen, see figure 2.19(c). The intensity of cone I is lower than background whereas cone II is higher than background.

These Kikuchi cones may be considered in relation to the Ewald sphere construction by a

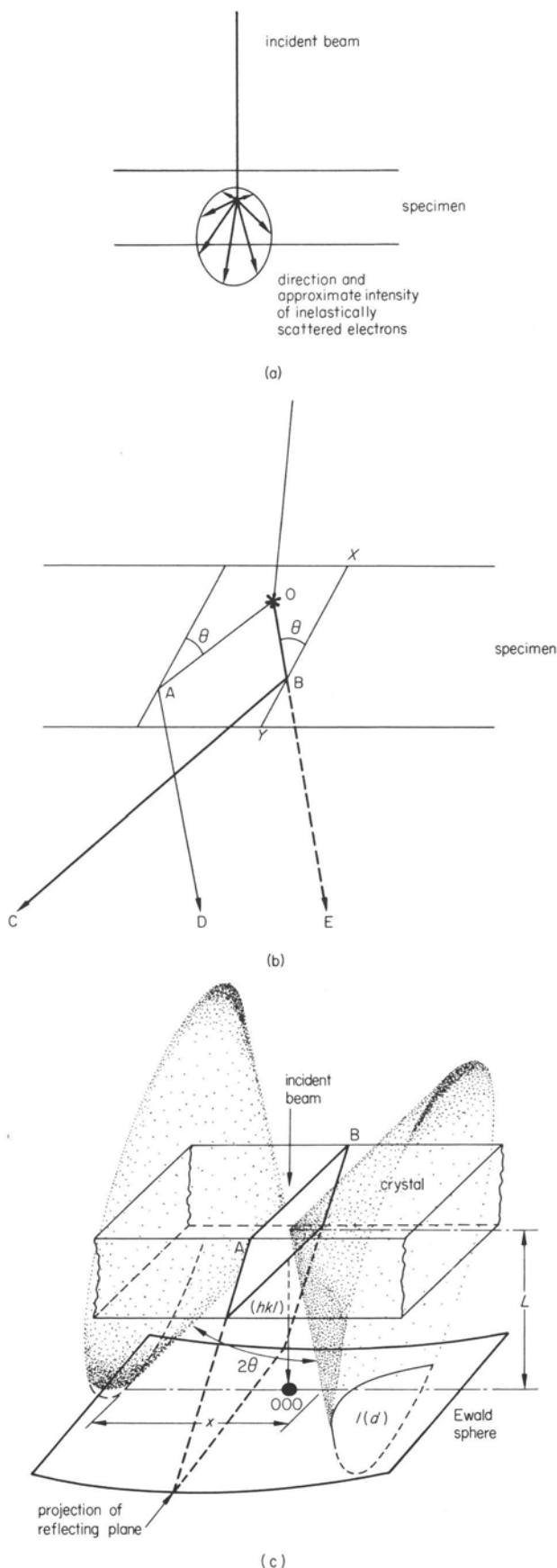


Figure 2.19 (a) A polar diagram of the intensity distribution of inelastically scattered electrons. (b) The reflection of these electrons by crystal planes to form Kikuchi cones shown in (c)

device similar to that in figure 2.17. The result is shown in figure 2.19(c) in which it may be seen that the cones intersect the Ewald sphere in hyperbolae with such large radii that they are effectively straight lines, one brighter than background and one darker than background. Since the diffraction pattern in the electron microscope is effectively a magnified picture of the surface of the Ewald sphere, see section 2.4, the Kikuchi lines are visible as parallel bright and dark pairs in the diffraction pattern (see B'B' and D'D' in figure 2.22). Furthermore they are parallel to the projection of the reflecting planes responsible. In effect the specimen acts as a monochromator and only selects those electrons that exactly satisfy the Bragg law $n\lambda' = 2d \sin \theta'$. Since typically the characteristic losses are of the order of < 50 eV, and $\lambda' \approx \lambda_{\text{incident}}$, the same reflecting sphere reciprocal lattice construction may be used to describe both spot and Kikuchi patterns.

Kikuchi patterns are very important in electron microscopy because the position of Kikuchi lines in the diffraction pattern is a more sensitive measure of the crystal orientation relative to the electron beam than the spot positions. The reason for this may be seen by considering the movement of the Kikuchi lines over the surface of the Ewald sphere if the specimen is tilted slightly, for example about the axis AB, figure 2.19(c). The cones of radiation behave as if directly attached to the specimen. Thus the Kikuchi line pair swing about the Ewald sphere radius $1/\lambda$ and move rapidly across the surface of the Ewald sphere. In contrast the reciprocal lattice only tilts about its origin, that is about a radius $g \approx (1/25)(1/\lambda)$. Thus tilting from s negative to $s = 0$, figure 2.16, only moves the spot on the surface of the Ewald sphere slightly closer to the origin, which is almost undetectable in a typical diffraction pattern. Kikuchi patterns are discussed more fully in sections 2.7.3, 2.8.1.

2.7 Indexing Diffraction Patterns

The procedure for indexing spot patterns, Kikuchi patterns and ring patterns is outlined below. *Indexing is always performed with the negative emulsion side up, that is as it is in the microscope, to preserve geometrical continuity in relating the specimen, its image and its diffraction pattern. For the same reason images are always analysed and printed emulsion side up.* Before discussing methods of indexing diffraction patterns it should be noted that accuracy of operation of the electron microscope is an important factor in determining the usefulness of a correctly indexed diffraction pattern. If the results are to be interpreted quantitatively, particular care must be taken in the selection of the

area for diffraction and focussing of image and diffraction pattern, section 1.5.1.

2.7.1 Ring Patterns

The radius of each ring is characteristic of the spacing of the reflecting planes in the crystal and the magnification settings of the microscope lenses. The latter can be calibrated as described in section 1.11.2.

Procedure for indexing ring patterns is as follows.

(a) When the identity of the material is known, we have the following.

- (1) Measure the ring diameters.
- (2) Determine the ratios of the squares of the diameters of the outer rings to that of the first or second (low-index) ring.
- (3) Check the ratios against a table of ratios of the interplanar spacings for the crystal structure of interest, see table 2.2.

(b) When the identity of the substance is unknown, we have the following.

- (1) Measure the diameter of the rings.
- (2) Convert the distances into interplanar spacings using the camera constant defined in section 1.5.1.
- (3) Use ASTM index* to identify the phase, starting with the most likely on the basis of the known constituents of the alloy.

Diffraction patterns from polycrystalline specimens are most commonly used either to calibrate the camera length (section 1.11.2) or to identify precipitates. If a precipitate cannot be identified using the ASTM index, procedures involving the more accurate techniques such as x-ray analysis, x-ray microanalysis are necessary to specify the phase present.

In identifying the crystal structure of unknown phases, the simplest approach is often to work out the ratios of the squares of the radii of the rings. These ratios are proportional to different numbers depending on the crystal structure, see table 2.2. Those crystal structures not included in this table are difficult to deal with and the student is referred to the books by Henry *et al.* (1951) and Vainshtein (1964) for further details.

2.7.2 Spot Patterns

Some spot patterns have high symmetry in which case **B** will be parallel to a low-index direction in the crystal and for simple crystal structures it may be often indexed by inspection. More complex spot patterns which may be recognised by their lack of symmetry require special procedures.

* ASTM index to the powder diffraction file (American Society for Testing Materials, Philadelphia, U.S.A.).

Table 2.2 Proportionalities of the ratios of the radii of ring patterns for different crystal structures

Crystal structure	Formula for interplanar spacing	Possible values of h, k, l for reflection (up to 20)	Criterion
simple cubic	$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} = \frac{N}{a^2}$	N an integer except 7 or 15	ratios of squares of radii $\propto N$
f.c.c.	$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} = \frac{N}{a^2}$	$N = 3, 4, 8, 11, 12, 16, 19, 20$	ratios $\propto N$
b.c.c.	$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} = \frac{N}{a^2}$	$N = 2, 4, 6, 8, 10, 12, 14, 16, 18, 20$	ratios $\propto N$
diamond structure	$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} = \frac{N}{a^2}$	$N = 2, 8, 11, 16, 19$	ratios $\propto N$
tetragonal	$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2}$	$h^2 + k^2 = 1, 2, 4, 5, 8, 9, 10, 13, 16, 17, 18, 20$	ratios frequently proportional to 2; use Bunn chart, see Henry <i>et al.</i> (1951)
hexagonal	$\frac{1}{d^2} = \frac{4}{3} \frac{h^2 + hk + k^2}{a^2} + \frac{l^2}{c^2}$	$h^2 + hk + l^2 = 1, 3, 4, 7, 9, 12, 13, 16, 19$	ratios frequently proportional to 3; use Bunn chart, see Henry <i>et al.</i> (1951)

There are two features which enable spot patterns to be indexed.

(1) The distance R_1 , figure 2.20(b), from the diffracted spot to the transmitted beam (centre spot). This parameter is characteristic of the interplanar spacing $d_{(h_1k_1l_1)}$ of the reflecting plane and the magnification due to the lens settings, that is camera constant λL , see section 1.5.1. However, the finite size of the diffracted spots together with small shifts in position produced by non-zero values of s (see figure 2.16 and sections 2.4, 2.6.3) limit the accuracy of determination of d_{hkl} .

(2) The angles between lines drawn from the centre spot to diffracted spots $(h_1k_1l_1)$, $(h_2k_2l_2)$, figure 2.20(b), are the angles between the planes. This arises because these lines correspond to $\mathbf{g}$ and are effectively parallel to plane normals because the errors introduced if $s \neq 0$ are very small, see figure 2.16.

The geometry of the relationship between the diffraction pattern and the reflecting planes in the specimen is illustrated in figure 2.20(e). The Bragg angle for 100 kV electrons ($\lambda = 0.037 \text{ \AA}$, $d_{200\text{Al}} = 2.02 \text{ \AA}$) is 30° . Consequently the reflecting planes are almost parallel to the incident beam direction $\mathbf{B}$, figure 2.20(e). Thus we may make the following conclusions.

(1) $\mathbf{B}$ is approximately the zone axis of the reflecting planes.

(2) Directions $\mathbf{R}_{h_1k_1l_1}$ in the diffraction pattern are approximately $(h_1k_1l_1)$ plane normals, that is $\mathbf{g}_{(h_1k_1l_1)}$ and may be translated into crystallographic directions in the surface of the specimen, see figure 2.20(e).

Two cases may be considered, namely when the diffraction pattern is (1) simple, that is the spots are all produced by planes in one zone, (2) complex with the spots produced from more than one zone. The method of indexing diffraction patterns for these two cases is described below.

2.7.2.1 Simple patterns—spots are produced by planes in one zone

Frequently these patterns may be easily recognised from their simplicity and their six-, four-, three- and two-fold symmetry. Indexed examples of such simple patterns are shown in appendix 4 for various crystal structures.

(a) *Indexing the pattern.* The indexing procedure involves two stages: (1) the determination of the general $\{hkl\}$ indices of the spot, that is the combination of h, k and l without definite values and signs; (2) assignment of specific (hkl) indices to the individual spots and the determination of the beam direction $\mathbf{B}$. For stage (1), one of two procedures, (i) and (ii), may be used.

(i) Camera constant method. Camera constant known, material known.* Measure the distance R of the spot from the centre spot, figure 2.20(a) (most accurately determined by measuring the total distance between several spots in this direction and dividing as necessary). Divide the camera constant λL (equation (1.11) by R and check the result against a list of d spacings.

(ii) Method of ratios. Camera constant unknown. Material known. Measure distances of spots from centre spot relative to that between the nearest spot and the centre. Check against tables of ratios of d spacings for low-index

* Note that, if the camera constant is known but the material is not, the procedure is the same as (i) above but the interplanar spacings are checked against likely ASTM cards and the angles between reflections must be correct to provide a cross-check, see stage (2). If both camera constant and material are unknown, the camera constant must first be determined, see section 1.11.2.

planes. Account must be taken of the occurrence of planes with the same d spacing, see table A4.1, appendix 4, for cubic case (that is $h_1^2 + k_1^2 + l_1^2 = h_2^2 + k_2^2 + l_2^2$).

For stage (2),* assignment of specific hkl indices follows one of two possible procedures.

The closest spot to the centre ($h_2k_2l_2$), figure 2.20(a), is arbitrarily indexed for a given combination of h , k and l , for example (111), ($\bar{1}\bar{1}1$), ($\bar{1}11$), etc. Consideration is then given to two other spots ($h_1k_1l_1$) and ($h_3k_3l_3$) and pairs of indices chosen such that when added vectorially they give ($h_2k_2l_2$), that is

$$\begin{aligned} h_2 &= h_1 + h_3 \\ k_2 &= k_1 + k_3 \\ l_2 &= l_1 + l_3 \end{aligned} \quad (2.21)$$

The angles ϕ_1 and ϕ_2 between lines from the centre spot to the two spots measured and the correct combinations of ($h_1k_1l_1$), ($h_2k_2l_2$), ($h_3k_3l_3$) may be checked using the formula

$$\cos \phi = \frac{h_1h_2 + k_1k_2 + l_1l_2}{(h_1^2 + k_1^2 + l_1^2)^{1/2}(h_2^2 + k_2^2 + l_2^2)^{1/2}} \text{ (cubic)} \quad (2.22)$$

or the equivalent formula for other crystal structures from appendix 1. Any remaining spots in the pattern may be indexed by addition of vectors, see figure 2.20(b), that is $h_3 = h_2 - h_1$, $k_3 = k_2 - k_1$, $l_3 = l_2 - l_1$ bearing in mind that spots opposite to an (hkl) spot and the same distance from the centre spot are indexed with opposite sign, that is ($\bar{h}\bar{k}\bar{l}$).

(b) *Determination of B*.* This may be done as follows.

(i) Simple analysis. The zone axis $[uvw] = z = B$, the beam direction, may be determined using the relations

$$\begin{aligned} u &= k_1l_2 - k_2l_1 \\ v &= l_1h_2 - l_2h_1 \\ w &= h_1k_2 - h_2k_1 \end{aligned} \quad (2.23a)$$

that is

$$\begin{array}{ccc} h_1 & | & k_1l_1 & h_1k_1 & | & l_1 \\ & | & \times & \times & | & \\ h_2 & | & k_2l_2 & h_2k_2 & | & l_2 \end{array}$$

where spot ($h_1k_1l_1$) is positioned anticlockwise around the centre spot relative to spot ($h_2k_2l_2$). Equivalently, expressing the result in terms of the g vectors corresponding to ($h_2k_2l_2$) and ($h_1k_1l_1$), that is g_2 and g_1 , respectively.

$$B = g_1 \wedge g_2 \quad (2.23b)$$

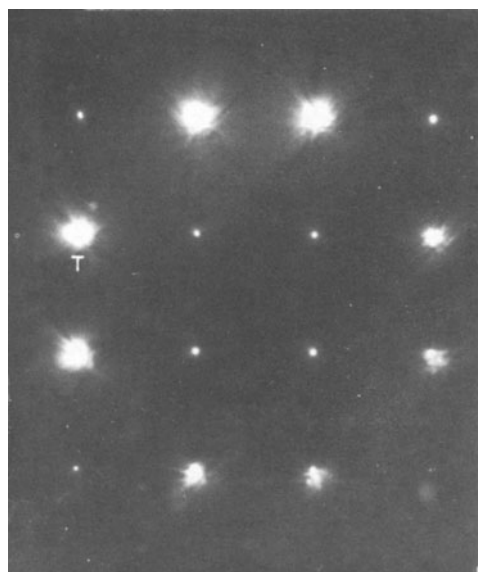
Using this anticlockwise indexing rule, B is defined as the upward drawn normal from the print of the diffraction pattern (negative emulsion side up), that is the upward drawn direction antiparallel to the incident electron beam. In fact figure 2.20(a) is a simple diffraction pattern from pure nickel ($a = 3.523 \text{ \AA}$). The distances in the diffraction pattern measured on the negative are $R_2 = 5.5 \text{ mm}$, $R_1 = 13.9 \text{ mm}$ and $R_3 = 14.25 \text{ mm}$ and angles $\phi_1 = 82^\circ$, $\phi_2 = 76^\circ$. The camera constant $L = 11.2 \text{ mm \AA}$ and, using $Rd = \lambda L$, the d spacings corresponding to R_1 , R_2 and R_3 are $2.038 \text{ \AA}$, $0.805 \text{ \AA}$ and $0.784 \text{ \AA}$ respectively. These values correspond to the interplanar spacings for {111}, {331} and {420} planes. Specific values of h , k and l for each reflection may be obtained by arbitrarily deciding that $h_2k_2l_2 = (111)$ and combining pairs of indices for $h_1k_1l_1$ and $h_3k_3l_3$ which add vectorially to give (111). Suitable pairs are ($\bar{3}31$) ($\bar{4}20$); ($\bar{3}31$) ($\bar{2}40$); ($\bar{3}13$) (402), etc. Choosing the first of these pairs, the angular relationships between the ($\bar{3}31$), ($\bar{4}20$) and (111) planes may be confirmed using equation (2.22) and the remainder of the diffraction pattern indexed vectorially, see figure 2.20(c). Using the zone law, equations (2.23) above, $B = [\bar{1}\bar{2}3]$.

An alternative final step instead of using equations (2.23) involves use of the stereogram. Draw a great circle through (111) pole on the stereogram such that the angle between (111) and ($\bar{2}\bar{1}0$) (that is ($\bar{4}20$) f.c.c. crystal) is 76° and that between ($\bar{3}31$) and (111) is 82° , see figure 2.20(d). The pole of this circle is $[\bar{1}\bar{2}3]$ which is the beam direction. This can be checked by using the relations (2.22), (2.23) and should be clear from figure 2.20(e) in which the zone axis of the reflecting planes is B .

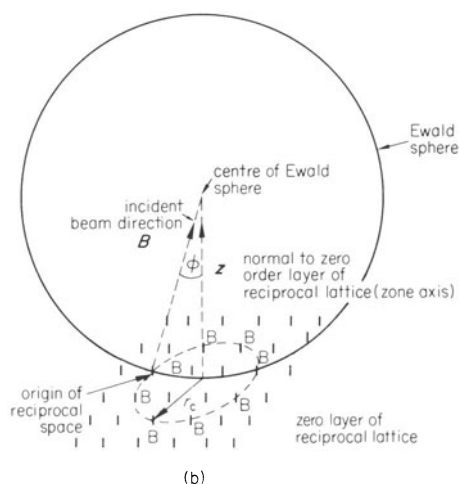
In the case of cubic structures the symmetry of the pattern may be directly compared with those standard patterns shown in appendix 4, using the distances from the centre spot to diffracted spots and angles between g vectors as a check. However, in the hexagonal system there are some difficulties in direct comparison with standard patterns because, for a given c/a , patterns are often very similar for different beam directions. Consequently, direct comparison with standard patterns may be unsatisfactory until a series has been generated with exactly the same camera length so that the two negatives may be superimposed. If not, patterns must be indexed using the above technique.

(ii) More accurate analysis: ring of bright spots. It is important to realise that, because of

* The approach given here is for cubic crystal structures.



(a)



(b)

Figure 2.21 (a) A spot pattern (ring of bright spots) with B close to $[100]$ in aluminium and (b) the equivalent Ewald sphere position in reciprocal space

streaking of the reciprocal lattice points perpendicular to the specimen surface, the spot pattern will remain visible when the specimen is tilted away from an exact zone axis. If equation (2.23) is used to determine the zone axis, this is no longer parallel to B and significant errors of up to 15° in an undeformed specimen and 30° in a deformed specimen can result (Laird *et al.*, 1966). However, this may be recognised from the difference in intensity of the spots. An example is shown in figure 2.21, in which the incident beam direction (figure 2.21(b)) is such that the Ewald sphere cuts the zero layer of reciprocal space in a circle whose centre is to the right of the origin. Thus $s = 0$ for a ring of bright spots including the transmitted spot T visible in the diffraction pattern, see figure 2.21(a). Then B is a few degrees ϕ away from the simple zone axis z corresponding to the spot

positions. This angle ϕ may be determined from the relation

$$\sin \phi = r_c \lambda \quad (2.24)$$

where r_c is the radius of the ring of bright spots. Thus B may be obtained to within $\pm 1^\circ$ because it lies ϕ° from z along the crystallographic direction corresponding to the diameter through the transmitted spot T of the ring of bright spots. In the case of the diffraction pattern in figure 2.21(a) $z = [100]$ and B is 3° from $[100]$ along the $[010]$ direction.

It is important to realise that, because of the 180° ambiguity in indexing diffraction patterns, discussed in detail in section 2.8.1, the direction of tilt of B away from the simple zone axis $[100]$ above is not known because two possibilities exist.

2.7.2.2 Complicated patterns—spots arising from different zones

Indexing the pattern. Many electron diffraction patterns from single-crystal specimens are not related to a single section through the reciprocal lattice, particularly in those materials with large lattice parameters and therefore closely spaced reciprocal lattice points. Two situations can occur.

(1) If the crystal is very thin, the streaks at the reciprocal lattice points will be correspondingly long, section 2.4 and figure 2.16. Consequently those from the next layer 1 above the 0 layer in reciprocal space, figure 2.13, will cut the Ewald sphere as it curves up out of the bottom layer. Referring to figures 2.13 and 2.17 it will be clear that this will introduce additional spots only at the edge of the diffraction pattern.

(2) If the crystal is thin, the diffraction pattern consists of strips of fragmentary arrays of spots when the incident beam is not near a simple low-index crystal direction. This situation should generally be avoided as it complicates indexing the pattern because each strip of spots comes from a different layer of the reciprocal lattice 0, 1, 2, etc., figure 2.13. The simplest method of avoiding it is to tilt the specimen until the incident beam is parallel to a low-index crystal direction.

In indexing spots from different layers of reciprocal space, which are known as Laue zones, the following relationships may be used:

$$\text{zero-order Laue zone } hu + kv + lw = 0 \quad (2.25)$$

$$\text{first-order Laue zone } hu + kv + lw = 1 \quad (2.26)$$

Normally a single-crystal pattern, such as that in figure 2.1, only contains spots from the zero-order Laue zone and equation (2.25) applies. However, if at the edge of such a pattern additional spots appear that cannot be indexed

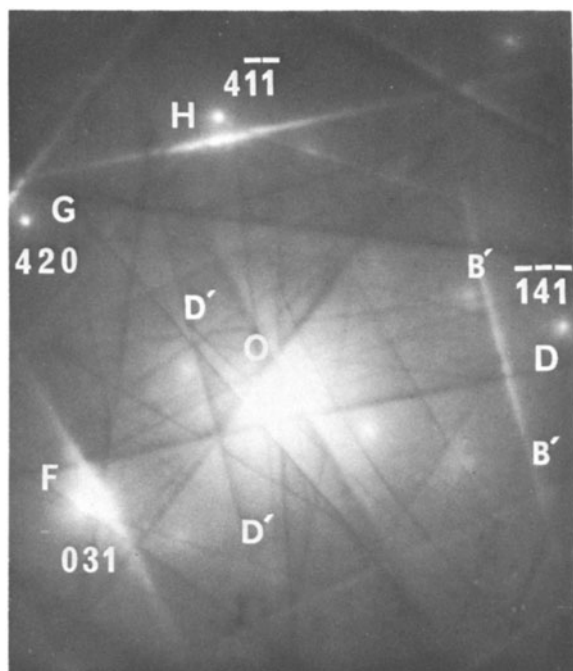


Figure 2.22 A combined spot and Kikuchi line pattern from fully annealed α -iron showing two zones of spots [Courtesy of C. A. Shell]

using the normal rules, section 2.7.2.1, then equation (2.26) must be used.

Note that equation (2.25) always applies, independent of the incident beam direction. However, the integer in equation (2.26) does change but the value can be readily found by inspection. Thus, for an f.c.c. crystal and $[uvw] = [111]$, $N = 0, 1, 2, 3$ etc. However, for $[uvw] = [112]$, $N = 0, 2, 4, 6$, etc. In practice the spots do not correspond exactly to their calculated positions because of the curvature of the Ewald sphere.

2.7.2.3 Imperfect patterns

It is often found that spot patterns are imperfect containing several spots from different zero-order

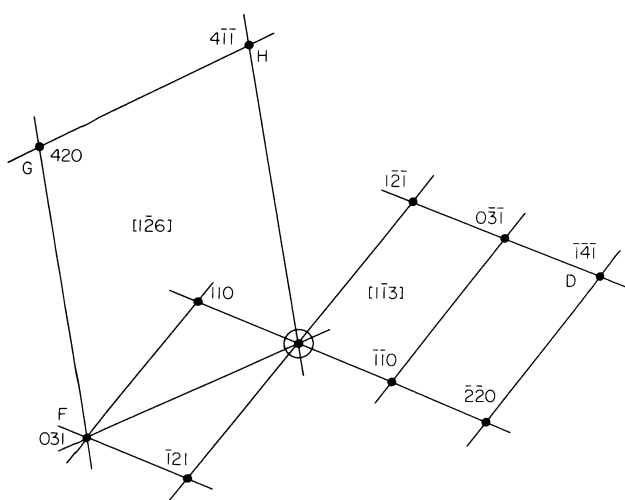


Figure 2.23 The indexed spots in figure 2.22

Laue zones, see, for example, figure 2.22 which clearly contains two sets of spots delineated in figure 2.23. This situation is particularly important because it removes a source of ambiguity in indexing the single Laue zone spot patterns which is discussed in detail later in section 2.8.1.

(a) *Indexing the pattern.* The procedure to be followed in indexing imperfect patterns has been outlined by Ryder and Pitsch (1968). There are two experimental cases to consider, see figure 2.24,

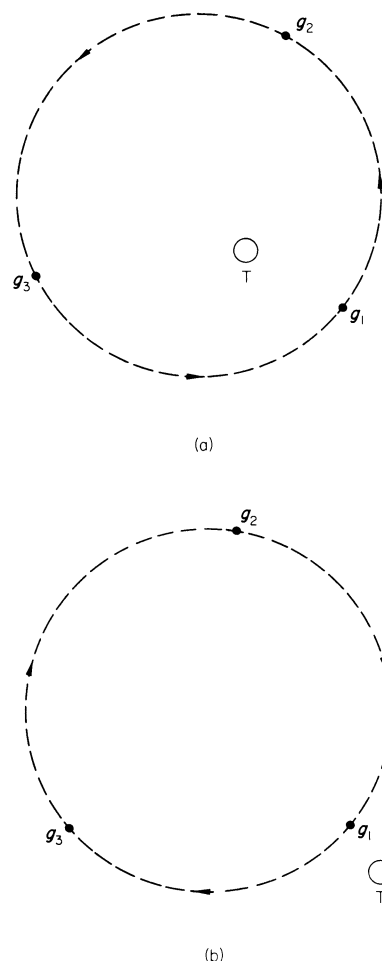


Figure 2.24 The method of indexing electron diffraction patterns for right-handed axes. g_1 , g_2 and g_3 are three strong reflections not all lying in the same zone. These spots are numbered (a) anticlockwise or (b) clockwise, depending upon whether the transmitted spot T lies inside or outside the circle through the spots

in which three vectors of interest, that is to bright spots, have been labelled g_1 , g_2 and g_3 . The two cases correspond to the transmitted spot T inside or outside a circle through g_1 , g_2 and g_3 . Physically these situations correspond respectively to the origin of the reciprocal lattice lying below or above the plane defined by the three spots g_1 , g_2 and g_3 . For these two cases the spots must be

labelled anticlockwise and clockwise respectively. The procedure is as follows.

(1) Evaluate the type $\{hkl\}$ of each spot on the basis of the camera constant or the method of ratios, section 2.7.2.1.

(2) Arbitrarily index one spot with specific (hkl) then label the spots clockwise or anticlockwise around the circle depending on the situation outlined in figure 2.24. The product $\mathbf{g}_1 \wedge \mathbf{g}_2$ may be used to determine the zone axes of each pair of points. All zone axes should be closely spaced on the stereogram, that is have compatible indices. For example, in figure 2.23, the zone axes for spots D, H; H, G; G, F are $[3, \bar{5}, 17]$; $[1, \bar{2}, 6]$; $[1, \bar{2}, 6]$; respectively.

(3) Check that the triple products $\mathbf{g}_1 \cdot (\mathbf{g}_2 \wedge \mathbf{g}_3)$ are positive ensuring consistency in indexing. If it is negative the sign of each reciprocal lattice vector must be reversed. Using this approach the SADP in figure 2.22 may be indexed as shown in figure 2.23.

(b) *Determination of B.* The 'beam direction' $\mathbf{B}$ may be determined using the relation

$$\mathbf{B} = |\mathbf{g}_1|^2 (\mathbf{g}_2 \wedge \mathbf{g}_3) + |\mathbf{g}_2|^2 (\mathbf{g}_3 \wedge \mathbf{g}_1) + |\mathbf{g}_3|^2 (\mathbf{g}_1 \wedge \mathbf{g}_2) \quad (2.27)$$

Each cross product corresponds to a zone axis close to $\mathbf{B}$ and the terms $|\mathbf{g}_1|^2$, etc., correspond to a weighting factor of this zone axis in relation to $\mathbf{B}$. For details, see Ryder and Pitsch (1968).

Using spots D, G, E of figure 2.23 then

$$\mathbf{B} = 18 \begin{vmatrix} i & j & k \\ 4 & 2 & 0 \\ 0 & 3 & 1 \end{vmatrix} + 20 \begin{vmatrix} i & j & k \\ 0 & 3 & 1 \\ \bar{1} & \bar{4} & \bar{1} \end{vmatrix} + 10 \begin{vmatrix} i & j & k \\ \bar{1} & \bar{4} & \bar{1} \\ 4 & 2 & 0 \end{vmatrix}$$

$$\mathbf{B} = 18(2i - 4j + 12k) + 20(i - j + 3k) + 10(2i - 4j + 14k)$$

$$\mathbf{B} = 76i; -132j; 416k$$

$$\mathbf{B} = 1; -1.737; 5.474$$

In this case the beam direction $\mathbf{B}$ is $1; -1.737; 5.474$.

2.7.3 Kikuchi Patterns

If $\mathbf{B}$ is a low-index direction in the crystal, Kikuchi patterns are simple with a high degree of symmetry. Figure 2.25 shows an example. These standard patterns may then be used to identify $\mathbf{B}$ by direct comparison. However, more complex patterns frequently occur and these may be recognised by their lack of symmetry.

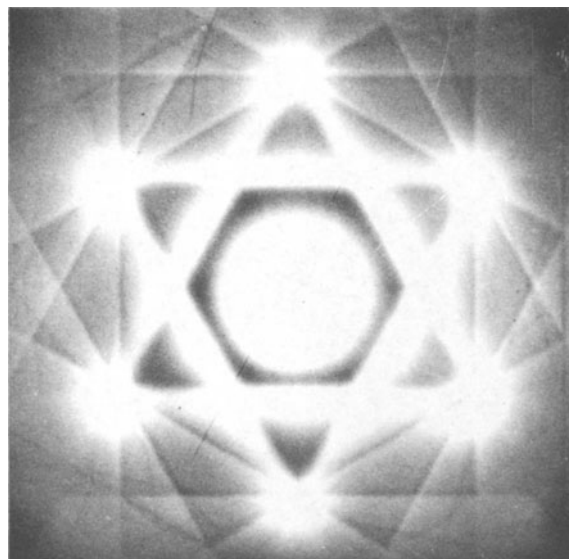


Figure 2.25 Kikuchi patterns with $\mathbf{B}[111]$ in aluminium (f.c.c.) [Courtesy of D. B. Williams]

The two features that enable a Kikuchi line pair (parallel black and white lines, section 2.6.3) to be indexed are as follows.

(1) Kikuchi line spacing in the diffraction pattern which is directly related to the spacing of the operative crystal planes by the equation $Dd = \lambda L$, where D is the spacing of the Kikuchi line pair.* There is no error arising because of deviation from the exact Bragg position comparable with that in spot patterns because the Bragg law is always exactly satisfied for Kikuchi lines—no deviation is permissible on the grounds of specimen shape. Any error arising from the difference in wavelength of the inelastically scattered electrons is less than the experimental error in measuring the Kikuchi line spacing.

(2) The angles between Kikuchi lines from different $\{hkl\}$ planes correspond to the angles between the planes because the Kikuchi lines are parallel to the trace of the planes, see figure 2.19(c).

2.7.3.1 Simple patterns—Kikuchi lines in the same zone

(a) *Indexing the pattern.* The procedure for indexing the pattern is identical with that outlined in section 2.7.2.1 for spot patterns except that the spacing D of the line pair is substituted in the camera constant relation to obtain d_{hkl} .

The angle between Kikuchi pairs is used to obtain the angle between the crystal reflecting planes to use in relation (2.22).

(b) *Determination of B.* In general if all Kikuchi lines are from one zero-order Laue zone only, $\mathbf{B}$

* It has been reported by Tan *et al.* (1971) that Kikuchi line spacing in very thick crystals is not characteristic of the interplanar spacing of the operative reflecting planes but such difficulties should not be encountered if spots are still visible in the diffraction pattern.

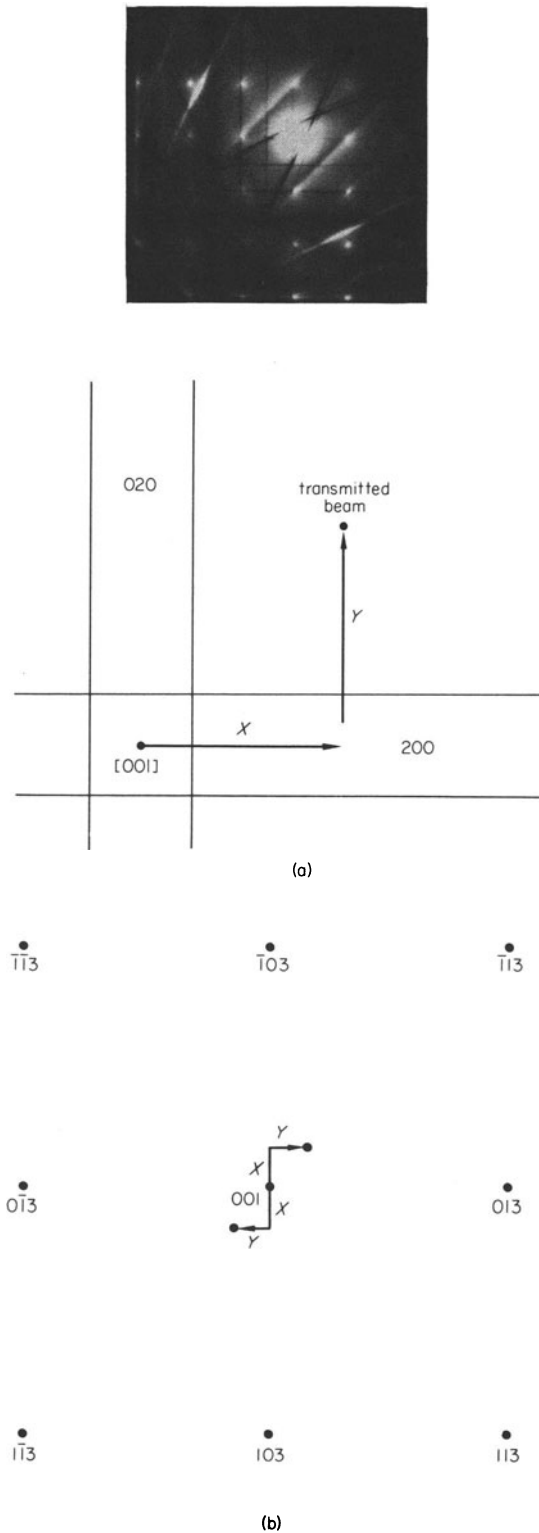


Figure 2.26 A diffraction pattern (a) with B close to, but $X^\circ + Y^\circ$ away from, $[100]$. Alternative and indistinguishable B are shown in (b) on the central portion of a $[100]$ stereogram

will be close to a simple zone axis and it will only be necessary to determine the deviation of B from it. Thus the approach is similar to the ring of bright spots analysis discussed in section 2.7.2.1(b) and the same 180° ambiguity is present in indexing the pattern, see section 2.8.1, so that there will be two possible directions of B .

An example of the determination of the angle between B and z is shown in figure 2.26 for which B is close to $[001]$ and the Kikuchi line pairs have been indexed as shown. Then B , defined as the location of the centre spot relative to the $[001]$ Kikuchi line centre is a tilt of X° about $[010]$ and Y° about $[100]$. In order to calibrate distance

against angle, the spacing D of a pair of Kikuchi lines is measured, then $\sin 2\theta = D/2L$, where L is the camera length. In the case of figure 2.26 $X^\circ = 2.1$, $Y^\circ = 2.1$, and the two alternative positions of B may be plotted on the stereogram as shown in figure 2.26(b).

2.7.3.2 Complex patterns—Kikuchi lines from different zones

(a) *Indexing the pattern.* In this case the most accurate analysis is that due to von Heimendahl *et al.* (1964) which requires a minimum of three non-parallel Kikuchi line pairs to obtain B unambiguously from one pattern. Three such pairs are shown schematically in figure 2.27. The dashed lines are the traces of the intersection of the $\{hkl\}$ planes producing the Kikuchi lines with the Ewald sphere, see figure 2.19(c). Therefore, points A, B and C correspond to the zone axis of intersection of the reflecting planes, see figure 2.27(b). Consequently the beam direction OO in figure 2.27(b) is specific angles ϕ_1 , ϕ_2 and ϕ_3 from

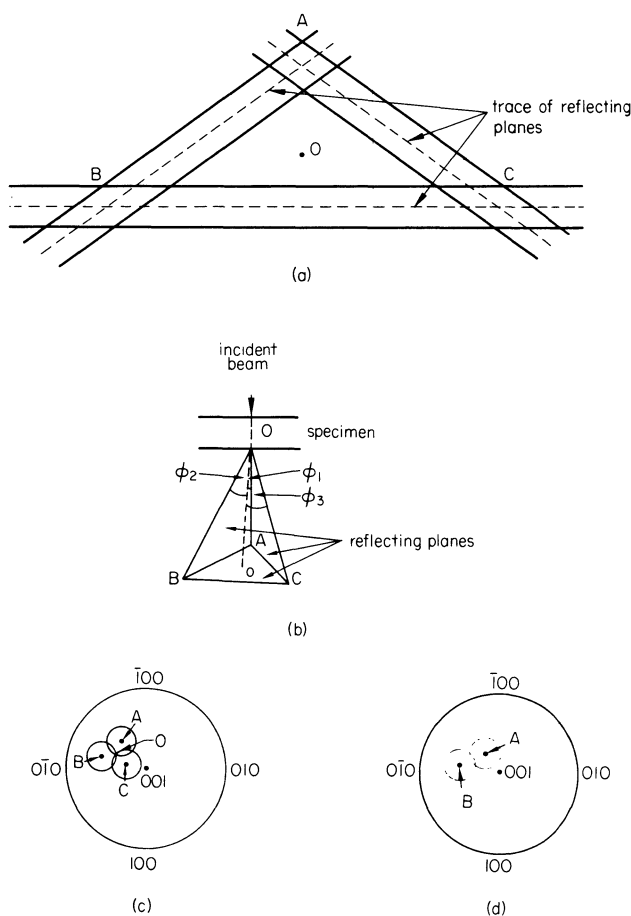


Figure 2.27 (a) Three pairs of Kikuchi lines whose medians, that is projections of reflecting planes, intersect at A, B, C, (b) the projection of the responsible reflecting planes relative to the specimen. (c) The projection of the incident B on the stereogram. (d) The same situation as (c) but for only two Kikuchi line pairs

each direction OA , OB , OC , the latter being indexed using the zone law equations (2.23). Thus the beam direction B is located on the stereogram, at the intersection of three small circles, radius ϕ_1° , ϕ_2° and ϕ_3° about poles A, B and C, see figure 2.27(c). (It should now be clear that two Kikuchi pairs give two possible values for the beam direction, see figure 2.27(d) and therefore three pairs are needed for an unambiguous result.) The values of ϕ_1 , ϕ_2 , ϕ_3 are obtained from the relation

$$\tan \phi = R/L \quad (2.28)$$

where R is the distance OA , OB , OC , etc., and L is the camera length. Alternatively and more accurately we may obtain the beam direction analytically. If the indices of B are u, v, w and p, q, r , etc., are the indices of the poles ABC, etc.

$$\cos \phi_1 = \frac{up_1 + vq_1 + wr_1}{(u^2 + v^2 + w^2)^{1/2}(p_1^2 + q_1^2 + r_1^2)^{1/2}} \quad (2.29)$$

Inserting values for ϕ and pqr we can solve the three simultaneous equations obtained for u, v, w . An example of the use of this approach is given below for the case of magnesium (c/a , 1.62) using the Kikuchi line picture in figure 2.28(a). By using the method of ratios, section 2.7.2.1, with the interline spacing and comparing with ratios of d spacings it can be shown that band 1 = $\{10\bar{1}1\}$ type, 2 = $\{20\bar{2}1\}$ type and 3 = $\{12\bar{3}1\}$ type. Then band 1 is arbitrarily chosen as $(01\bar{1}1)$. We now use the angles between bands which can be calculated using equation (A2.6), appendix 2, or using tables of angles. Possible angles between $\{01\bar{1}1\}$ and planes of the $\{20\bar{2}1\}$ and $\{12\bar{3}1\}$ types are shown in table 2.3.

The measured angles between bands $\widehat{1, 2}$ and $\widehat{1, 3}$ are 57° and 75° respectively so they are $(20\bar{2}1)$ or $(\bar{2}201)$ and $(3\bar{2}11)$ or $(3\bar{1}21)$ respectively.

The possible angles between these bands are

$$(20\bar{2}1) \text{ and } (3\bar{2}11) = 123.81^\circ$$

$$(20\bar{2}1) \text{ and } (3\bar{1}21) = 19.12^\circ$$

$$(\bar{2}201) \text{ and } (3\bar{2}11) = 19.12^\circ$$

$$(\bar{2}201) \text{ and } (3\bar{1}21) = 123.81^\circ$$

and comparison with measured angles give two possibilities

	band 1	band 2	band 3
A	$(01\bar{1}1)$	$(20\bar{2}1)$	$(3\bar{1}21)$
B	$(01\bar{1}1)$	$(\bar{2}201)$	$(3\bar{2}11)$

These alternatives A and B may be resolved using the double stereogram in figure 2.28(b) which is based on figure A2.5, appendix 2, and plotting the positions of the planes. The sense of rotation of the alternatives A and B determined

from the stereogram leads to the two possible distributions of Kikuchi lines shown in figure 2.28(c). Clearly **B** is correct and we can index the intersections *x*, *y* and *z* of the projections of the planes producing Kikuchi bands as $[1\bar{2}16]$, $[0\bar{1}12]$ and $[1\bar{5}49]$ respectively.

(b) *Determination of B.* The distances between the centre of the pattern and poles *x*, *y* and *z* are measured and converted into degrees using the fact that $2\theta(\bar{3}211) = \text{spacing of the } (\bar{3}211) \text{ Kikuchi pair}$. We find that the beam direction **B** is 3.1° from $[1\bar{5}49]$, 11.1° from $[1\bar{2}16]$ and 6.5° from $[0\bar{1}12]$.

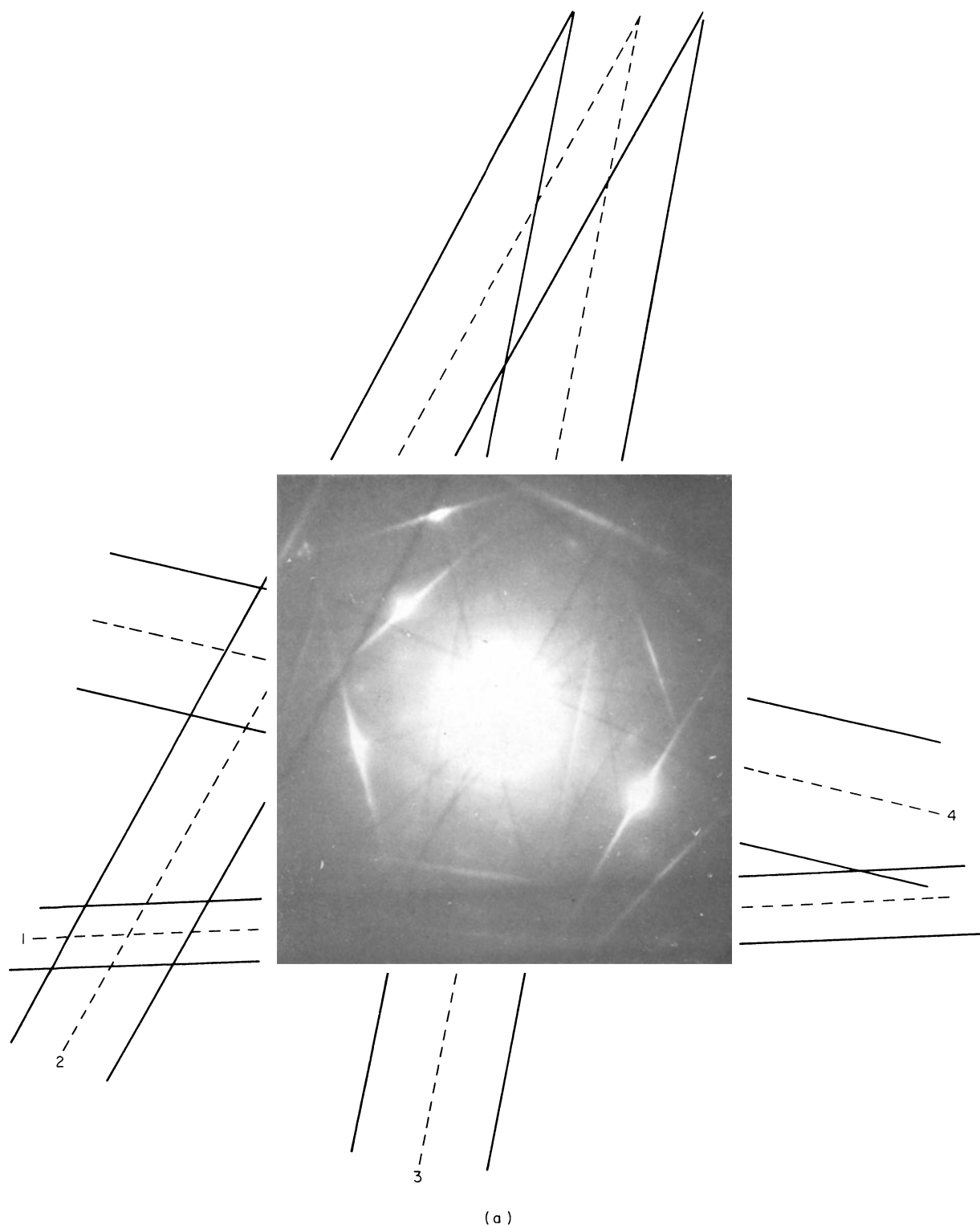


Figure 2.28 (a) A diffraction pattern from magnesium with Kikuchi line pairs marked, (b) the equivalent double stereogram showing projection of planes and directions, see appendix 2. (c) Possible Kikuchi line arrangements, (d) correctly indexed, and (e) the stereogram with **B** indicated

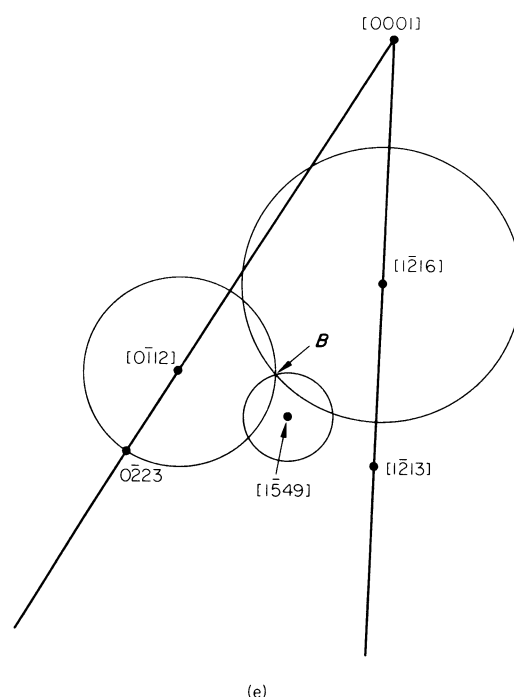
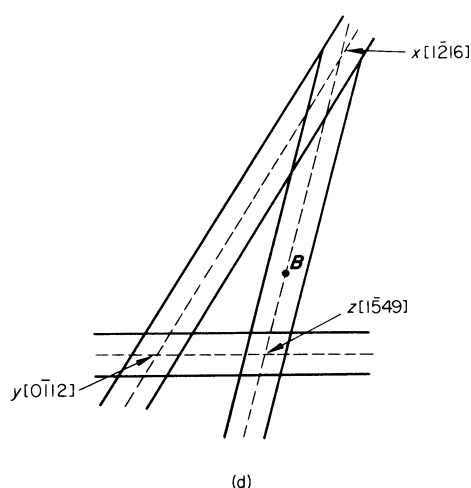
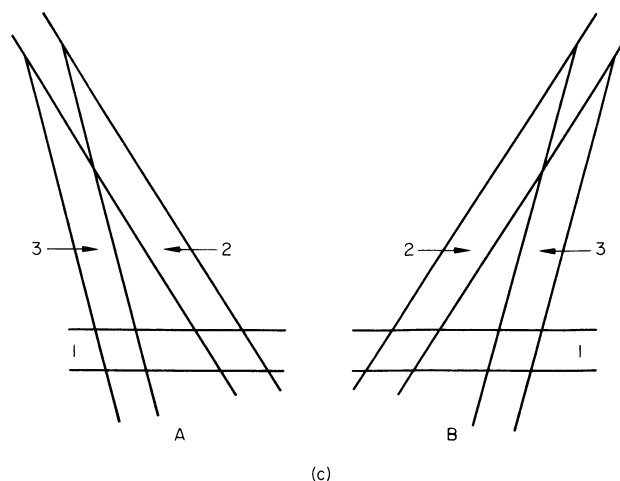
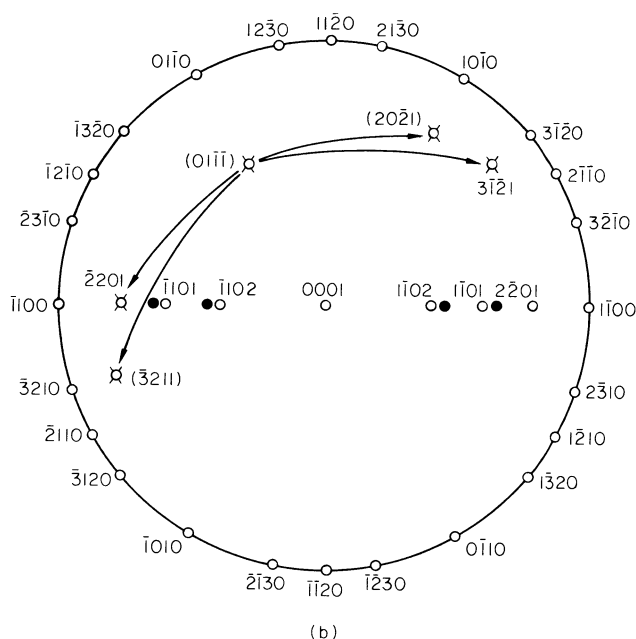


Figure 2.28 (continued)

There are now two alternatives. Small circles may be constructed on the direction stereogram to plot **B**: a section is shown in figure 2.28(e). Alternatively the indices of **B** may be determined accurately by solving the simultaneous equations of the form of equation (A2.6), appendix 2. If this is done the indices of **B** are 1, -6.024, 5.04, 13.98.

Alternatively, Thomas (1970) has pointed out that, if **B** is exactly between two intersecting

Kikuchi pairs, the indices of **B** may be found by calculating the zone axis of the two bands. In figure 2.28(a), **B** is almost at the centre of Kikuchi pairs 3 and 4 and the indices of 4 are (1232). Using the zone law $\mathbf{B} = [\bar{1}438]$ and the angle between the two beam directions determined above is 2° . Such errors arise because the centre spot is not exactly at the centre of the crossing Kikuchi pairs and because the error in measuring **B**, figure 2.28(a),

Table 2.3 Angles between (0111) and planes of the form {2021} and {1231}

Kikuchi lines 1 and 2, figure 2.28(a)	Kikuchi lines 1 and 3, figure 2.28(a)
(0111) and (0221) = 13.16°	(0111) and (1231) ($\bar{1}321$) = 24.47°
(0111) and (2021) ($\bar{2}201$) = 56.79°	(0111) and (2131) ($\bar{2}311$) = 41.68°
(0111) and (0221) = 136.71°	(0111) and ($\bar{3}211$) ($3\bar{1}21$) = 75.12°
(0111) and ($\bar{2}201$) ($\bar{2}021$) = 107.91°	(0111) and ($\bar{3}121$) ($3\bar{2}11$) = 94.01°
	(0111) and ($\bar{2}131$) ($2\bar{3}11$) = 124.06°
	(0111) and ($\bar{1}231$) ($1\bar{3}21$) = 136.34°

is significant when the Kikuchi lines cross at a small angle.

2.7.3.3 Kikuchi maps

Kikuchi maps described by Levine *et al.* (1966) and Okamoto *et al.* (1967) are useful because they can help to eliminate the trial and error stage of indexing Kikuchi patterns described in the previous section. Furthermore, an approximate estimate of $\mathbf{B}$ to within $\sim 2^\circ$ can be rapidly obtained, and the ambiguity of indexing diffraction patterns may be removed, see section 2.8.1. Kikuchi maps consist of a montage of Kikuchi patterns taken with different $\mathbf{B}$ positioned in the corresponding place in the unit triangle of the stereogram such that all the Kikuchi lines join up to produce a 'road map' of the unit triangle. The method of making Kikuchi maps is discussed in appendix 5 which also includes maps for specific crystal structures. Examples of the use of Kikuchi maps are described in sections 2.8.1, 2.10.1, 2.11.2 involving tilting the specimen in a known sense so that $\mathbf{B}$ is always known within the unit triangle; tilting from one specific $\mathbf{B}$ to another; measurement of the deviation from the Bragg position and removal of the ambiguity in indexing diffraction patterns.

The approximate determination of $\mathbf{B}$ involves the location of the diffraction pattern on the Kikuchi map by inspection. If the position of this point on the map is shown to be $\mathbf{B}$ in the schematic diagram in figure 2.29, then the (hkl) indices of the pole $\mathbf{B}$ are given by $h:k:l = \cos \beta : \sin \beta \cos \alpha : \sin \alpha \sin \beta$. In view of the errors associated with Kikuchi maps ($\sim \pm 2^\circ$), see appendix 5, similar errors can be expected in the determination of $\mathbf{B}$. Alternatively the angular displacement of the located $\mathbf{B}$ from three specific low-index poles, for example $[112]$, $[110]$, $[100]$, etc., may be measured in the Kikuchi map. $\mathbf{B}$ may then be located on the stereogram at the intersection of three small circles centred at these low-index poles and having the correct angular radii as described in the example shown in figure 2.28 (previous section). If greater accuracy is required, equations (2.29) may be solved as described previously.

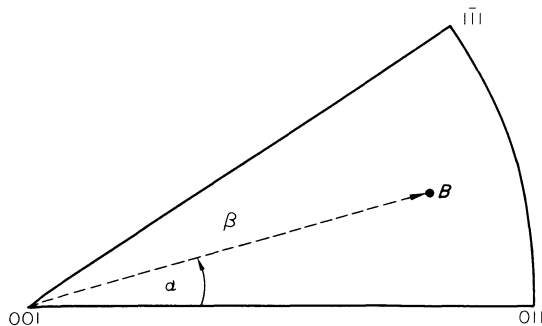


Figure 2.29 The determination of $\mathbf{B}$ from the Kikuchi map

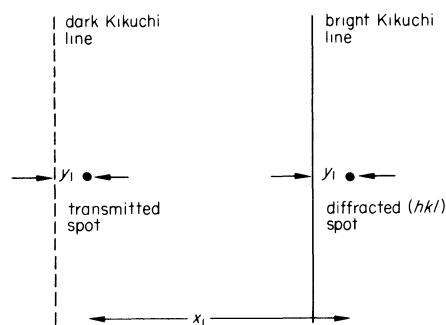


Figure 2.30 The parameters in equation (2.30)

2.7.3.4 Combinations of Kikuchi lines and spots

The approach outlined in section 2.7.2.3 may be employed to index the lines and spots using the rules of Ryder and Pitsch (1968) to obtain consistent indexing.

The beam direction $\mathbf{B}$ may be determined by using the equation

$$\mathbf{B} = \alpha_1 |\mathbf{g}_1|^2 (\mathbf{g}_2 \wedge \mathbf{g}_3) + \alpha_2 |\mathbf{g}_2|^2 (\mathbf{g}_1 \wedge \mathbf{g}_3) + \alpha_3 |\mathbf{g}_3|^2 (\mathbf{g}_1 \wedge \mathbf{g}_2) \quad (2.30)$$

due to Ryder and Pitsch. Here $\alpha_1 = (x_1 + 2y_1)/x_1$, where x_1 and y_1 are the parameters shown in figure 2.30. The sign of x is always positive but the sign of $y = \text{sign of } s$, that is positive if the Kikuchi line is outside the spot and negative if inside, see section 2.10.2. Using the Kikuchi line pairs corresponding to the spots D, G, F of figure 2.22, a value of $\mathbf{B} = 1; -1.611; 5.108$ is obtained.

2.7.4 Summary of Procedures for Indexing Single-crystal Patterns

2.7.4.1 Spot patterns—identity of material known

(1) Measure the distance R from the centre spot of three diffraction spots which form a parallelogram.

(2) Convert these distances into interplanar (d) values via the camera constant. (Camera constant method, section 2.7.2.1.) Alternatively if the camera constant is not known use the method of ratios (section 2.7.2.1).

(3) Using a table of interplanar spacings for the particular substance, correlate the measured d values with actual d values and thus obtain the indices $\{hkl\}$ for each spot.

(4) Measure the angles (ϕ) between the chosen diffraction spots.

(5) Arbitrarily index one of the closest-in spots with particular (hkl) indices.

(6) Choose specific combinations of h , k and l for two additional spots which may be added vectorially to obtain the first arbitrary (hkl) value in (5) above.

(7) Use equation (2.22), or similar for specific crystal structure (section A1.8) to check the angular

relationships between the three spots. Use equations (2.23), to obtain **B**. Completely index the remaining spots by (a) continuing a sequence of indices along a prominent direction, and (b) adding two sets of indices of prominent directions, equation (2.21). Cross-check answers with equations (2.22) and (2.23). All the indices should satisfy the condition (2.25) near the centre of the pattern, but on moving outwards it may be necessary to change to equation (2.26), etc.

2.7.4.2 Spot patterns—identity of material unknown

(1) Obtain a number of patterns using different **B** parallel to simple zone axes, that is simple patterns with a high spot density. For preference do this with high tilt specimen holder tilting a known amount about specific reflections, see section 2.10.1.3.

(2) Measure all distances of spots from centre and check common types of reflection.

(3) Use symmetry properties of diffraction patterns with the above information to estimate type of structure, cubic, tetragonal, hexagonal, etc., using the rules in table 2.2.

(4) Choose a simple pattern with the maximum symmetry (six-, four-, three-fold) and attempt to index it by trial and error using measured *d* spacings and interplanar angles together with the formula in appendix 1, and rules of indexing, section 2.7.2.1.

(5) Progress through remaining patterns until all are completely self-consistent, both in terms of indexing and applied angle of tilt to move from one **B** to another.

2.7.4.3 Kikuchi lines—identity of material known

As for spot patterns, see section 2.7.4.1, (1)–(4), using the Kikuchi line pair spacing instead of spot spacing.

Arbitrarily index one Kikuchi line pair then use approach in section 2.7.3.1 to index remaining pattern and determine **B** to the accuracy required.

2.7.4.4 Kikuchi lines—identity of material unknown

This situation is not recommended alone, but is a useful check on the spot approach, section 2.7.4.2, above using the real crystal symmetry of Kikuchi patterns (see section 2.6.3) with **B** as a simple zone axis.

2.7.5 Accuracy of the Determination of **B**

The following discussion assumes that the methods outlined in section 1.5.1 have been followed to maximise the accuracy with which the SADP have been obtained.

If all of the spots in the diffraction patterns are of uniform intensity, that is **B** very close to simple zone axis, for example figure 2.1, and the approach outlined in section 2.7.2.1 is employed, **B** is accurate to $<0.5^\circ$. The intensity of the spots are a maximum when $s = 0$. For exact **B**, all first-order spots should have the same intensity.

If the ring of bright spots approach, section 2.7.2.1(b), is used to determine the angle between **B** and a simple zone axis the angle may be determined to $<0.5^\circ$. However, the error in **B** is $>1^\circ$ because of inaccuracies in determining the position of the tilt axis.

If care is not taken to obtain a uniformly intense set of spots, errors of up to 15° in **B** are possible for very thin annealed specimens. Errors of up to 30° can occur for heavily cold-worked specimens (Laird *et al.*, 1966). Using the approach of von Heimendahl *et al.* (1964), errors of $\pm 0.1^\circ$ are

Table 2.4 Estimates of the indices of the 'beam direction' [*uvw*] of the α -iron diffraction pattern in figure 2.22 using various methods; the spots are labelled as in figure 2.22, Δr is the deviation in minutes from the mean of the determinations by the accurate method of Pumphrey and Bowkett (1970a)

(a) Spots and Kikuchi lines

Spots	<i>u</i>	<i>v</i>	<i>w</i>	Δr
F, D	1.0	−1.66891	5.30684	29.3'
D, H	1.0	−1.60502	5.06929	3.6'
D, G	1.0	−1.59678	5.03801	4.9'
H, G	1.0	−1.59517	5.06052	1.0'
H, F	1.0	−1.59001	5.05594	3.3'
G, F	1.0	−1.59450	5.07019	3.5'
mean	1.0	−1.59630	5.05879	—

(b) Zones of spots

Spots	<i>u</i>	<i>v</i>	<i>w</i>	Δr
G, F	1	−2	6	115'
F, D	1	−1	3	416'
D, G	1	−2	7	195'
D, H	3	−5	17	92'

(c) Ryder and Pitsch analyses

(i) Spots

(ii) Spots plus Kikuchi lines

Spots	<i>u</i>	<i>v</i>	<i>w</i>	Δr
D, H, F (i)	1.0	−1.66667	5.23810	23.4'
(ii)	1.0	−1.60783	5.09697	4.7'
D, H, G (i)	1.0	−1.66667	5.06667	41.1'
(ii)	1.0	−1.60689	5.08781	3.7'
D, G, F (i)	1.0	−1.73684	5.47368	48.2'
(ii)	1.0	−1.61106	5.10809	6.0'
mean (ii)	1.0	−1.60859	5.09762	4.8'

(d) Kikuchi line analysis

Kikuchi centres	<i>u</i>	<i>v</i>	<i>w</i>	Δr
HG, DH, DG	1.0	−1.57584	5.00552	7.2'
HG, DH, FD	1.0	−1.59859	5.09570	7.0'

usually obtained although this can increase very significantly if two of the Kikuchi centres and the origin are nearly collinear.

Using spot/Kikuchi line combinations as outlined in section 2.7.3.4, errors of $\pm 0.1^\circ$ in B are to be expected.

2.7.6 Practical Comparison Between Methods of Analysing Diffraction Patterns

The methods outlined in sections 2.7.2 and 2.7.3 have all been applied to figure 2.22 by Pumphrey (1970) which has enabled estimates of the errors in determination of the beam direction to be obtained for a specific case. The results are shown in table 2.4.

In this case, the technique using spots and Kikuchi lines devised by Pumphrey and Bowkett (1970a, b) has been used as a standard, that is to obtain a mean value with which to compare the other approaches. The mean was obtained by averaging all values in table 2.4(a) except the first, and all values of Δr represent the deviation of the measurement from this mean. Referring to table 2.4 one may make the following conclusions.

- (1) The errors are approximately as predicted.
- (2) The large errors using the zones of spots approach, table 2.4(b), arise because the spots are of different intensities and B cannot be exactly equal to a zone axis defined by the spots.
- (3) The deviations arising using the Ryder and Pitsch analysis, table 2.4(c) are smaller than the method in table 2.4(b), even when only the spots are used. Clearly this method is to be preferred.

2.7.6.1 Consideration of further sources of error in determining B

Such factors as spherical aberration, errors in lattice parameter or electron wavelength, errors in measurement of the SADP (provided reasonable care is taken to measure an enlarged print) do not significantly influence the accuracy of the methods discussed in sections 2.7.2, 2.7.3. However they may be important if higher accuracy is required, see Pumphrey and Bowkett (1970b). However, significant errors can arise from tilting the specimen through large angles because the direction of g may be incorrect. At high angles of specimen tilt, the direction of g is accurate at $s = 0$ but not if $s \neq 0$. Errors of up to $\pm 5^\circ$ are possible because the reciprocal lattice point is streaked at an angle to the incident beam direction and not parallel to it as it is at zero specimen tilt. This effect is only important if combined Kikuchi line/spot analysis is used, see section 2.7.3.4. Even in this case, provided that Kikuchi lines a long way outside

their spots are not used, the error introduced is less than the errors in measurement. If the dark (hkl) Kikuchi line is closer than $\sim g/4$ to the centre spot in the diffraction pattern, measurements may be safely made (Pumphrey, 1970).

2.7.7 Limitations in the Usefulness of the Various Methods of Determination of B

The value of B is an average over the region selected by the diffraction aperture. If the specimen is bent or has a high defect density the Kikuchi lines become diffuse and become undetectable. Consequently, Kikuchi line analysis techniques are often not applicable to materials which have been deformed or have undergone phase transformations and spot patterns must be used.

Furthermore, each technique has limitations of its own, often of a purely practical nature. For example, the methods of Ryder and Pitsch (1968) or von Heimendahl *et al.* (1964) may be difficult to apply to a particular problem because it is often difficult to obtain a second zone of spots in both phases of a two-phase material for a given tilt setting of the specimen.

2.8 Uniqueness in Indexing Diffraction Patterns

Table 2.5 is a summary of those types of analysis for which a 'unique' solution to single-crystal diffraction patterns is and is not required. Provided the rules outlined in sections 2.7.2, 2.7.3 are followed, there are only two sources of ambiguity in indexing single-crystal diffraction patterns. Both must be removed for unique indexing.

The most significant difficulty is the 180° ambiguity which arises because every diffraction pattern can be indexed by choosing a positive or negative set of indices using the rules in section 2.7.2, compare figure 2.31(a), (b). This reversal of indexing does not change B because $g_1 \wedge g_2 = B = -g_1 \wedge -g_2$. A secondary problem, coincidence ambiguity, may arise if different $\{hkl\}$ planes have the same interplanar spacing, for example $\{355\}$ and $\{173\}$ in the cubic crystal structure. However, this problem may be easily solved by tilting the specimen.

2.8.1 The 180° Ambiguity

This effect is present in all diffraction patterns that contain only spots or Kikuchi lines from one Laue zone of the reciprocal lattice.

It arises because the small Bragg angle θ combined with the deviation $\Delta\theta$ (or s) over which significant diffraction occurs, section 2.4, enables reflection to occur simultaneously from both sides of the reflecting plane. Consequently there are

Table 2.5 Analyses that do and do not require unique indexing of diffraction patterns

Unique indexing required	Unique indexing not required
accurate orientation relationships, for example grain–grain see section 2.11.2; some precipitate–matrix, section 2.11.1	precipitate shape, size and density measurements
determination of the Burgers vector and sense of dislocation loop, sections 3.4.3, 3.5	orientation relationships between precipitate and matrix when epitaxial, that is axis/angle pairs not required, section 2.11.2
determination of the Burgers vector and line direction of dislocations using computer simulation, section 3.4.3	habit plane determinations
	determinations of crystal system
	confirmation of identity of second phases
	determination of specimen thickness
	the applications in part IV
	determination of nature and sense of stacking faults, π , δ and α - δ boundaries, sections 3.6–3.9
	detection of dislocation pairs and dipoles, sections 3.4.10, 3.4.11
	identification of cavities, gas bubbles, sections 3.12, 3.13
	determination of the sense of precipitate strain fields and small dislocation loops, sections 3.5.6, 3.11
	magnetic domain boundary studies, section 3.23
	Moiré pattern studies, section 3.21

always two spots symmetrically disposed about the centre spot which are indexed (hkl) ($\bar{h}\bar{k}\bar{l}$), see section 2.7.2. Thus any diffraction pattern may be indexed by reversing the indices without altering $\mathbf{B}$, compare figure 2.31(a), (b).

The importance of the 180° ambiguity may be seen with the aid of figure 2.31(c), (d). If it is required to determine the orientation of the crystal producing the diffraction pattern relative to another, two different answers will be obtained for the two methods of indexing the pattern, because

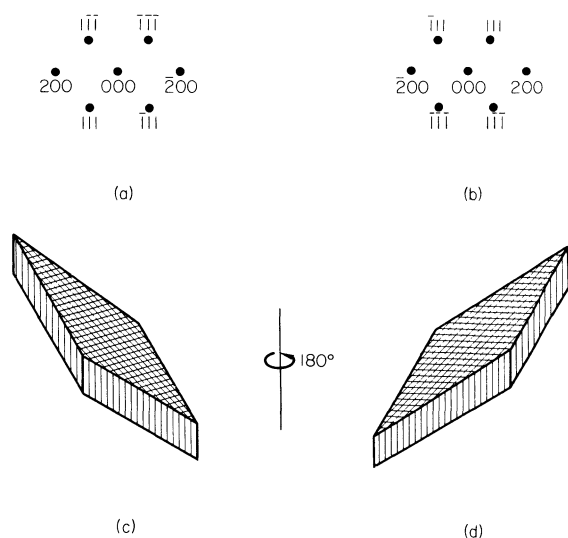


Figure 2.31 The influence of reversing the indices on the spot pattern in (a), (b) on the effective positions of the specimen (c), (d)

they are equivalent to rotating the crystal 180° about $\mathbf{B}$ as shown in figure 2.31(c), (d).

The 180° ambiguity may be removed as follows.

(1) For a single diffraction pattern; by the use of the Ryder and Pitsch analysis (section 2.7.2.2) provided the triple product $\mathbf{g}_1 (\mathbf{g}_2 \wedge \mathbf{g}_3)$ is set positive, or the analysis of von Heimendahl (section 2.7.3.2).

(2) By tilting the specimen (Otte *et al.*, 1964; Vaughan and Silcock, 1967) from a simple $\mathbf{B}$ ($[011]$, $[111]$, etc.) and noting the direction of movement of either the Kikuchi lines (section 2.6.3.3) or ring of bright spots (section 2.7.2.1(b)). This is equivalent to determining in which unit triangle $\mathbf{B}$ lies using a Kikuchi map.

The Ryder and Pitsch (1968) analysis relies on the presence in the diffraction pattern of more than one zero-order Laue zone of spots which fixes $\mathbf{B}$ uniquely, for example in figure 2.22 $\mathbf{B}$ lies between $[1\bar{2}6]$ and $[1\bar{1}3]$. The same result may be produced by tilting the specimen between two readily identifiable $\mathbf{B}$ provided the sense of tilt is known, that is using a Kikuchi map (Otte *et al.*, 1964; Vaughan and Silcock, 1967). The procedure is as follows.

(1) Tilt the specimen to a recognisable simple beam direction, $\mathbf{B}$ $[112]$, $[001]$, $[111]$, etc., for f.c.c. or b.c.c. materials.

(2) Tilt a known amount in a known direction until a simple recognisable diffraction pattern is

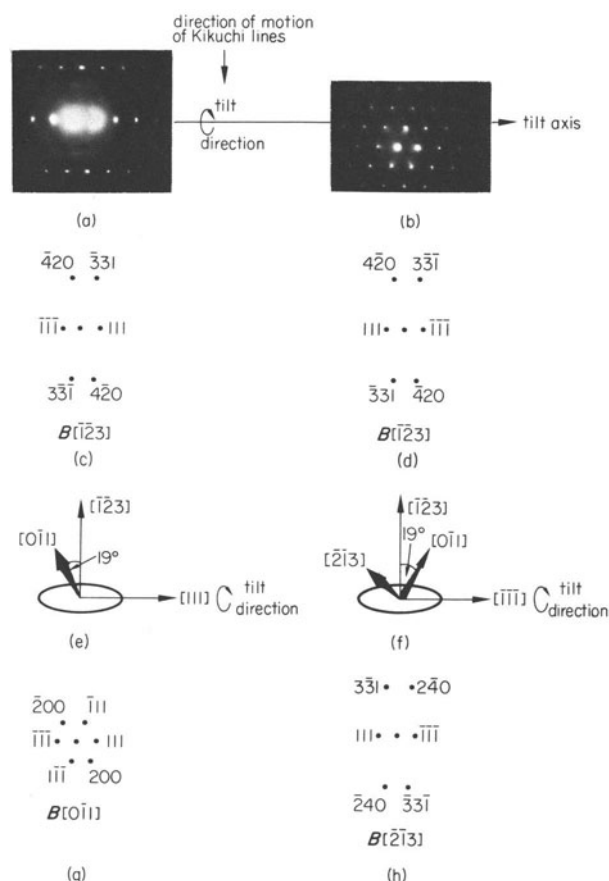


Figure 2.32 Unique indexing of two diffraction patterns (a), (b) obtained by tilting $\sim 19^\circ$ from (a) to (b) about the common (111) reflection. (c), (e), (g) and (d), (f), (h) are possible alternative sequences of indexing and tilting events but only the former is consistent with (a), (b)

obtained, for example with a $[011]$ or an $[112]$ beam direction. This fixes the unit triangle containing B and all subsequent tilting operations.

The sense of tilt within the diffraction pattern may be determined by observing the direction of motion of the Kikuchi lines or ring of bright spots, that is we are tilting in a known sense within a Kikuchi map. The schematic diagram in figure 2.32 demonstrates why the 180° ambiguity has been removed. Figure 2.32(a), (b) shows the diffraction patterns obtained during a tilting experiment performed on Nimonic 75 (f.c.c.). Initially B is $[\bar{1}\bar{2}3]$ and the patterns may be indexed using the rules in section 2.7.2.1 as shown in figure 2.32(c). A specimen tilt of 19° about $[111]$ produces B $[0\bar{1}1]$ and it was observed that the Kikuchi lines moved in the direction shown. Figure 2.32(c)–(h) shows the two possible descriptions of the sequence of events. (c) and (d) are the alternative indexing possibilities for the diffraction pattern in figure 2.32(a). (e) and (f) describe the equivalent situations at the specimen which is represented by the disc. From the known sense of tilt of the specimen (from the direction of movement of the

Kikuchi lines, see section 2.6.3) we must either bring $[0\bar{1}1]$ or $\sim[\bar{2}\bar{1}3]$ to B producing the diffraction patterns in figure 2.32(g), (h) respectively. Only the former is consistent with the experimental result.

It is important to realise that, if a particular analysis requires the removal of the 180° ambiguity, only the Ryder and Pitsch (1968), the von Heimendahl *et al.* (1964), or the known tilt Kikuchi map analysis are applicable. All other indexing systems described in sections 2.7.2, 2.7.3 contain the 180° ambiguity.

Alternative satisfactory combinations of indices exist for figure 2.32(a) because of initial arbitrariness in choice of indices, stage (1) in section 2.7.2.1. However, these alternatives will simply specify a beam direction with different combinations of $\langle 123 \rangle$ and $\langle 011 \rangle$ for figure 2.32(a) and (b), relative to a different unit triangle in the stereogram.

2.8.2 Coincidence Ambiguity

This type of ambiguity occurs infrequently in highly symmetrical cubic crystal structures. Certain relatively high-index orientations in one spot pattern can be indexed in two completely different ways, that is with different zone axes which are not connected by any crystal symmetry relation. Both indexed patterns will be in agreement with all the indexing rules but only one is correct. This situation arises because high-index planes in the cubic system may have the same d spacing, that is $h_1^2 + k_1^2 + l_1^2 = h_2^2 + k_2^2 + l_2^2$ even though (hkl) values are very different, see, for example, (355) and (173) and others in table A6.2, appendix 6. If sufficient of these reflecting planes lie in a given zone, coincidence ambiguity will occur. As an example, consider the case in figure 2.33. In this case $h^2 + k^2 + l^2 = 59$ for (355) and $(\bar{1}73)$ reflections and the patterns may be indexed as $B = [\bar{1}28]$ or $[2\bar{4}7]$.

Coincidence ambiguity may be easily removed by tilting the specimen in a known sense using the Kikuchi map, see section 2.7.3.3. Alternatively

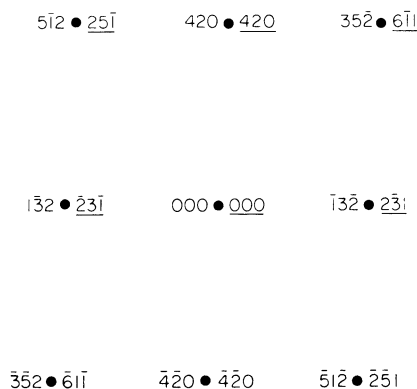


Figure 2.33 A schematic diagram of a spot pattern from a b.c.c. single crystal indexed with $B[\bar{1}28]$, indices underlined, or $B[2\bar{4}7]$, remaining indices

indexing additional spots from other Laue zones which are present in the pattern may provide the solution.

Other examples of B for which identical electron

diffraction patterns are obtained are f.c.c. $[\bar{7} \ 6 \ \bar{5}]$ and $[2 \ 5 \ 9]$; $[1 \ \bar{1} \ 13]$ and $[\bar{10} \ 5 \ 7]$; $[259]$ and $[\bar{5}67]$; b.c.c. $[\bar{1}28]$ and $[24\bar{7}]$; $[5 \ 7 \ 11]$ and $[\bar{1} \ 5 \ 13]$; $[7 \ 9 \ 13]$ and $[3 \ 11 \ 13]$.

PART III. USES OF ELECTRON DIFFRACTION PATTERNS FROM SINGLE CRYSTALS: THE BASIC DIFFRACTION PATTERN

2.9 General Introduction

Correctly indexed diffraction patterns enable a wide range of experiments to be performed and considerable information obtained about the specimen, see sections 2.6.1–2.6.3. In general, Kikuchi patterns provide more accurate information than spot patterns, but this section provides examples of typical uses of both in metallurgical studies. The simple case of ring patterns is not described here, although examples are included in section 4.12.2.

2.10 Specimen Tilting Experiments

2.10.1 Tilting to Specific Orientations

For quantitative studies of crystal defects and orientation relationships between phases the availability of high-angle specimen tilting facilities ($\pm \geq 30^\circ$) and the ability to use them is essential. If large angles of tilt are available ($> \pm 30^\circ$) data can be gathered in a fraction of the time necessary if tilt is limited to $\pm 5^\circ$ about one or two axes. In obtaining quantitative information three types of tilting are required.

(a) Tilt to obtain a succession of two beam conditions, that is only one strongly diffracted beam in addition to the transmitted beam, for example analysis of crystal defects.

(b) Tilt through a large angle ($\sim 30^\circ$) maintaining specific reflection conditions, for example stereo pairs.

(c) Tilt from one known specimen orientation to another, for example determination of orientation relationships between phases.

Consider these three types of tilting experiment separately.*

2.10.1.1 A succession of two-beam conditions

When analysing a crystal defect, for example determination of the Burgers vector of a dislocation line, it is necessary to obtain a series of images of the same field of view with a number of specific reflecting planes operating separately. If the specimen has been tilted so that B is close to a major zone axis Z , the specimen may be further tilted a small amount $\sim 5\text{--}10^\circ$ about a series of different

axes in the diffraction pattern to obtain a series of two-beam conditions, see figure 2.34, using the double tilt type holder (two orthogonal tilt axes). In the EM300 with the specimen height adjusted for axis-centred tilting (section 1.4.4) no lateral movement of the specimen occurs. Consequently, tilting can be performed in the selected area diffraction mode, only occasionally returning to the image mode to check if specimen movement has occurred.

2.10.1.2 Stereo microscopy

Views of the same area of the specimen from different directions (that is different B) can be used in a stereo viewer to obtain a three-dimensional picture of the contents of a thin foil. This technique is very useful in analysing dislocation configurations which are difficult to visualise on one print where only a projection of the dislocation array is visible. Furthermore, the depth of a defect in the foil may be measured very accurately ($\pm 10 \text{ \AA}$), see section 1.6, which is important in determining the sense of the strain field of small defects, see sections 3.5.6, 3.11.

Tilting from one B to another is best carried out in the selected area diffraction mode using the double tilt holder. Since the form of the image is sensitively dependent on the diffracting conditions it is essential to duplicate two-beam conditions before and after tilting, that is same g and s . If identical dynamical two-beam conditions are not operative the detail in the image is lost and the stereographic effect is destroyed. The procedure to produce a stereographic pair is as follows.

(1) The specimen is tilted into two-beam conditions such that the crystal defect under investigation is clearly visible and the diffraction pattern and bright field image photographed, using small values of s for maximum contrast.

(2) The double tilt controls are operated viewing the SADP on the screen to retain the same operative reflection yet tilt through a large angle. In thicker parts of the crystal good imaging conditions coincide with a bright Kikuchi line AA' running very close to the diffraction spot and a dark Kikuchi

* Refer to sections 2.7.2, 2.7.3 for the techniques of indexing the diffraction patterns used in this section.

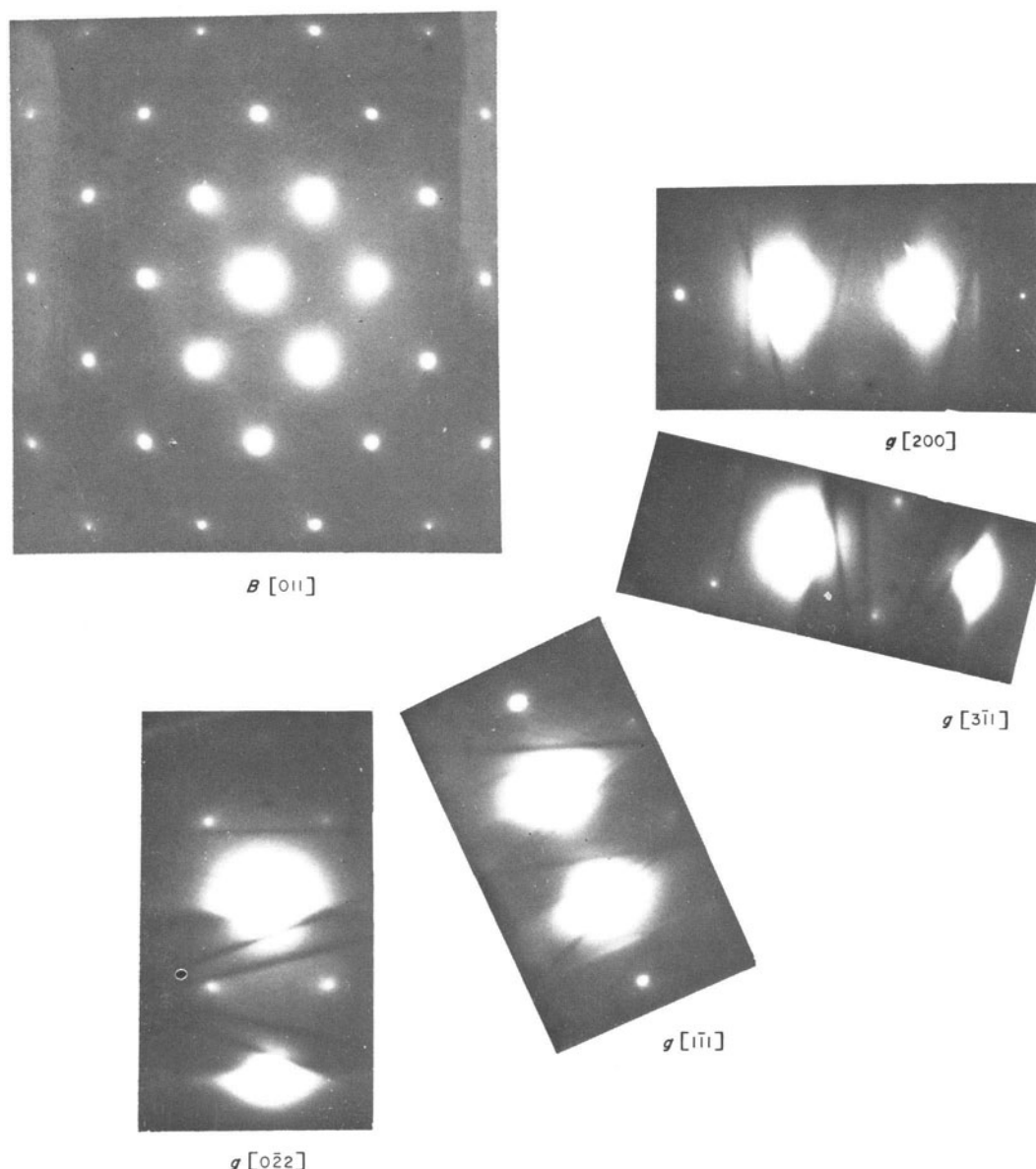


Figure 2.34 A series of two-beam diffraction patterns suitable for forming strong images required in section 3 near $B = [011]$ in an f.c.c. crystal structure

line BB' similarly close to the transmitted spot T in figure 2.35. The specimen is tilted so that this Kikuchi line pair does not move significantly relative to the associated spots.

(3) After tilting through a large angle, estimated from the tilt drive movement, the same two beam conditions with the same values of s are re-established to retain image detail. See figure 2.35. Small adjustments of the tilts are made to regain the original positions of the Kikuchi lines associated with the spots corresponding to the operative reflection. Note that the amount of tilt selected depends on the thickness of the foil and the accuracy of any depth measurement required, see section 1.6.

(4) The micrograph and diffraction pattern are then photographed and the amount of tilt accurately measured from the two diffraction patterns,

determining B using Kikuchi lines, see section 2.7.3. It should be apparent on consulting section 2.4 that this tilting procedure is effectively equivalent to rotating the specimen about the reflecting plane normal, g .

Kikuchi maps, section 2.7.3.3 and appendix 5, are particularly useful for these experiments since both the sense and approximate value of tilt can be determined directly from the map.

2.10.1.3 From one specific orientation to another

If we have a foil with beam direction close to a major zone axis in the crystal, i.e. $[110]$, it is often of interest to tilt to another major orientation, for example $[111]$. Such a requirement is most likely for Burgers' vector determinations, sections 3.4.3, 3.4.5, when specific reflecting planes are required;

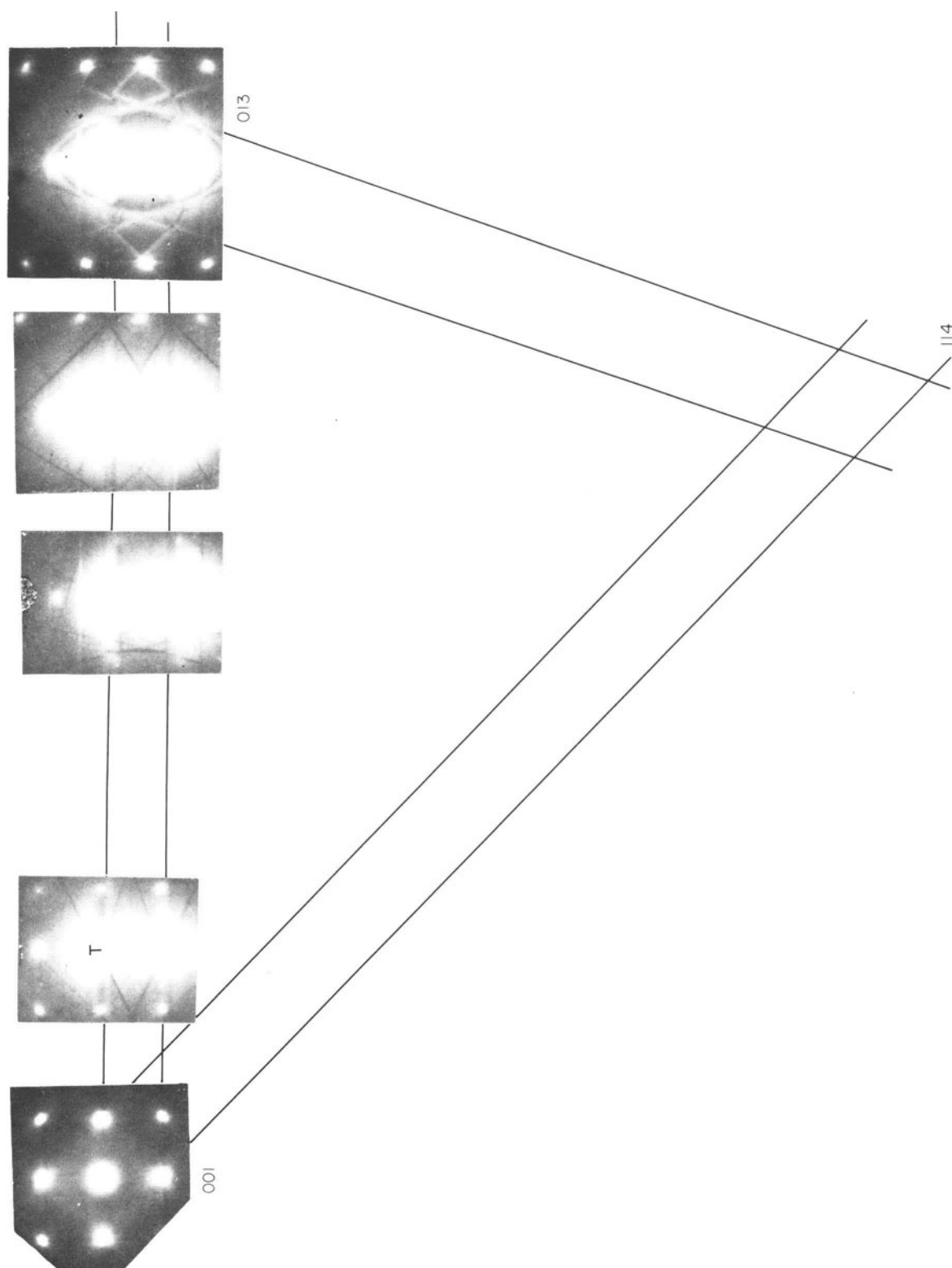


Figure 2.35 A sequence of diffraction patterns obtained on tilting from $B[001]$ to $[013]$

for determination of the sense of dislocation loops section 3.5; or for the determination of orientation relationships. The easiest method is to decide which crystallographic plane is common to both zones and then to tilt about the normal to this plane because it is perpendicular to both zone axes. In practice a strong reflection is obtained in the first diffraction pattern from the common plane, that is Kikuchi line passing through the bright spot. Then, while viewing the diffraction pattern in the microscope, the specimen should be tilted keeping the same reflection strongly operating until the diffraction pattern corresponding to the required zone is observed. A simple spot pattern may be used if the specimen is thin, simply keeping the diffracted spot bright. However, it is often easier to rely on a Kikuchi pattern, determining B by comparison with the Kikuchi map, section 2.7.3.3, appendix 5, and then using the Kikuchi map like a road map to follow B during tilting. Thus the specimen may be tilted from one B to another by tilting in a known sense so that B progresses down a Kikuchi line pair. This technique is very similar to that described for a stereo pair because the specimen is being tilted about g , see figure 2.35.

A typical example of this type of operation is shown in figure 2.36 for spot patterns. This figure shows a unit triangle in an f.c.c. material with diffraction patterns obtained with B parallel to low-index crystal directions. Inspection of the patterns shows that it is possible to tilt from an $[001]$ to an $[011]$ B about a $[200]$ g vector. Table 2.6 shows suitable axes for tilting the specimen from one B to another in cubic crystal structures.

Table 2.6 Tilt axes for tilting from one B to another in f.c.c. and b.c.c. crystal structures

Beam direction B	Common reflection (tilt axis)	
	f.c.c.	b.c.c.
$[001]$ to $[011]$, $[013]$, $[012]$	200	200
$[001]$ to $[111]$, $[113]$, $[112]$	220	110
$[011]$ to $[112]$, $[123]$	$1\bar{1}1$	222
$[011]$ to $[113]$	422	$2\bar{1}1$
$[111]$ to $[123]$, $[012]$	242	$12\bar{1}$
$[111]$ to $[013]$	462	$2\bar{3}1$
$[001]$ to $[133]$	620	$3\bar{1}0$
$[011]$ to $[123]$, $[122]$	$1\bar{1}1$	222
$[011]$ to $[122]$	022	$0\bar{1}1$

When tilting from one zone axis to another the direction of rotation should be noted so that once the initial diffraction pattern is indexed, for example B $[001]$ rather than $[010]$, the sense of tilt in the crystal can be determined within the unit triangle. Methods of determining the sense of tilt are described in sections 2.7.3.3 and 2.8.1.

2.10.2 Control of the Sign and Value of s

As shown in section 2.6.3 a dark Kikuchi line is associated with the transmitted spot while a bright line occurs near a strongly diffracted spot. It is the position of the bright Kikuchi line, either inside or outside the associated diffraction spot, that enables the sign and value of s to be determined.

For Kikuchi lines, the Bragg law is always exactly satisfied and there is no relaxation of the Bragg condition arising from specimen shape. Consequently for small specimen tilts one can discuss the position on the Ewald sphere of the Kikuchi cones shown in figure 2.19(c) by assuming that the bright cone always intersects the centre of the reciprocal lattice point. This position will be the same in the diffraction pattern which is simply a magnified view of the surface of the Ewald sphere. Consider the following three conditions.

(1) $s = 0$, figure 2.37(a). The Ewald sphere cuts the centre of the reciprocal lattice streak and the bright Kikuchi line runs through the spot.

(2) s negative, figure 2.37(b), the Kikuchi line lies inside the spot because the centre of the reciprocal lattice streak lies below the Ewald sphere.

(3) s positive, figure 2.37(c), the Kikuchi line lies outside the spot because the centre of the reciprocal lattice streak lies above the Ewald sphere. This is the most common imaging condition because the BF image exhibits maximum contrast and maximum penetration is obtained, see figure 3.3.

Since the spacing of the bright and dark Kikuchi lines is always the same for the same camera constant the dark line follows the bright line as shown in figure 2.37.

The value of s may be obtained by measuring the distance of the bright Kikuchi line from its associated diffraction spot. Referring to figure 2.30

$$s = \frac{y_1}{dL} \quad \text{or} \quad \frac{y_1 \lambda}{x_1 d^2} \quad (2.30)$$

where L is the camera length, λ is the wavelength of the electrons and d is the interplanar spacing of the reflecting planes.

2.10.3 Determination of the Sign of B

As pointed out in section 2.7.2, single-crystal diffraction patterns are symmetrical with six-, four-, three- and two-fold symmetry. Under such conditions it is impossible to distinguish between $\pm B$ because each pattern is identical when turned over. However, in some cases, for example $B \langle 123 \rangle$ (f.c.c., b.c.c.) the pattern is asymmetric. Figure 2.20(a) shows a B $[123]$ pattern which, if it were turned over, that is B $[1\bar{2}3]$, would be asymmetric in

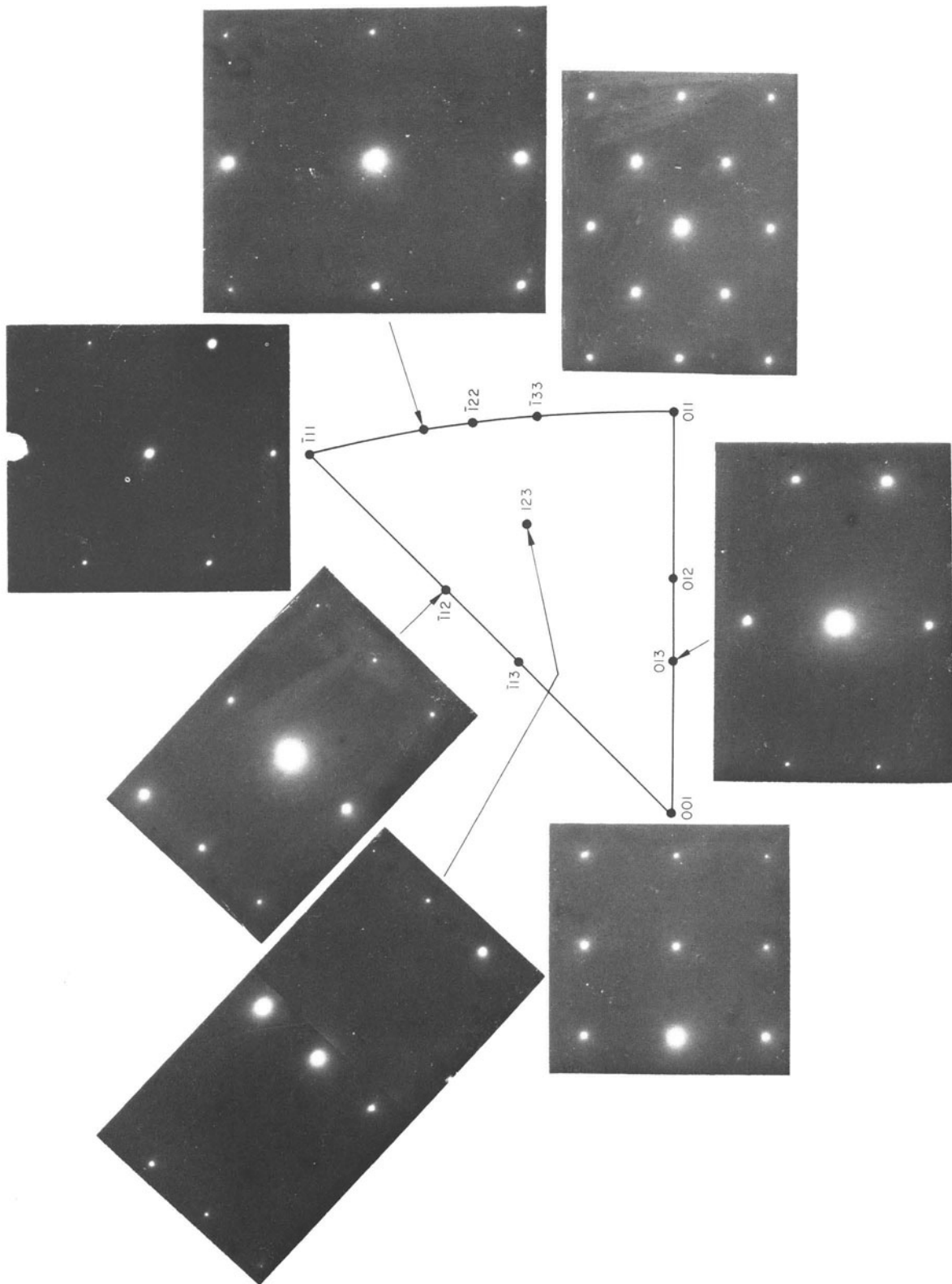


Figure 2.36 A series of diffraction patterns showing exact **B** within the unit stereographic triangle (f.c.c.). Note the common reflections

the opposite sense. Thus $\pm \mathbf{B}$ may be determined but the 180° ambiguity remains, see section 2.8.1.

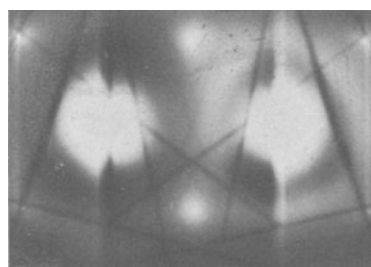
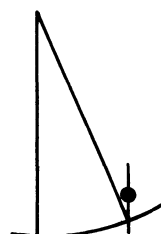
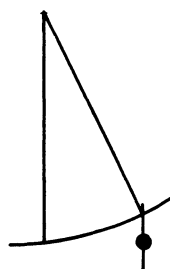
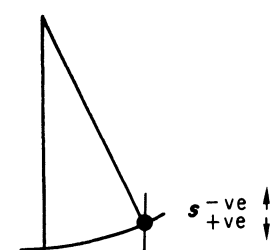
Duggan and Segall (1971) have pointed out that Kikuchi line patterns may also be used to determine the sign of $\mathbf{B}$ because they are often asymmetric, even when the spot patterns are symmetrical, for example $[\bar{1}25]$ and $[1\bar{2}5]$, see figure 2.38(a), (b). It is clear that the converging Kikuchi line pair XX' and YY' converge in a different sense in the two photographs. This difference in symmetry between Kikuchi and spot patterns arises because, unlike the spot pattern which usually consists only of spots from the zero-order Laue zone for a low-index $\mathbf{B}$ (see section 2.7.2), the Kikuchi pattern contains dark lines from reflections in the first order Laue zone. These arise because the defect and excess cones are projected into the surface of the Ewald

sphere. The Kikuchi line pattern thus exhibits the proper symmetry of the crystal not the pseudo-symmetry of the spot pattern that arises from the 180° ambiguity described in section 2.8.1.

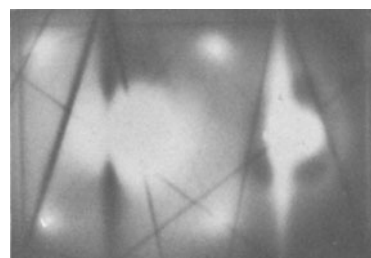
These lines also enable the correct symmetry of the crystal to be ascertained. An example of the Kikuchi diffraction pattern $\mathbf{B} = [111]$ in aluminium is shown in figure 2.25. Compare with the spot pattern in appendix 4. The spot pattern shows pseudo-six-fold symmetry arising from the 180° ambiguity but the Kikuchi pattern has the correct three-fold symmetry of the crystal structure.

2.11 Orientation Relationship Determination

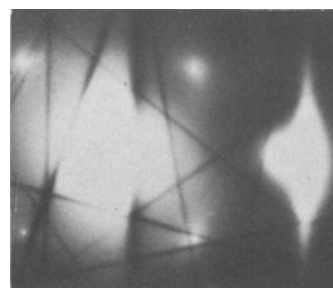
Orientation relationships between phases are generally expressed in one of the following two ways.



(a)

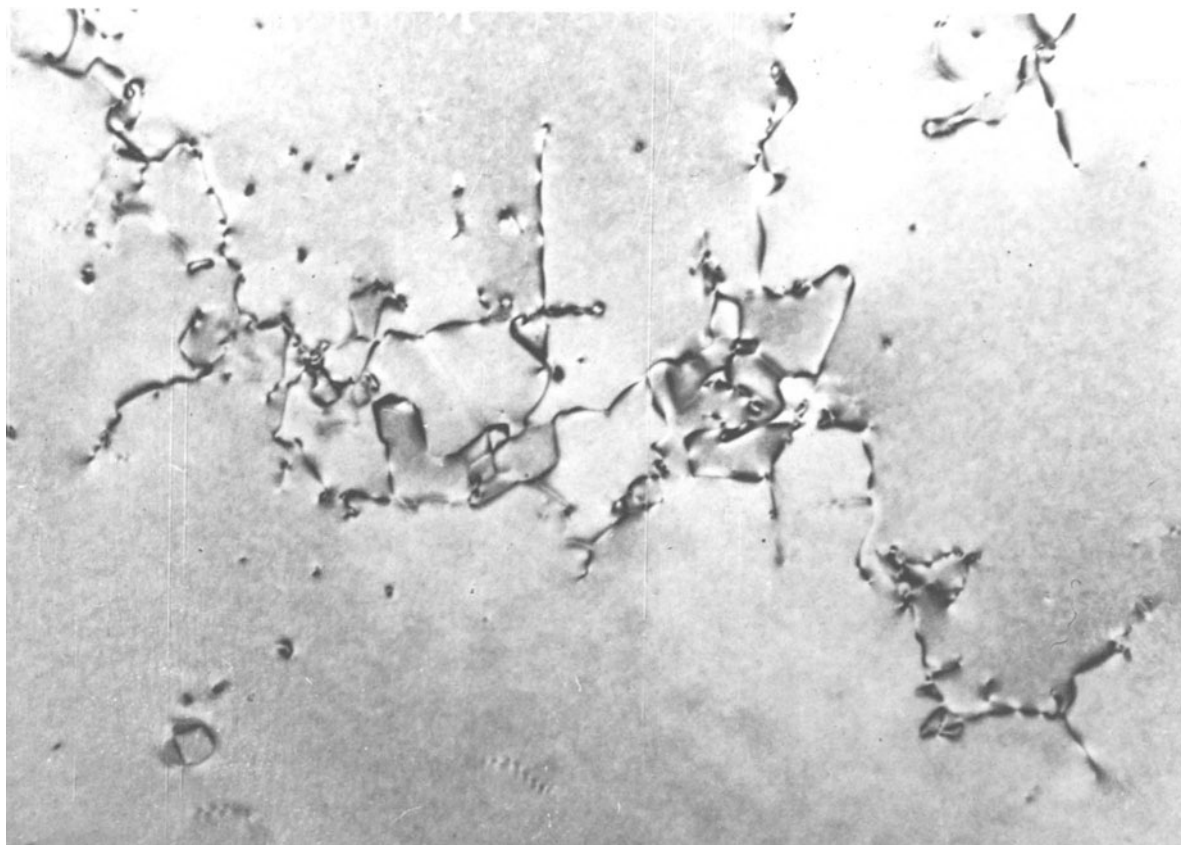


(b)

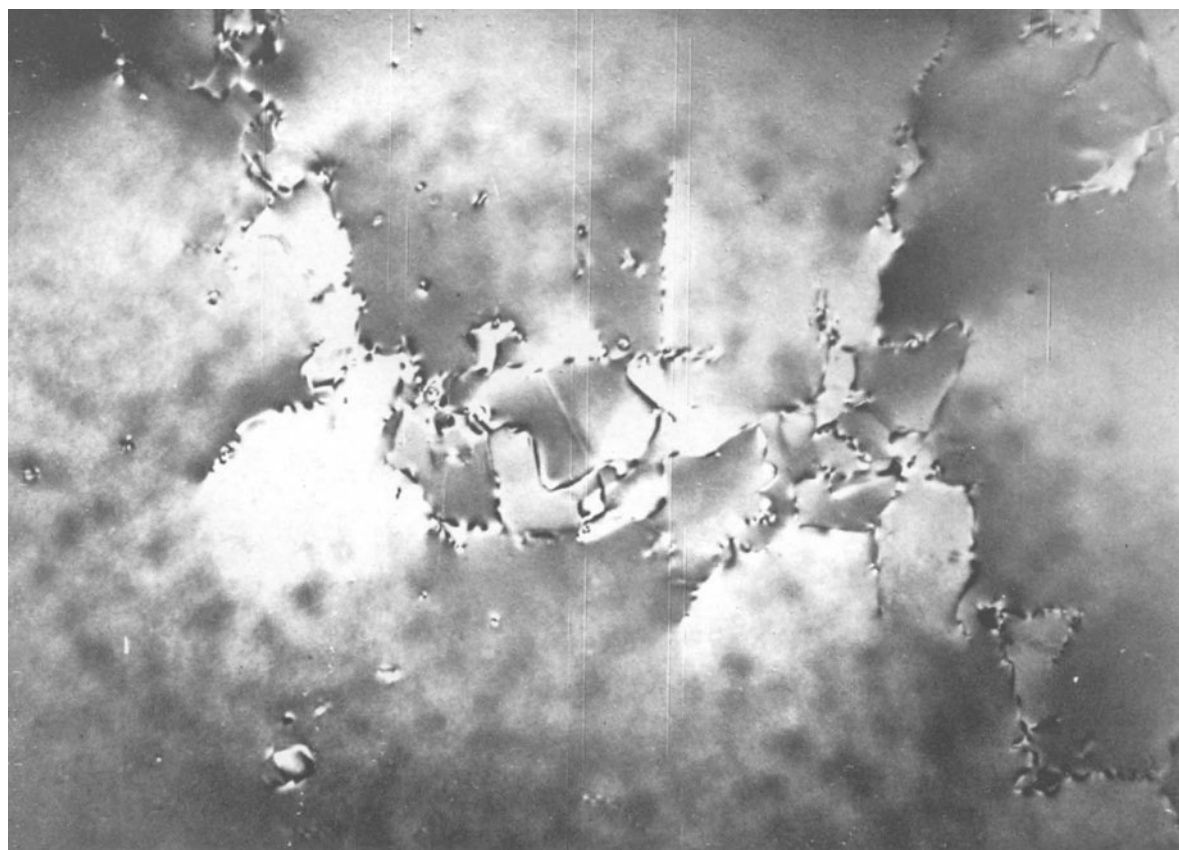


(c)

Figure 2.37 Diffraction patterns showing (a) $s = 0$, (b) s negative, (c) s positive, with the equivalent positions of the reciprocal lattice point and the Ewald sphere. The bright Kikuchi line (a) runs through the diffracted spot, (b) is inside and (c) is outside it. (d)–(f) show images of dislocations taken under the diffracting conditions corresponding respectively to (a)–(c)

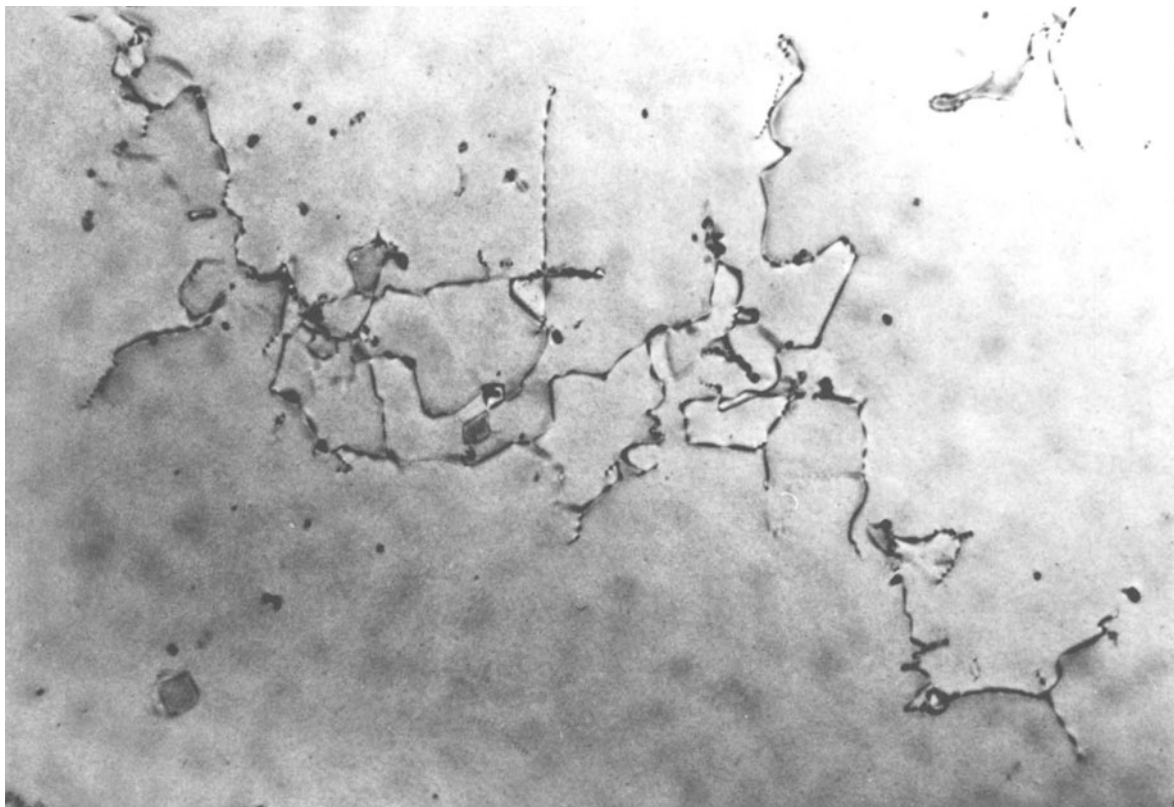


(d)



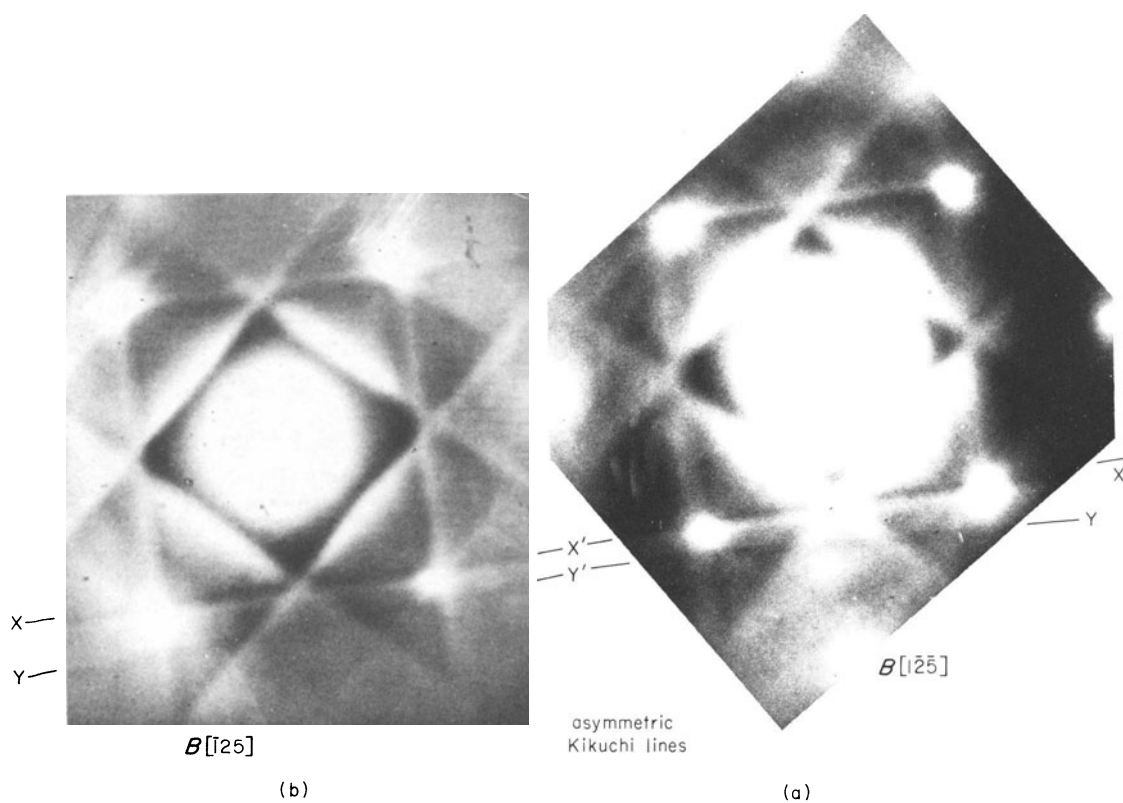
(e)

Figure 2.37 (continued)



(f)

Figure 2.37 (continued)

Figure 2.38 Kikuchi line patterns for (a) $B[1\bar{2}5]$, (b) $B[\bar{1}25]$ in f.c.c. crystal structure. Note the opposite sense of the converging black Kikuchi lines XX' , YY'

(a) A pair of parallel directions in a pair of parallel planes. Such a method of expression is normally used for coherent or semicoherent precipitate–matrix orientation relationships which are usually simple and almost exact. The orientation relationships between phases in a phase transformation are also expressed in this way, for example the Kurdjumov–Sachs (1930) relationship for the formation of martensite in low-carbon steels $(111)_\gamma // (110)_M$ and $[\bar{1}\bar{1}0]_\gamma // [\bar{1}\bar{1}1]_M$ where M is martensite and γ is austenite.

(b) As a matrix describing the orientation relationship between two crystals. Physically this corresponds to an axis/angle pair, that is a rotation ϕ about a common (invariant) direction which makes the two sets of axes of the two lattices parallel.

The technique used to determine the orientation relationships between two phases depends upon the type of orientation relationship, the accuracy of information required, and the limitations imposed by the specimen. Two approaches are commonly used.

(1) Spot patterns from both phases may be used either because strain effects prevent the formation of Kikuchi lines (for example carbide precipitates in martensite) or because the crystallographic relationship is particularly simple (coherent precipitates). In the latter case the habit plane and shape of the precipitate often provide a check of the orientation relationships.

(2) Kikuchi lines may be employed either to improve the accuracy of the orientation relationship determination or because the spot pattern is clearly an inaccurate representation of the beam direction. Since the quality of Kikuchi patterns deteriorates if the lattice is strained this technique has limited application. Nevertheless, it is suitable for grain–grain or subgrain–subgrain orientation relationships in annealed materials and for phase orientation relationships with large second-phase particles such as directionally solidified eutectics or large grain boundary particles.

The procedures for determining orientation relationships either in terms of parallel directions and planes or as an axis/angle pair will now be described separately.

2.11.1 Parallel Directions and Planes

Two types of situation may be distinguished for convenience. The first arises when either an individual large particle is being studied or all the precipitate particles within the SAD aperture have

exactly the same orientation relationship. The second occurs either when the precipitates contributing to the SADP have different variations of the same orientation relationship or when different closely intermingled precipitates are present. These two cases will be treated separately.

2.11.1.1 All precipitates have the same orientation relationship

These are usually simple orientation relationships and may often be obtained from a single diffraction pattern like that in figure 2.39 containing both precipitate (weak) and matrix (strong) reflections. The principles of using such a diffraction pattern to obtain an orientation relationship are the following.

(a) Index the patterns using the rules outlined in section 2.7.2 to enable **B** to be specified in both phases. Thus these directions are parallel in both phases. *N.B.* Because of the errors in determining **B** discussed in section 2.7.5 exact **B** must be obtained at least in one phase (usually the matrix) by tilting the specimen until all matrix spots are equally intense.

(b) Rows of parallel matrix and precipitate spots in the pattern indicate parallel planes (within $\pm 5^\circ$) because as pointed out in section 2.7.2 all reflecting planes are almost parallel to the electron beam (small Bragg angle $< 1^\circ$).

The full procedure is as follows.

(1) Determine the identity of the precipitate if unknown, using either extraction replicas, or suitable x-ray techniques.

(2) Obtain a simple diffraction pattern including both precipitate and matrix spots but with all matrix spots uniformly intense, that is **B** as close as possible to the zone axis.

(3) Photograph the diffraction pattern.

(4)* Tilt the specimen in a known direction (see section 2.10.1) in the matrix to produce different simple matrix **B** in the same area and obtain photographs.

(5) Fully index each diffraction pattern, both matrix and precipitate spots.

(6) Determine the zone axis **B** in both precipitate and matrix for each pattern.

(7) Plot the superimposed stereograms corresponding to the matrix and precipitate **B** for each diffraction pattern and construct the poles of the important planes.

(8) Quote the orientation relationship in terms of parallel directions **B** and planes from parallel

* This step may not be possible if the specimen tilt is limited. Under these circumstances several different areas, each in a different grain with a different **B**, may be used. However, this approach is not completely satisfactory because it does not allow step (9) to be performed. It is only sensible to use this latter approach to confirm a simple orientation relationship. New or complex relationships should be determined using (4) above for complete self-consistency.

rows of indexed spots in both phases unless lower-index planes and directions are parallel. If they are, quote them.

(9) Check for crystallographic equivalence of the diffraction patterns obtained with the two different matrix **B**. This step is not essential (see footnote on previous page) but will be found to be

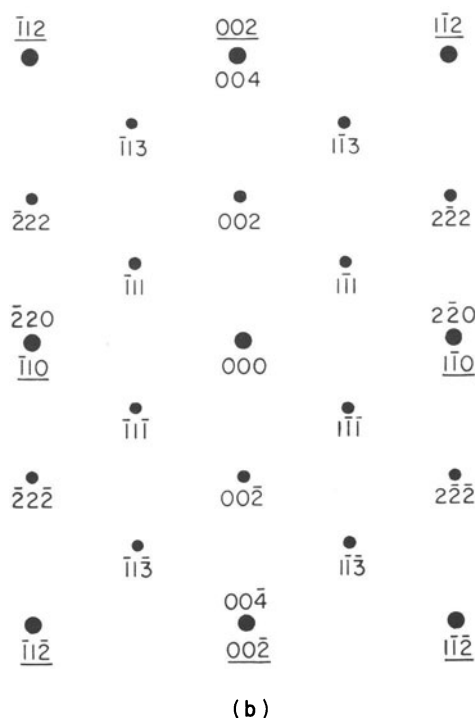
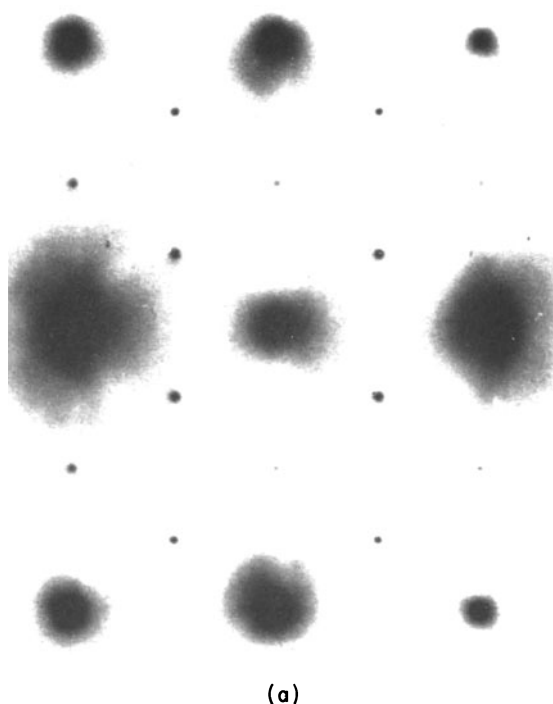


Figure 2.39 A spot pattern (a) containing matrix spots (indexed 002, etc.) and Fe₂Si/Ti precipitate spots (indexed 004, etc.). The equivalent superimposed stereograms are shown in (b) [Courtesy of D. H. Jack]

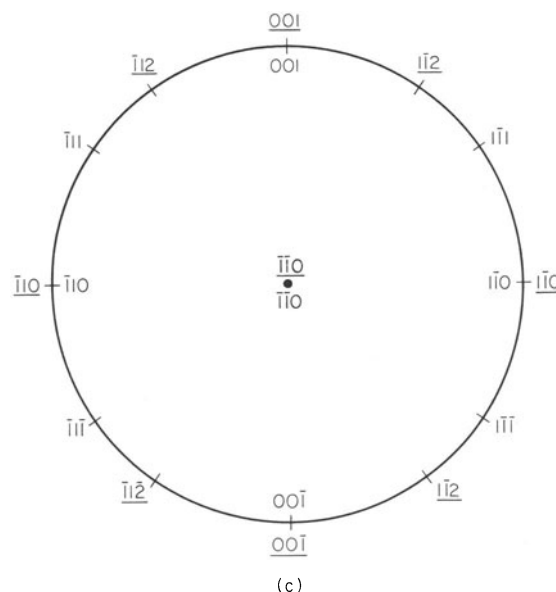


Figure 2.39 (continued)

a useful check of self-consistency, particularly when complex crystal structures are involved, because careful tilting using the approach outlined in section 2.10.1 will remove the 180° ambiguity described in section 2.8.1.

The electron diffraction pattern in figure 2.39(a) was taken from the region shown in figure 2.51(a) of an Fe–Si–Ti alloy aged for 16 hours at 600 °C and may be used as an example of an orientation relationship determination. It may be indexed as shown in figure 2.39(b) using the lattice parameters of b.c.c. α -iron (2.866 Å) and the ordered f.c.c. $\text{Li}_2\text{Fe}_2\text{TiSi}$ intermetallic compound (5.714 Å). The zone axes of the patterns are $[\bar{1}\bar{1}0]_{\alpha\text{-iron}}$ and $[\bar{1}\bar{1}0]_{\text{Fe}_2\text{TiSi}}$ and the (002) reflecting planes in both phases are parallel. The superimposed stereograms are shown in figure 2.39(c) with the important poles located. Using this stereogram the orientation relationship may be expressed as

$$\begin{aligned} [\bar{1}\bar{1}0]_{\text{matrix}} // [\bar{1}\bar{1}0]_{\text{precipitate}} \\ (002)_{\text{matrix}} // (002)_{\text{precipitate}} \end{aligned}$$

This orientation relationship was checked for consistency using other diffraction patterns from this area with different **B** in the matrix $[100]$ and $[111]$, see point (4) above.

Note that such an orientation relationship is general, not specific and may be quoted with different equivalent combinations of $\langle uvw \rangle$ or $\{hkl\}$ depending upon how the pattern is indexed because of initial arbitrariness and the 180° ambiguity described in section 2.8.1. In more complex cases (Vaughan and Silcock, 1967) it may be necessary to remove the 180° ambiguity by tilting the specimen into specific orientations as described in section 2.8.1. The accuracy of orientation

relationships determined by this method is $\sim 1^\circ$ provided B almost exactly parallel to zone axes are used (equalise of diffracted spot intensity). If this is not done, errors of $\pm 15^\circ$ may easily occur particularly for low-index zones (Laird *et al.*, 1966).

Double diffraction effects, described in section 2.16.1, are common when B is almost exactly parallel to low-index zone axes, and introduce a complicating factor. An example is shown in figure 2.58(a) which has been taken from an Ni–Ti–C alloy containing fine precipitates of TiC. The lattice parameter of the matrix is 3.5326 Å while that of TiC, which has the NaCl structure, is 4.329 Å. The matrix diffraction pattern can be indexed $B = [100]$, see figure 2.57(b), and B in the basic TiC pattern is also $[100]$, see figure 2.57(c), showing the $\{111\}$ fundamental spots. The additional clusters of spots in figure 2.58(b) arises because of double diffraction, as described in section 2.16.1, reproducing the symmetry of the basic $[100]$ matrix pattern around each matrix spot.

When double diffraction effects are thought to be present the analysis procedure is outlined in section 2.16.1.

In some materials, specific orientation relationships may be expected, see table 2.7. Superimposed stereograms for these orientation relationships are shown in appendix 9, and may be used to construct a series of standard patterns for low-index B for comparison with experimental patterns.

It is important to note that a single agreement from one electron diffraction pattern only demonstrates consistency with a particular orientation relationship and does not define it completely. In practice, two different B parallel to simple zone axes should be obtained by tilting the specimen a known amount in a known direction as outlined in section 2.8.1 to remove the 180° ambiguity. If this is not possible, a large number of different

particles must be studied with different foil normals to make certain that all particles are consistent with a particular orientation relationship. The exact number of determinations of this latter type required for consistency increases with the number of phases present and the complexity of the orientation relationship. Typical numbers would be 30–300 depending on the complexity of the system under study.

2.11.1.2 Several variants of one orientation relationship or several phases present

Complex diffraction patterns frequently occur in these cases because coexisting different precipitates increase the number of spots present. Furthermore, even if there is only one precipitate present, there may be several variants of the orientation relationship within the selected area aperture which may also increase the number of spots present. Thus if there is a precipitate with a $\{111\}$ habit plane and an orientation relationship

$$(111)_{\text{matrix}} // (hkl)_{\text{precipitate}} \\ [1\bar{1}0]_{\text{matrix}} // [uvw]_{\text{precipitate}}$$

there will be four variants of this corresponding to precipitates on different $\{111\}$ and a further three for each $\langle 110 \rangle$ in (111) corresponding to precipitates with $[uvw] //$ the three $\langle 110 \rangle$ producing a total of twelve variants. In some cases where the precipitate has a simple crystal structure, the spots in the diffraction patterns from each variant may superimpose. However, for more complex precipitates this may not be true. An example of this latter case are described in section 4.9.2.

Each case must be treated on its merits but where possible it is recommended that the problem should be simplified by the following.

Table 2.7 Common orientation relationships between phases

Type of relationship	Phases	Parallel planes
Pitsch (1962)	cementite/ferrite	(100) cementite // (3 $\bar{1}$ 1) ferrite (010) cementite // (131) ferrite (001) cementite // (2 $\bar{1}$ 5) ferrite
Bagaryatski (1950)	cementite/ferrite	(100) cementite // (0 $\bar{1}$ 1) ferrite (010) cementite // (1 $\bar{1}$ 1) ferrite (001) cementite // (211) ferrite
Bain (1924)	b.c.c./f.c.c. materials	(100) b.c.c. // (100) f.c.c. (01 $\bar{1}$) b.c.c. // (010) f.c.c. (011) b.c.c. // (001) f.c.c.
Nishiyama (1934), Wasserman (1933)	b.c.c./f.c.c. materials	(001) b.c.c. // (0 $\bar{1}$ 1) f.c.c. ($\bar{1}$ 10) b.c.c. // ($\bar{1}$ 11) f.c.c. (110) b.c.c. // (211) f.c.c.
Kurdjumov and Sachs (1930)	b.c.c./f.c.c. materials	(011) b.c.c. // (111) f.c.c. (11 $\bar{1}$) b.c.c. // (10 $\bar{1}$) f.c.c. (2 $\bar{1}$ 1) b.c.c. // ($\bar{1}$ 2 $\bar{1}$) f.c.c.

(1) Age for longer periods, so that individual particles may be studied to determine the orientation relationship, provided a change in precipitate type does not occur. For a worked example see section 4.9.1.

(2) Use extensively the centred dark field (CDF) imaging technique described in section 1.5.2 with precipitate reflections. This determines whether two types of precipitate with different shapes occur and which spots arise from each, and which spots arise from the same variant of the orientation relationship. For a worked example see section 4.9.2.

(3) Predict the positions of spots in the diffraction pattern as far as possible for low-index matrix **B** to compare with experimental SADP. In many cases the known crystal structure of likely precipitating phases may be used to predict likely orientation relationships and habit planes by testing for planes in which lattice spacings match.

Prediction of SADP is a particularly important part of analysis of complex situations. Here a stereographic approach due to Sass (1969a) is outlined for the case of variants of the ω phase precipitate in β matrix titanium-base alloys.

The ω phase has the hexagonal structure (see section 4.13.2) with the orientation relationship

$$(0001)_{\omega} // \{111\}_{\beta}, \quad \langle 1\bar{2}10 \rangle_{\omega} // \langle 1\bar{1}0 \rangle_{\beta}$$

Since there are four matrix $\{111\}$ planes there will be four fundamentally different orientations of the ω phase. Within each case of $(0001)_{\omega} // (111)_{\beta}$ each orientation produces its own diffraction spots. Then there will be three possible combinations of $\langle 1\bar{2}10 \rangle_{\omega} // \langle 1\bar{1}0 \rangle_{\beta}$, but these diffraction spots superimpose on the others from each $(0001)_{\omega} // \{111\}_{\beta}$ variant.

As pointed out in section 2.7.2.1 the reflecting planes of the matrix are almost parallel to **B** and therefore **B** is the zone axis of the reflecting planes. Consequently the reflecting plane normals (**g** vectors) all lie on the great circle pole **B**. The question is simply which variants of ω produce poles on this great circle and where do they lie relative to the matrix reflections?

First an $[0001]$ stereographic projection is constructed for the plane normals of the ω phase, based on its lattice parameters. This projection is then rotated as described in appendix 1 to correspond to one variant of the above orientation relationship, that is $(0001)_{\omega} // (111)_{\beta}$; $[\bar{1}2\bar{1}0]_{\omega} // (\bar{1}10)_{\beta}$ in a $[001]_{\beta}$ cube projection. These two projections are shown in figure 2.40(a), (b). Let us now synthesise the diffraction pattern to be expected for **B** $[110]_{\beta}$. The $[110]_{\beta}$ great circle is drawn on figure 2.40(b) and the plane normals lying on this

great circle are immediately obtained, leading to the schematic matrix pattern shown in figure 2.40(c). The ω plane normals which lie on this great circle for this particular variant of ω are obtained by superimposing figure 2.40(a) on figure 2.40(b) with $[0001]$ lying on $[111]$ and $[\bar{1}2\bar{1}0]$ lying on $[\bar{1}10]$. This produces the great circle on the ω projection corresponding to the **B** $[110]_{\omega}$ and is drawn in figure 2.40(a). Clearly only $\pm(2021)$ and $\pm(\bar{1}2\bar{1}0)$ reflections occur lying parallel to (001) and $(\bar{1}10)$ reflecting planes. When the interplanar spacing of these ω reflections is taken into account, the position of the $\pm(2021)$ and $\pm(\bar{1}2\bar{1}0)$ spots may be drawn and it is found they superimpose on (002) and $(\bar{1}10)$ matrix reflections. Consequently the ω spots arising from the $(001)_{\omega} // (111)_{\beta}$, $[\bar{1}2\bar{1}0]_{\omega} // [\bar{1}10]_{\beta}$ variant are those shown in figure 2.40(d). The spots from the remaining variants may be

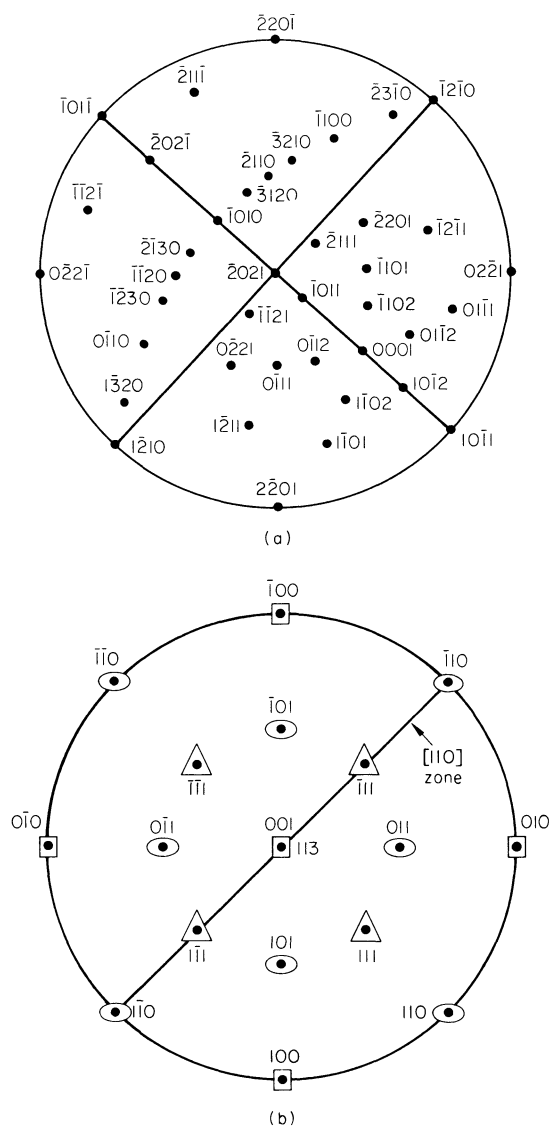


Figure 2.40 Stereographic projections for (a) $[hkl]$ plane normals for the ω phase and (b) the b.c.c. matrix, (c) shows the matrix spots for **B** $[110]_{\beta}$ in the matrix together with (d)–(g) the spots from different variants of the ω precipitate

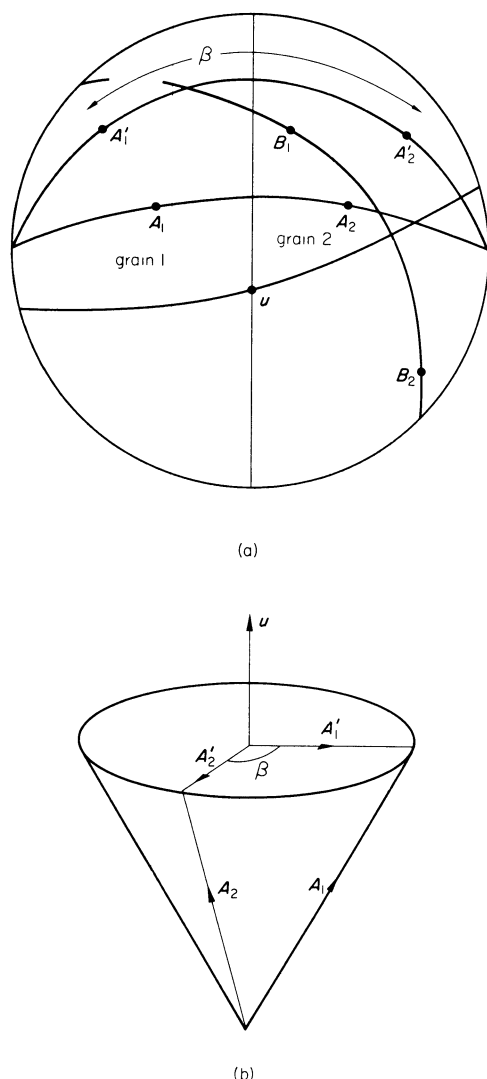


Figure 2.41 Graphical determination of the axis/angle pair for a grain boundary for two pairs of parallel beam directions $A_1 // A_2$ and $B_1 // B_2$. The directions are plotted on the standard stereogram. (a) The conventional definition of the angle of misorientation is shown in (b)

parallel directions B_1 and B_2 may also be constructed, knowing the sense of tilt from A_1 to B_1 , and the bisecting great circle drawn. The axis u lies at the intersection of the great circles. The angle of rotation β is conventionally taken as the angle of rotation of a direction in the invariant plane (that is that plane perpendicular to u). This is determined graphically by projecting A_1 and A_2 onto this plane (see figure 2.41(b)) and determining the angle between the two. The projection is performed using the techniques obtained in appendix 1 and is shown as A'_1 and A'_2 . The angle β may be determined by measurement along this great circle. The accuracy of this method is limited to $\pm 2^\circ$ at best by the stereographic projection.

An example of the application of the Goux analysis to the case of a grain boundary in an austenitic stainless steel is shown in figure 2.42.

Two pairs of diffraction patterns (a), (b) are from grains 1 and 2, then (c) and (d) from grains 1 and 2 after an angular tilt of 26.5° . The diffraction patterns in figure 2.42(a), (b) could be unambiguously indexed with B $[110]$ and $[\bar{1}\bar{1}0]$ by noting the direction of tilting from B $[321]$ and $[3\bar{2}1]$ patterns obtained in each grain. By noting the direction of tilt from the movement of Kikuchi lines (see section 2.8.1) the second patterns could be indexed as B close to $[332]$ and $[2\bar{5}1]$ in grains 1 and 2 respectively which corresponded closely to the measured angle of tilt. These zone axes and the directions of tilt are plotted in figure 2.42(e). The application of the Goux analysis leads to an axis/angle pair close to $[\bar{1}01]/110^\circ$ as shown.

This pair may be compared with the most accurate computed values for B shown in table 2.8 obtained using the analysis of Pumphrey and Bowkett (1970a). These give rise to an axis/angle pair $[-263.07, -1.0, +263.85]/109.34^\circ$ which is within 0.2° of a twin relationship $[\bar{1}01]/109.47^\circ$ (Brandon *et al.*, 1964).

By computation

axis/angle pair are

$$[-263.07 - 1.0 + 263.85]/109.34^\circ$$

Compare

axis/angle pair for twin $[\bar{1}01]/109.47^\circ$

This example is a useful indication of the errors that can arise if exact B are not used. Note that initial arbitrariness described in section 2.7.2.1 could have enabled different indices to be chosen for B in figure 2.42(a), (b) by using different pairs of unit triangles in the stereogram which were equivalent in terms of the sense of tilt from $B \langle 123 \rangle$. The present cases were chosen so that A_1, A_2 could be close to 90° which maximises the accuracy of the determination.

2.12 Twinning

Diffraction patterns from twins in cubic crystal structures have been discussed in detail by a number of workers, see below, but hexagonal crystal structures have received relatively little attention.

2.12.1 Origin of the Extra Spots

A twin is simply one part of a crystal that is oriented with respect to another according to a symmetry rule. Extra spots arise in the SADP because the twin in the thin foil is oriented differently from the matrix relative to the incoming electron beam. This situation is shown schematically in

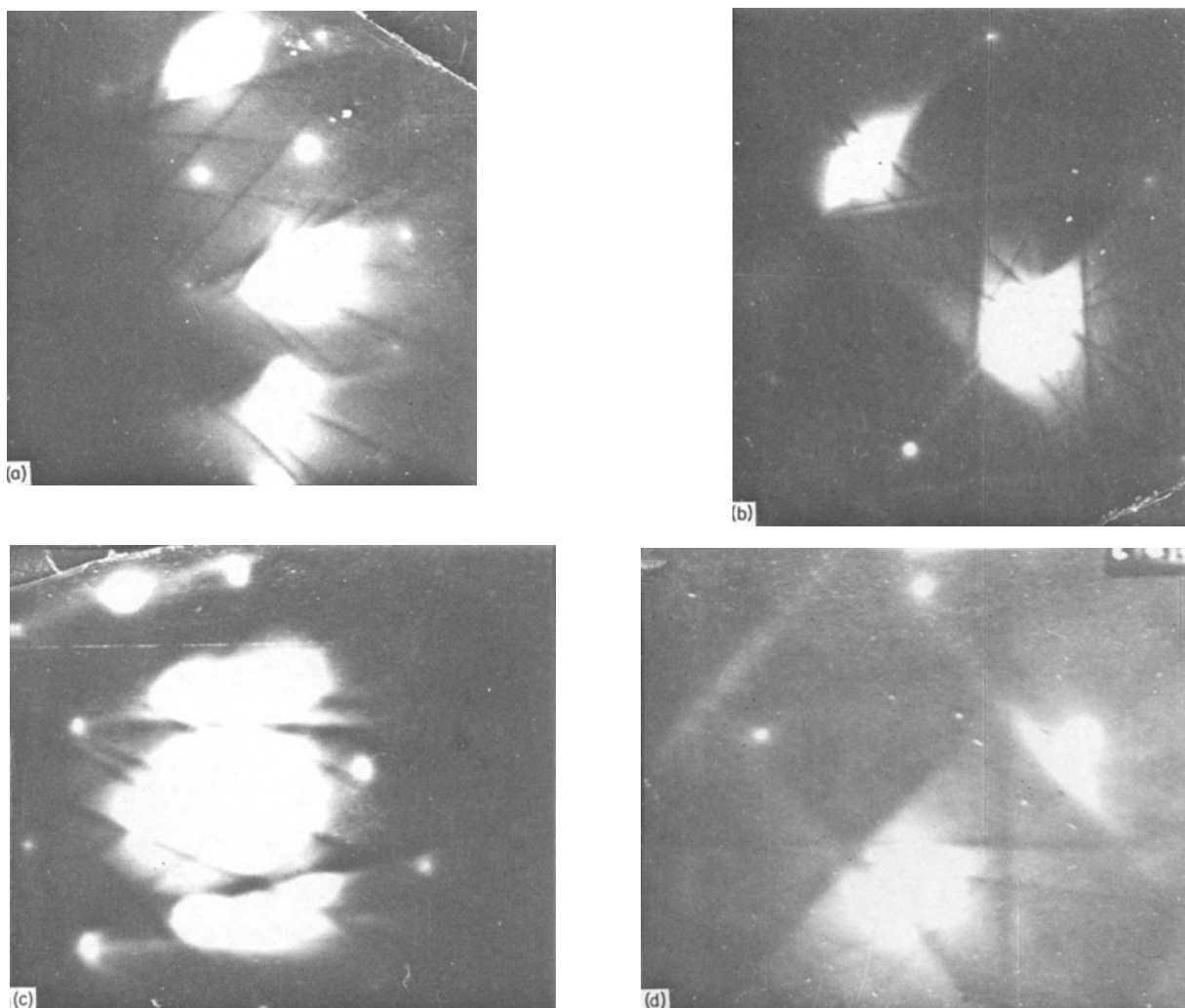


Figure 2.42 (a), (c) diffraction patterns from grains 1 and 2 respectively. (b), (d) diffraction patterns from the same grains after 26.5° tilt [Courtesy of P. H. Pumphrey]

figure 2.43(a) with one set of reflecting planes shown changing direction within the twin.

The most frequently occurring symmetry rule, but not the only one, describes the twin as the mirror image of the crystal structure of the matrix across a certain crystallographic plane called the twinning plane, as shown schematically in figure 2.43(a). The simplest examples are the deformation or annealing twins that occur in some f.c.c. metals; figure 2.43(b) shows the atomic positions in the lattice in such a twin. Here the plane of the paper is $(1\bar{1}0)$ and the planes labelled A, B, C are (111) perpendicular to the paper. In effect the whole lattice above the C plane has been sheared by an amount η_1 , the distance between the old atom position outlined by the dashed lines, and the new ones outlined by the continuous lines. Here the twin is a mirror image of the matrix in a (111) plane (the twin plane). Thus the stacking sequence across the twin plane is also a mirror image ABCABC; BACBACBA.

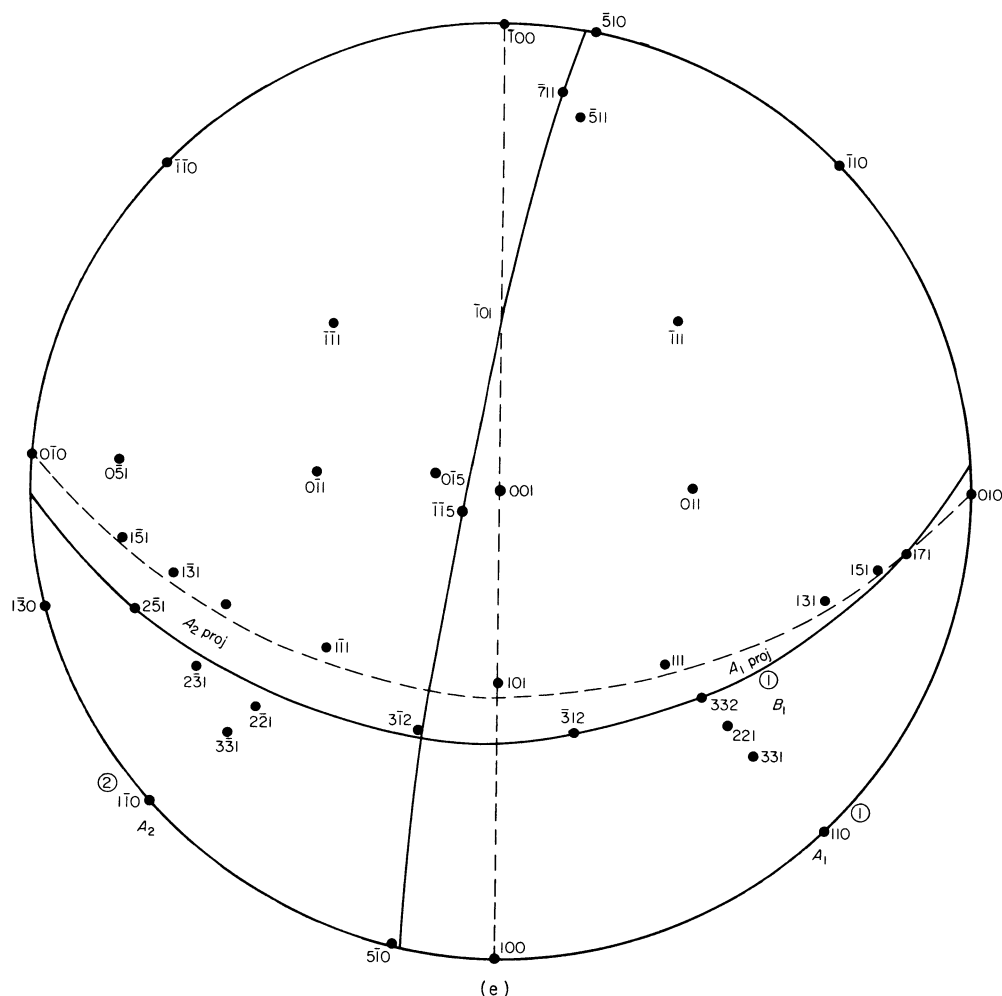
The crystallographic relationship of the twin to,

the matrix is described by four parameters K_1, K_2, η_1, η_2 which may be understood with reference to the schematic diagram in figure 2.43(c). Here the twin plane is K_1 and the twinned material above it has been distorted by a shear η_1 relative to the matrix below it. The plane K_2 which contains the direction η_2 is sheared through the angle ϕ by the twinning process but is not distorted, that is $\eta_2 = \eta'_2$. The K_2 plane is known as the second undistorted plane. In metals and alloys all four twinning elements are rational, the twin is called a compound twin, and subsequent discussion is limited to this type. Table 2.9 contains a list of the commonly occurring twin elements in metals and alloys.

2.12.2 The Distribution of Twin Spots in the Diffraction Pattern

2.12.2.2 Cubic crystal structures

If the twin–matrix orientation relationship is described by a simple reflection across K_1 then



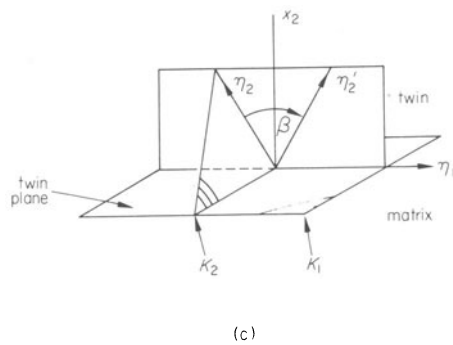
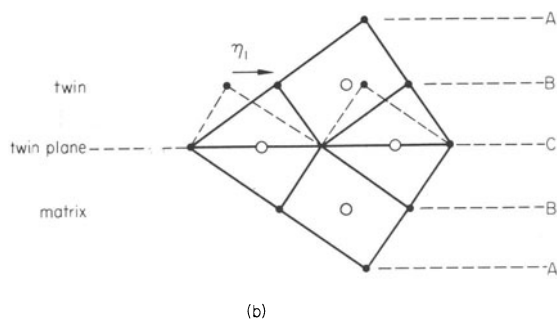
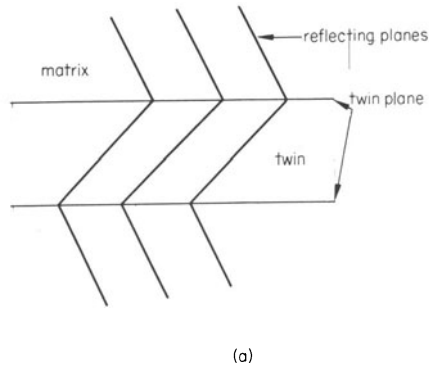


Figure 2.43 (a) plan view of a twin in a thin foil, (b) atom positions for a $(\bar{1}10)$ section of a twin on (111) in the f.c.c. crystal structure. ABC stacking of (111) is reversed inside the twin. (c) The geometry of the twin parameters η_1 , η_2 , K_1 , K_2

(b) K_1 randomly oriented relative to B . In this case the existence of twins is not immediately recognisable from the symmetry of the electron diffraction pattern. Even for a simple B only a few additional reflections may occur and CDF images may be used to check that the spots arise from the twins.

There are a number of approaches to solve this type of SADP. They are usually based on comparison of predicted patterns with experimental

Table 2.9 Twinning elements of the common metals and graphite

Material	Lattice	K_1	η_1	K_2	η_2
α -Fe V Nb W Mo Cr	b.c.c.	$\{112\}$	$\langle \bar{1}\bar{1}1 \rangle$	$\{11\bar{2}\}$	$\langle 111 \rangle$
Cu Au Ag	f.c.c.	$\{111\}$	$\langle 11\bar{2} \rangle$	$\{11\bar{1}\}$	$\langle 112 \rangle$
Zn Cd Co Mg Zr Ti Be	hex.	$\{10\bar{1}2\}$	$\langle \bar{1}011 \rangle$	$\{10\bar{1}\bar{2}\}$	$\langle 10\bar{1}1 \rangle$
Co Re Zr graphite	hex.	$\{11\bar{2}1\}$	$\langle 11\bar{2}\bar{6} \rangle$	$\{0001\}$	$\langle 11\bar{2}0 \rangle$
Mg	hex.	$\{10\bar{1}1\}$	$\langle \bar{1}012 \rangle$	$\{1013\}$	$\langle 30\bar{3}2 \rangle$
Zr Ti	hex.	$\{11\bar{2}2\}$	$\langle 11\bar{2}\bar{3} \rangle$	$\{11\bar{2}4\}$	$\langle 22\bar{4}3 \rangle$

patterns and therefore frequently involve computation of patterns or geometric analysis of the reciprocal lattice.

(i) Construction of a model of the twinned reciprocal lattice (Pashley and Stowell, 1963). For simple twins the approach is quite useful.

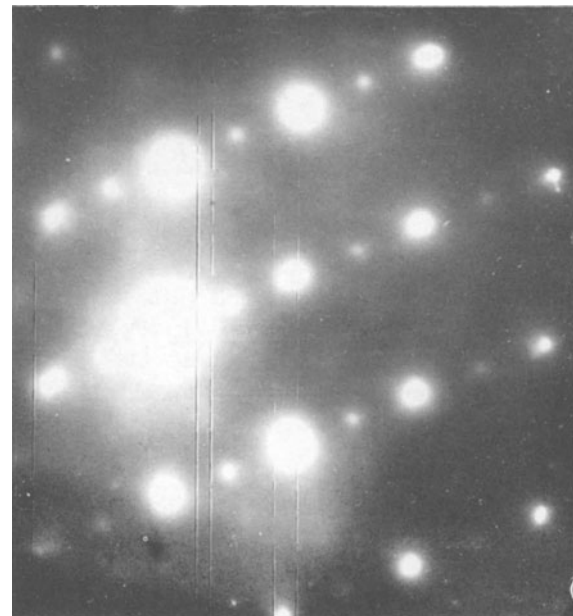
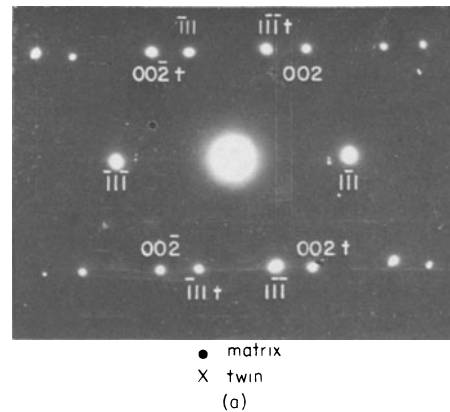


Figure 2.44 Twin patterns from (a) f.c.c. crystal structure $B[110]$, twinned on (111) [Courtesy of K. N. Melton] (b) Ordered $L1_0$ structure $B[201]$ twinned on (112) [Courtesy of J. L. Derclaye]

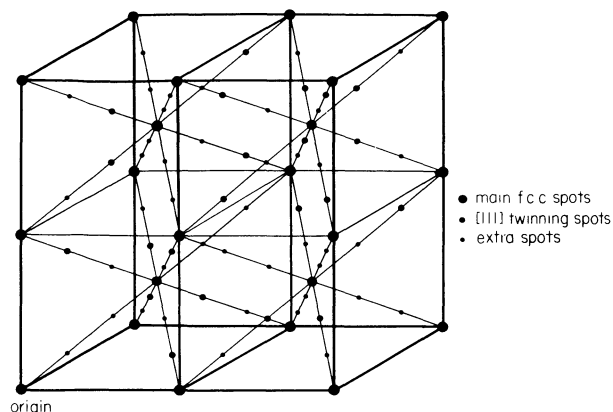


Figure 2.45 The location of twin spots in reciprocal space for twinning on $\{111\}$ and including double diffraction in an f.c.c. crystal structure [after Pashley and Stowell (1963)]

Figure 2.45 shows a reciprocal lattice model for the f.c.c. matrix (large spots) with twin spots (intermediate spots) and double diffraction spots (small spots). Thus all g_{111} in reciprocal space are divided into three. Two important conclusions can be drawn.

(1) Not all exact B will have extra spots in the diffraction pattern unless the foil is very thin or buckled, for example $B [100]$ has no spots but, if buckled, spots may appear as shown in figure 2.46 depending on which twin variants are present.

(2) Twin spots will occur at $n \times g/3$ in $\langle 111 \rangle$ in the diffraction pattern and may be easily recognised from their position, see figure 2.44(a).

Using this approach it is possible to plot standard electron diffraction patterns from the model for a particular combination of twinning elements and B . These may be compared directly with experimental patterns.

(ii) Use of a twin stereogram Kelley (1965) has pointed out that, whilst f.c.c. metals twin on $\{111\}$ planes in $\langle 112 \rangle$ directions, b.c.c. metals twin on $\{112\}$ planes in $\langle 111 \rangle$ directions, producing equivalent results. Consequently it is possible to construct a twinned

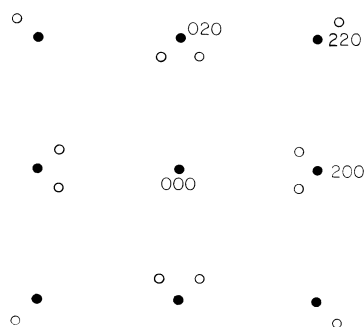


Figure 2.46 The position of twin spots with $B[001]$ in f.c.c. crystal structure for a thin buckled foil

stereogram, figure 2.47, containing twin and matrix poles for both twinning on $\{111\}$ for f.c.c. $\{112\}$ b.c.c. The equivalence of the twin processes in the f.c.c. and b.c.c. structures means that, for example, figure 2.47, $B [001]$ would be $B [22\bar{1}]$ twin. The procedure for analysing diffraction patterns is very rapid using this approach.

(1) Index the matrix pattern to determine the type of B using the rules in section 2.7.2.

(2) Locate all possible types of B on the stereogram to ascertain which twin zones are within $5-10^\circ$ of B . Thus, if B_{matrix} is $\langle 110 \rangle$ then B_{twin} could be $\langle 110 \rangle$ or $\langle 311 \rangle$.

(3) Index the extra spots on the basis of these zones using the rules in section 2.7.2 or by drawing great circles, pole B_{twin} in figure 2.47.

(iii) Calculation of twin patterns. Electron diffraction patterns from twinned crystals have been derived analytically by Meieran and Richman (1963), Johari and Thomas (1964), Crocker (1965), Wayman and Bullough (1966), and Calbick and Marcus (1967).

Meieran and Richman (1963) have produced a table of the location of twin spots in reciprocal space which is reproduced in table 2.10 in the modified form due to Andrews *et al.* (1971). The positions of the twin spots are described in terms of the matrix axes. Consequently they may be entered on the matrix pattern immediately, but will only be present if they satisfy the zone, equation (2.23).

Johari and Thomas (1964) have given details of how twin spots can be calculated for f.c.c. and b.c.c. metals. These calculations have been extended by Wayman and Bullough (1966) who include double diffraction effects and have derived the result in figure 2.45. They also show that for $\{112\}$ twins in b.c.c. metals doubly diffracted spots occur displaced parallel to $\langle 112 \rangle$ by an amount $\frac{1}{3}g \langle 112 \rangle$. This effect has been observed in internally twinned martensite by Patterson and Wayman (1966).

2.12.2.2 Hexagonal crystal structures

The simplest method of identifying twin spots in electron diffraction patterns is the stereographic technique described by Partridge (1967). This approach is similar to that described by Kelly (1965) and involves construction of a (0001) stereogram containing both matrix and twin poles. The positions of these twin poles may be constructed by rotating the (0001) projection about the normal to the plane containing η_1 and η_2 , figure 2.43(d) through an angle 2ϕ where ϕ is now the angle between K_1 and (0001) . The sense of rotation is such as to reduce ϕ . Rotations for twins in titanium are shown in table 2.11.

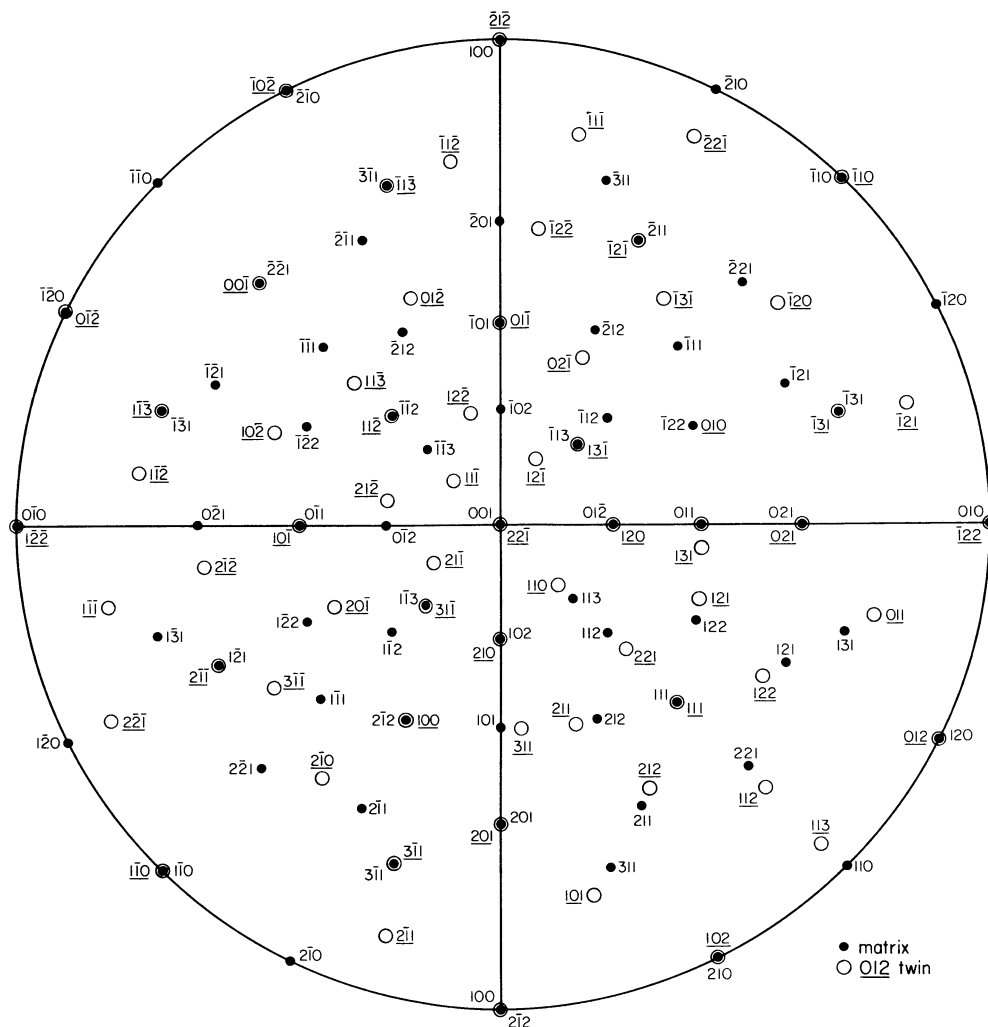


Figure 2.47 A superimposed matrix/twin stereogram for twinning in body- and face-centred cubic crystals [after Kelly (1965)]

A rational plane in the matrix may become a rational or an irrational plane in the twin. In figure 2.48(a) the basal plane in titanium, after twinning on $(10\bar{1}2)$, becomes a $(\bar{1}010)$ plane in the twin inclined 5° to the basal plane in the parent.

Directions in K_1 are unchanged in position and have the same type indices after twinning, for example $[\bar{1}011]$, $[\bar{1}\bar{2}10]$ for the $(10\bar{1}2)$ twin. Planes in η_1 zones are also unchanged in position and have similar indices after twinning. Such planes are parallel in twin and parent, and are listed for the three twins in table 2.12.

Figure 2.48 contains combined stereograms for $(10\bar{1}2)$, $(\bar{1}\bar{1}21)$ and $(\bar{1}\bar{1}22)$ twinning in titanium. Possible reflections from any twin occur as low-index reflections nearest to the great circle corresponding to the pole B in the matrix.

As an example of the prediction of a diffraction pattern, consider a situation in which B is $[0001]$ in the matrix for which the diffraction pattern will consist of reflections from planes whose poles lie in the great circle corresponding to the (0001) plane

of projection, see section 2.7.2.1. Using figure 2.48(a) the twin crystal will give rise to reflections from planes whose poles are close to the (0001) great circle. The nearest poles are those on the $(\bar{1}010)$ twin zone, namely $(\bar{1}\bar{2}10)$, $(1\bar{2}\bar{1}2)$, $(\bar{1}\bar{2}\bar{1}4)$, (0001) , etc. Consequently the diffraction pattern in figure 2.49(a) may be constructed using the approach outlined in section 2.7.2.1. Similarly the diffraction patterns for $(11\bar{2}1)$ and $(11\bar{2}2)$ twins for B $[0001]$ in the matrix may be obtained, see figures 2.49(b), (c). Clearly all three twin planes may be readily distinguished in practice by tilting the specimen such that B is $[0001]$ in the matrix and deriving the positions of the twin poles for other types of $\{10\bar{1}2\}$, $\{\bar{1}\bar{1}21\}$ and $\{\bar{1}\bar{1}22\}$ twins. Confirmation of the twin plane may be obtained by trace analysis, see section 2.14.1.

As for the f.c.c. case in the previous section, special cases arise when the coherent twin boundary (K_1 plane) is either parallel or normal to the foil surface.

For all twins, when η_1 and K_1 are parallel to B

Table 2.10 Location of twin spots in reciprocal space, f.c.c. and b.c.c. crystal structures

× denotes the $h_1k_1l_1$ and $h_2k_2l_2$ reflections which do not occur in b.c.c. structures				Twinning across (21 $\bar{1}$) (that is as in b.c.c. structures) →				← Twinning across (1 $\bar{1}$ 1) (that is as in f.c.c. structures)				× denotes the $h_1k_1l_1$ and $h_2k_2l_2$ reflections which do not occur in f.c.c. structures				× denotes the $h_1k_1l_1$ and $h_2k_2l_2$ reflections which do not occur in b.c.c. structures				Twinning across (21 $\bar{1}$) (that is as in b.c.c. structures) →				← Twinning across (1 $\bar{1}$ 1) (that is as in f.c.c. structures)				× denotes the $h_1k_1l_1$ and $h_2k_2l_2$ reflections which do not occur in f.c.c. structures				
h_1	k_1	l_1		h_2	k_2	l_2		h_1	k_1	l_1		h_1	k_1	l_1		h_2	k_2	l_2		h_1	k_1	l_1		h_2	k_2	l_2		h_1	k_1	l_1		
	0	1	1	0	1	1						×				2	-2	0		$\frac{2}{3}$	$-\frac{2}{3}$	$\frac{2}{3}$		-2	0	-2						
	0	-1	1		$-\frac{1}{3}$	$-\frac{1}{3}$		0	1	-1		×				0	1	3		$\frac{4}{3}$		$-\frac{2}{3}$		0	3	1		×				
	0	1	-1		$-\frac{1}{3}$	$-\frac{1}{3}$		0	-1	1		×				0	-1	3						0	3	-1		×				
	1	0	1		$-\frac{1}{3}$	$-\frac{1}{3}$		-1	1	0		×				0	3	1						0	1	3		×				
	-1	0	1		1	1		1	1	0		×				0	-3	1						0	1	-3		×				
	1	0	-1		-1	-1		-1	-1	0		×				3	0	1						-3	1	0		×				
	1	1	0		-1	0		-1	0	1		×				-3	0	1						3	1	0		×				
	-1	1	0		$-\frac{1}{3}$	$-\frac{1}{3}$		1	0	1		×				1	0	3						-1	3	0		×				
	1	-1	0		$-\frac{1}{3}$	$-\frac{1}{3}$		-1	0	-1		×				-1	0	3						1	3	0		×				
×	1	1	1		$-\frac{1}{3}$	$-\frac{1}{3}$		-1	1	1						1	3	0						-1	0	3		×				
×	-1	1	1		$-\frac{1}{3}$	$-\frac{1}{3}$		1	1	1						-1	3	0						1	0	3		×				
×	1	-1	1		1	1		-1	1	-1						3	1	0						-3	0	1		×				
×	1	1	-1		$-\frac{1}{3}$	$-\frac{1}{3}$		-1	-1	1						-3	1	0						3	0	1		×				
	0	0	2		$\frac{4}{3}$	$\frac{4}{3}$		0	2	0						×	1	1	3		1	1	3		-1	3	1					
	0	2	0		$-\frac{1}{3}$	$-\frac{1}{3}$		0	0	2						×	-1	1	3					1	3	1						
	2	0	0		$-\frac{1}{3}$	$-\frac{1}{3}$		-2	0	0						×	1	-1	3					-1	3	-1						
	1	1	2		$-\frac{1}{3}$	$-\frac{1}{3}$		-1	2	1		×				×	1	1	-3		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-1	-3	1					
	-1	1	2		1	2		1	2	1		×				×	1	3	1		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-1	1	3					
	1	-1	2		$-\frac{1}{3}$	$-\frac{1}{3}$		-1	2	-1		×				×	1	-3	1		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-1	1	-3					
	1	1	-2		$-\frac{1}{3}$	$-\frac{1}{3}$		-1	-2	1		×				×	1	3	-1		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-1	-1	3					
	1	2	1		-1	1		-1	1	2		×				×	3	1	1		-1	-1	-1		-3	1	1					
	-1	2	1		$-\frac{1}{3}$	$-\frac{1}{3}$		1	1	2		×				×	-3	1	1		1	3	-1		3	1	1					
	1	-2	1		$-\frac{1}{3}$	$-\frac{1}{3}$		-1	1	-2		×				×	3	-1	1		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-3	1	-1					
	1	2	-1		$-\frac{1}{3}$	$-\frac{1}{3}$		-1	-1	2		×				×	3	1	-1		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-3	-1	1					
	2	1	1		$-\frac{1}{3}$	$-\frac{1}{3}$		2	-1	-1		×				×	2	2	2		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-2	2	2					
	2	-1	1		$-\frac{1}{3}$	$-\frac{1}{3}$		-2	1	-1		×				×	-2	2	2		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		2	2	2					
	-2	1	1		$-\frac{1}{3}$	$-\frac{1}{3}$		-2	1	1		×				×	2	-2	2		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-2	2	-2					
	2	1	-1		-2	-1		-2	-1	1		×				×	2	2	-2		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-2	-2	2					
	0	2	2		0	2		0	2	2							1	2	3		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-1	3	2		×			
	0	2	-2		$-\frac{1}{3}$	$-\frac{1}{3}$		0	-2	2							-1	2	3		1	3	2		1	3	2		×			
	2	0	2		$-\frac{1}{3}$	$-\frac{1}{3}$		-2	2	0							1	-2	3		3	-1	2		-1	3	-2		×			
	2	0	-2		-2	-2		-2	-2	0							1	2	-3		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-1	-3	2		×			
	2	2	0		-2	0		-2	0	2							4	-1	1		0	-3	3		-4	1	-1		×			
	2	1	3		$-\frac{1}{3}$	$-\frac{1}{3}$		-2	3	1		×					4	1	-1		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-4	-1	1		×			
	-2	1	3		2	3		2	3	1		×					0	3	3		0	3	3		0	3	3		×			
	2	-1	3		-1	3		-2	3	-1		×					0	3	-3		-4	1	-1		0	-3	3		×			
	2	1	-3		$-\frac{1}{3}$	$-\frac{1}{3}$		-2	3	-1		×					3	0	3		1	-1	4		-3	3	0		×			
	3	2	1		$-\frac{1}{3}$	$-\frac{1}{3}$		-3	1	2		×					3	0	-3		-3	-3	0		-3	-3	0		×			
	-3	2	1		$-\frac{1}{3}$	$-\frac{1}{3}$		3	1	2		×					3	3	0		-3	0	3		-3	0	3		×			
	3	-2	1		-1	3		-2	3	-1		×					3	0	-3		-3	0	3		-3	0	3		×			
	3	2	-1		-3	-1		-3	-1	2		×					3	3	0		-3	0	3		-3	0	3		×			
	2	3	1		-2	-1		-2	1	3		×					3	-3	0		1	-4	1		-3	0	-3		×			
	-2	3	1		$-\frac{1}{3}$	$-\frac{1}{3}$		2	1	3		×					3	3	1		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-3	1	3					
	2	-3	1		2	-3		-2	1	-3		×				×	3	3	1		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		3	1	3					
	2	3	-1		$-\frac{1}{3}$	$-\frac{1}{3}$		-2	-1	3		×				×	-3	3	1		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-3	1	-3					
	3	1	2		$-\frac{1}{3}$	$-\frac{1}{3}$		-3	2	1		×				×	3	3	-1		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-3	-1	3					
	-3	1	2		$-\frac{1}{3}$	$-\frac{1}{3}$		3	2	-1		×				×	3	1	3		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-3	3	1					
	3	-1	2		1	-2		-3	2	-1		×				×	-3	1	3		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-3	3	1					
	3	1	-2		-3	-2		-3	-2	1		×				×	3	-1	3		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-3	3	-1					
	3	3	2		-1	2		-1	2	3		×				×	3	-1	3		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-3	-3	1					
	-1	3	2		$-\frac{1}{3}$	$-\frac{1}{3}$		1	2	3		×				×	3	1	-3		$-\frac{1}{3}$	$-\frac{1}{3}$	$-\frac{1}{3}$		-							

Table 2.11 Rotations for twins in titanium

Twin plane K_1	Rotation axis (plane of shears normal)	Angle of rotation
$\{10\bar{1}2\}$	$\langle\bar{1}2\bar{1}0\rangle$	95°
$\{\bar{1}122\}$	$\langle\bar{1}100\rangle$	$63^\circ 58'$
$\{\bar{1}121\}$	$\langle\bar{1}100\rangle$	$34^\circ 54'$

all twin spots are coincident with matrix spots and only one pattern is obtained. Coincident spots for the three twin systems are given in table 2.12.

When the electron beam is parallel to other directions in K_1 , the twin spots can be obtained by rotation of the parent pattern 180° about an axis normal to the twin boundary to produce a mirror image of the parent pattern across the twin boundary. This is shown in figure 2.50(a), (b) for a beam parallel to $[1\bar{1}00]$ and for reflections from two different twin modes belonging to the $[1\bar{1}00]$ zone. The twins give rise to identical patterns and can only be distinguished by the difference in angle between the twin and parent patterns in each

case. A similar situation exists when the beam is normal to the plane of shear of two reciprocal $\{10\bar{1}2\}$ twins.

When K_1 is normal to the electron beam and for small twinning shears, that is $\{10\bar{1}2\}$, $\{1122\}$ twins, the twin and parent diffraction patterns are similar. However, the twin spots will be displaced slightly from the parent spots. Two examples of indexing twin patterns in titanium are described in section 4.13.1, in which the approach described in this section is used.

As an alternative to this stereographic method, the positions of twin spots may be calculated using the program of Johari and Thomas (1964). Double diffraction spots may be calculated using the approach of Wayman and Bullough (1966). These workers showed that the position of the double diffracted reflections depends on c/a , and, since Miller–Bravais $(hkil) = (hkl)$ Miller then

$$(h''k''l'')_{\text{doubly diffracted}} = (h'k'l')_{\text{matrix}} + \frac{L}{r^2} (hkl)_{\text{twin}}$$

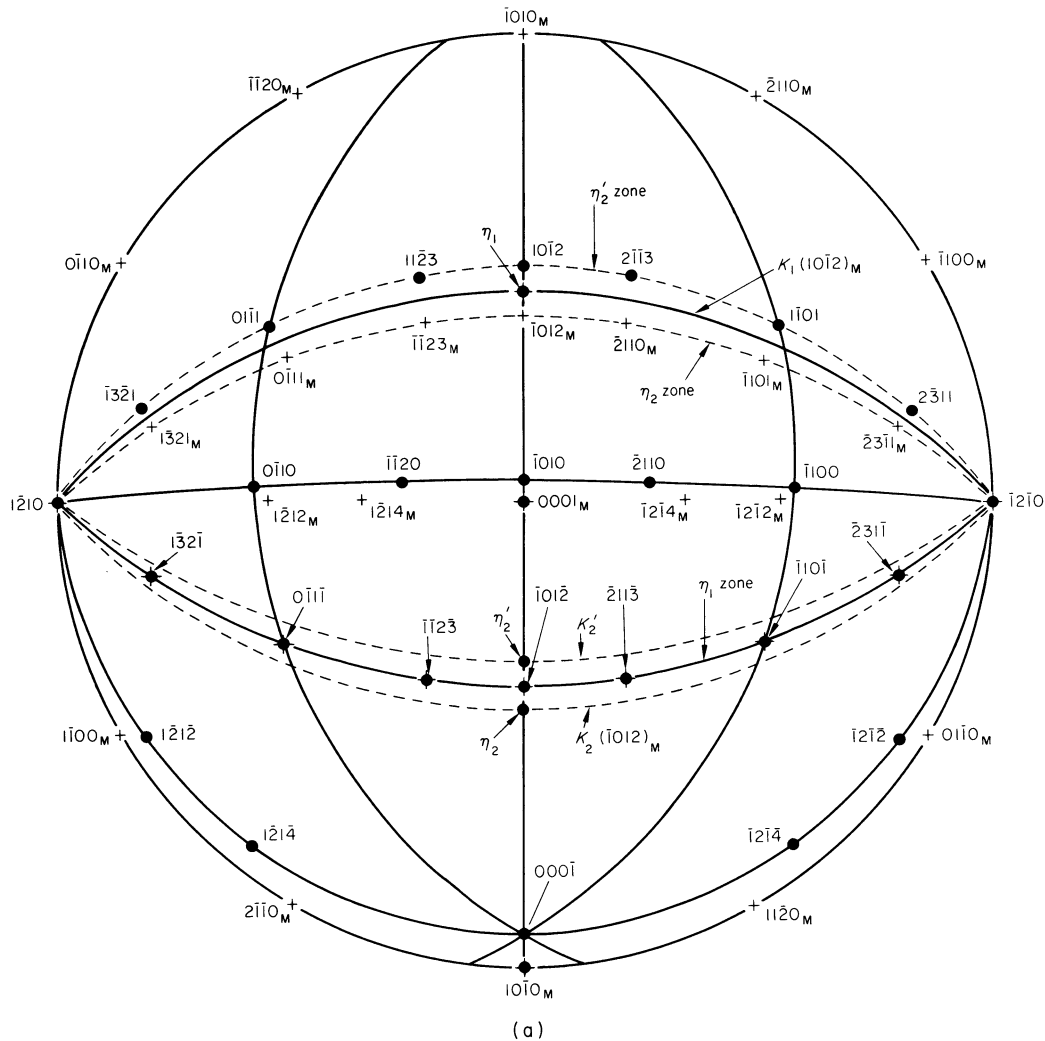


Figure 2.48 Superimposed matrix/twin stereograms for (a) $(10\bar{1}2)$, (b) $(\bar{1}\bar{1}21)$ and (c) $(\bar{1}\bar{1}22)$ twinning in titanium. Also shown are K_1 , K_2 , K'_2 , η_1 , η_2 , η'_2 after twinning and the zones for the last three axes: + parent; • twin; ✦ coincident poles [From Partridge (1967), courtesy of *Metallurgical Reviews*]

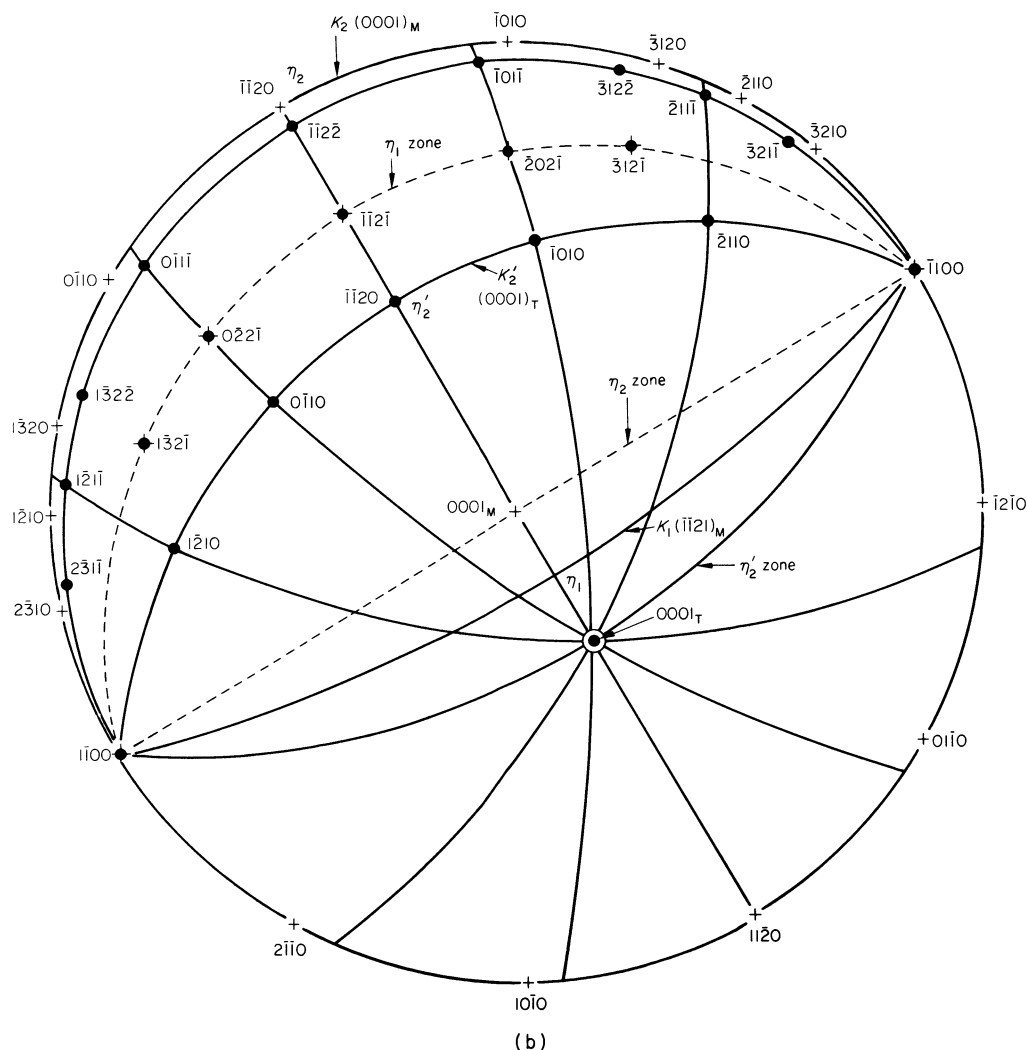


Figure 2.48 (continued)

where $(h'k'l')$ matrix is the matrix spot giving rise to the doubly diffracted spot and

$$r^2 = \frac{4h^2}{3} + \frac{4k^3}{3} + \frac{4hk}{3} + \frac{l^2a^2}{c^2} \quad (2.31)$$

and

$$L = \left(\frac{8h}{3} + \frac{4k}{3}\right)x_h + \left(\frac{4k}{3} + \frac{8k}{3}\right)x_k + \frac{21a^2}{c^2}x_l \quad (2.32)$$

where x_h, x_k, x_l are the Miller indices for the (hkl) matrix reflections.

2.13 Second Phases

2.13.1 Identification

In this context the complete determination of the crystal structure of a new phase is not implied, rather confirmation that the phase is one for which the crystal structure has been previously characterised, usually by x-ray diffraction.

Second phases such as precipitates are generally most easily identified using carbon extraction replica techniques combined with electron diffraction to produce a ring pattern, see section 2.6.1. The ring pattern may be indexed using the approach outlined in section 2.7.1 with the aid of ASTM* index cards. Figure 2.51 shows an extraction replica of Fe₂TiSi precipitates in a quenched Fe–1.7 at% Ti–7 at% Si alloy aged for 1 hour at 700 °C. The ring electron diffraction pattern taken from an extraction replica is shown in figure 2.18(a) which

Table 2.12 Coincident reflections in twin and parent matrix for titanium

Twin plane (K_1)	(10 $\bar{1}$ 2)	($\bar{1}$ 121)	($\bar{1}$ 122)
Zone axis (η_1)	[$\bar{1}$ 011]	[11 $\bar{2}$ 6]	[11 $\bar{2}$ 3]
Indices of planes in the parent	(1 $\bar{2}$ 10)	(1 $\bar{1}$ 00)	(1 $\bar{1}$ 23)
	(2 $\bar{3}$ 10)	(1 $\bar{3}$ 21)	(2 $\bar{3}$ 11)
	(1 $\bar{1}$ 01)	(0 $\bar{2}$ 21)	(1 $\bar{2}$ 11)
	(10 $\bar{1}$ 2)	($\bar{1}$ 121)	($\bar{1}$ 322)
	(2 $\bar{1}$ 13)		(0 $\bar{1}$ 11)

* ASTM index to the powder diffraction file (American Society for Testing Materials, Philadelphia, U.S.A.).

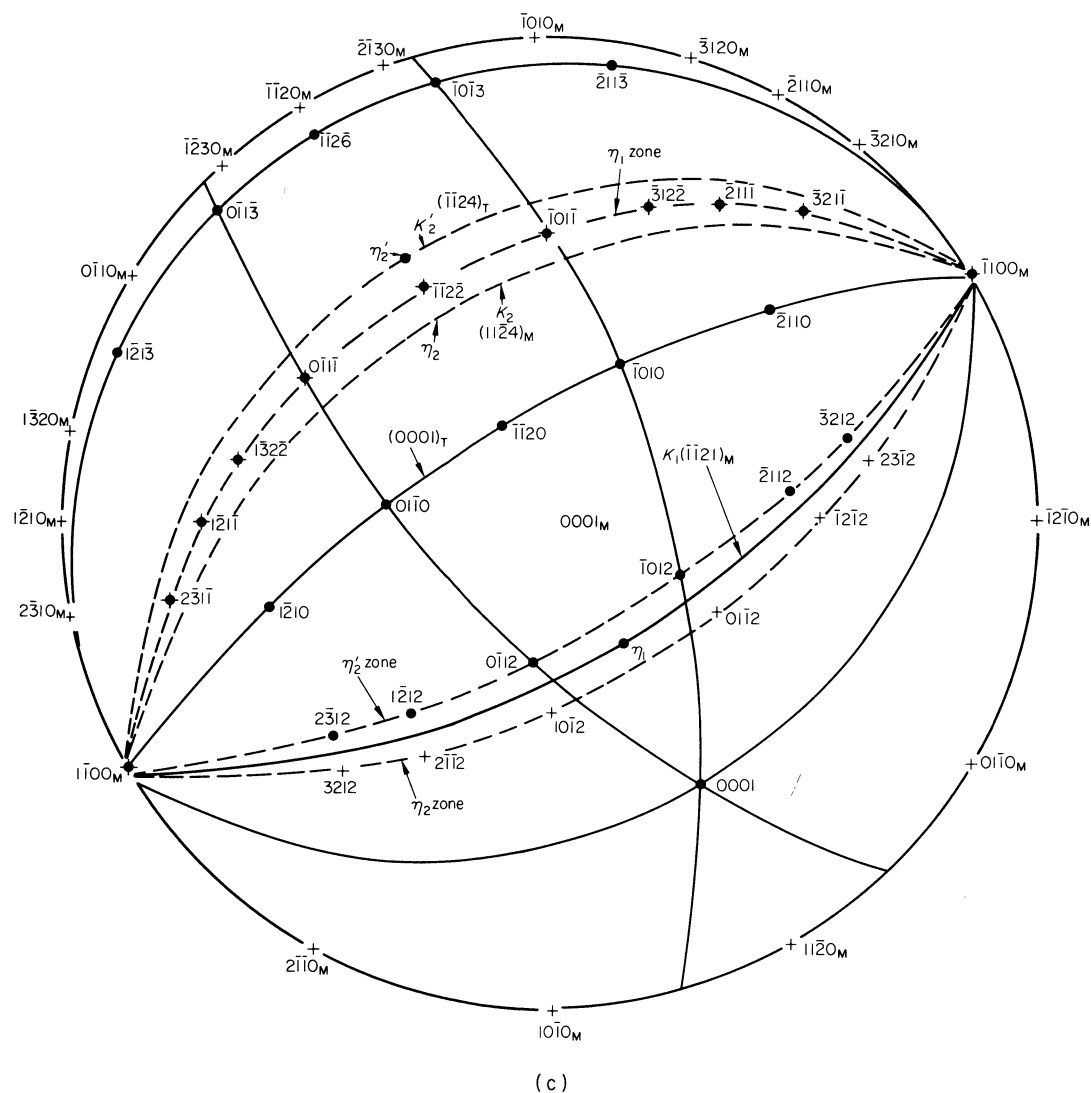


Figure 2.48 (continued)

has been indexed as shown using the rules described in section 2.7.1. In some cases, it may be necessary to use a built-in standard so that identification of the phase is independent of the camera constant because this parameter cannot always be calibrated to the necessary accuracy. Section 1.11.2 summarises the various techniques that may be used. After indexing the pattern the lattice parameter may be compared with that in the ASTM index card* for that phase.

It is possible to identify second phases from single-crystal electron diffraction patterns. However, a large number of beam directions must be obtained with the aid of the specimen tilt facility so that all reflections are obtained to enable the crystal structure to be unambiguously identified. Note that, because of double diffraction effects, this approach can present difficulties when small particles are present (see section 2.16.1). Furthermore, streaking of reciprocal lattice points because of elastic strain or precipitate shape effects may

complicate interpretation of SADPs, as described in sections 2.18.1, 2.18.2. Finally, if two phases are present, further complications will occur. Suggestions for overcoming these difficulties are listed in sections 2.18.1, 2.18.2 and worked examples are described in sections 4.9.2, 4.12.6.

Single-crystal patterns can be very useful in identifying phases for which electron diffraction patterns have already been published by direct comparison for specific **B**. This approach is very useful when weak superlattice reflections are present because these are often only visible for **B** exactly parallel to zone axes and are very difficult to detect in ring patterns from extraction replicas and in x-ray diffraction patterns.

Structure determination of unknown second phases in the electron microscope is difficult because a complete map of the reciprocal lattice must be constructed by tilting the specimen. Complicating effects can arise from double diffraction and from streaking of reciprocal lattice

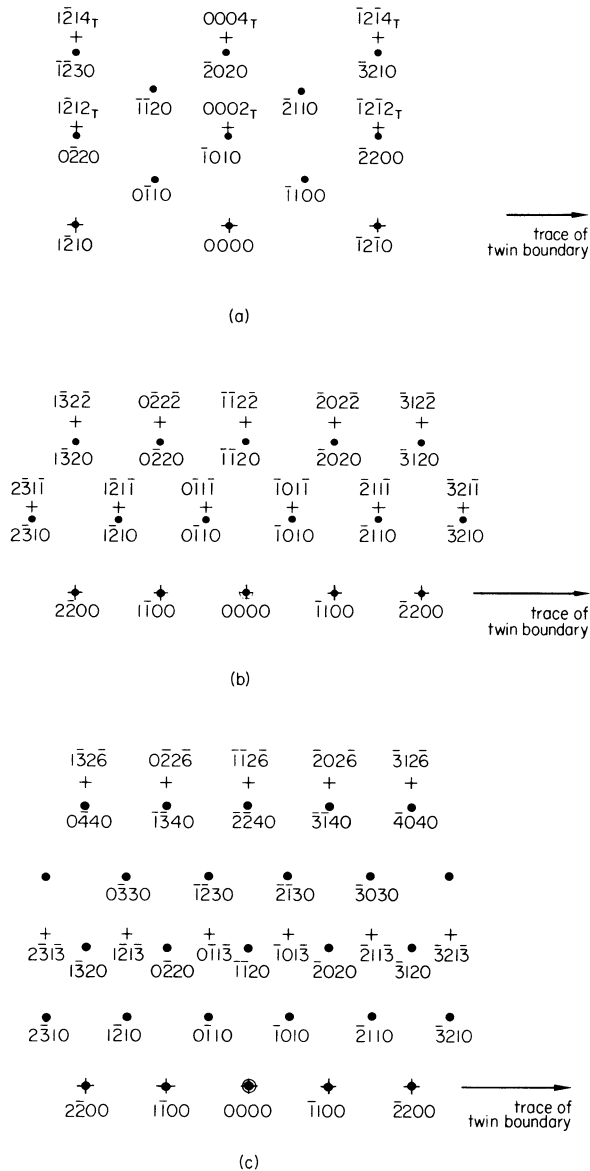


Figure 2.49 The positions of twin spots in the (0001) matrix diffraction pattern in titanium, (a) $(10\bar{1}2)$ twin, (b) $(\bar{1}\bar{1}21)$ twin, (c) $(\bar{1}\bar{1}22)$ twin: • parent; + twin; ✕ coincident, ○ forbidden reflections [From Partridge (1967), courtesy of *Metallurgical Reviews*]

points. Furthermore, quantitative intensity measurements cannot be made in the same way as for x-rays. Nevertheless, structure determinations have been reported in a number of phases. However, it is important to use other techniques such as x-ray analysis or nuclear magnetic resonance to check the conclusions. The techniques of structure determination are complex and have been reviewed by Vainshtein (1964) and the reader is referred to this book for further detailed discussion.

2.13.2 Determination of Crystal System

It has been pointed out in section 2.10.3 that, because Kikuchi lines exhibit Laue crystal symmetry, they may be used in principle to determine

the crystal system to which a phase belongs. However, such determination is generally simpler using alternative x-ray techniques. Some success may be obtained in the electron microscope where large angles of specimen tilt are readily available.

2.13.3 Determination of Differences in Lattice Parameter Between Precipitate and Matrix

In phase transformations, involving the formation of a new phase with a different lattice parameter from the parent phase, it may be possible to measure the difference in lattice parameter from the Kikuchi pattern. As pointed out in section 2.6.3, Kikuchi line spacing is a more accurate measure of the lattice parameter than spot spacing. However, in some cases Kikuchi lines may be too diffuse because of matrix strain. Figure 2.52 shows a

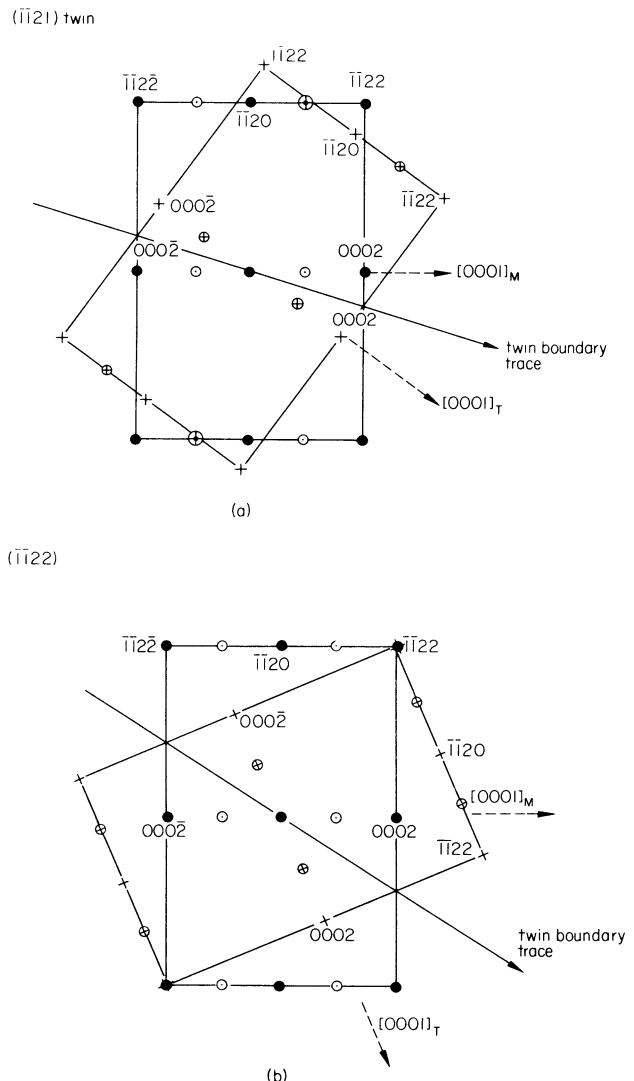


Figure 2.50 Diffraction patterns produced when $B[1\bar{1}00]$ matrix K_1 is parallel to and η_1 normal to the electron beam (a) $(\bar{1}\bar{1}21)$ twin, (b) $(\bar{1}\bar{1}22)$ twin [From Partridge (1967), courtesy of *Metallurgical Reviews*]

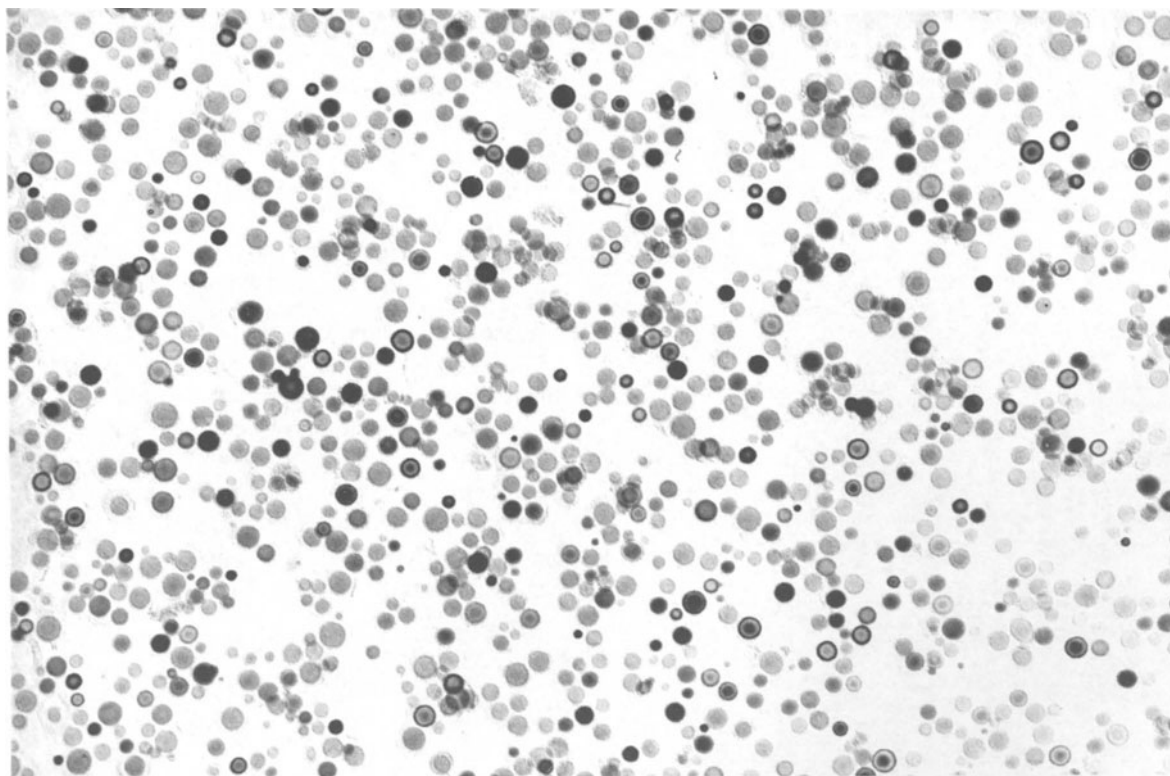


Figure 2.51 Extraction replica showing Fe_2TiSi precipitates [Courtesy of D. H. Jack]

Kikuchi pattern from an f.c.c. Ni–38.5 at% Fe–3.04 at% V–2.4 at% C alloy containing a fine dispersion of coherent ‘matrix dot’ vanadium carbide particles, lattice parameter 4.14 Å. The difference in lattice parameter indicated by the Kikuchi line pairs is ~4% but the accuracy is not much higher than that from spot measurement because of the diffuse lines.

Kikuchi line spacing is also useful in determining, for example c/a ratio in martensitic transformations provided the transformation does not induce a high density of crystal defects so as to make Kikuchi lines too diffuse.

2.13.4 Detection of the Initial Stages of Phase Transformations

Kikuchi line splitting discussed above occurs during the early stages of precipitation or order–disorder transformations before significant effects are visible in the image (van Torne and Thomas, 1966) provided there is a change in lattice parameter. Thus this effect may be used to detect the early stages of such reactions. It is also possible that Kikuchi lines may be used to detect the beginning of spinodal decomposition, perhaps by the presence of satellite lines although this effect has not yet been reported. To assist in indexing Kikuchi lines in cases where several phases are present, dark field images, see section 1.5.2, may be formed using

the bright Kikuchi lines. However, these are normally low-quality images because of chromatic aberration.

2.14 Crystallographic Information

2.14.1 Habit Plane Determination

Two cases may be distinguished, namely when a planar defect does or does not intersect one surface of the foil. These cases are treated separately.

2.14.1.1 Defect intersects the foil surface

Trace analysis of the plane of a crystal defect is based on determination of the direction of intersection of the habit plane of the defect and the angle between the habit plane and the foil normal. An example is shown in figure 2.53 in which a very thin planar precipitate exists in a thin foil of

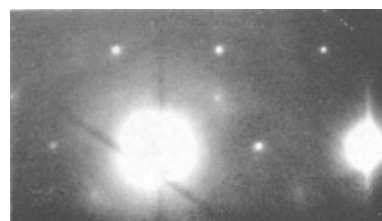
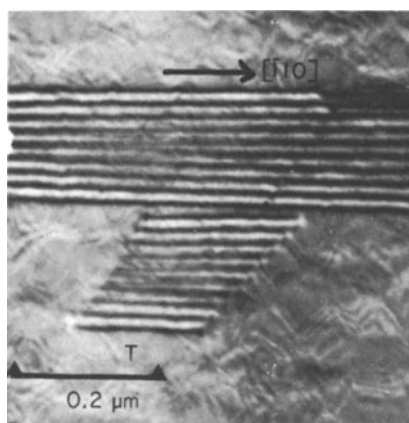
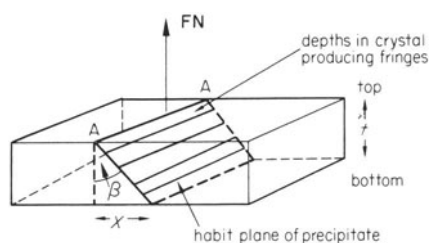


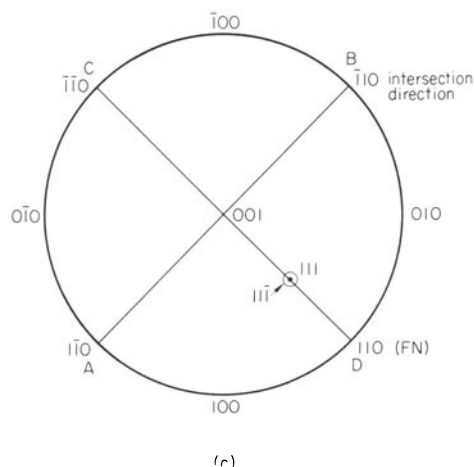
Figure 2.52 SADP from quenched and aged Ni–38.5 wt% Fe–3.04 wt% V–2.4 wt% C showing split Kikuchi lines



(a)



(b)



(c)

Figure 2.53 Trace analysis of a planar precipitate in vanadium carbide: (a) BF image $FN[110]$, (b) diagram of geometry of precipitate within foil, (c) stereogram describing the crystallography of (a)

boron-doped vanadium carbide. The precipitate lies on an inclined plane as shown in figure 2.53(b) and is visible, as described in section 3.6.3, as fringes occurring at specific depths in the crystal and running parallel to the line of intersection of the foil surface and habit plane. If the angle β , the foil normal FN , the crystallographic direction of intersection of the habit plane and the foil plane are known, the indices of the habit plane may be determined. In figure 2.53(a) the foil normal

FN is $[110]$ and is within 4° of B . The operative reflection is (220) with $s = 0$ and the direction of intersection of the defect and foil planes is $[\bar{1}10]$. The foil thickness t is obtained from the number of fringes which at $s = 0$ correspond to $\frac{1}{2}\xi_g$, see section 3.6.3, because these images are known to behave like stacking faults (Hollox *et al.*, 1972). Thus the foil thickness $= 6\xi_g \approx 1650 \text{ \AA}$. The projected width of the defect plane into the foil plane is $2350 \text{ \AA}$. Consequently $\tan \phi$, angle between foil plane and defect plane, $= \frac{1650}{2350}$ that is $\phi = 35^\circ$.

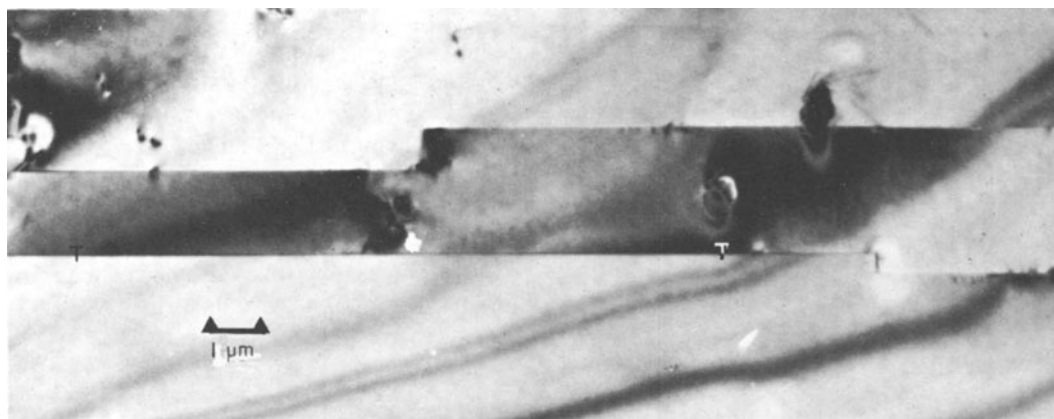
Using the stereogram, figure 2.53(c), great circle AB represents all of the directions lying in the foil plane with foil normal FN $[110]$, and $[\bar{1}10]$ is the direction of intersection of the defect plane with the foil plane, that is the direction common to both planes. The defect plane normal must be perpendicular to $[\bar{1}10]$ and therefore lies in the great circle CD at 35° to the $[110]$ foil normal, that is it is either $[111]$ or $[\bar{1}\bar{1}\bar{1}]$. Comparison of bright and dark field photographs shows that T , figure 2.53(a), is the top of the foil and the defect plane normal is $[111]$, see section 3.6.2.3.

The procedure for performing a trace analysis is as follows.

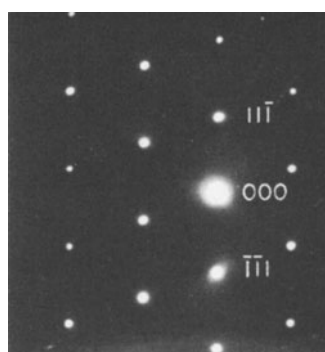
- (1) Locate the area of interest.
- (2) Obtain a SADP within $\pm 2^\circ$ of zero specimen tilt in order to determine the foil normal FN .
- (3) Obtain a two-beam image (for definition see section 3.2.1) with B within $\pm 5^\circ$ of FN with $s = 0$ at a known magnification.
- (4) Determine the thickness of the specimen by counting fringes or any of the techniques described in appendix 16.
- (5) Determine the crystallographic direction $[uvw]$ of intersection of the habit plane and the foil surface.
- (6) Measure the projected width of the defect, X , figure 2.53(b), and determine the angle β .
- (7) Draw the great circle pole $[uvw]$ on the stereogram and measure angle β from FN on the great circle.
- (8) Determine the sense of inclination of the defect plane relative to the foil plane by either tilting the specimen in a known sense (see section 2.10.1) or comparing BF and DF images if asymmetric like a stacking fault (see section 3.6.2.3).

Using this approach errors in the habit plane of $\pm 5^\circ$ may occur primarily because of errors in ξ_g which is only accurate to $\pm 10\%$ even in pure metals and because of experimental errors in setting $s = 0$.

It is very important to realise that, in a modern electron microscope which has facilities for tilting the specimen through large angles, a trace analysis



(a)



(b)

Figure 2.54 (a) A twin boundary TT in austenitic stainless steel, tilted edge on, (b) diffraction pattern from matrix [Courtesy of A. Jones]

must be performed with **B** and **FN** as close as possible, that is a small angle of tilt from the zero position. Limited tilting only $\pm 5^\circ$ is allowed to enable good two-beam conditions to be obtained. If the specimen is significantly tilted from this position, errors arise because directions are projected into a diffraction pattern (a plane in the reciprocal lattice) that is not parallel to the foil plane. Thus in relating a picture and a diffraction pattern taken at large angles of tilt using the above analysis very significant errors can be introduced.

Pashley and Presland (1959) have analysed the effect of specimen tilting on trace analysis of habit planes. They showed that the most serious errors occur when the planar defect is inclined at a large angle to the electron beam and the axis of tilt makes a large angle with the trace direction obtained at zero specimen tilt.

As an alternative approach in electron microscopes such as the EM300, where large angles of specimen tilt are available, it is often useful to tilt the specimen such that the habit plane of the defect lies parallel to the electron beam, that is minimum width of the image. Figure 2.54 shows a typical example of such an analysis for a twin boundary in austenitic stainless steel. The boundary

was set parallel to the tilt axis in the tilt/rotation stage of the EM300, and **FN** determined from a SADP taken at zero tilt. Then the specimen was tilted in a known direction until the boundary plane width was a minimum. In the diffraction pattern at the angle of tilt the $(\bar{1}\bar{1}1)$ reflection was very strong and, after allowing for image rotation (see section 1.11.3), **g** $[111]$ was within 1° of perpendicular to the line of the twin boundary. Furthermore, the angle of tilt measured on the goniometer stage was within 1° of the angle between **FN** and $(\bar{1}\bar{1}1)$. Consequently the habit plane may be concluded to be $(\bar{1}\bar{1}1)$ which is the coherent twin plane in f.c.c. materials, see table 2.9.

Using the above approach the procedure is as follows.

- (1) Select an area of interest.
- (2) Determine **FN** by obtaining a SADP at zero tilt from the area of interest.
- (3) Set the tilt axis parallel to the line of intersection of the foil and defect planes (see section 1.11.5.4).
- (4) Tilt the specimen in a known sense until the defect is edge on (minimum image thickness).
- (5) Obtain image and SADP at this tilt and

analyse as above using measured angle of tilt on goniometer and the presence of a bright reflection with g perpendicular to the trace of the plane.

(6) Note that for maximum accuracy a series of pictures should be taken at different angles of tilt and the projected width of the boundary measured to determine the position of minimum image width. Errors are generally $\pm 0.5^\circ$ and may be realistically estimated to $\pm 1-2^\circ$ for most similar experiments. Significant errors will always arise because the image of the boundary is influenced by the diffracting conditions and may remain constant over several degrees of tilt.

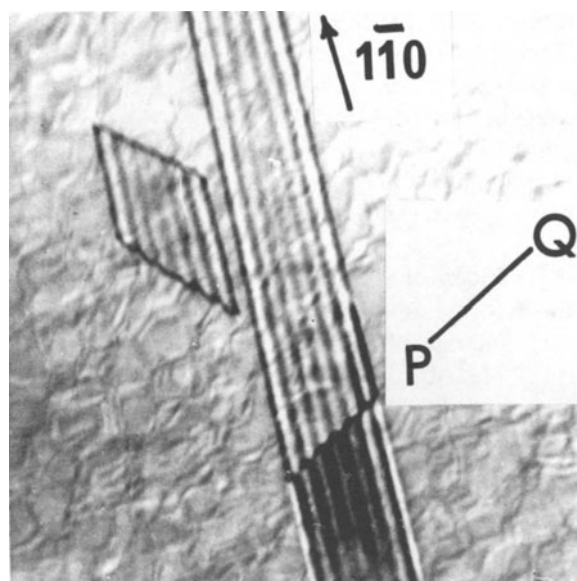
2.14.1.2 Defect contained within the foil

Typical examples of this type of defect are dislocation loops, small planar precipitates, etc. The most effective approach is to determine first the shape of the defect by tilting the specimen until the habit plane is approximately perpendicular to B . The sequence of events will then depend upon the shape of the defect. If it is large with well-defined sides like the Frank loop in figure 3.15 the directions of two of the sides of the defect may be determined using the approach outlined in the section 2.14.2 which will define the habit plane. Alternatively, if the defect is a round disc or has an irregular shape, the approach of tilting in a known direction by a measured amount until the defect is edge on may be used. In such an experiment a low-index tilt axis is selected by tilting about a bright spot as described in section 2.10.1 with g in the foil plane. Then the procedure is the same as that outlined above in section 2.14.1.1. However, the errors may be quite large for small plate precipitates or loops because of difficulties in deciding when exactly edge on, particularly when strain field contrast occurs. Weak beam images (section 1.5.3) or precipitate CDF images (section 1.5.2) may improve the accuracy of such analyses.

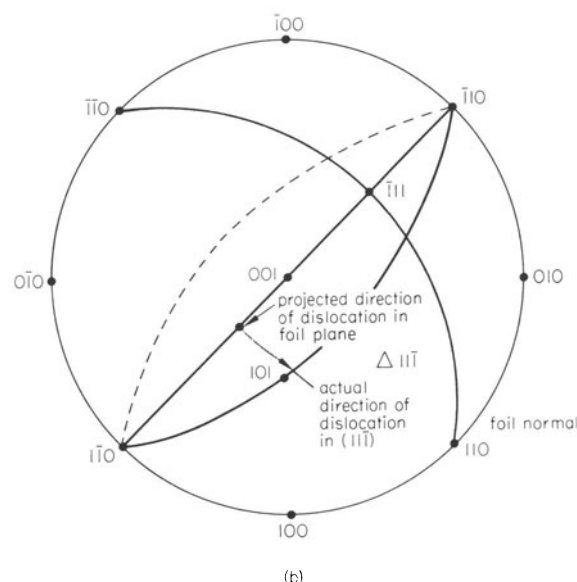
A number of alternative methods of habit plane analysis, particularly for small loops, have been reported. For example Hudson and Makin (1963) determined the $\{021\}$ habit plane of diamond-shaped loops in a quenched Al-1% Mg alloy using trace analysis of the major axis of the loops. However, the approach outlined above is recommended as the most self-consistent both because of its relatively high accuracy and because of the removal of the 180° ambiguity described in section 2.8.1 if the sense of tilt is known from the Kikuchi map.

2.14.2 Line Direction u of a Dislocation

If the plane in which the dislocation lies is known then the determination of its line direction is relatively simple. Figure 2.55(a) shows overlapping



(a)



(b)

Figure 2.55 Line direction determination for dislocation PQ associated with planar precipitate in vanadium carbide, (a) BF image, (b) stereographic analysis

precipitates on the $(11\bar{1})$ plane of vanadium carbide (determined by trace analysis plus dark field to obtain the sense of inclination of the plane. Here the foil normal is $[110]$ and B is close to $[110]$. The $(11\bar{1})$ plane intersects the (110) plane parallel to $[1\bar{1}0]$ and the projected direction of PQ into the diffraction pattern is 61° clockwise from $[1\bar{1}0]$. This is drawn in the stereogram in figure 2.55(b) and is within 1° of $[101]$ in the defect plane $(11\bar{1})$ following the rules for projecting directions, see appendix 1.

If the habit plane of the dislocation is not known, for example an isolated single dislocation, the line direction of the dislocation may be determined

from three photographs with different beam directions in the crystal. Figure 2.56(a), (b), (c) shows three such pictures of a dislocation in magnesium ($c/a = 1.2$). In the case of this hexagonal material we work in terms of projections of plane normals but do not index them as directions. The procedure is as follows.

(1) Index the diffraction patterns and mark traces of a suitable strongly reflecting plane on each micrograph.

(2) Measure the angle θ between the plane chosen and the normal to the projected line direction of the dislocation for all three cases.

(3) Plot these normals on the stereogram using the exact beam direction B for the micrographs and the angles θ .

(4) Draw a great circle through the normals. The pole of this great circle is the line direction u of the dislocation in the crystal. The accuracy of this determination of u depends on (a) the accuracy with which B is indexed, note if B is close to a zone axis the specimen is normally tilted $\sim 5^\circ$ away from the zone axis to obtain two-beam conditions, and (b) the amount of tilt from the zero tilt position which can produce errors of up to 5° in the direction of g due to the tilting of the spike parallel to the foil normal in reciprocal space.

Provided B is indexed to $< 1^\circ$ and g directions are transferred when s is very small or at small angles of tilt from zero, u may be determined to $\pm 5^\circ$.

Exact beam directions B for figure 2.56(a), (b), (c) have been constructed on the stereogram in figure 2.56(d) close to beam directions $[\bar{1}213]$, $[0001]$, $[\bar{1}\bar{1}23]$, respectively. Directions A, B, C normal to the directions of the dislocation have been constructed by measuring the angles from the directions marked in figure 2.56(a), (b), (c) along great circles corresponding to the three values of B . The line direction u is then shown to be 4° from $[01\bar{1}0]$.

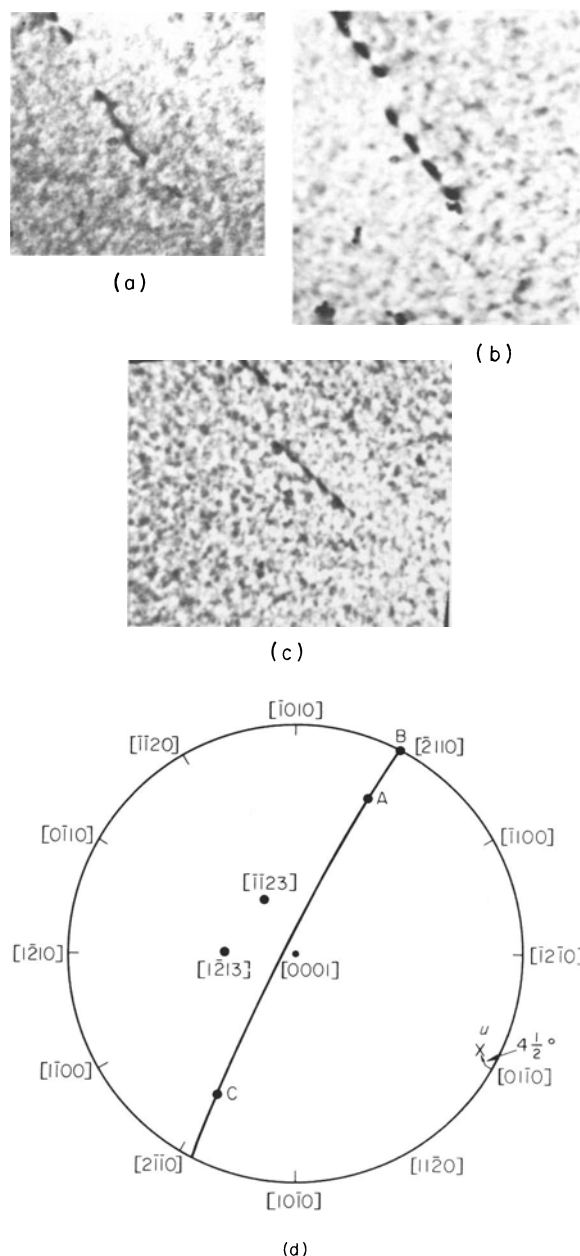


Figure 2.56 Line direction determination for a dislocation in magnesium. (a)–(c) BF images with different B , (d) stereographic analysis

PART IV. USES OF DIFFRACTION PATTERNS FROM SINGLE CRYSTALS: THE FINE STRUCTURE IN THE PATTERN

2.15 General Introduction

The fine structure such as streaks or satellite spots present in many diffraction patterns contains a great deal of information about the defect structure of thin foils. However, some caution must be used in interpreting these effects because a number of factors can contribute to the detail of the diffraction pattern, for example precipitate shape or coherency strains, composition fluctuations, the

presence of interfaces, etc. Different factors can produce similar effects and, in complicated microstructures such as occur in commercial materials, several types of crystal defect may be present within the selected area aperture and therefore contribute to the resulting complex diffraction pattern. Consequently it is essential to correlate the individual features of the diffraction pattern with image detail making extensive use of centred dark field images (section 1.5.2) formed with these

features. Thus a quantitative picture may be obtained of the defect density/morphology and identity in the thin foil in relation to the detailed structure in the SADP.

Four features of fine structure may be readily recognised.

- (1) Extra spots.
- (2) Spot splitting and satellite spots.
- (3) Streaks.
- (4) Walls of intensity.

These will now be considered in detail.

2.16 Extra Spots

The most common sources of extra spots in diffraction patterns are second-phase particles, twins, martensite plates, etc. These spots may be used to check the crystal structure and lattice parameter of the phase and its orientation relationship with the matrix using the techniques outlined earlier in sections 2.11, 2.12, 2.13. However, extra spots may also arise from double diffraction and atomic ordering processes which are discussed below.

2.16.1 Double Diffraction

This is one of the commonest sources of extra spots, particularly in two-phase alloys, and the process involved may be understood with reference to the idealised case shown in figure 2.57. Here a beam of electrons is incident on a sandwich specimen consisting of two layers, crystals 1 and 2. The Bragg angle is exactly satisfied in the upper crystal 1 for a set of closely spaced planes (spacing X_1). However, the geometry is such that the diffracted beam is incident on crystal 2 at the Bragg angle for a set of widely spaced planes spacing X_2 . Thus it is re-diffracted back in a direction almost parallel to the transmitted beam. As a result three spots appear in the diffraction pattern.

- (1) A transmitted spot T.
- (2) A diffracted spot D_1 from crystal 1 and distance $\propto 1/X_1$ from the centre spot.
- (3) A diffracted spot D_2 (from the incident diffracted beam D_1 on crystal 2) distant $\propto 1/X_2$ from spot D_1 .

In practice in a thin foil the relaxation of the third Laue condition means that the incident beam will probably also excite reflection D_3 distance $\propto 1/X_2$ from the transmitted beam if $X_2 \approx X_1$ and four spots will actually occur.

2.16.1.1 The distribution of spots during double diffraction

The distribution of spots arising from double diffraction may be determined using the two

fundamental units of the diffraction pattern, that is the diffraction patterns from each crystal for the specific B involved. These are shown schematically in figure 2.57(b), (c) for $B \langle 100 \rangle$ in a cubic material. The procedure is as follows.

(1) Superimpose the two patterns with the centre spots together to produce the basic patterns expected from both crystals without double diffraction, figure 2.57(d).

(2) Each diffracted beam in crystal 1 is an incident beam for crystal 2; consequently determine the location of the spots arising from double diffraction by superimposing the characteristic diffraction

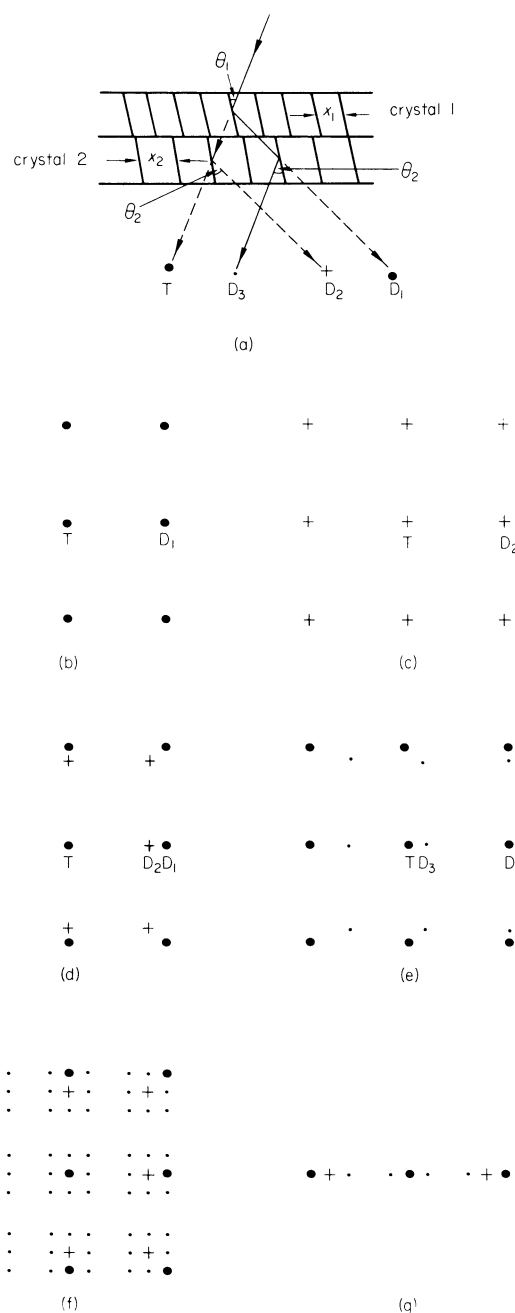
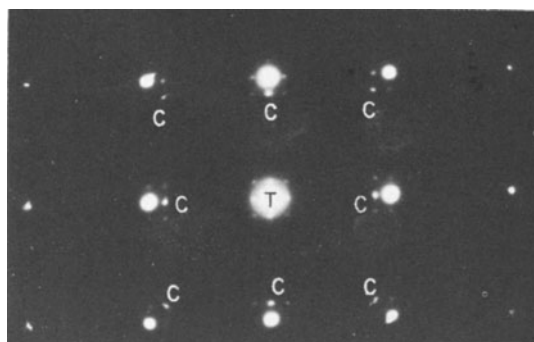


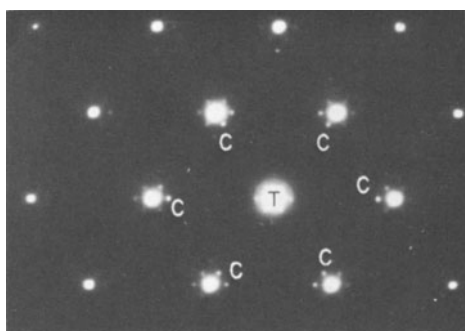
Figure 2.57 A schematic illustration of double diffraction

pattern from crystal 2 on that of crystal 1, but with the origin of the former displaced to the strongly diffracted spot D_1 in crystal 1. This situation is shown in figure 2.57(e).

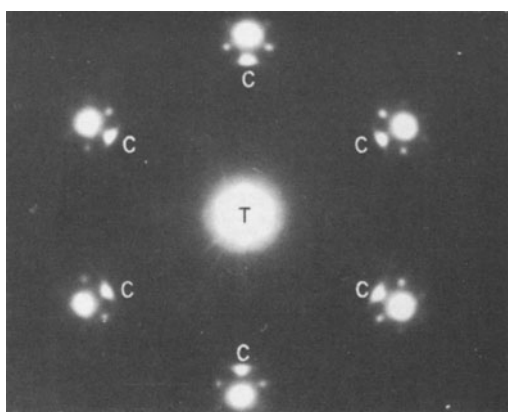
(3) Repeat this process for all strongly diffracted beams in crystal 1 to obtain the result in figure 2.57(f). Examples for $B = [100]$, $[110]$ and $[111]$ are shown in figure 2.58. In all cases the matrix is



(a)



(b)



(c)

Figure 2.58 Examples of double-diffraction effects in SADP taken from quenched and aged Ni-4.47 at% Ti-1.96 at% C alloy: (a) $B[100]$, (b) $B[110]$, (c) $B[111]$. The spots marked T are the transmitted spots, those marked C are the TiC spots, the brightest spots are matrix and the remainder are double diffraction [Courtesy of R. B. Scarlin]

Ni with $a = 3.517 \text{ \AA}$ and TiC precipitate (NaCl structure $a = 4.329 \text{ \AA}$) with a cube-cube orientation relationship.

Note that in perfect crystals double diffraction does not lead to the formation of additional reflections because the combination of any two reflections always leads to an existing third reflection, for example b.c.c. $(111) + (200) = (311)$. This is true for f.c.c. and b.c.c. crystal structures because both may be referred to a primitive base where the combination of two or more lattice points leads to a fundamental translation vector or a multiple thereof. However, in both the diamond and close-packed hexagonal (c.p.h.) crystal lattices the above rule is not true. Thus for these materials double diffraction does lead to the occurrence of spots not predicted by the structure factor. Table 2.13 shows the additional reflections arising from double diffraction for specific B in the c.p.h. case. Compare with the standard diffraction patterns in appendix 4.

The spots in table 2.13 may be obtained by adding vectorially existing reflections in the particular reciprocal lattice plane, for example $(10\bar{1}0) + (\bar{1}011) = (0001)$. Clearly, for $B [10\bar{1}0]$, $[0001]$, $[12\bar{1}1]$ extra reflections cannot arise from double diffraction, see figure A4.6, appendix 4. Images formed with 'forbidden' reflections in hexagonal and diamond crystal structures have no special features and may be used for defect analysis procedures outlined in section 3.

2.16.1.2 Detection of double diffraction in the electron microscope

It is frequently necessary to determine whether particular diffraction spots arise from double diffraction. Since the double diffraction process arises from the symmetry of the diffraction pattern, it can be useful to tilt the specimen $\sim 10^\circ$ about the axis containing a bright spot to produce two-beam conditions, that is only one strongly diffracted beam. If the spot of interest arises from double diffraction it may disappear or if part of the intensity arises from this process it should become less intense. Note, however, that, if a line of systematic reflections (hkl) , $(2h 2k 2l)$, $(3h 3k 3l)$, etc., is excited, double diffraction may still occur,

Table 2.13 Extra reflections in c.p.h. crystal structures arising from double diffraction for different B

Reflection	Beam direction B
$\{0001\}$	$[2\bar{1}10]$
$\{0003\}$	
$\{2\bar{1}1\}$	$[\bar{1}2\bar{1}3] [\bar{1}2\bar{1}6] [5\bar{1}43]$
$\{11\bar{2}3\}$	$[01\bar{1}1] [\bar{1}2\bar{1}1]$
$\{3033\}$	$[01\bar{1}1] [01\bar{1}2] [5\bar{1}43] [7253]$
$\{1125\}$	$[\bar{7}253]$

see figure 2.57(g). Consequently considerable care must be taken if this approach is used.

It is also possible to detect spots arising from double diffraction using CDF images. If for example the precipitate particle as thick as the foil, the double diffraction process will be confined to the interface which will be bright in a CDF image using a double diffraction spot. Figure 2.59(a) shows a CDF image obtained with the double diffracted spot in figure 2.59(b). The foil contains a large twin T and only the twin-matrix interface is visible in the CDF image, see Pashley and Stowell (1963). In contrast, if a particle is completely contained within the foil there will be no obvious difference between dark field images with double diffraction and precipitate reflections—the precipitate particle will be bright in both cases.

2.16.2 Long-range Order

It was pointed out in section 2.3.1 that superlattice reflections may arise from long-range ordering and that the intensity of these reflections is related to the difference in atomic scattering factors of the ordering elements. Typical diffraction patterns for simple cases are shown in appendix 4.

2.16.2.1 The distribution of spots in the electron diffraction pattern

The position of the extra spots in the diffraction pattern depends on the lattice parameter of the new unit cell created by the ordering process.

2.16.2.2 The detection of long-range order in the electron microscope

Frequently it is possible to detect long-range order more easily in the electron microscope than using x-ray techniques. A number of workers have reported detailed studies of long-range order. For example, the VC phase field has been studied by Venables *et al.* (1968), Hannink *et al.* (1971) and Lewis *et al.* (1972). It has been shown that two compounds V_8C_7 (cubic) and V_6C_5 (trigonal) both form characteristic electron diffraction patterns. A similar approach has been used to characterise the structure of Nb and Ta containing ordered interstitials (van Landuyt, 1964; van Landuyt *et al.*, 1966a; Villagrana and Thomas, 1965).

The fact that the spots arise from ordering may be checked by CDF using superlattice reflections which should show up the domains. Alternatively, the antiphase domain boundaries will be visible if the domain size is very large, see figure 3.33. However, if the domain size is the same as the grain size, dark field images of superlattice reflections should show no specific image characteristics.

This situation has been reported for Ti_3Sn by Blackburn and Williams (1967) and Jones and Edington (1972).

2.16.2.3 Quantitative information available

The unit cell of the ordered phase may be specified by obtaining self-consistent indexing with different B . An example for ordered V_6C_5 is shown in section 4.14.1. However, in general, intensity measurements cannot be made in the electron diffraction pattern. Consequently the atomic positions within the unit cell cannot be specified. To do so requires correlating the results with those of other techniques such as nuclear magnetic resonance or x-rays.

2.17 Spot Splitting and Satellite Spots

Spot splitting or the occurrence of satellite spots can occur because of the presence of planar defects such as stacking faults, line defects such as dislocations, spinodal decomposition or regular arrays of crystal defects.

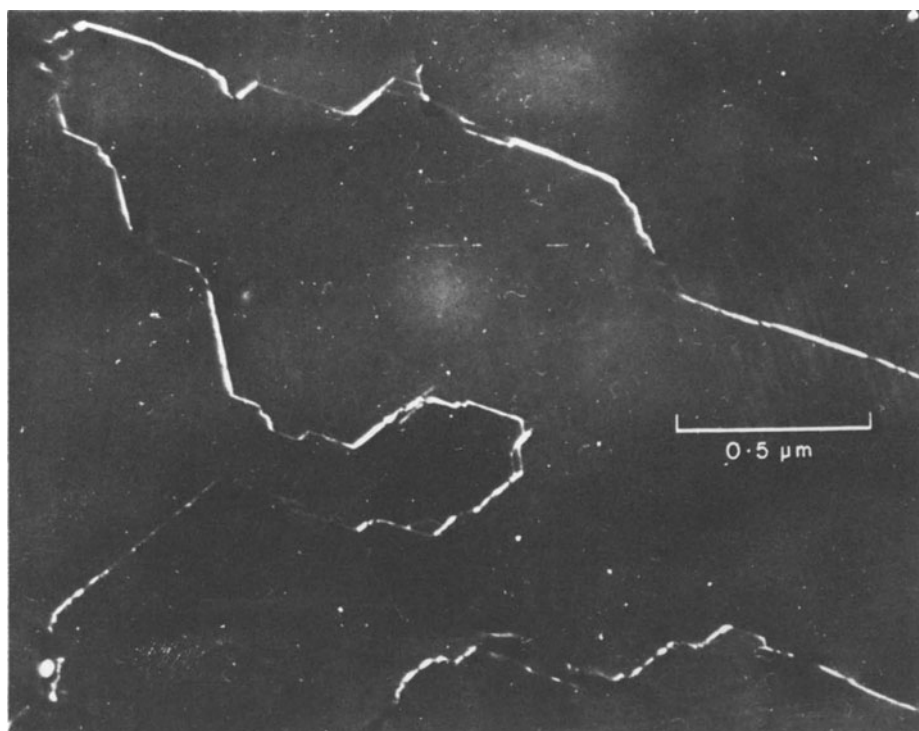
2.17.1 Planar Crystal Defects

In a series of papers, Gevers, Amelinckx and their coworkers have made detailed theoretical and experimental studies of satellites near both the transmitted and diffracted spots arising from single planar defects inclined at an angle ϕ to the foil surface for zero foil tilt. The individual cases considered included stacking faults (Gevers *et al.*, 1966, 1967a, b, 1969), domain boundaries (Gevers *et al.*, 1968; de Ridder *et al.*, 1970a, b, c), grain and twin boundaries (de Ridder *et al.*, 1968) and wedge-shaped crystals (de Ridder *et al.*, 1968, 1970d; Serneels *et al.*, 1971). A number of satellites are predicted theoretically and observed experimentally in carefully focussed and exposed diffraction patterns. It has been shown that the number, position and intensity of the satellites depends on both the defect involved and the diffracting conditions employed, and anomalous absorption* for the material involved. The results given here are based on the simple two-beam theory which is adequate to obtain some quantitative information about the specimen.

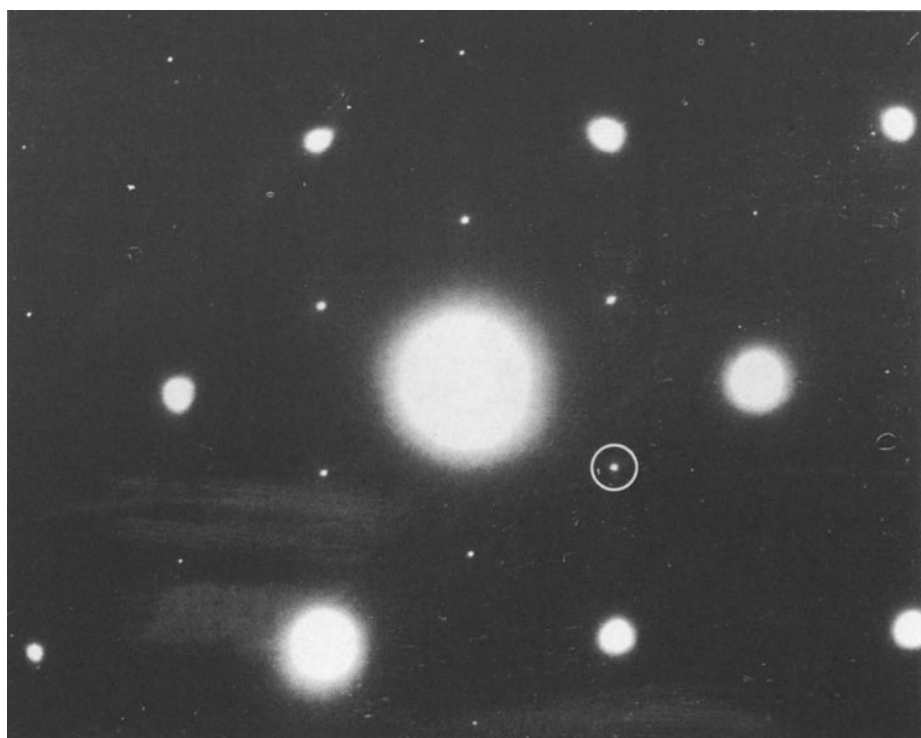
2.17.1.1 The general form of satellites

The main features of the satellites associated with the diffracted spot may be understood with reference to figure 2.60 which contains both schematic diagrams of each defect configuration in the thin foil, together with the schematic intensity distribution around the reciprocal lattice point. In each case rods of intensity occur around the reciprocal lattice point, the details of which

* Anomalous absorption is described in section 3.2.2.



(a)



(b)

Figure 2.59 (a) A CDF image from an evaporated gold film using the double diffracted spot circled in the SADP, (b). Note that only the twin boundary is visible, that is the site of the double diffraction [From Pashley and Stowell (1963), courtesy of *The Philosophical Magazine*]

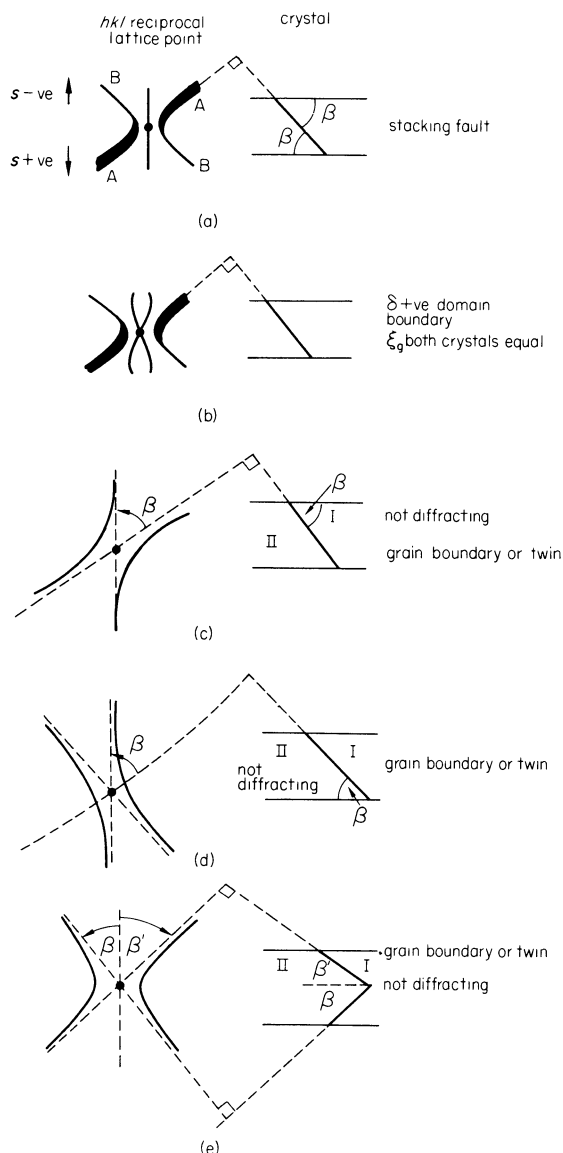


Figure 2.60 The distribution of satellites at an (hkl) reciprocal lattice point for various types of boundary. The thicker lines show the more intense streaks

depend upon the particular case under consideration. As a result satellites occur in SADP at positions which depend on the sign and magnitude of s . The most useful conclusions of this work are summarised in table 2.14.

2.17.1.2 The detection of satellites

In practice satellites are visible over a wide range of values of s if exact focussing and long exposure (1–2 minutes condenser 2 almost fully over-focussed) are used. It is very important that the diffraction pattern must be *exactly* focussed and that condenser astigmatism should be corrected or the diffraction spots may be seriously distorted.

Note that the maximum satellite intensity for stacking faults is predicted for values of $s\xi_g^* = 0.5$ which is a useful experimental condition to aim for when trying to detect satellites. It is also important to realise that the visibility of the satellites for δ boundaries depends on the difference in axial ratios of the two domains. The maximum spacing $\Delta\mu$ of the two branches is $\Delta g \sin \phi$. Consequently, for very small axial ratios (BaTiO₃) only one spot is visible because Δg will be too small. However, for Ni₄Mo, c/a 0.986, these can be separated. Finally, very thin foils $\lesssim 2\xi_g$ should be avoided because complicating effects may occur.

2.17.2 Periodic/modulated Structures

Periodic variations in interplanar spacings, structure factor, or a well-defined periodic distribution of crystal defects such as antiphase domain boundaries or dislocations can lead to the occurrence of satellites in the electron diffraction pattern.

2.17.2.1 Spinodal decomposition

Decomposition of supersaturated solid solutions within the spinodal region of the equilibrium diagram produces composition fluctuations in the elastically soft directions in the crystal,[†] see Cahn (1968) for a review. Satellite reflections are produced in the direction of the composition fluctuation with contributions from both the lattice parameter variation (Daniel and Lipson, 1943a, b; Guinier, 1959; Hirsch *et al.*, 1967) and the scattering factor variation (Hargreaves, 1951).

The important characteristics of the electron diffraction pattern may be understood with reference to figure 2.61. Here a composition variation is shown schematically to occur along the $[100]$ direction normal to the (100) plane. The reciprocal lattice contains extra points symmetrically disposed about the (hkl) reflection as shown in figure 2.61(b) such that, if the Ewald sphere is in the position shown, that is $s \approx 0$, figure 2.61(c), two satellite spots occur on either side of the main (hkl) spot as shown in figure 2.61(d). Figure 2.61(b) is in fact a complete description of the distribution of satellites in reciprocal space for Cu–Ni–Fe alloys with composition modulation along $\langle 100 \rangle$. Satellites occur symmetrically located in $\langle 100 \rangle$ about each lattice point except the origin.

In general electron diffraction patterns have the following characteristics.

- (1) The spacing of the satellites is dependent only on the wavelength λ of the modulation, not on the order of reflection.

* $w = s\xi_g$.

[†] In cubic crystal structures the direction of modulation will be $\langle 100 \rangle$ or $\langle 111 \rangle$ depending upon whether the factor $2C_{44} - C_{11} + C_{12}$ is positive or negative respectively. Commonly the direction of modulation is $\langle 100 \rangle$.

Table 2.14

Defect	Major features of reciprocal lattice point indicated by simple theory	Quantitative information available (close to zero specimen tilt to minimise errors)
stacking fault (α), or antiphase domain boundary (APB, π)	rods of intensity occur (figure 2.60(a)) which at $s \neq 0$ are perpendicular to the defect plane (Gevers <i>et al.</i> , 1966; van Landuyt <i>et al.</i> , 1966a). The intensity of the rods is greater at A than B. At $s \neq 0$ the line joining the satellite to the diffracted spot is perpendicular to the line of intersection of the stacking fault plane and the foil surface (van Landuyt <i>et al.</i> , 1966b)	the sense of inclination of the stacking fault or APB is obtainable if the diffraction pattern and positive print are superimposed allowing for image rotation such that the diffracted spot is at the centre of the stacking fault. Then the line joining the main spot to the satellite points towards the bottom of the fault. This effect has been used to determine the extrinsic/intrinsic nature of stacking faults by van Landuyt <i>et al.</i> (1966b), see section 3.6.2.3 for an example. The angle of inclination of the fault (APB) may be measured to within $\sim \pm 5^\circ$ using the distance μ between the main spot and the strong satellite. Using the formula $\mu = \sigma \tan \beta$ where $\sigma = \frac{1}{\xi_g} (1 + w^2)^{1/2} \quad \text{and} \quad w = s \xi_g$
δ boundary defined in section 3.8	rods of intensity occur (figure 2.60(b)) (Gevers <i>et al.</i> , 1968; de Ridder <i>et al.</i> , 1970a, b, c) which have the same general features as the stacking fault case, provided the extinction distances in both domains are the same (which they usually are). The split centre-rod cannot be detected in experimental diffraction patterns	the sign of s may be determined using the rule: $s > 0$ the most intense inner satellite 2, figure 2.60(b), lies closest to the main spot, which is on the same side of the halfway point between the two main spots as the bright outer fringe of the image. The reverse is true for $s < 0$. This rule applies for any value and sign of Δs and δ . The angle of inclination may be measured approximately using the relation $\mu = s \tan \beta$ for maximum s if the extinction distances in both domains are the same, $1/t^2 \ll \delta^2$, and the other satellites are used. Note that this method avoids the use of extinction distances which may be difficult to compute, accuracy $\sim \pm 5^\circ$
high-angle grain-twin boundaries	the reciprocal lattice point is split into two rods as shown in figure 2.60(c), (d), the sense depending upon which part of the crystal is diffracting (de Ridder <i>et al.</i> , 1968). At $s = 0$ one rod is asymptotic to the foil normal and the other to the defect plane normal. Thus at $s \neq 0$ the line joining the satellite to the main spot is perpendicular to the line of intersection of the boundary plane and the foil surface	the angle β between the boundary plane and the foil plane may be obtained under kinematical conditions (large s) from $\mu = s \tan \beta$, independent of which half of the crystal is diffracting, accuracy $\pm 1^\circ$
wedge-shaped crystals	the reciprocal lattice point has two associated rods asymptotic to the normals to the wedge surface, figure 4.60(e) (de Ridder <i>et al.</i> , 1968). Two satellites are predicted at $s = 0$. The line joining them is normal to the edge of the specimen	the angle of the wedge may be obtained from the formulae $\mu = s (\tan \beta + \tan \beta')$ at large values of s

(2) There should not be satellites on the transmitted beam but they have been observed (Woodilla and Averbach, 1968) possibly because of double diffraction.

(3) The direction of splitting of the satellites in a single-crystal electron diffraction pattern from a material with a cubic crystal structure will be $\langle 100 \rangle$ or $\langle 111 \rangle$ depending upon the positive or negative value of $2C_{44} - C_{11} + C_{12}$.

For cubic crystal structures the wavelength may be calculated from the expression of Daniel and Lipson (1943b) modified for electron diffraction

$$\lambda = \frac{ah}{h^2 + k^2 + l^2} \frac{g}{\Delta g} \quad (2.33)$$

where $g/\Delta g$ is the ratio of the distances from the

zero-order reflection and satellite to the hkl spot, see figure 2.61(d). In measuring $g/\Delta g$ from electron diffraction patterns it will be found that the ability to resolve the satellites is influenced by the beam direction. Furthermore, non-exact beam directions, for example (200) and $(\bar{2}00)$ fundamental reflections with different intensities, that is small differences in s can introduce asymmetry of intensity of the satellites. Consequently, for $\langle 100 \rangle$ modulation foils should be tilted to produce B exactly $\langle 100 \rangle$ or $\langle 110 \rangle$ with satellites of equal intensity along $\langle 100 \rangle$ on opposite sides of the transmitted beam. Under these circumstances the maximum deviation of the measurement from the mean is $\pm 5\%$. A typical electron diffraction pattern and example of the determination of λ is shown in figures 4.25, 4.26. Note that considerable overfocussing of condenser

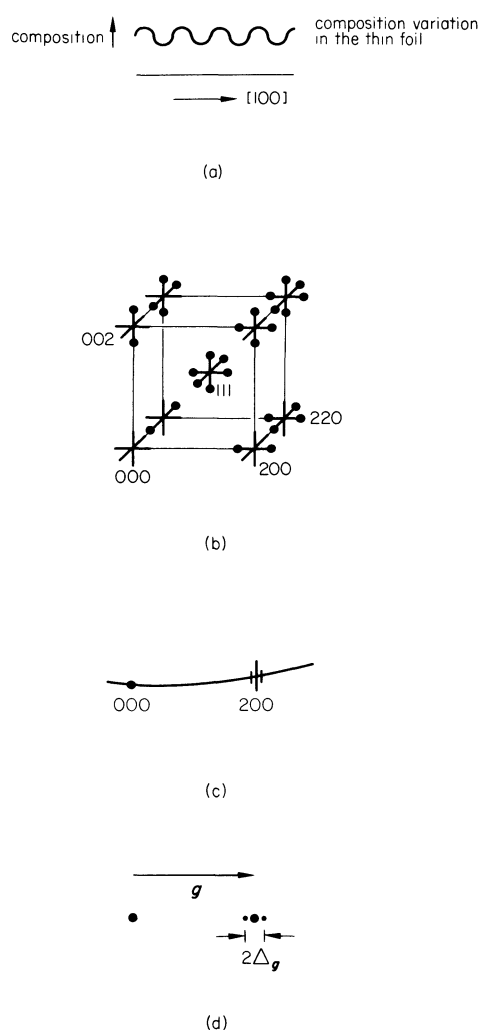


Figure 2.61 A schematic illustration of satellite spots formed if thin foils have composition modulations: (a) modulation, (b) position of satellites in reciprocal space, (c) Ewald sphere construction and (d) position of satellites in diffraction pattern

2, accurate focussing of the pattern, and exposures of ~ 1 min are necessary to obtain good reproducible results.

It is possible to estimate the maximum composition difference in the modulation direction by measurement of intensities in the electron diffraction pattern. However, the results are only approximate because of complications from inelastic scattering and the non-linear response of the photographic emulsion to the electrons (Woodilla and Averbach, 1968).

Satellite reflections in electron diffraction patterns have been reported in the following alloys, Cu–Ni–Fe (Butler and Thomas, 1970; Butler, 1971), Al–Ni–Co (Granovsky *et al.*, 1968), Ticonal X (Kuizhnik *et al.*, 1968), Co–Cr–Ta (Drapier and Coutsouradis, 1968), Cu–Ti (Saito and Watanabe, 1967), Au–Ni (Woodilla and Averbach, 1968),

Co–Ti (Thompson and Edington, 1970). These have all been associated with spinodal decomposition.

2.17.2.2 Regular arrays of antiphase domain boundaries

A particularly well-defined case that has received considerable attention is that of the ordered compound AuCuII. The crystal structure of the material may be described in terms of a unit cell 10 f.c.c. cells in length with an APB every 5 unit cells along the a axis, see figure 2.62(a). If an electron diffraction pattern B [001] is obtained from a region of the crystal with the long axis of this unit cell parallel to [100] and [010] the diffraction pattern contains a large number of satellites, figure 2.62(b). The schematic diagram in figure 2.62(c) shows the satellites produced by the periodic array of APBs from just one orientation of the long cell. The primary satellites occur on the superlattice reflections $\{110\}$ type indexed on the original unit cell. Glossop and Pashley (1959) showed that double diffraction can account for the presence of the satellites on the transmitted and the fundamental spots, but Ogawa *et al.* (1958) showed that there was also a contribution to these spots from the periodicity of the structure which they ascribed to the small change in interplanar spacing occurring at the APB. This represents a good example of the complications arising from double diffraction. Similar satellite effects have been reported in Cu₃Pd (Watanabe and Ogawa, 1956), Au₃Mn (Watanabe, 1958) and AgMg₃ (Fujiwara *et al.*, 1958).

2.17.2.3 Regular dislocation array

It has recently been pointed out by Balluffi *et al.* (1972a, b) that regular dislocation arrays at thin-film bicrystal interfaces produce weak satellite reflections. Work in this area is still proceeding and the amount of quantitative information available from measurements on the satellites is not certain.

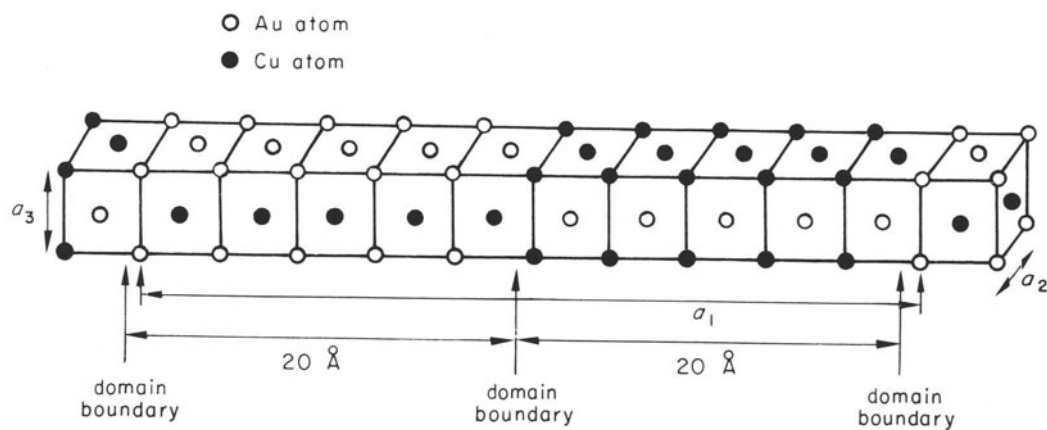
An example showing satellites arising from a simple dislocation array is shown in figure 2.63. The geometry of the situation is shown schematically in figure 2.63(a), in which the dislocation array is present at the interface between two overlapping single-crystal films. Satellites are present in the diffraction pattern, figure 2.63(c), (d), and lie in a row perpendicular to the dislocation lines. Note that, because of the likelihood of double diffraction, careful tilting of exact two-beam conditions with no additional reflections excited is essential to eliminate double diffraction and be certain of the origin of the satellites.

Final confirmation of the analysis of the origin

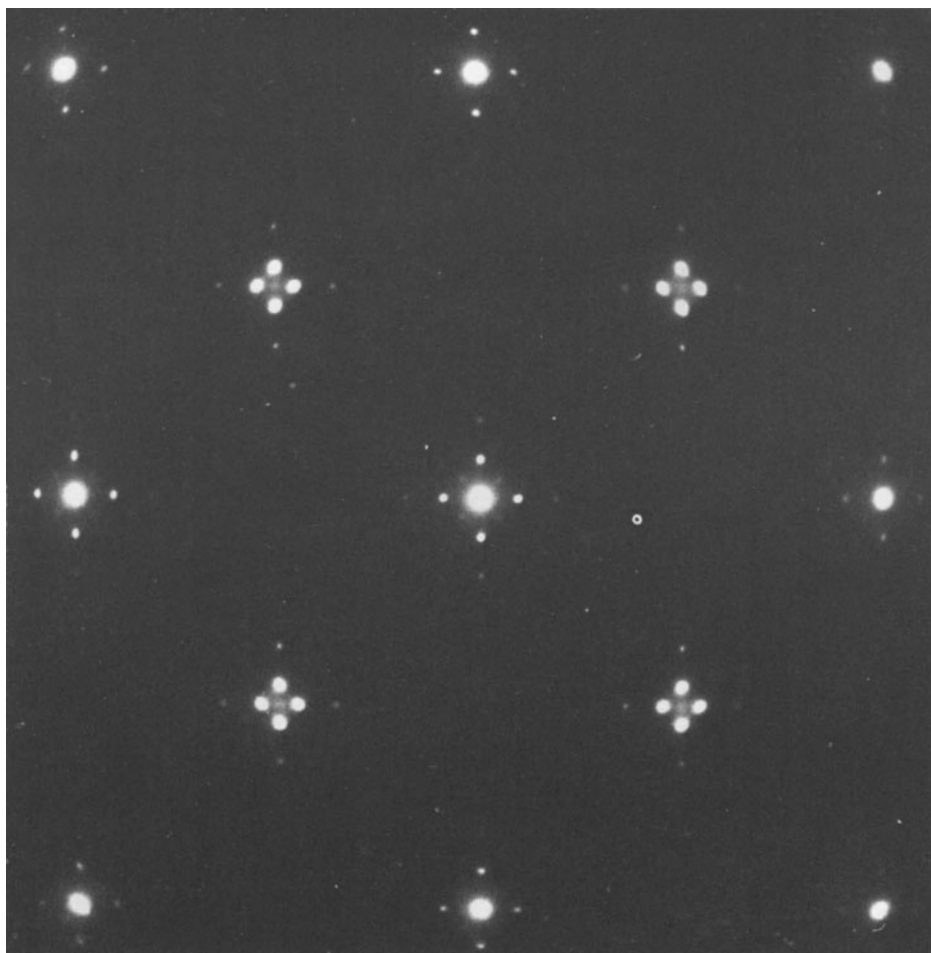
of the satellites may be obtained from electron diffraction patterns from regions of overlapping crystals that do not weld to form a dislocation grid. In this case for the non-welded region the diffraction pattern does not contain satellites.

2.17.2.4 Detection of satellites

The experimental conditions outlined in section 2.17.1.2 for the operation of the microscope should be followed. In addition careful control of the



(a)



(b)

Figure 2.62 (a) Unit cell of the periodic antiphase structure CuAuII, (b) SADP, (c) the position of satellites produced from one orientation of the unit cell. The film has (001) orientation [From Pashley and Presland (1959), courtesy of *The Philosophical Magazine*]

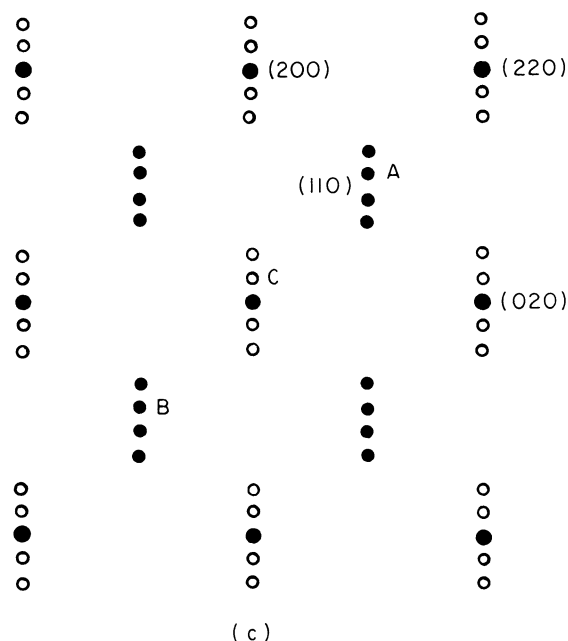


Figure 2.62 (continued)

number of operative reflections and the value of s is essential.

2.17.3 Magnetic Domains

If the electron microscope is operated in the normal way with the single-phase ferromagnetic specimen in the high-field region of the objective lens the specimen is fully magnetised. Under these conditions magnetic domains are not observed and there is no spot splitting in the diffraction pattern. However, when the specimen is not in the high-field region of the objective lens, all spots including the transmitted spot are split if the diffraction pattern is taken from an area containing two magnetic domains. This arises because the different magnetisation in the two domains reflects the whole electron diffraction pattern in different directions. For two domains in zero energy positions the spots split into two joined by a straight streak which is always perpendicular to the domain wall.

The amount of spot splitting may be used to calculate the thickness (t) of the specimen (Warrington *et al.*, 1962).

2.18 Streaks

Streaks can arise in diffraction patterns because of modifications to the shape of reciprocal lattice points arising from either the shape of crystal defects or the lattice strain associated with them.

2.18.1 Shape Effects

Streaks are already present in reciprocal space perpendicular to the foil surface and running through each reciprocal lattice point because of the relaxation of the third Laue condition produced by the shape of the thin foil, section 2.4. A similar effect is produced if the specimen contains a high density of crystallographically oriented defects such as precipitates. Thus the intensity distribution at a reciprocal lattice point is increased in directions perpendicular to the thin dimension of the particle. Consequently, the type of streak observed depends on both the crystal defect present and the diffracting conditions.

2.18.1.1 Precipitates

(a) *Origin of the streaks.* The intensity distribution at a reciprocal lattice point arising from specific precipitate shapes is shown in figure 2.64. Either rods, spheres or discs of intensity can arise. Using a simple kinematical approach the approximate dimensions of these streaks may be shown to be $<g$. The approximate angular distribution $d\phi$ of the streak is λ/t' , where t' is the width of the crystal normal to the beam. For a disc-shaped grain precipitate (GP) zone 10 Å thick $d\phi \approx 3.7 \times 10^{-3}$ radians (compare Bragg angle $\theta \approx 10^{-2}$ radians). Consequently the streak only runs a fraction of the distance between reciprocal lattice points. Figure 2.64(e) shows streaks arising from

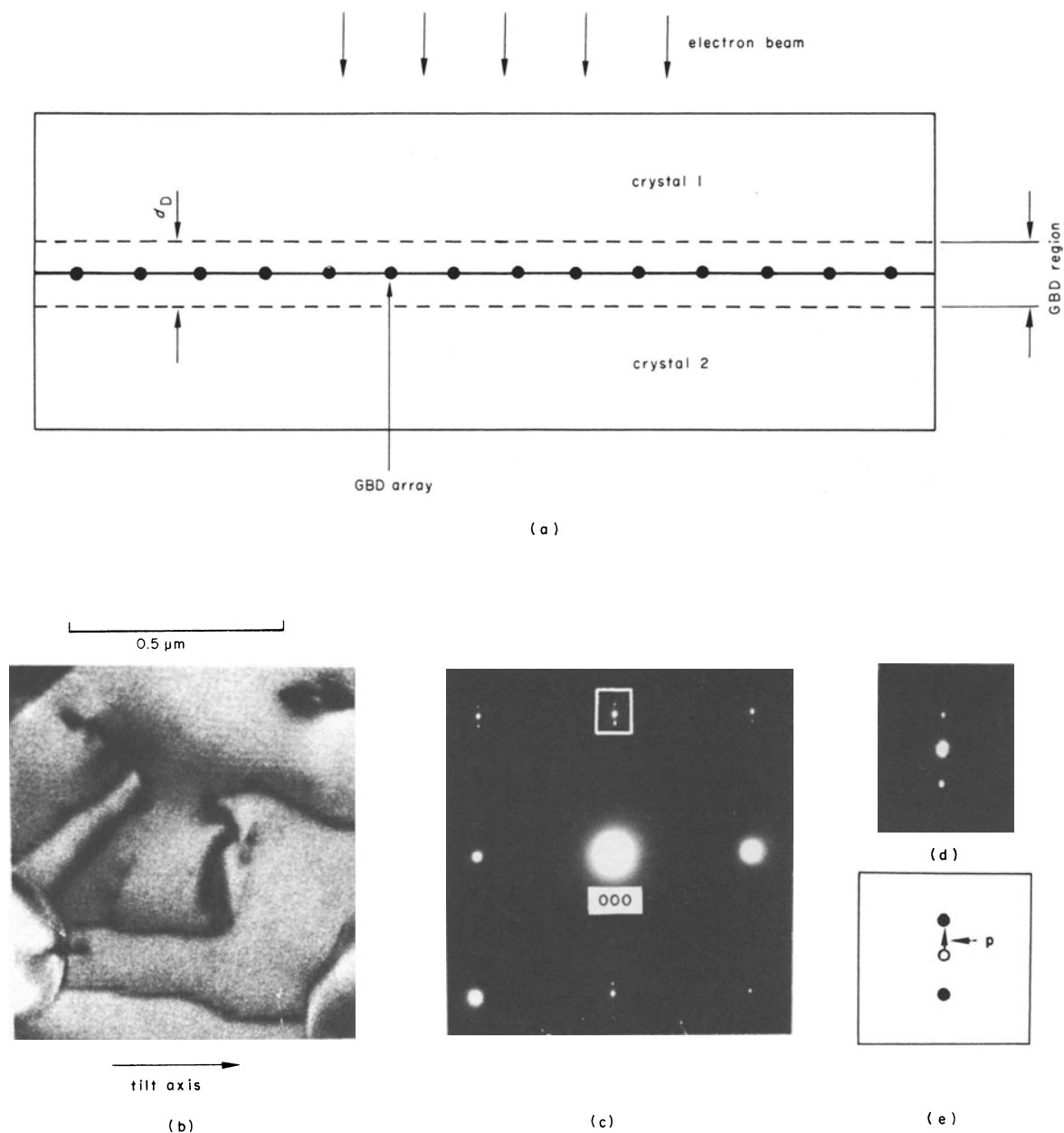


Figure 2.63 (a) Schematic drawing of bicrystal specimen showing grain boundary dislocation (GBD) region. (b) Pure symmetric low-angle [001] tilt boundary in gold observed at normal incidence. Grain boundary plane is $\sim(010)$. Tilt angle = 8.4° . Bright field image with several strong operating reflections. Finely spaced edge GBDs are parallel to [001] tilt axis. (c) SADP corresponding to (b). (d) Enlargement of the $(200)_{1,2}$ region enclosed in white in (c). (e) Schematic diagram of (d). Open circle corresponds to superimposed matrix reflections; filled circles correspond to the GBD diffraction spots [From Balluffi *et al.* (1972a), courtesy of *The Philosophical Magazine*]

GP zones on $\{100\}$ in a nitrided Fe–Mo alloy shown in figure 2.64(f). These run completely through each spot forming a cross-grid and are much larger than the kinematical theory would predict, possibly because of dynamical diffraction effects, the high density of precipitates, or misfit strain effects.

(b) *The distribution of streaks in the diffraction pattern.* Because the electron diffraction pattern is effectively a section through reciprocal space, the overall effect is to produce either streaks or satellites. The relationship between a single array

of plate precipitates normal to the foil surface, the shape of the reciprocal lattice points and the diffraction pattern is shown in figure 2.65(a).

If a number of habit planes were involved, reciprocal lattice points would be streaked in several directions as shown in figure 2.65(b), (c).

When $s = 0$ the precipitate streak in the plane of the Ewald sphere is visible in the diffraction pattern. However, when $s \neq 0$ the Ewald sphere cuts the precipitate streaks at an angle forming satellites.

For sheets of intensity in reciprocal space such

as those arising from rod precipitates, see figure 2.64(d), the streaks will be straight if the sheets are parallel to B . However, if the sheets of intensity in reciprocal space are not parallel to the electron beam a curved streak is formed, because of the curvature of the Ewald sphere. Figure 2.66 shows an example of curved streaks running through the origin of a SADP taken from an Al–Cu–Mg alloy. In this case laths of the S phase are present which create a wall of diffuse intensity in (001) plane of reciprocal space. However, B is not exactly parallel to $[001]$ and, as predicted, a curved streak is observed.

The shape of the precipitate particle during the early stages of ageing may be obtained from analysis of streaks in the SADP. However, considerable care must be taken because of the following.

- (1) Sections through discs of intensity in reciprocal space are visible as streaks.
- (2) More than one precipitate morphology may be present if the material is a complex commercial alloy, for example commercial Al–Zn–Mg, section 4.9.2.

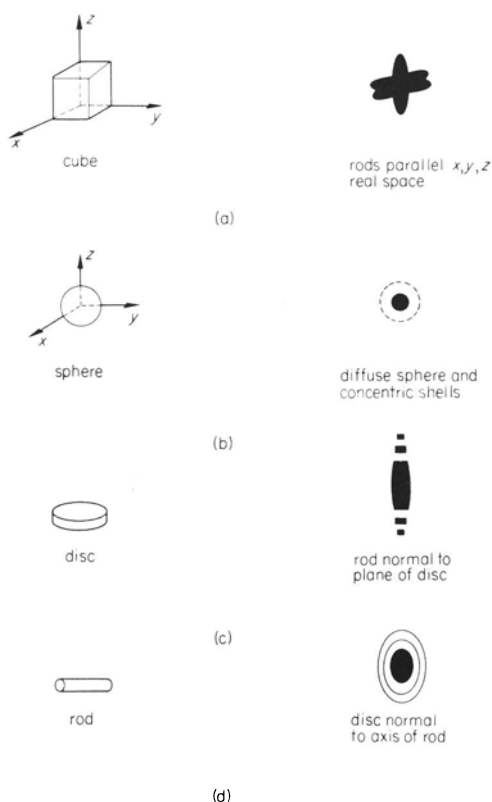
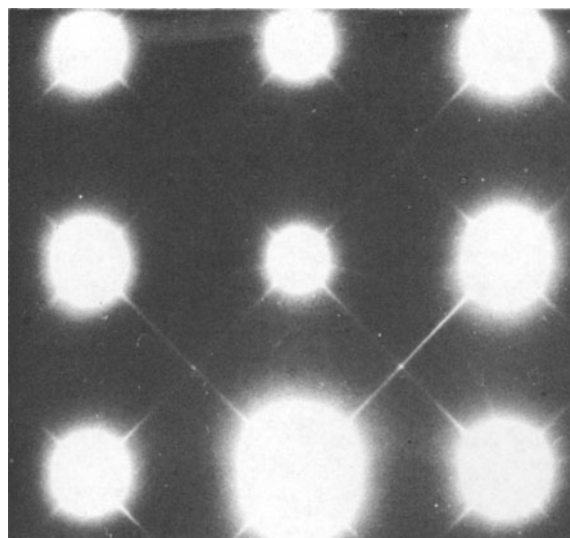
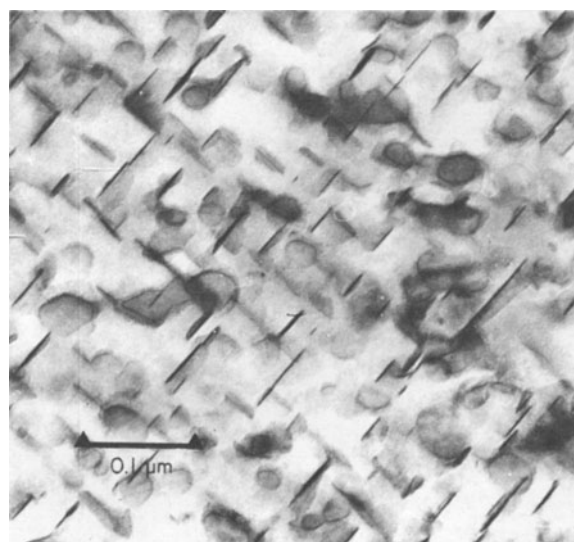


Figure 2.64 (a)–(d) The shapes of reciprocal lattice points for different shapes of precipitate, (e) SADP and (f) BF image from Fe–2.9 at% Mo, nitrided in 7:93 $\text{NH}_3:\text{H}_2$ at 580 °C for 10 hours to 2.5 at% N, and H_2 reduced at 580 °C for 80 hours [From Driver *et al.* (1972), courtesy of *The Philosophical Magazine*]



(e)



(f)

Figure 2.64 (continued)

- (3) Streaks from elastic strain may complicate the pattern (section 2.18.2).

If the situation is simple, that is only one precipitate morphology is present, several long-exposure SADPs must be obtained with different low-index B , preferably from the same area to determine the three-dimensional nature of the streaking in the reciprocal lattice. Then the difference between the different types of precipitate morphology may be detected.

If the precipitate is a plate, its habit plane may be ascertained because it will be perpendicular to the streaks. The precipitate dimensions cannot be obtained from the size of the streak for the reasons apparent from figure 2.64(e) and the discussion above.

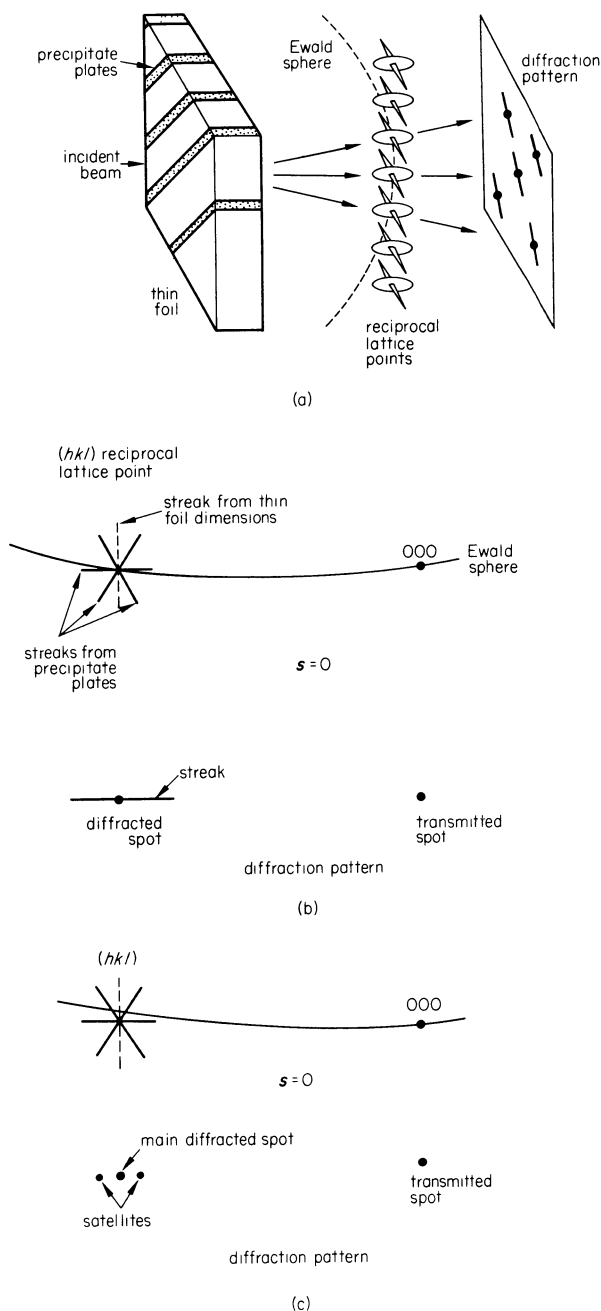


Figure 2.65 (a) Streking of reciprocal lattice points arising from precipitate plates lying parallel to the electron beam, (b) streaks visible $s = 0$, (c) pseudo-satellites $s \neq 0$

2.18.1.2 Stacking faults

As pointed out in section 2.17.1, the intensity distribution at reciprocal lattice points arising from stacking faults are approximately streaks normal to the habit plane. For high densities of faults these are easily visible in diffraction patterns and the habit planes may be obtained using the approach outlined above or using trace analysis, section 2.14.1, or the approach in section 2.17.1 to determine the angle between foil and fault plane.

2.18.1.3 Twins

The shape effect approximation leads to the conclusion that streaks normal to the K_1 plane, see figure 2.43(c), would be expected in the diffraction pattern and have been observed for twinned martensite by Kelly and Nutting (1960) and Tadaki and Shimizu (1970).

Using the approach outlined above for precipitates the habit plane (coherent twin plane) may be obtained because it is perpendicular to the streaks.

2.18.1.4 Dislocations

Willaime *et al.* (1966) have pointed out that dislocations influence the shape of diffracted spots. Effectively the spot acquires a disc-type halo perpendicular to the line direction of the dislocation. It was demonstrated experimentally for a high density of long parallel dislocations in the plane of the foil but was shown to be a very faint effect.

2.18.1.5 Surface films

Streking arising from surface films on thin foils has been reported for Au-Ni alloys by Woodilla and Averbach (1968). If the electropolishing process produces such a surface film care must be taken in interpreting the diffraction pattern.

2.18.2 Elastic Strain Effects

Guinier (1959) has shown that elastic distortion of the matrix arising from a coherent misfitting precipitate produces diffuse scattering in reciprocal space in the direction of the distortion. Often diffuse scattering arises simultaneously with shape effects (Al-Cu, Ni-Al alloys) and they are difficult to separate because they occur in the same direction.

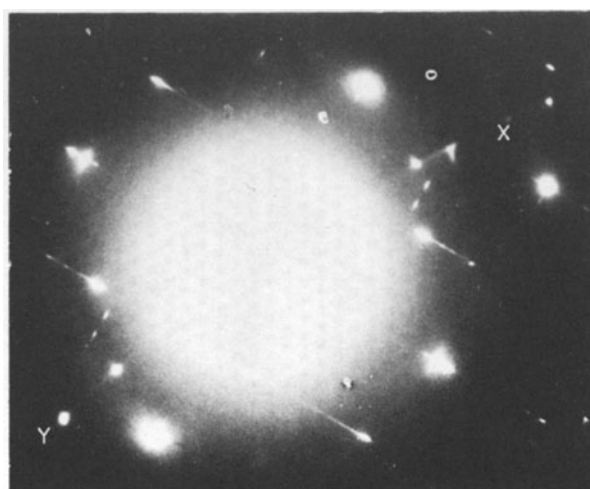


Figure 2.66 An example of a curved streak XY in a quenched Al-2.7 wt% Cu-1.36 wt% Mg alloy aged for 5 days at 190 °C [Courtesy of G. C. Weatherly]

However, Tanner (1966) showed that it was possible to do so for GP zones in Cu-2% Be alloys. In this case, GP zones are plates on $\{100\}$ matrix planes producing shape streaks in reciprocal space in $\langle 100 \rangle$. However, the coherency strains are shear strains in $\langle 110 \rangle^*$ (Tyapkin and Gavrilova, 1964) and produce streaks parallel to $\langle 110 \rangle$ in reciprocal space.

The main characteristics of elastic strain effects were described by Guinier and obtained for Cu-Be by Tanner (1966). These are as follows.

(1) The size of the streak increases with increasing order of g (this is difficult to observe because s increases with increasing order of g and the specimen must be tilted to $s = 0$ for each reflection).

(2) There are no streaks on the transmitted spot.

(3) There are no streaks present along the distortion directions that lie in the reflecting plane. Thus, for $\langle 110 \rangle$ strain fields, all $\langle 110 \rangle$ distortions will be represented within the selected area aperture. Each reciprocal lattice point will have some streaks missing, that is $\{111\}$ type will have three out of six missing $\{200\}$ type will have two out of six missing, and $\{220\}$ type one of six missing, see figure 2.67.

It is important to realise that the occurrence of double diffraction effects whereby a diffracted beam can act as an incident beam can complicate the above effects. For example, it can create streaks on the transmitted beam by diffracting back from a strongly diffracted beam. However, Tanner was able to overcome this factor by using very thin regions of the crystal.

In the case of Cu-Be, Tanner (1966) has related the form of the cross-hatching observed in the bright field image to the shear strain field. He showed (figure 2.68) that for $\{111\}$ reflections only parallel streak images were formed whereas for $\{200\}$ reflections a cross-hatching pattern was formed for B parallel to $\langle 110 \rangle$. Only some parts of the strain field are visible for individual reflections because of extinction as described above. Furthermore $\langle 110 \rangle$ project parallel into $\{110\}$ planes (see section 3.11.3 and Tanner (1966) for further details). However, this approach may not always be used as a guide particularly if the precipitates are large enough to contribute to the image by diffraction themselves.

Note that streaks in the plane of the reciprocal lattice perpendicular to B will only be observed at $s = 0$, that is exact B , see figure 2.65(b). If $s \neq 0$, satellite spots will be observed because the Ewald

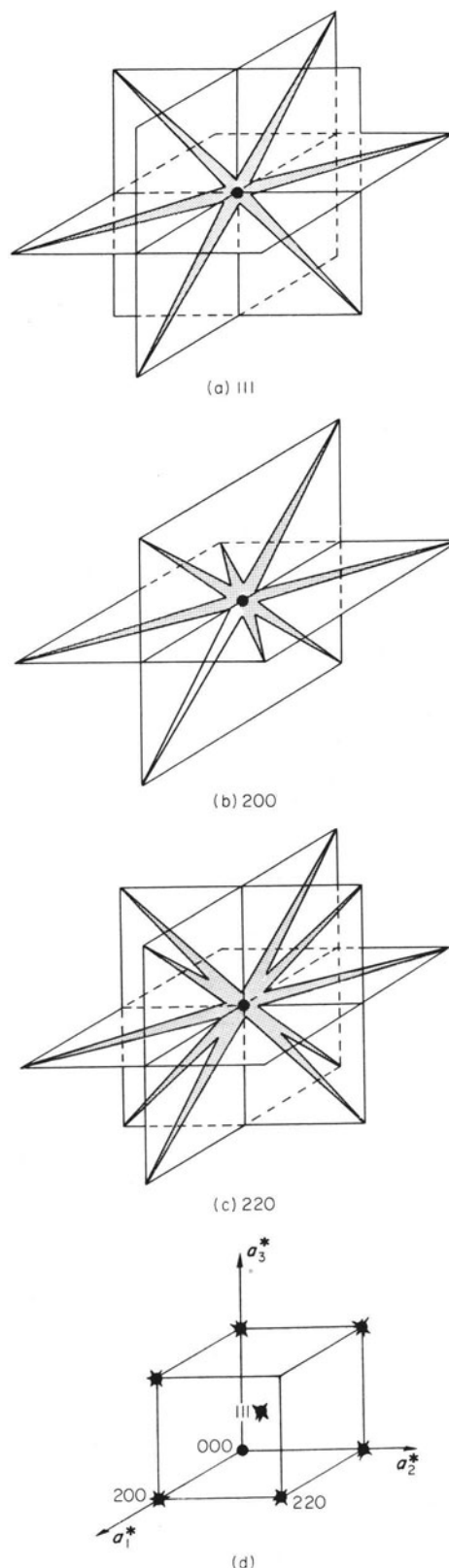
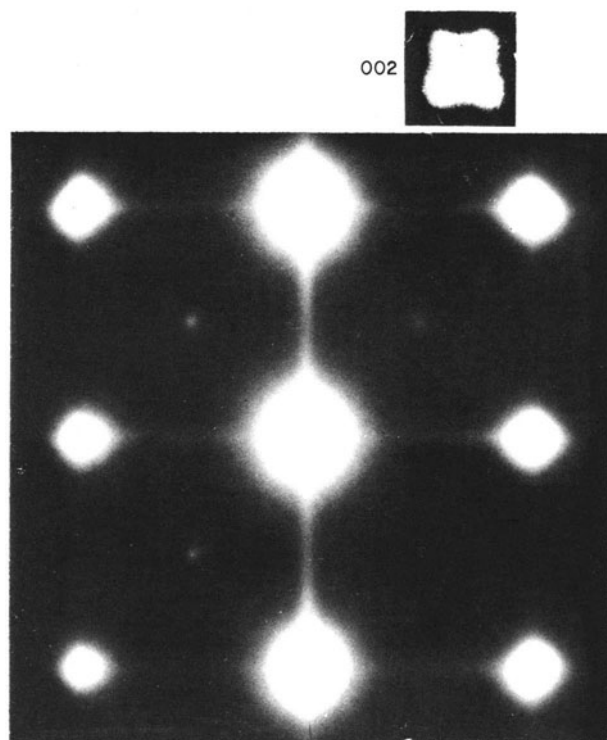
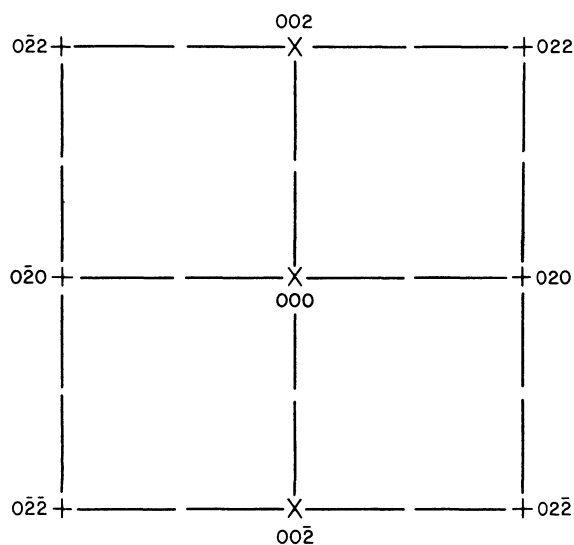


Figure 2.67 Schematic representation of streaks produced by strain field effects along $\langle 110 \rangle$ for GP zones on $\{100\}$ in Cu-Be alloys for three different reflections [From Tanner (1966), courtesy of *The Philosophical Magazine*]

* The reason for the occurrence of this type of shear coherency strain in Cu-Be but not in Al-Cu is not at present clear. Initially it was attributed to elastic anisotropy of the Cu-rich matrix (Tanner, 1966) but more recently this has been rejected in favour of complex strain field interactions from the very closely spaced particles (Tanner, 1968; Fillingham *et al.*, 1972; Phillips and Tanner, 1973).



(a)



(b)

Figure 2.68 (a) The SADP from Cu-2 wt % Be aged at 200 °C for 30 min. Only $\langle 100 \rangle$ streaks are visible on the print but the positions of the $\langle 110 \rangle$ streaks are shown in (b) which is a tracing from the original negative. Inset shows the $[0\bar{1}1]$ and $[011]$ streaks at the (002) reflection [From Tanner (1966), courtesy of *The Philosophical Magazine*]

sphere cuts both the streaks due to the thinness of the foil and the additional streaks running out of the plane in reciprocal space, figure 2.65(c).

2.18.3 Detection of Streaks

The experimental conditions outlined for the observation of satellites in section 2.17.1.2 should

be followed except that the condenser lens potentiometer is only defocussed ~ 3 coarse clicks for a 1 min exposure. B exactly parallel to low-index zone axes should be used. This is particularly important for the case of rods in reciprocal space because their diameter is very small and non-zero values of s (a) can cause some streaks in the plane of the diffraction pattern to disappear; and (b) can create additional satellite spots by sectioning streaks in reciprocal space that run in directions out of the plane of the diffraction pattern.

It is important to realise that there are a large number of precipitates inside the SADP aperture, so all habit planes will be represented. However, if two morphologies are present, for example commercial alloys, the interpretation of the diffraction pattern may be difficult.

For complete analysis the procedure is as follows.

- (1) Obtain long-exposure SADP from the same area with different known B to map the intensity distribution in reciprocal space.
- (2) Where possible obtain centred DF images using several precipitate reflections for all simple B to check the shape of the particles.

2.19 Diffuse Scattering Effects

A completely quantitative description of diffuse scattering is not available at present. Consequently, this section is confined largely to a summary of experimental observations.

2.19.1 Streaks in Reciprocal Space

Solid solutions with short-range order usually exhibit diffuse intensity maxima in electron diffraction patterns. Often these diffuse maxima at B , figure 2.69, occur near the position A , figure 2.69, that the superlattice reflections would have if the material were long-range ordered. Thus the occurrence of these intensity maxima may be explained in terms of broadening of superlattice reflections from the small domain size or in terms of the statistical models of Clapp and Moss (1968).

However, additional diffuse maxima occur at positions other than the long-range order positions in Ni_4Mo (Okamoto and Thomas, 1971a, b), Au_3Mn (Tanner *et al.*, 1968) and Au_3Cu (Tanner *et al.*, 1968). There is currently some discussion on the explanation of these effects in terms of the statistical model of Clapp and Moss (1971) and the microdomain model with non-conservative APB within the domains. However, it is important to be aware that, in studying such effects, accurate mapping out of the weak intensity distributions in reciprocal space requires the examination of a large number of different B and careful tilting close to these B . Furthermore, a large number of

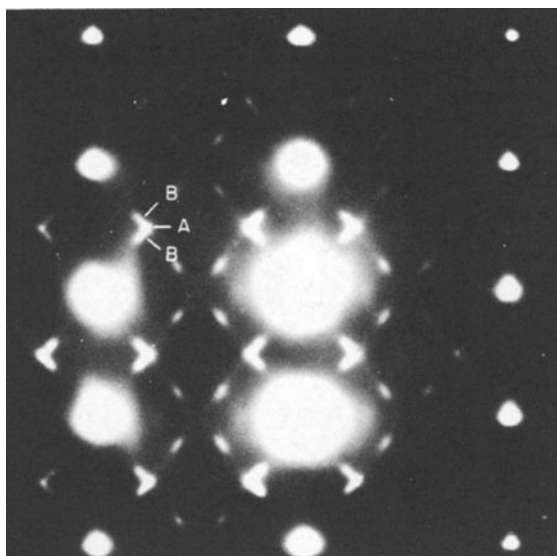
$t = 10$ minutes

Figure 2.69 Electron diffraction patterns of as-quenched Ni_4Mo aged at 650°C . $[121]$ orientation after 10 min ageing. The Ni_4Mo superlattice spots are broadened in the $\langle 2\bar{1}0 \rangle$ directions [From Okamoto and Thomas (1971a), courtesy of *Acta Metallurgica*]

photographs taken with condenser 2 almost completely overfocussed and 1–2 min exposures is necessary because these effects are often not

visible on the screen. Note that the interpretation of CDF images taken from these diffuse streaks can be uncertain if the domain size $\lesssim 10 \text{ \AA}$, see section 3.18.

2.19.2 Sheets in Reciprocal Space

There have been several reports of streaks of diffuse scattering in well-focussed long-exposure SADP (condenser 2 completely overfocussed 1–2 min exposure). These correspond to walls in reciprocal space and it is important not to confuse them with streaks arising from shape effects. They are normally very faint and diffuse.

Four types of wall have been described.

2.19.2.1 Thermal diffuse scattering

Walls of diffuse intensity have been reported by Honjo *et al.* (1964) in a number of fully annealed metals. These walls lie on $\{110\}$ for Al, Au, Si and Ge and $\{111\}$ for $\alpha\text{-Fe}$ and have been ascribed to directional thermal vibration. These walls are manifested in SADP as faint streaks, see figure 2.70, running in different crystallographic directions depending on B .

In most specimens this effect may be lost under superimposed particle shape effects, etc. However, if the fine detail of long-exposure electron diffraction

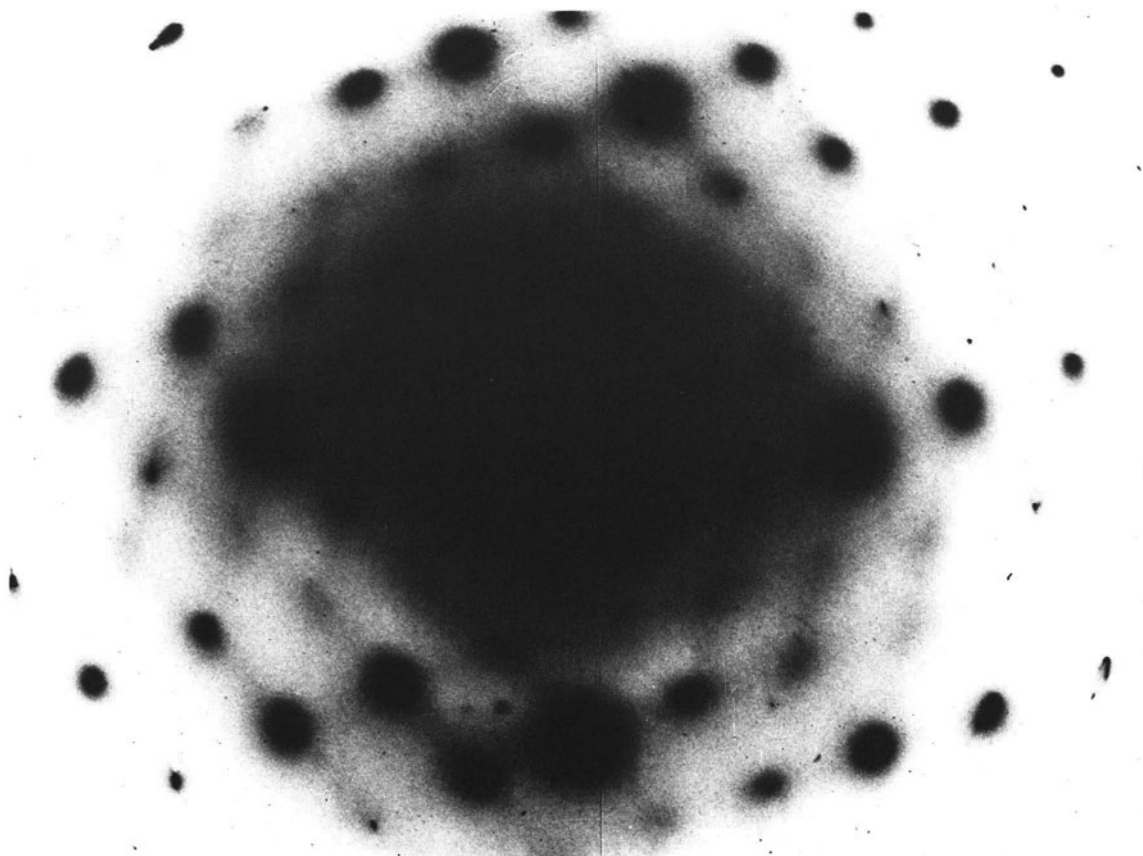


Figure 2.70 Thermal diffuse scattering effects in fully recrystallised aluminium $B \sim [110]$ [From Honjo *et al.* (1964), courtesy of *Journal of the Physical Society of Japan*]

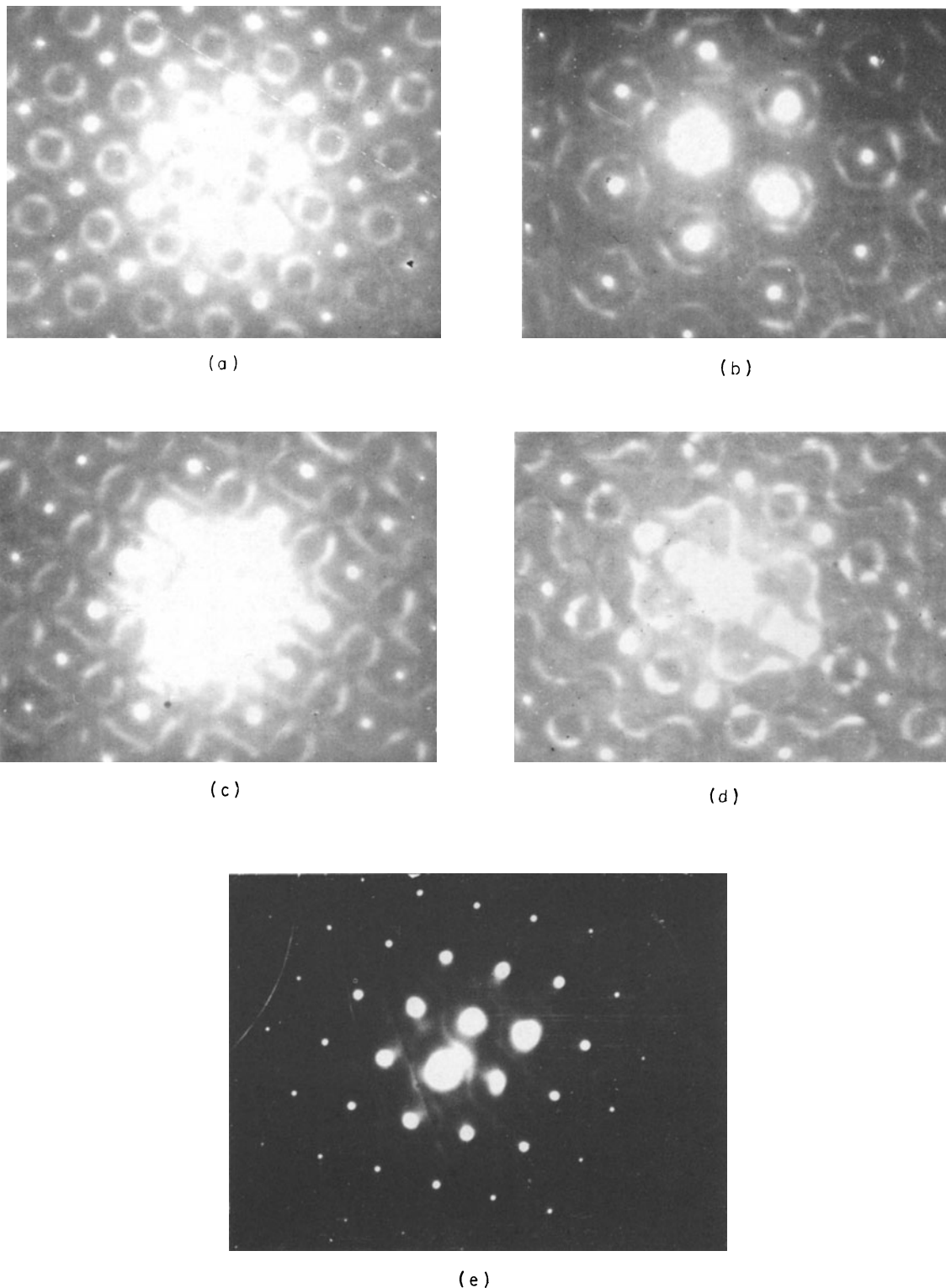


Figure 2.71 Diffuse scattering effects in short-range ordered vanadium carbide *B*: (a) [100], (b) [111], (c) [013], (d) [114], (e) [110]

patterns is under study it should not be ignored. In this context streaks from thermal diffuse scattering have two characteristics that may be used to identify them.

(1) Their intensity increases with increasing temperature.

(2) Since they arise from the Ewald sphere

cutting walls of intensity they are (a) much stronger for simple *B* where the walls may be cut almost perpendicularly, and (b) present over a range of directions that vary with *B* in a way in which may easily be determined by exposing a series of diffraction patterns obtained by tilting through a range of *B*.

2.19.2.2 β - ω phase mixtures

In certain Ti, Zr and Hf alloys the β b.c.c. phase is retained on quenching to room temperature, and a fine dispersion of a metastable phase ω is often formed in the β matrix either during the quench or on subsequent ageing treatment. The structure of the ω phase is a distorted form of the β phase with hexagonal symmetry. SADPs taken from alloys containing the ω phase often contain diffuse streaks parallel to specific crystallographic directions depending on $\mathbf{B}$, for example $\mathbf{B} \langle 111 \rangle$ streaks parallel to $\langle 110 \rangle$, see figure 4.43(d). Sass (1969b) has shown by careful tilting experiments that these correspond to sheets of intensity parallel to $\langle 111 \rangle$ in reciprocal space. Silcock (1958) and Sass (1969b) consider these sheets to be produced by scattering from rows of particles parallel to $\langle 111 \rangle$. Alternatively, Perkins *et al.* (1970) suggest that the cause is a large-amplitude atomic vibration parallel to $\langle 111 \rangle$ or mechanical instabilities in $\langle 111 \rangle$ (de Fontaine, 1970).

McCabe and Sass (1971) showed that, in T-V alloys, the lines of 'diffuse' intensity become sharper at lower V concentrations and the above criteria for identification of these streaks would not be satisfactory in this case. A check of the form of the image would be necessary to eliminate shape effects.

2.19.2.3 MC carbides

Curved diffuse streaks corresponding to corrugated sheets in reciprocal space have been reported in SADPs taken from off-stoichiometric MC carbides (Billingham *et al.*, 1972; Lewis *et al.*, 1972).

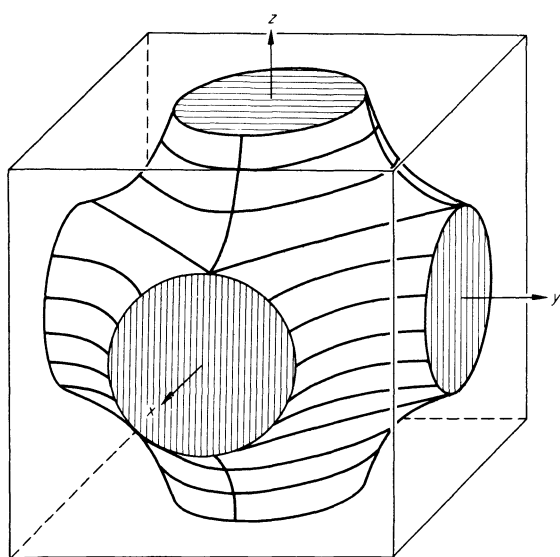


Figure 2.72 The distribution of diffuse intensity in reciprocal space corresponding to figure 2.71 [From Sauvage and Parthé (1972), courtesy of *Acta Crystallographica*]

Figure 2.71 shows a series of diffraction patterns with different $\mathbf{B}$ exhibiting this diffuse scattering. Figure 2.72 shows the distribution of the diffuse scattering in reciprocal space, interpreted by Sauvage and Parthé (1973) in terms of short-range ordering of vacancies.

2: Recommended Reading

- Andrews, K. W., Dyson, D. J., and Keown, S. R. (1971). *Interpretation of Electron Diffraction Patterns*, Hilger, London.
- Ball, C. J. (1971). *An Introduction to the Theory of Diffraction*, Pergamon Press, Oxford.
- Henry, M. F. N., Lipson, H., and Wooster, W. A. (1951). *Interpretation of X-ray Photographs*, Macmillan, New York.
- Hirsch, P. B., Howie, A., Nicholson, R. B., Pashley, D. W., and Whelan, M. J., (1967). *Electron Microscopy of Thin Crystals*, Butterworths, London.
- Rymer, T. B. (1970). *Electron Diffraction*, Methuen, London.
- Vainshtein, B. K. (1964). *Structure Analysis by Electron Diffraction*, Pergamon Press, Oxford.

2: References

- Andrews, K. W., Dyson, D. J., and Keown, S. R. (1971). *Interpretation of Electron Diffraction Patterns*, Hilger, London.
- Bagaryatski, Yu. A. (1950). *Dokl. Akad. Nauk SSSR*, **73**, 1161.
- Bain, E. C. (1924). *Trans. A.I.M.E.*, **70**, 25.
- Balluffi, R. W., Sass, S. L., and Schober, T. (1972a). *Phil. Mag.*, **26**, 585.
- (1972b). Private communication.
- Billingham, J., Bell, P. S., and Lewis, M. H. (1972). *Acta Cryst.*, **A28**, 602.
- Blackburn, M. J., and Williams, J. C. (1967). *Trans. A.I.M.E.*, **239**, 287.
- Brandon, D. G., Ralph, B., Ranganathan, S., and Wald, M. S. (1964). *Acta Met.*, **12**, 813.
- Butler, E. P. (1971). *Metal Sci. J.*, **5**, 8.
- Butler, E. P., and Thomas, G. (1970). *Acta Met.*, **18**, 347.
- Cahn, J. W. (1968). *Trans. A.I.M.E.*, **242**, 166.
- Calbick, C. J., and Marcus, R. B. (1967). *Acta Cryst.*, **A23**, 12.
- Clapp, P. C., and Moss, S. C. (1968). *Phys. Rev.*, **171**, 754.
- (1971). *Phys. Rev.*, **171**, 764.
- Crocker, A. G. (1965). *Trans. A.I.M.E.*, **233**, 17.
- Daniel, V., and Lipson, H. (1943a). *Proc. Roy. Soc. A*, **181**, 368.
- (1943b). *Proc. Roy. Soc. A*, **182**, 378.
- Drapier, J. M., and Coutsouradis, D. (1968). *Cobalt*, **39**, 63.

- Driver, J. H., Unthank, D. C., and Jack, K. H. (1972). *Phil. Mag.*, **26**, 1227.
- Duggan, B. J., and Segall, R. L. (1971). *Acta Met.*, **19**, 317.
- Fillingham, P. J., Leamy, H. J., and Tanner, L. E. (1972). *Electron Microscopy and Structure of Materials* (eds. G. Thomas, R. M. Fulrath and R. M. Fisher), University of California Press, California, p. 163.
- de Fontaine, D. (1970). *Acta Met.*, **18**, 275.
- Fujiwara, K., Hirabayashi, M., Watanabe, D., and Ogawa, S. (1958). *J. Phys. Soc. Japan*, **13**, 167.
- Gevers, R., van Landuyt, J., and Amelinckx, S. (1966). *Phys. Stat. Sol.*, **18**, 343(a).
- (1967a). *Phys. Stat. Sol.*, **21**, 393.
- (1967b). *Phys. Stat. Sol.*, **23**, 549.
- (1968). *Phys. Stat. Sol.*, **26**, 577.
- Gevers, R., Serneels, R., van Landuyt, J., and Amelinckx, S. (1969). *Phys. Stat. Sol.*, **31**, 681.
- Glossop, A. B., and Pashley, D. W. (1959). *Proc. Roy. Soc. A*, **250**, 132.
- Goux, C. (1961). *Bull. du Cercle d'Etudes des Métaux*, **8**, 185.
- Granovsky, E. B., Fridman, A. A., Pashkov, P. P., and Umansky, Y. A. S. (1968). *Proc. 4th European Reg. Conf. on Electron Microscopy, Rome*, vol. 1, Tipografia Poliglotta Vaticana, Rome, p. 439.
- Guinier, A. (1959). *Sol. State Phys.*, **9**, 293.
- Hannink, R. H. J., Murray, M. J., and Packer, M. E. (1971). *Phil. Mag.*, **24**, 1179.
- Hargreaves, M. E. (1951). *Acta Cryst.*, **4**, 301.
- von Heimendahl, M., Bell, W., and Thomas, G. (1964). *J. Appl. Phys.*, **35**, 3614.
- Henry, M. F. N., Lipson, H., and Wooster, W. A. (1951). *Interpretations of X-ray Photographs*, Macmillan, New York.
- Hirsch, P. B., Howie, A., Nicholson, R. B., Pashley, D. W., and Whelan, M. J. (1967). *Electron Microscopy of Thin Crystals*, Butterworths, London.
- Hollox, G. E., Rowcliffe, D. J., and Edington, J. W. (1972). *Electron Microscopy and Structure of Materials* (eds. G. Thomas, R. M. Fulrath and R. M. Fisher), University of California Press, California, p. 1116.
- Honjo, G., Kodera, S., and Kitamura, N. (1964). *J. Phys. Soc. Japan*, **19**, 351.
- Hudson, B., and Makin, M. J. (1963). *Phil. Mag.*, **8**, 553.
- International Tables for X-ray Crystallography* (1962). Kynoch Press, London.
- Johari, O., and Thomas, G. (1964). *Trans. A.I.M.E.*, **230**, 597.
- Jones, P. J., and Edington, J. W. (1972). *Met. Trans.*, **3**, 2706.
- Kelly, P. M. (1965). *Trans. A.I.M.E.*, **233**, 264.
- Kelly, P. M., and Nutting, J. (1960). *Proc. Roy. Soc. A*, **259**, 45.
- Kikuchi, S. (1928). *Japanese J. Phys.*, **5**, 83.
- Kuizhnik, E., Kraposhin, V., Livshitz, B., and Linetsky, Y. (1968). *Proc. 4th European Reg. Conf. on Electron Microscopy, Rome*, vol. 1, Tipografia Poliglotta Vaticana, Rome, p. 473.
- Kurdjumov, G., and Sachs, G. (1930). *Z. Physik*, **64**, 325.
- Laird, C., Eichen, E., and Bitler, W. R. (1966). *J. Appl. Phys.*, **37**, 2225.
- van Landuyt, J. (1964). *Phys. Stat. Sol.*, **6**, 957.
- van Landuyt, J., Gevers, R., and Amelinckx, S. (1966a). *Phys. Stat. Sol.*, **13**, 467.
- (1966b). *Phys. Stat. Sol.*, **18**, 167.
- Levine, E., Bell, W. L., and Thomas, G. (1966). *J. Appl. Phys.*, **37**, 2141.
- Lewis, M. H., Billingham, J., and Bell, P. S. (1972). *Electron Microscopy and Structure of Materials* (eds. G. Thomas, R. M. Fulrath and R. M. Fisher), University of California Press, California, p. 1084.
- McCabe, K. K., and Sass, S. L. (1971). *Phil. Mag.*, **23**, 957.
- Meieran, E. S., and Richman, H. M. (1963). *Trans. A.I.M.E.*, **227**, 1044.
- Nishyama, Z. (1934). *Sci. Rep. Tokoku Univ.*, **64**, 325.
- Ogawa, S., Watanabe, D., Watanabe, H., and Komoda, T. (1958). *Acta Cryst.*, **11**, 872.
- Okamoto, P. R., Levine, E., and Thomas, G. (1967). *J. Appl. Phys.*, **38**, 289.
- Okamoto, P. R., and Thomas, G. (1971a). *Mat. Res. Bull.*, **6**, 45.
- (1971b). *Acta Met.*, **19**, 825.
- Otte, H. M., Dash, J., and Schaake, H. F. (1964). *Phys. Stat. Sol.*, **5**, 527.
- Partridge, P. G. (1967). *Met. Rev.*, **12**, No. 118, 169.
- Pashley, D. W., and Presland, A. E. B. (1959). *J. Inst. Met.*, **87**, 419.
- Pashley, D. W., and Stowell, M. J. (1963). *Phil. Mag.*, **8**, 1605.
- Patterson, R. L., and Wayman, C. M. (1966). *Acta Met.*, **14**, 347.
- Perkins, A. J., Yaffe, P. E., and Heheman, R. F. (1970). *Met. Trans.*, **1**, 2785.
- Phillips, V. A., and Tanner, L. E. (1973). *Acta Met.*, **21**, 441.
- Pitsch, W. (1962). *Acta Met.*, **10**, 79.
- Pumphrey, P. H. (1970). *Ph.D. Thesis*, University of Cambridge.
- Pumphrey, P. H., and Bowkett, K. M. (1970a). *Phys. Stat. Sol.*, **2a**, 339.
- (1970b). *Phys. Stat. Sol.*, **3a**, 375.
- de Ridder, R., van Landuyt, J., Gevers, R., and Amelinckx, S. (1968). *Phys. Stat. Sol.*, **30**, 797.
- (1970a). *Phys. Stat. Sol.*, **38**, 747.
- (1970b). *Phys. Stat. Sol.*, **40**, 271.
- (1970c). *Phys. Stat. Sol.*, **41**, 519.
- (1970d). *Phys. Stat. Sol.*, **42**, 645.

- Ryder, P. L., and Pitsch, W. (1968). *Phil. Mag.*, **18**, 807.
- Saito, K., and Watanabe, R. (1967). *J. Phys. Soc. Japan*, **22**, 681.
- Sass, S. L. (1969a). *Trans. A.I.M.E.*, **245**, 1836.
- (1969b). *Acta Met.*, **17**, 813.
- Sauvage, M., and Parthé, E. (1972). *Acta Cryst.*, **A28**, 607.
- Serneels, R., de Ridder, R., Gevers, R., and Amelinckx, S. (1971). *Phys. Stat. Sol. B*, **45**, 123.
- Silcock, J. M. (1958). *Acta Met.*, **6**, 481.
- Tadaki, T., and Shimizu, K. (1970). *Trans. Japanese Inst. Met.*, **11**, 44.
- Tan, T. Y., Bell, W. L., and Thomas, G. (1971). *Phil. Mag.*, **24**, 417.
- Tanner, L. E. (1966). *Phil. Mag.*, **14**, 111.
- (1968). *Phys. Stat. Sol.*, **30**, 685.
- Tanner, L. E., Clapp, P. C., and Toth, R. S. (1968). *Mat. Res. Bull.*, **3**, 855.
- Thomas, G. (1970). *Modern Diffraction and Imaging Techniques in Materials Science* (eds. S. Amelinckx, R. Gevers, G. Remaut and J. van Landuyt), North-Holland, Amsterdam, p. 159.
- Thompson, M. N., and Edington, J. W. (1970). *Proc. 7th Intern. Congr. for Electron Microscopy, Grenoble*, vol. 2, Société Française de Microscopie Electronique, Paris, p. 545.
- van Torne, L. I., and Thomas G. (1966). *Acta Met.*, **14**, 621.
- Tyapkin, Yu. D., and Gavrilova, A. V. (1964). *Sov. Phys.—Cryst.*, **9**, 166.
- Vainshtein, B. K. (1964). *Structure Analysis by Electron Diffraction* (transl. E. Feigl and J. A. Spink), Pergamon Press, Oxford.
- Vaughan, D., and Silcock, J. M. (1967). *Phys. Stat. Sol.*, **20**, 725.
- Venables, J. D., Kahn, D., and Lye, R. G. (1968). *Phil. Mag.*, **18**, 177.
- Villagrana, R. E., and Thomas, G. (1965). *Phys. Stat. Sol.*, **9**, 499.
- Warrington, D. H., Rodgers, J. M., and Tebble, R. S. (1962). *Phil. Mag.*, **7**, 1783.
- Wasserman, G. (1933). *Arch. Für das Eisenhutt*, **16**, 647.
- Watanabe, D. (1958). *J. Phys. Soc. Japan*, **13**, 535.
- Watanabe, D., and Ogawa, S. (1956). *J. Phys. Soc. Japan*, **11**, 226.
- Wayman, C. M., and Bullough, R. (1966). *Trans. A.I.M.E.*, **236**, 1711.
- Willaime, C., Delavignette, P., Gevers, R., and Amelinckx, S. (1966). *Phys. Stat. Sol.*, **17**, K173.
- Woodilla, J. E., and Averbach, B. L. (1968). *Acta Met.*, **16**, 255.

APPENDIX 1.

BASIC CRYSTALLOGRAPHY

A1.1 Introduction

In order to perform electron microscope studies it is necessary to understand the basic principles of crystallography set out below.

It is the regular arrangement of atoms in space which constitutes the distinguishing feature of the crystalline state. These regular arrays, called crystal structures, which are characterised in various different ways, give rise to the internal and

external symmetry of the structure. The translational symmetry leads to the concept of a lattice (an array of atoms at points in space with identical surroundings). Arising from considerations of this type we arrive at fourteen distinct Bravais lattices, figure A1.1. These fall into the seven fundamental crystal classes listed in table A1.1. Most metals are either cubic, hexagonal or tetragonal in structure, see table A1.3. However, many non-metallic materials have more complex structures.

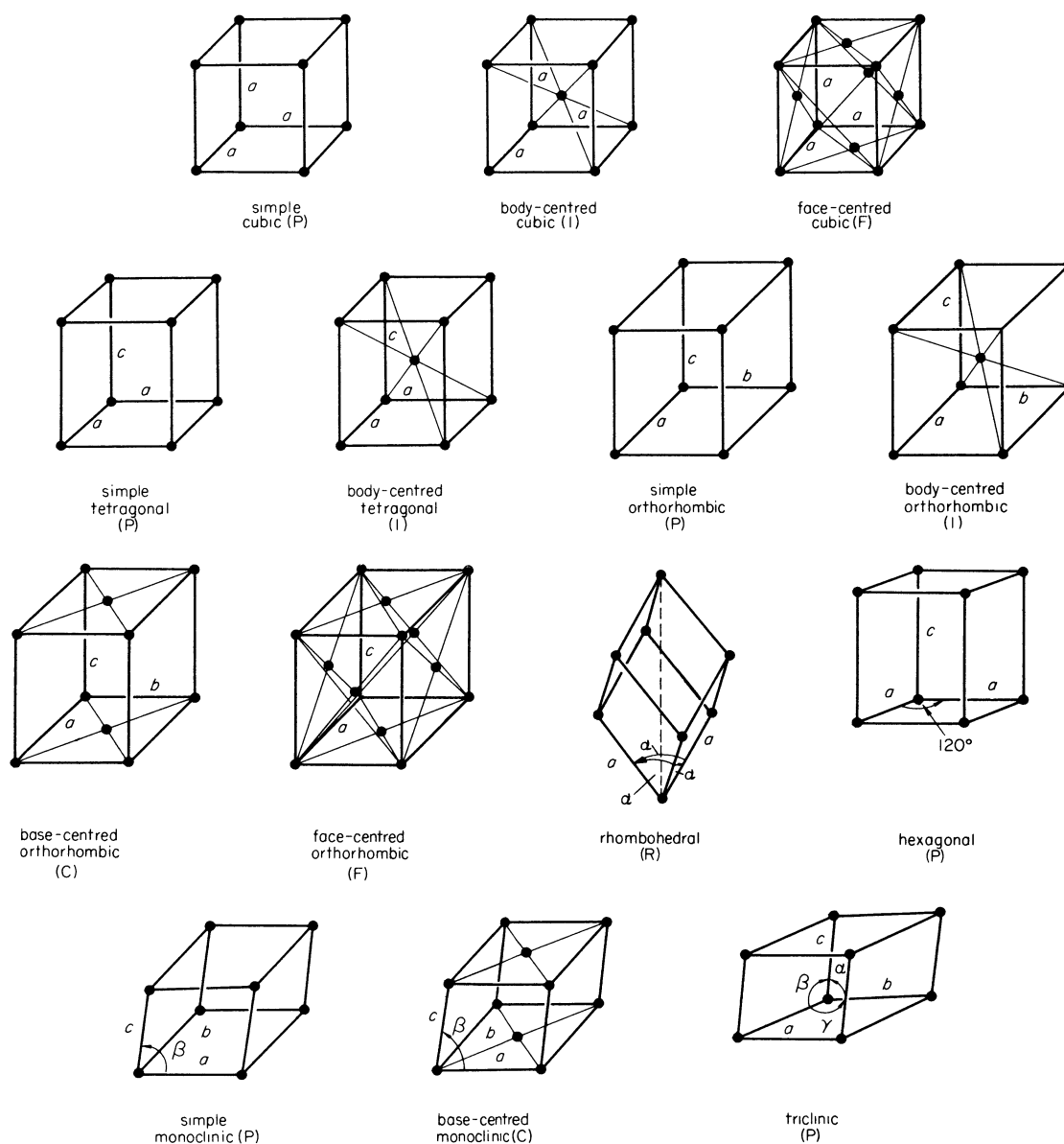


Figure A1.1 The fourteen Bravais lattices

Table A1.1 Crystallographic formulae for interplanar spacings,

Crystal system		Interplanar spacing of the (hkl) plane
cubic	$a = b = c$ $\alpha = \beta = \gamma = 90^\circ$	$\frac{1}{d^2} = \frac{1}{a^2} (h^2 + k^2 + l^2)$
tetragonal	$a = b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	$\frac{1}{d^2} = \frac{1}{a^2} (h^2 + k^2) + \frac{1}{c^2} l^2$
orthorhombic	$a \neq b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	$\frac{1}{d^2} = \frac{1}{a^2} h^2 + \frac{1}{b^2} k^2 + \frac{1}{c^2} l^2$
hexagonal	$a = b \neq c$ $\alpha = \beta = 90^\circ; \gamma = 120^\circ$	$\frac{1}{d^2} = \frac{4}{3a^2} (h^2 + hk + k^2) + \frac{1}{c^2} l^2$
rhombohedral	$a = b = c$ $\alpha = \beta = \gamma < 120^\circ \neq 90^\circ$	$\frac{1}{d^2} = \frac{1}{a^2} \frac{(1 + \cos \alpha) \{ (h^2 + k^2 + l^2) - (1 - \tan^2 \frac{1}{2} \alpha)(hk + kl + lh) \}}{1 + \cos \alpha - 2 \cos^2 \alpha}$
monoclinic	$a \neq b \neq c$ $\alpha = \gamma = 90^\circ \neq \beta$	$\frac{1}{d^2} = \frac{1}{a^2} \frac{h^2}{\sin^2 \beta} + \frac{1}{b^2} k^2 + \frac{1}{c^2} \frac{l^2}{\sin^2 \beta} - \frac{2hl \cos \beta}{ac \sin^2 \beta}$
triclinic	$a \neq b \neq c$ $\alpha \neq \beta \neq \gamma$	$\frac{1}{d^2} = \frac{1}{V^2} (s_{11}h^2 + s_{22}k^2 + s_{33}l^2 + 2s_{12}hk + 2s_{23}kl + 2s_{31}lh)$ where $V^2 = a^2b^2c^2(1 - \cos^2 \alpha - \cos^2 \beta - \cos^2 \gamma + 2 \cos \alpha \cos \beta \cos \gamma)$ and $s_{11} = b^2c^2 \sin^2 \alpha$ $s_{22} = a^2c^2 \sin^2 \beta$ $s_{33} = a^2b^2 \sin^2 \gamma$ $s_{12} = abc^2(\cos \alpha \cos \beta - \cos \gamma)$ $s_{23} = a^2bc(\cos \beta \cos \gamma - \cos \alpha)$ $s_{31} = ab^2c(\cos \gamma \cos \alpha - \cos \beta)$

A1.2 Indexing Planes

Planes in any one of the fourteen Bravais lattices can be indexed in the same way. Axes are chosen defining a unit cell x , y and z at angles α , β and γ with the unit translation distances a , b and c (figure A1.2(a)).

A plane is defined in terms of its intercepts on these axes. For example, in figure A1.2(b) the plane cuts the axes at a/h , b/k and c/l where h , k and l are the Miller indices of the plane. Thus, in figure A1.2(c), the plane cuts the axes at unit translation distances in the x and y axes and two translation distances in the z axis. The index of the plane is then OX/OX , OY/OY and OZ/OW , that is $11\frac{1}{2}$ which is rationalised to 221, the hkl indices for the plane, that is the intercepts

in the plane are a/h , b/k and c/l . A negative intercept results in a negative component of the Miller index written as $\bar{h}$.

Formulae for the angles between different (hkl) planes in the fourteen Bravais lattices are given in table A1.1.

A1.3 Indexing Lattice Directions

For any Bravais lattice, such as that shown in figure A1.3, the direction OA has the indices $[121]$, that is the path from O to A involves moving 1 unit translation parallel to x , 2 parallel to y and 1 parallel to z . The other directions $[01\bar{1}]$ and $[\bar{1}10]$ are self-evident. The symbol $\bar{1}$ means one translation in the negative direction. The general symbol

angles, and angles between directions for the seven crystal systems [After Andrews *et al.* (1971)]

Angle ϕ between $(h_1 k_1 l_1)$ and $(h_2 k_2 l_2)$	Angle ρ between directions $[u_1 v_1 w_1]$ and $[u_2 v_2 w_2]$
$\cos \phi = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{\{(h_1^2 + k_1^2 + l_1^2)(h_2^2 + k_2^2 + l_2^2)\}^{1/2}}$	$\cos \rho = \frac{u_1 u_2 + v_1 v_2 + w_1 w_2}{\{(u_1^2 + v_1^2 + w_1^2)(u_2^2 + v_2^2 + w_2^2)\}^{1/2}}$
$\cos \phi = \frac{\frac{1}{a^2}(h_1 h_2 + k_1 k_2) + \frac{1}{c^2} l_1 l_2}{\left[\left\{ \frac{1}{a^2}(h_1^2 + k_1^2) + \frac{1}{c^2} l_1^2 \right\} \left\{ \frac{1}{a^2}(h_2^2 + k_2^2) + \frac{1}{c^2} l_2^2 \right\} \right]^{1/2}}$	$\cos \rho = \frac{a^2(u_1 u_2 + v_1 v_2) + c^2 w_1 w_2}{[\{a^2(u_1^2 + v_1^2) + c^2 w_1^2\} \{a^2(u_2^2 + v_2^2) + c^2 w_2^2\}]^{1/2}}$
$\cos \phi = \frac{\frac{1}{a^2} h_1 h_2 + \frac{1}{b^2} k_1 k_2 + \frac{1}{c^2} l_1 l_2}{\left\{ \left(\frac{1}{a^2} h_1^2 + \frac{1}{b^2} k_1^2 + \frac{1}{c^2} l_1^2 \right) \left(\frac{1}{a^2} h_2^2 + \frac{1}{b^2} k_2^2 + \frac{1}{c^2} l_2^2 \right) \right\}^{1/2}}$	$\cos \rho = \frac{a^2 u_1 u_2 + b^2 v_1 v_2 + c^2 w_1 w_2}{\{(a^2 u_1^2 + b^2 v_1^2 + c^2 w_1^2)(a^2 u_2^2 + b^2 v_2^2 + c^2 w_2^2)\}^{1/2}}$
$\cos \phi = \frac{h_1 h_2 + k_1 k_2 + \frac{1}{2}(h_1 k_2 + k_1 h_2) + \frac{3}{4} \frac{a^2}{c^2} l_1 l_2}{\left\{ \left(h_1^2 + k_1^2 + h_1 k_1 + \frac{3}{4} \frac{a^2}{c^2} l_1^2 \right) \left(h_2^2 + k_2^2 + h_2 k_2 + \frac{3}{4} \frac{a^2}{c^2} l_2^2 \right) \right\}^{1/2}}$	$\cos \rho = \frac{u_1 u_2 + v_1 v_2 - \frac{1}{2}(u_1 v_2 + v_1 u_2) + \frac{c^2}{a^2} w_1 w_2}{\left\{ \left(u_1^2 + v_1^2 - u_1 v_1 + \frac{c^2}{a^2} w_1^2 \right) \left(u_2^2 + v_2^2 - u_2 v_2 + \frac{c^2}{a^2} w_2^2 \right) \right\}^{1/2}}$

convert to corresponding hexagonal indices (see appendix 2) and use the above two formulae

$\cos \phi = \frac{\frac{1}{a^2} h_1 h_2 + \frac{1}{b^2} k_1 k_2 \sin^2 \beta + \frac{1}{c^2} l_1 l_2 - \frac{1}{ac} (l_1 h_2 + l_2 h_1) \cos \beta}{\left\{ \left(\frac{1}{a^2} h_1^2 + \frac{1}{b^2} k_1^2 \sin^2 \beta + \frac{1}{c^2} l_1^2 - \frac{2h_1 l_1}{ac} \cos \beta \right) \left(\frac{1}{a^2} h_2^2 + \frac{1}{b^2} k_2^2 \sin^2 \beta + \frac{1}{c^2} l_2^2 - \frac{2h_2 l_2}{ac} \cos \beta \right) \right\}^{1/2}}$	$\cos \rho = \frac{a^2 u_1 u_2 + b^2 v_1 v_2 + c^2 w_1 w_2 + ac(w_1 u_2 + u_1 w_2) \cos \beta}{\{(a^2 u_1^2 + b^2 v_1^2 + c^2 w_1^2 + 2acu_1 w_1 \cos \beta) \times (a^2 u_2^2 + b^2 v_2^2 + c^2 w_2^2 + 2acu_2 w_2 \cos \beta)\}^{1/2}}$
$\cos \phi = \frac{F}{A_{h_1 k_1 l_1} A_{h_2 k_2 l_2}}$	$\cos \rho = \frac{L}{I_{u_1 v_1 w_1} I_{u_2 v_2 w_2}}$
where $F = h_1 h_2 b^2 c^2 \sin^2 \alpha + k_1 k_2 a^2 c^2 \sin^2 \beta + l_1 l_2 a^2 b^2 \sin^2 \gamma$ $+ abc^2(\cos \alpha \cos \beta - \cos \gamma)(k_1 h_2 + h_1 k_2)$ $+ ab^2 c(\cos \gamma \cos \alpha - \cos \beta)(h_1 l_2 + l_1 h_2)$ $+ a^2 b c(\cos \beta \cos \gamma - \cos \alpha)(k_1 l_2 + l_1 k_2)$	where $L = a^2 u_1 u_2 + b^2 v_1 v_2 + c^2 w_1 w_2$ $+ bc(v_1 w_2 + w_1 v_2) \cos \alpha$ $+ ac(w_1 u_2 + u_1 w_2) \cos \beta$ $+ ab(u_1 v_2 + v_1 u_2) \cos \gamma$
and $A_{hkl} = \{h^2 b^2 c^2 \sin^2 \alpha + k^2 a^2 c^2 \sin^2 \beta + l^2 a^2 b^2 \sin^2 \gamma$ $+ 2hkabc^2(\cos \alpha \cos \beta - \cos \gamma)$ $+ 2hlab^2 c(\cos \gamma \cos \alpha - \cos \beta)$ $+ 2kla^2 bc(\cos \beta \cos \gamma - \cos \alpha)\}^{1/2}$	and $I_{uvw} = (a^2 u^2 + b^2 v^2 + c^2 w^2$ $+ 2bcvw \cos \alpha$ $+ 2cawu \cos \beta$ $+ 2abuv \cos \gamma)^{1/2}$

for a direction is $[uvw]$ where $r = OA$, figure A1.3, $= ua + vb + wc$.

Consequently, r is a vector with components u , v and w along the axes.

Formulae for the angles between different $[uvw]$ directions in the fourteen Bravais lattices are given in table A1.1.

A1.4 Plane Normals

In the cubic crystal system only the direction normal to the plane (hkl) has indices $[hkl]$, for example $[111]$ is the (111) plane normal. In all other crystal systems this is not true and table A1.2 gives the formulae for determining the indices of the directions $[uvw]$ normal to the plane (hkl) and vice versa.

A1.5 Zones and the Zone Law

Any two lattice planes intersect in a line which can be defined by the directional indices $[uvw]$. This is the axis for a prism of planes with this common direction. The planes are known as the zone of planes and the long axis is the zone axis, z , given the symbol $[UVW]$. The zone indices for any pair of planes, that is $(h'k'l')$ and (hkl) can be obtained in the following way:

$$\begin{array}{c|ccc|c} h & k & l & h & k & l \\ & \times & \times & \times & & \\ h' & k' & l' & h' & k' & l' \end{array}$$

that is $U = kl' - k'l$, $V = lh' - l'h$, $W = hk' - h'k$. This is the cross product between plane indices or the directions of the plane normal.

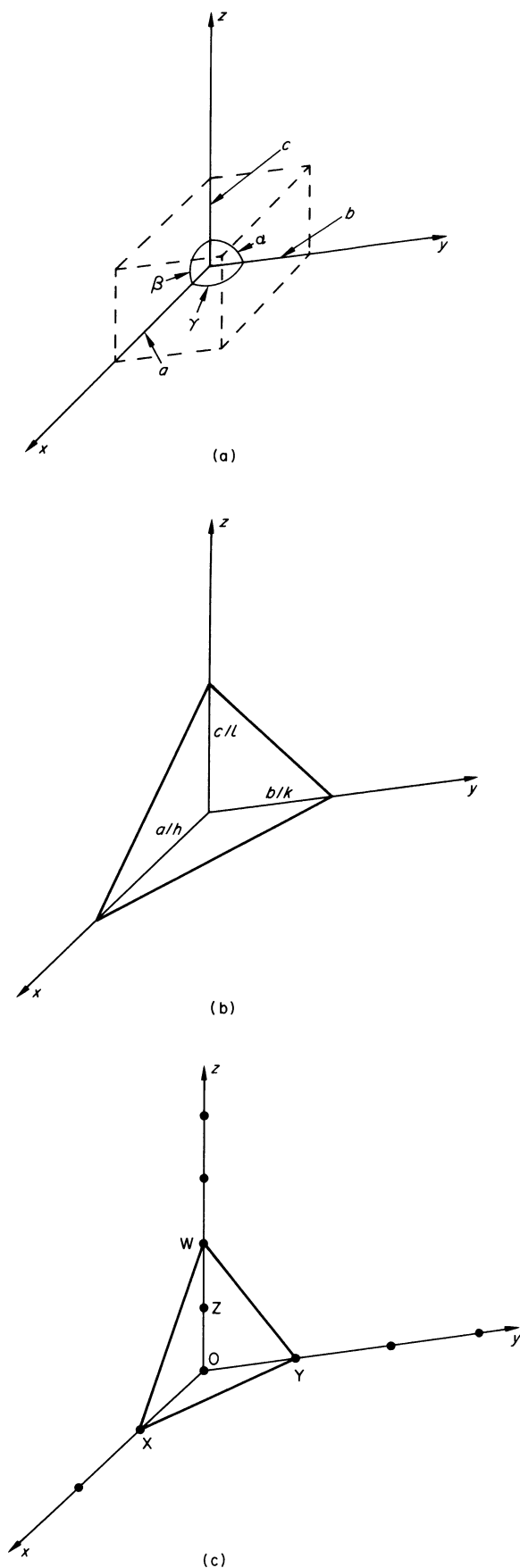


Figure A1.2 (a) The axes x , y , z defining the unit cell (dashed lines). (b) A general plane intersecting the axes. (c) A (221) plane

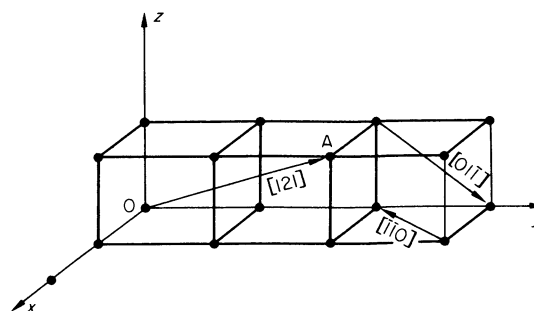


Figure A1.3 Crystal directions, OA is $[121]$

If it is necessary to find out if a plane (hkl) lies in a zone $[UVW]$ then the condition is

$$hU + kV + lW = 0$$

and this is called the Weiss zone law. This is essentially the condition that the normal to the plane (hkl) is perpendicular to $[UVW]$ direction.

Notation is as follows:

- (111) means single set of parallel planes
- $\{111\}$ means equivalent planes of the type, that is (111), $(1\bar{1}1)$, etc.
- $[111]$ means a single zone axis or direction
- $\langle 111 \rangle$ means directions of equivalent type

A1.6 Stereographic Projection

Although other methods of projection of the three-dimensional crystal into two dimensions exist, the stereographic projection is the most common way of describing crystals. This is because the projection preserves angular truth. The advantage of working with the stereographic projection lies in the ease and rapidity of performing those crystallographic analyses necessary in the electron microscope.

Imagine a crystal at the centre of a sphere, see figure A1.4, with plane normals drawn from the centre of the sphere to its surface. In figure A1.5, one of these plane normals P is shown projected into the equatorial plane about the south pole of the sphere. Normally only those planes above the equator are projected. For planes underneath, the north pole is used as the projection point, indicated by open circles in the projection. The stereographic projection of the cubic crystal in figure A1.4 with $[001]$ parallel to the south–north direction SN and $[010]$ parallel to OD , is shown in figure A1.6, each point being indexed as the normal to a particular plane.

Standard projections for cubic crystal structures are shown in figure A1.12 with different planes in the centre of the stereogram.

It is necessary to be able to measure the angles between planes using the stereographic projection. The normals to any two crystal planes, for example

Table A1.2 Formulae defining the indices of the direction $[uvw]$ perpendicular to plane (hkl) for the seven crystal systems [After Andrews *et al.* (1971)]

Crystal system	Equations for finding $[uvw]$ given (hkl)	Equations for finding (hkl) given $[uvw]$
cubic	$\frac{u}{h} = \frac{v}{k} = \frac{w}{l}$	$\frac{h}{u} = \frac{k}{v} = \frac{l}{w}$
tetragonal	$\frac{u}{h} = \frac{v}{k} = \frac{w(c)^2}{l(a)^2}$	$\frac{h}{u} = \frac{k}{v} = \frac{l}{w(c/a)^2}$
orthorhombic	$\frac{u}{h} a^2 = \frac{v}{k} b^2 = \frac{w}{l} c^2$	$\frac{h}{ua^2} = \frac{k}{vb^2} = \frac{l}{wc^2}$
hexagonal	$\frac{u}{2k+h} = \frac{v}{h+2k} = \frac{2w(c)^2}{3l(a)^2}$	$\frac{h}{2u-v} = \frac{k}{2v-u} = \frac{l}{2w(c/a)^2}$
rhombohedral	$\frac{u}{h \sin^2 \alpha + (k+l)(\cos^2 \alpha - \cos \alpha)} = \frac{v}{k \sin^2 \alpha + (l+h)(\cos^2 \alpha - \cos \alpha)} = \frac{w}{l \sin^2 \alpha + (h+k)(\cos^2 \alpha - \cos \alpha)}$	$\frac{h}{u + (v+w) \cos \alpha} = \frac{k}{v + (w+u) \cos \alpha} = \frac{l}{w + (u+v) \cos \alpha}$
monoclinic	$\frac{u}{hb^2c^2 - lab^2c \cos \beta} = \frac{v}{kc^2a^2 \sin^2 \beta} = \frac{w}{la^2b^2 - hab^2c \cos \beta}$	$\frac{h}{ua^2 + wca \cos \beta} = \frac{k}{vb^2} = \frac{l}{uca \cos \beta + wc^2}$
triclinic	$\frac{u}{hs_{11} + ks_{12} + ls_{13}} = \frac{v}{hs_{12} + ks_{22} + ls_{23}} = \frac{w}{hs_{13} + ks_{23} + ls_{33}}$ ($s_{11} = b^2c^2 \sin^2 \alpha$, etc; $s_{12} = s_{21} = abc^2 (\cos \alpha \cos \beta - \cos \gamma)$, etc.)	$\frac{h}{ua^2 + vab \cos \gamma + wca \cos \beta} = \frac{k}{uab \cos \gamma + vb^2 + wbc \cos \alpha} = \frac{l}{uca \cos \beta + vbc \cos \alpha + wc^2}$

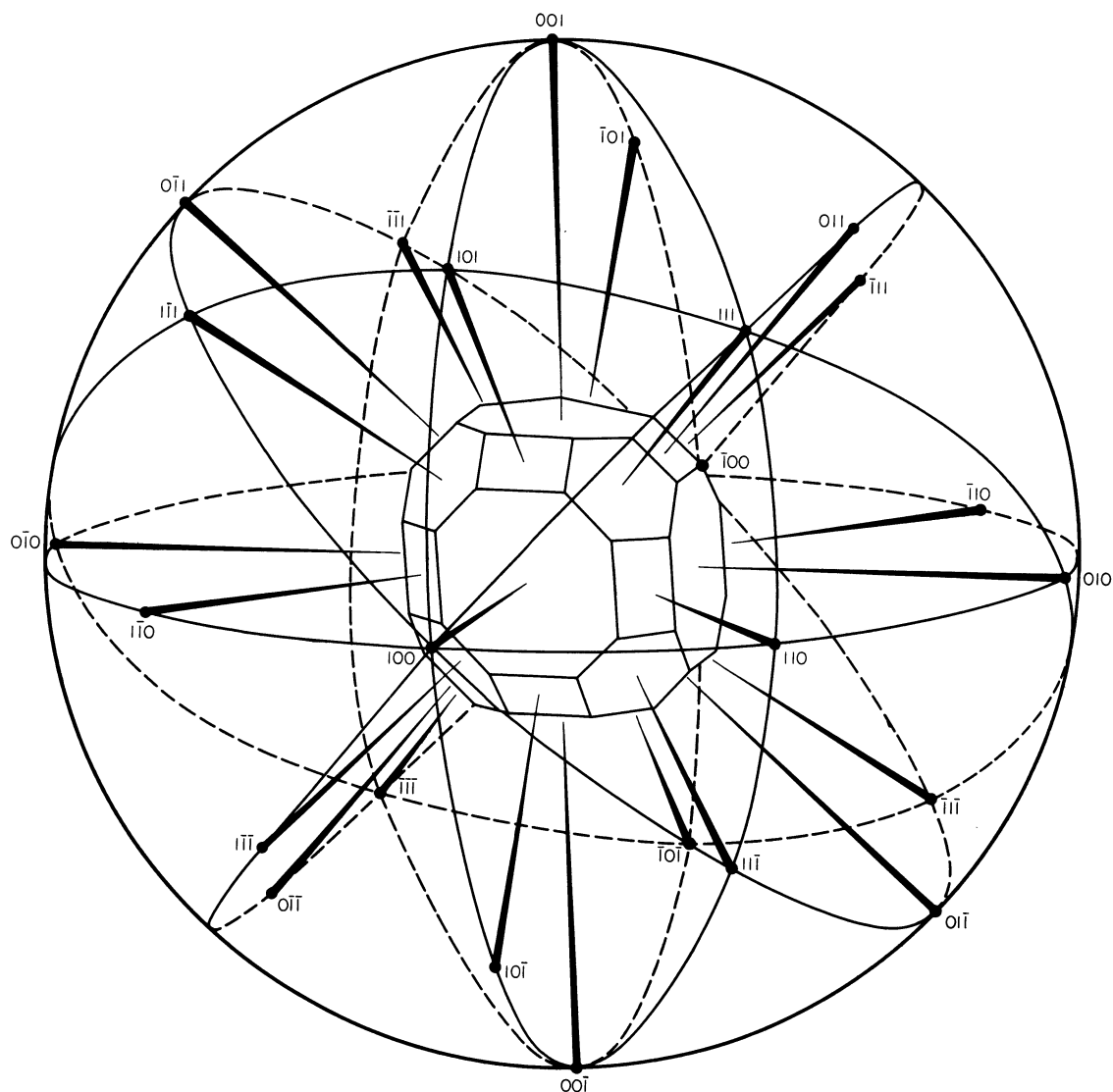


Figure A1.4 A crystal with cubic crystal structure situated at the centre of a sphere

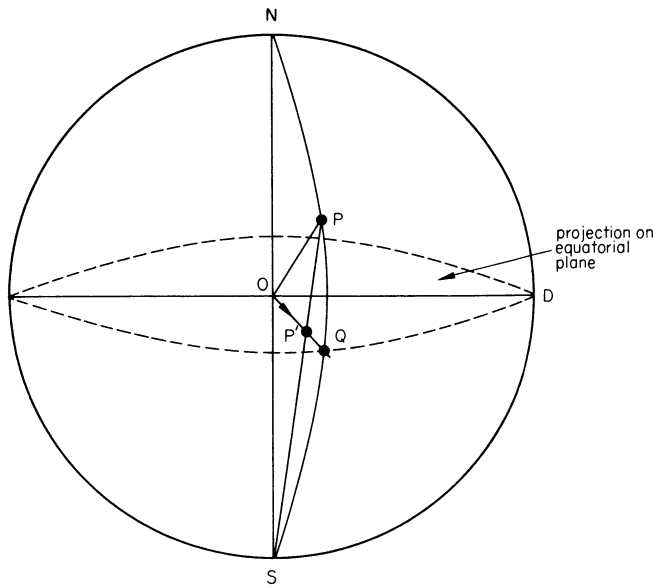


Figure A1.5 The projection of a plane normal OP into the equatorial plane about the south pole S

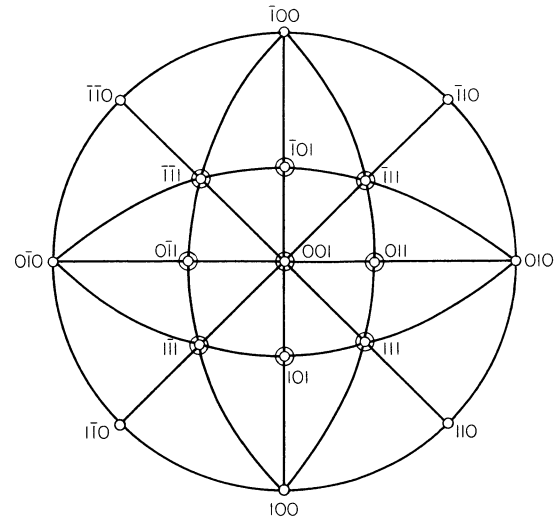


Figure A1.6 The stereographic projection for a cube crystal with $[001]$ parallel to the south north direction in the sphere of figure A1.4. $[010]$ is parallel to east-west

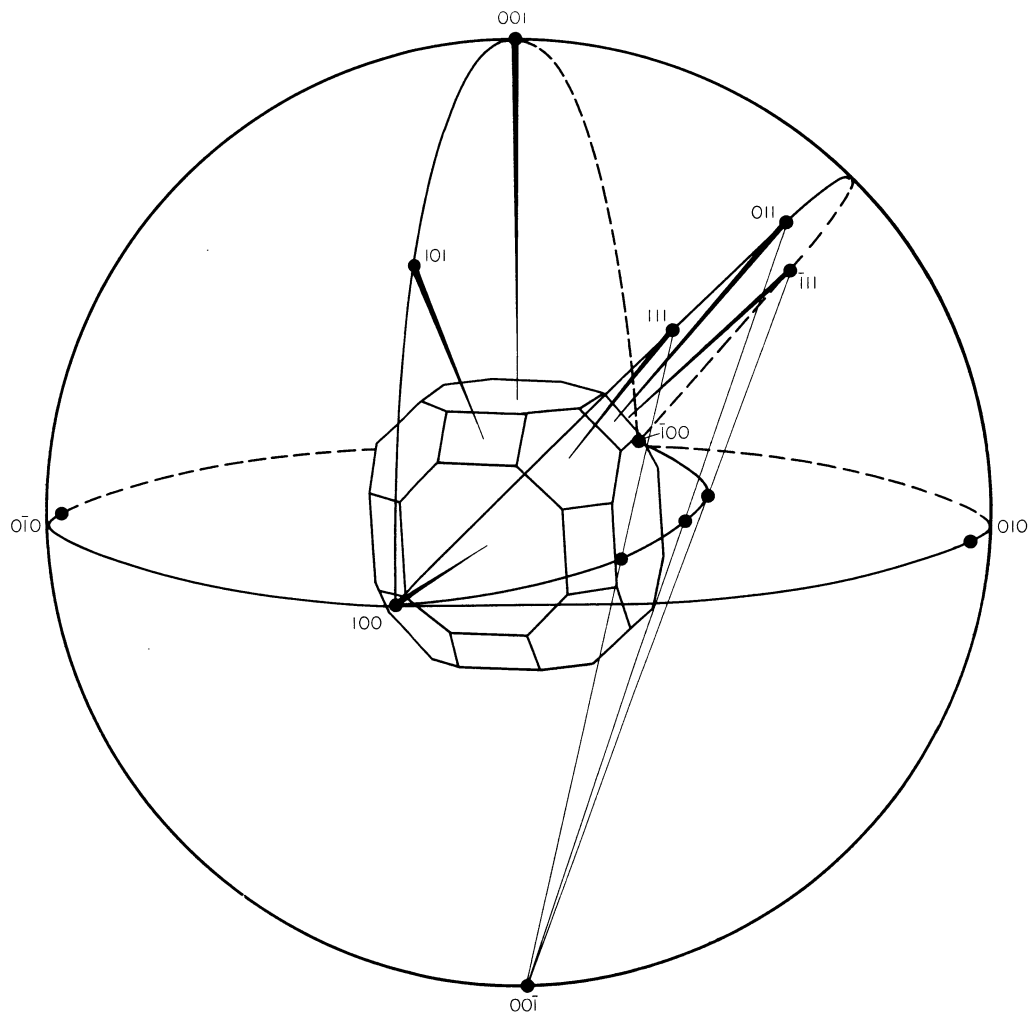


Figure A1.7 A cubic crystal at the centre of a sphere showing that the stereographic projection of (100) , (111) , (011) , $(\bar{1}11)$, $(\bar{1}00)$ and (100) , (101) , (001) lie on great circles, the latter being a diameter

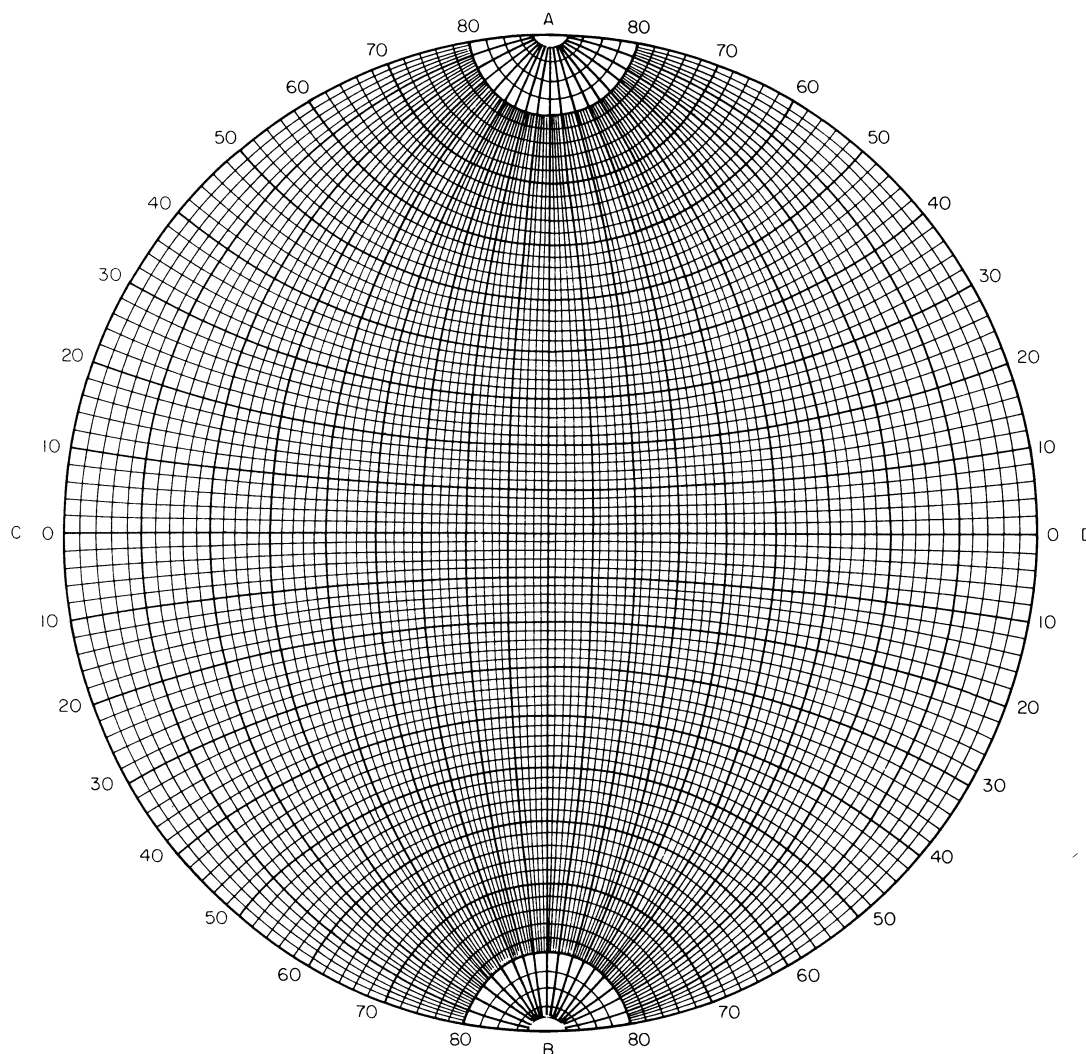


Figure A1.8 A Wulff net divided into two degree divisions

(111) and (011), figure A1.7, define a plane that passes through the centre of the sphere and intersects its surface in a great circle, that is one whose diameter is that of the sphere. The angle between (111) and (011) is proportional to the length of the arc of the great circle defined by their normals. Figure A1.7 shows that this great circle projects as an arc on a diameter.* Consequently, it is possible to measure the arc angle between (111) and (011) in terms of the distance along this arc between the plane normal projections. The device for doing this is a Wulff net, shown in figure A1.8. This consists of two sets of arcs. The first is an array of great circles on the same diameter AB. The second is a series of arcs centred on A and B such that their separation along any great circle corresponds to the same angle.† Thus to use the Wulff net to measure an angle between the projected plane normals P_1 and P_2 , figure A1.9(a), the net is rotated about its centre until P_1 and P_2 lie on the

same great circle, figure A1.9(b). The angle can then be read as shown.

The diameter CD on the Wulff net, figure A1.8, is a great circle and angles may be measured along it.

A1.7 Useful Manipulations with the Stereographic Projection and Wulff Net

In all of the following manipulations the Wulff net is used with its centre at the centre of the projection.

(1) To measure angles between any two planes or directions. This has been covered at the end of the previous section.

(2) To find the pole of a great circle. This is the projection of the axis of the zone of planes whose normals lie on the great circle. The Wulff net is aligned to superimpose on the great circle and the pole is constructed 90° along CD from the intersection of the great circle with CD, figure A1.10(a).

* Note that the great circle (100), (101), (001), figure A1.7, projects onto a diameter.

† Clearly the Wulff net corresponds to the projection of lines of latitude and longitude on the earth into the plane of the Greenwich meridian using the point 90° west on the equator as the projecting point.

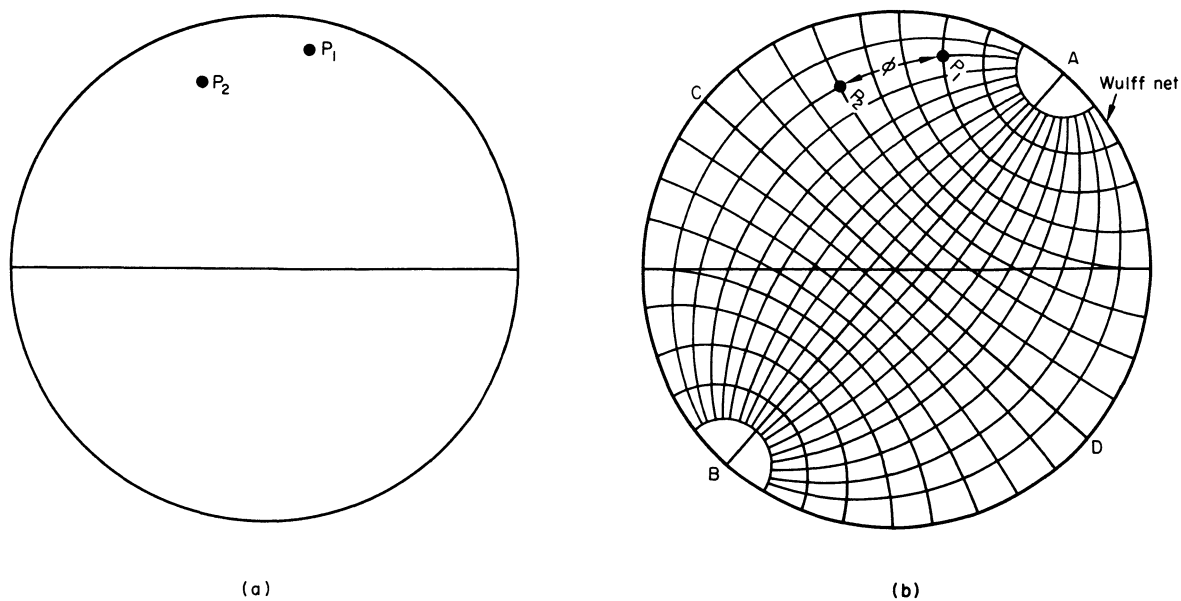


Figure A1.9 The measurement of the angle ϕ between poles P_1 and P_2 in the stereographic projection, using the Wulff net

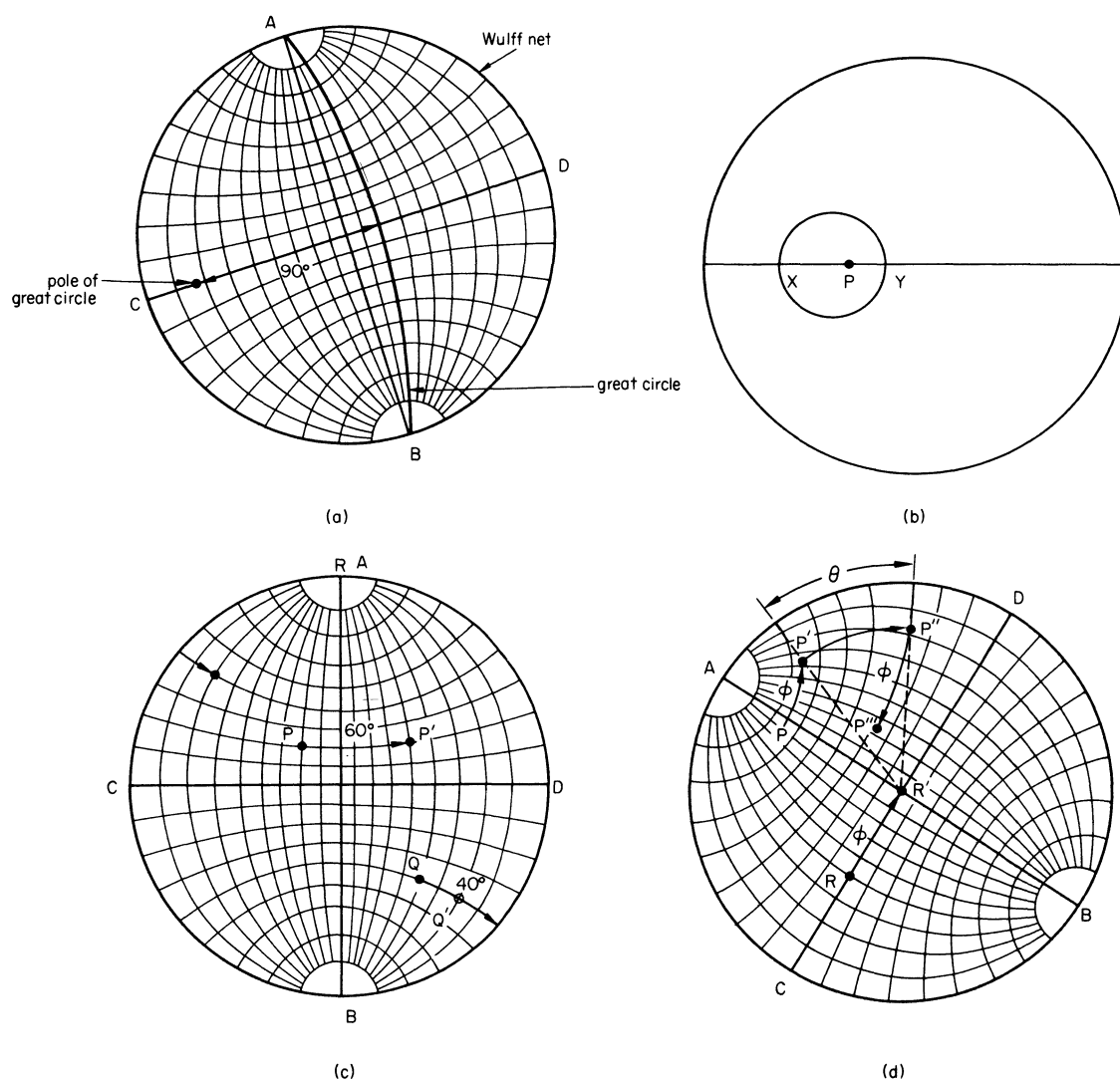


Figure A1.10 The use of the Wulff net (a) to find the pole of a great circle, (b) to construct a small circle about a pole P , (c) to rotate poles P and Q about a direction R on the edge of the stereogram, and (d) to rotate pole P about an axis R that does not lie on the edge of the stereogram

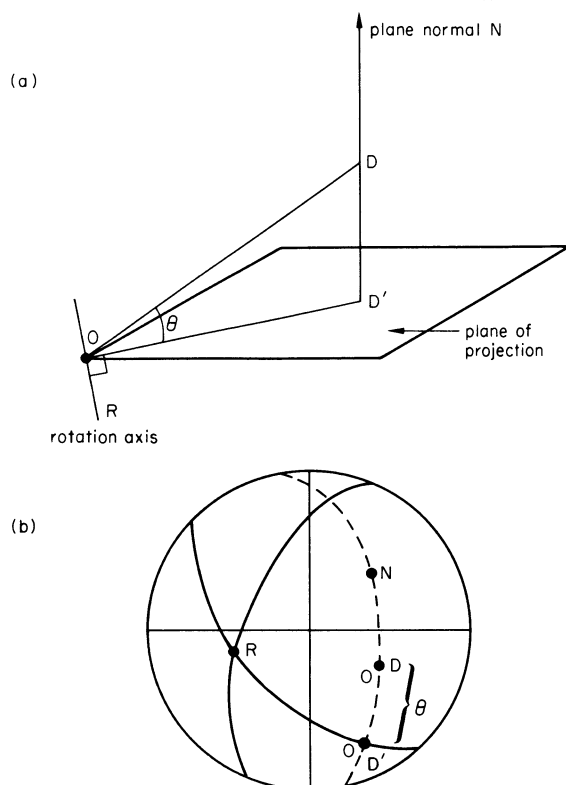


Figure A1.11 The projection of a particular crystal direction OD into a given plane (a) in real space, (b) on the stereographic projection

(3) To find the great circle corresponding to a pole. This is the reverse of manipulation (2).

(4) To construct a small circle about a pole. This corresponds to the projections of all plane normals at a given angle to the pole. Rotate the Wulff net so that either AB or CD cuts the pole. Measure the necessary angle from the pole P in figure A1.10(b) in both directions along either AB or CD to give the points X and Y. Bisect the line XY and draw the circle with XY as a diameter. Note that the geometric centre of this circle is not P.

(5) To rotate poles about an axis in the plane of projection, see figure A1.10(c). To rotate poles P and Q through 60° , in figure A1.10(c) about a pole R lying in the perimeter of the stereogram, set the A of the stereogram at R and measure along the arc shown the necessary amount to the positions P' and Q'. Note that Q' now must be regarded as projected from the north pole, see section A1.6, and is shown as an open circle, that is 'underneath' the stereogram.

(6) To rotate a pole through an angle θ about an axis R not on the perimeter of the stereogram. This procedure is shown in figure A1.10(d). Here R lies as shown and the net is rotated until CD cuts R. R and P are rotated by ϕ about AB as in (5) until R lies in the centre at R' and P moves to P'. Then P' is rotated to P'' by the required amount (θ) about R'; then R' is rotated back to R about AB and P'' moves to P'''.

(7) To project a given direction OD into a

specific plane, normal N, see figure A1.11(a). In three dimensions this involves rotating the direction OD down into the plane required, about an axis OR lying in the projection plane and perpendicular to OD, see figure A1.11(a). This is performed on the stereogram by drawing great circles corresponding to the plane normal N, and OD. These will intersect at R, a direction perpendicular to N and OD, see figure A1.11(b). Consideration of figure A1.11(a) will show that N, OD and OD' are all perpendicular to R. Thus D' always lies at the intersection of great circles with poles R and OD. The same result can be obtained by moving R to the centre then following the procedure outlined in (6).

A1.8 Useful Crystallographic Formulae for Various Crystal Structures

In interpreting electron diffraction patterns it is particularly useful to have available tables of interplanar spacings and angles between planes for the crystal structure of interest. These may be generated by computation from the formulae in table A1.1. Useful values of interplanar spacings and angles are listed in appendix 6 for the cubic and hexagonal crystal structures. The definitions of a , b , c , α , β , γ in table A1.1 are given for each crystal structure, illustrated in figure A1.1. Table A1.2 contains formulae for obtaining the indices of directions normal to planes and vice versa. Finally, table A1.3 lists crystal structures and lattice parameters of the elements which are crystalline at room temperature.

Appendix 1: Recommended Reading

Further information on crystallography and the use of the stereographic projection can be found in a number of books, including the following.

- Gay, P. (1972). *The Crystalline State, An Introduction*, Oliver and Boyd, Edinburgh.
- Johari, O., and Thomas, G. (1969). *The stereographic projection and its applications. Techniques of Metals Research* (ed. R. F. Bunshah), Interscience, New York.
- Kelly, A., and Groves, G. W. (1970). *Crystallography and Crystal Defects*, Longmans, London.
- Phillips, F. C. (1963). *An Introduction to Crystallography*, Longmans, London.
- Smaill, J. S. (1972). *Metallurgical Stereographic Projections*, Hilger, London.

Appendix 1: References

- Andrews, K. W., Dyson, D. J., and Keown, S. R. (1971). *Interpretation of Electron Diffraction Patterns*, Hilger, London.
- Barrett, C. S., and Hassalski, T. B. (1968). *Structure of Metal and Alloys*, McGraw-Hill, New York.

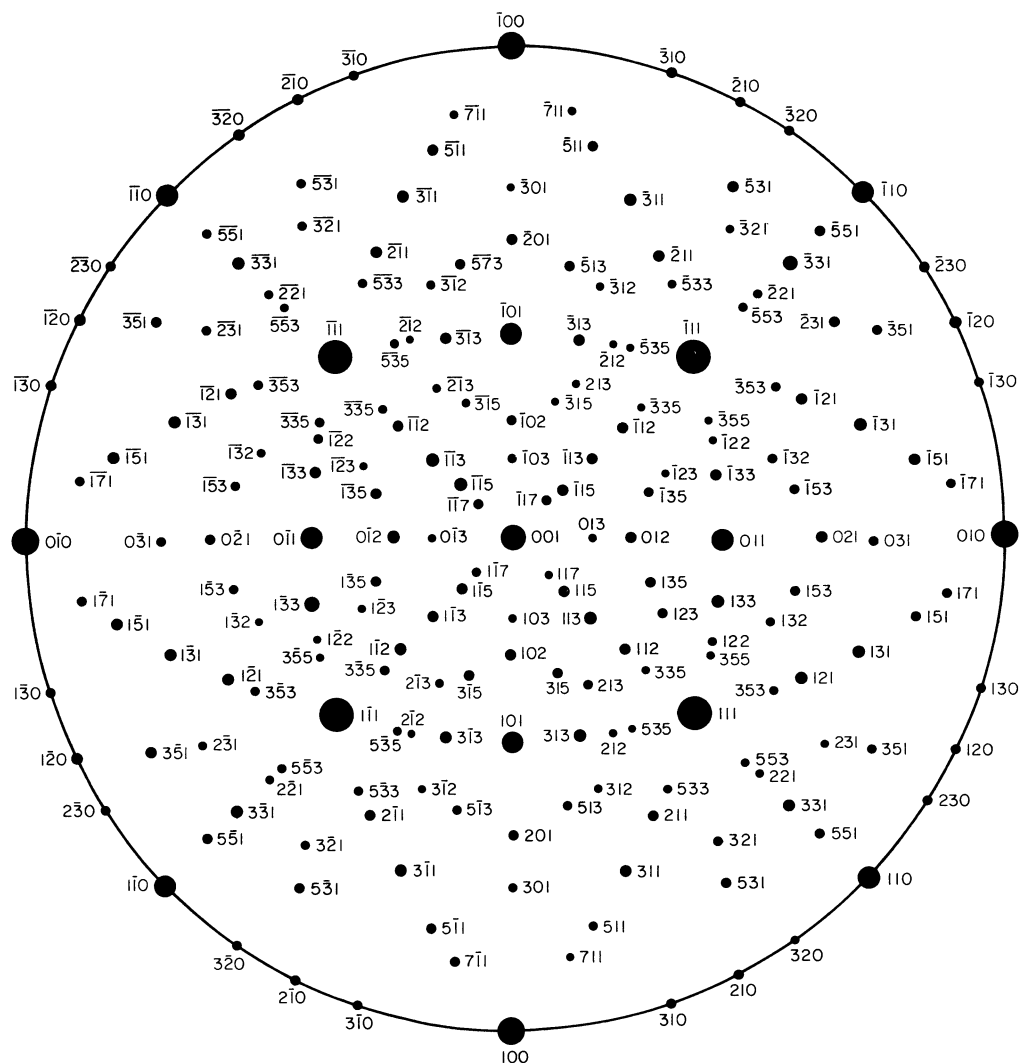


Figure A1.12 Standard stereographic projections for cubic structures: (a) (001); (b) (110); (c) (111); (d) (112)

Table A1.3 The lattice parameters and crystal structures of the elements crystalline at room temperature [After Barrett and Hassalski (1968)]

Element: form (transformation temp. °C)	Temp. (°C)	Structure	Lattice constants		c (Å) (α or β)
			a (Å)	b (Å)	
aluminum (Al)	25	f.c.c.	4.0496		
antimony (Sb)	25	rhomb.	4.5067		57°6'27"
	26	rhomb.	4.307	(hex. axes)	11.273
				z = 0.2335	
	4 °K	rhomb.	4.3007	z = 0.2336	11.222
arsenic (As)		rhomb.	4.131		α = 54°10'
barium (Ba)	R.T.	b.c.c.	5.019		
beryllium, α (Be)	20	c.p.h.	2.2856		3.5832
β? > 1250	1250	b.c.c.	2.55		
bismuth (Bi)	25	rhomb.	4.546		11.862
			(hex. axes) z = 0.2339		
	78 °K	rhomb.	4.535	z = 0.2341	11.814
	4 °K	rhomb.		z = 0.2340	11.862
cadmium (Cd)	21	c.p.h.	2.9788		5.6167
calcium, α (Ca)	18	f.c.c.	5.582		
γ 464 to m.p.	~ 500	b.c.c.	4.477		
carbon, diamond	20	cubic	3.5670		
graphite, α	20	hex.	2.4612		6.707

Table A1.3 (continued)

Element: form (transformation temp. °C)	Temp. (°C)	Structure	Lattice constants		c (Å) (α or β)
			a (Å)	b (Å)	
carbon (<i>contd.</i>)					
graphite, β		rhomb.	2.4612		10.061
caesium (Cs)	-10	b.c.c.	6.14		
chromium (Cr)	20	b.c.c.	2.8846		
cobalt, α (Co)	18	c.p.h.	2.506		4.069
β stable ~450 to m.p.	18	f.c.c.	3.544		
copper (Cu)	20	f.c.c.	3.6147		
	0	f.c.c.	3.6029		
gallium (Ga)	20	orthorhomb.	4.5258	4.5198	7.6602
germanium (Ge)	25	cubic	5.6576		
gold (Au)	25	f.c.c.	4.0788		
hafnium α (Hf)	24	c.p.h.	3.1946		5.0511
indium (In)	R.T.	tetrag.	4.5979	(f.c. cell)	4.9467
	R.T.	tetrag.	3.2512	(b.c. cell)	4.9467
iodine (I)	26	orthorhomb.	4.79	7.25	9.78
iridium (Ir)	26	f.c.c.	3.8389		
iron, α (Fe)	20	b.c.c.	2.8664		
γ 911 to 1392	916	f.c.c.	3.6468		
δ 1392 to m.p.	1394	b.c.c.	2.9322		
lead (Pb)	25	f.c.c.	4.9502		
magnesium (Mg)	25	c.p.h.	3.2094		5.2105
manganese, α (Mn)	25	cubic	8.9139		
β 742 to 1095	25	cubic	6.315		
γ 1095 to 1133	1095	f.c.c.	3.862		
δ 1133 to m.p.	1134	b.c.c.	3.081		
molybdenum (Mo)	20	b.c.c.	3.1468		
nickel (Ni)	18	f.c.c.	3.5236		
niobium (Nb)	20	b.c.c.	3.3007		
(columbium)					
palladium (Pd)	22	f.c.c.	3.8907		
platinum (Pt)	20	f.c.c.	3.9239		
plutonium, α (Pu)	21	monoclin.	6.1835	4.8244	10.973
				10.463	7.859
β 122 to 206	190	monoclin.	9.284	$\beta = 101.81^\circ$	$\beta = 92.13^\circ$
γ 206 to 319	235	orthorhomb.	3.159	5.768	10.162
δ 319 to 451	320	f.c.c.	4.637		
δ' 451 to 485	477	tetrag.	3.339		4.446
ϵ 476 to m.p.	490	b.c.c.	3.636		

APPENDIX 2.

CRYSTALLOGRAPHIC TECHNIQUES FOR THE INTERPRETATION OF TRANSMISSION ELECTRON MICROGRAPHS OF MATERIALS WITH HEXAGONAL CRYSTAL STRUCTURE

A2.1 Introduction

The geometrical interpretation of transmission electron micrographs of hexagonal close-packed metals is more complicated than the equivalent interpretation of cubic metals for three reasons. Firstly, prominent zone axes are not in general normal to prominent planes, so a simple diffraction pattern commonly corresponds to a non-rational foil plane; secondly, there is no simple relationship between the plane ($hkil$) and the direction $[hki]$; and, thirdly, the Miller–Bravais system of indexing directions in this crystal structure is not easy to visualise. The analysis of transmission electron micrographs obtained from hexagonal materials is based on a number of equations representing some important geometrical relationships between planes, directions, etc. These equations are first derived and then applied to particular problems.

A2.2 Crystallographic Relationships for the Hexagonal Lattice

As is well known, in the Miller–Bravais notation the hexagonal system is described by four axes, three of which are coplanar. The three coplanar axes, labelled a_1 , a_2 and a_3 , lie in the basal plane of the lattice and are 120° apart. The fourth axis is normal to this plane, and the right-hand rule applies for labelling the direction of the axes. The Miller–Bravais indices of a plane ($hkil$) are then the ratios of the reciprocals of the intercepts of the plane on the four axes (figure A2.1). If the intercepts on the axes a_1 , a_2 , a_3 are respectively a/h , a/k and $-a/i$ it follows from elementary geometry that

$$i = -(h + k) \quad (\text{A2.1})$$

One of the three coplanar axes is therefore strictly unnecessary, and in fact is included only to demonstrate the symmetry of the crystal system. In some cases ($hkil$) indexing is written as ($hk\cdot l$) where $i = \cdot$.

However, the labelling of directions is less obvious. Again, as in the cartesian representation of a direction in figure A1.3, the direction is represented as a line joining the origin of the coordinate system to a point in space, and the direction indices $[uvw]$ are the indices of the end point of the line. However, u , v and t now represent successive displacements parallel to a_1 , a_2 and a_3 and are chosen so that equality (A2.1) is satisfied. This produces a representation which is now non-cartesian as figure A2.2 demonstrates, and the t index is no longer a dummy. To obtain the equivalent components of the direction in the three-index system related to the non-coplanar axes a_1 , a_2 and c , where now the indices can be regarded as components of a vector in a skew three-space, we now set the unit vector along the axes as a , a and c , where a and c are the lattice spacings for the appropriate metal and c/a is the axial ratio. The magnitude of direction OR in the basal plane (figure A2.2) can be found by taking the square root of the scalar product of the vector

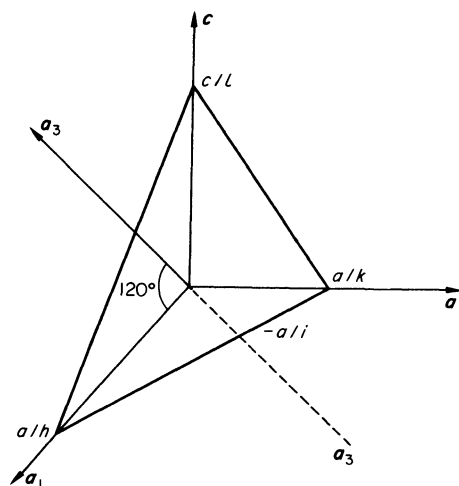


Figure A2.1 The Miller–Bravais notation for planes

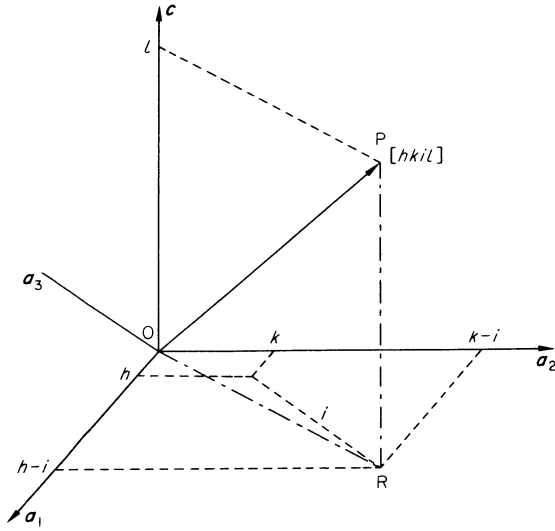


Figure A2.2 The Miller-Bravais notation for directions

specifying OR with itself and is given by

$$OR = \{3a^2(u^2 + v^2 + uv)\}^{1/2}$$

where a is the interatomic spacing in the basal plane. A distance OP (figure A2.2) that has a component out of the basal plane has a magnitude

$$OP = \{3a^2(u^2 + v^2 + uv) + c^2w^2\}^{1/2} \quad (A2.2)$$

A2.2.1 Angles between Two Directions, ϕ

The angle between two crystallographic directions is found by means of the cosine law

$$Z^2 = X^2 + Y^2 - 2XY \cos \phi$$

where X , Y and Z are magnitudes of vectors and ϕ is the angle between X and Y , as seen in figure A2.3.

Directions X and Y are known and Z can be found.* The magnitudes can be determined by the

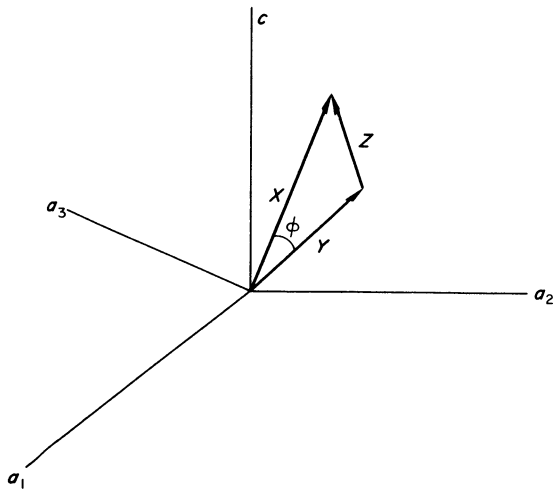


Figure A2.3 Angles between directions

* From equation (A2.1).

formula for distance. The cosine of the angle between X and Y is given by

$$\cos \phi = \frac{Dd + Ee + \frac{1}{2}(De + Ed) + \frac{1}{3}Gg(c/a)^2}{\{D^2 + E^2 + DE + (G^2/3)(c/a)^2\}^{1/2} \times \{d^2 + e^2 + de + (g^2/3)(c/a)^2\}^{1/2}} \quad (A2.3)$$

where $X = [DEFG]$ and $Y = [defg]$.

A2.2.2 Indices $[defg]$ of the Normal to the Plane $(hkil)$

The direction $[d, e, f, g]$ perpendicular to plane (h, k, i, l) can be found by forming the scalar product of the vector representing d, e, f, g with two vectors in (h, k, i, l) (figure A2.4).

Direction $[d, e, f, g]$ intersects (h, k, i, l) at a point which is some multiple n times ad, ae, af and cg . Since the intercepts of the plane with the axes are known, vectors AX , BX and CX can be found, for example

$$AX = (2nda + nea - a/h)a_1 + (2nea + nda)a_2 + ngc c$$

The direction d, e, f, g will have components such that

$$OX = (2nda + nea)a_1 + (2nea + nda)a_2 + ngc c$$

Forming the scalar product of AX and OX and setting it equal to zero, since the two vectors are perpendicular to each other, gives

$$OX \cdot AX = 4(nda) + 4(nea) + \frac{1}{2}(nda)(nea) - \frac{3}{2} \frac{(nda)a}{h} + (ngc)^2 = 0$$

Analogous equations can be formed for BX and CX .

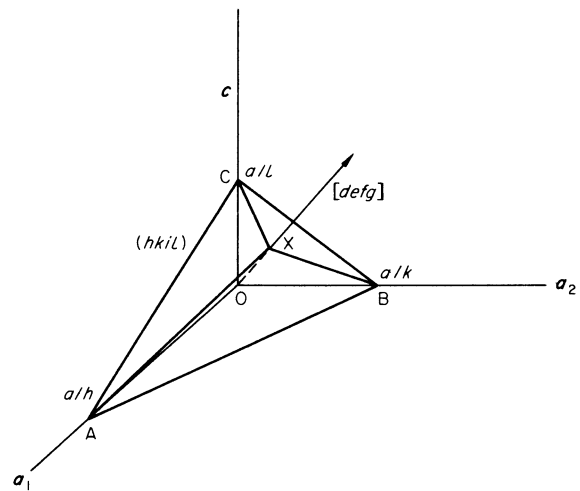


Figure A2.4 Indices of a plane normal

One of the indices, d, e, f, g , may be arbitrarily chosen. This is usually done in such a way as to make the indices the smallest integer values. The index d will be set equal to h .

The normal to the plane (h, k, i, l) will have indices

$$[defg] = h, k, i, \frac{3}{2}(a/c)^2 l \quad (\text{A2.4})$$

A2.2.3 Directions $[wxyz]$ Lying in a Plane ($hkil$)

This expression is easily calculated by taking the scalar product of $[wxyz]$ with the plane normal and setting equal to zero.

The condition becomes

$$uh + vk + ti + wl = 0 \quad (\text{A2.5})$$

This expression can also be used to calculate the planes containing a given direction. While it seems that relations (A2.4) and (A2.5) appear to require knowledge of each other, this is not in fact so. Three directions lying in a plane can immediately be constructed from the knowledge of the intercepts made by the plane on the axes.

A2.2.4 Angle ϕ between Two Planes

The angle between two planes is the same as the angle between their normals, so combining (A2.3) and (A2.4) the cosine of the angle ϕ between two planes ($hkil$) and ($defg$) is given by

$$\cos \phi = \frac{hd + ke + \frac{1}{2}(he + kd) + \frac{3}{4}lg(a/c)^2}{\{h^2 + k^2 + hk + \frac{3}{4}l^2(a/c)^2\}^{1/2} \times \{d^2 + e^2 + de + \frac{3}{4}g^2(a/c)^2\}^{1/2}} \quad (\text{A2.6})$$

This is identical with the expression (calculated by using different techniques) given in standard crystallographic texts. The angle between a direction and a plane can similarly be calculated by using (A2.3) and (A2.4).

A2.2.5 Direction of the Intersection of Two Planes

This is easily calculated by applying (A2.5) to both planes: the direction $[wxyz]$ which satisfies (A2.5) for both planes is plainly the direction of their line of intersection.

A2.3 Stereographic Manipulations in the Hexagonal Lattice

A number of geometrical calculations such as those outlined above can be performed on a double stereogram on which are represented both the poles of planes and crystallographic directions. Of course, it is necessary to orient the plane and direction projections relative to one another, and in the hexagonal system since $[0001]$ is normal to (0001) and $[hki0]$ is normal to $(hki0)$, it is easiest

to use a standard basal projection with the direction and plane indices coincident at the centre and the rim. In effect such a stereogram is a chart for transforming from one index system to another and, viewed in this light, is quite general. The concept of the double stereogram can be applied to any crystal system. A general point in such a stereogram may be regarded as a plane (properly, the projection of the pole of the plane) or as a direction and indexed accordingly. It follows therefore that the projection of a plane and the direction normal to it coincide, as usual, but they have different indices.

To determine Burgers' vectors, slip planes, and the geometry of dislocation interactions, it is necessary to perform a small number of simple operations using the double stereogram. In general geometrical operations are performed in the foil plane, which is determined from a diffraction pattern as described below.

A2.3.1 Indexing Diffraction Patterns

As pointed out in section 2.2.2.1, each spot in the diffraction pattern corresponds to a set of planes, almost parallel to the electron beam. The spots are indexed ($hkil$) using the procedure in section 2.7.2, but substituting relation (A2.6) for (2.22). Vector addition may be used to simplify indexing of the complete pattern, once the initial indexing of two spots has been accomplished. The zone axis $[uvw]$ of the pattern may be obtained from the relations

$$\left. \begin{aligned} u &= l_2(2k_1 + h_1) - l_1(2k_2 + h_2) \\ v &= l_1(2h_2 + k_1) - l_2(2h_1 + k_2) \\ w &= 3(h_1k_2 - h_2k_1) \\ t &= -(u + v) \end{aligned} \right\} \quad (\text{A2.7})$$

where the indexed spot ($h_2k_2l_2$) is positioned anticlockwise relative to ($h_1k_1l_1$). As an alternative to relations (A2.7), relation (2.23a) may be used with the Miller ($hkil$) indices to give notional values for u', v', w' which may be converted to the correct u, v, t, w by the relations

$$\left. \begin{aligned} u &= \frac{1}{3}(2u' - v') \\ v &= \frac{1}{3}(2v' - u') \\ t &= -\frac{1}{3}(v' + u') \\ w &= w' \end{aligned} \right\} \quad (\text{A2.8})$$

All spots in the pattern must satisfy relation (A2.5).

Having determined the beam direction B , it may be required to determine the foil plane (if zero specimen tilt). Unlike the cubic case, the plane normal to B does not have the same indices as B .

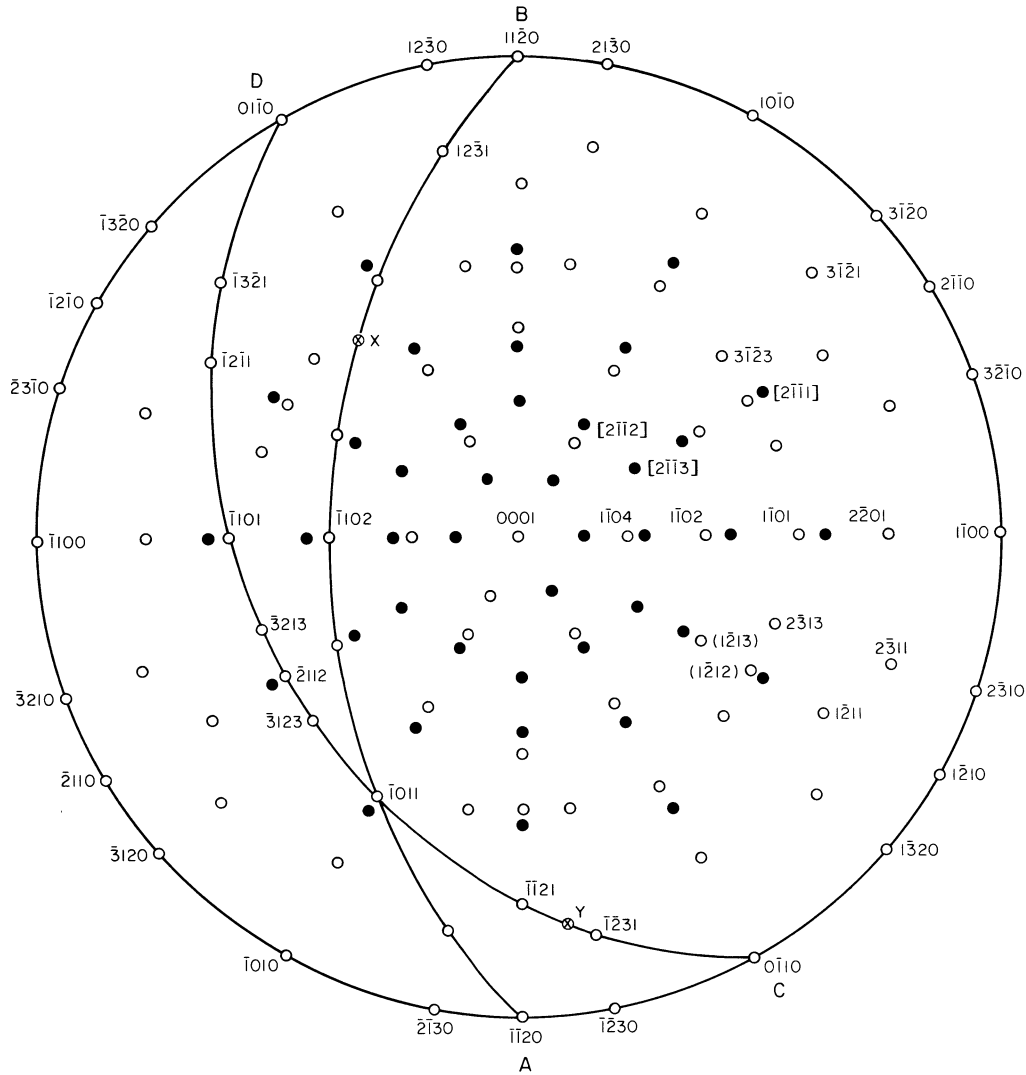


Figure A2.5 A double stereogram for axial ratio: $c/a = 1.62$; ○ planes; ● directions. For all $hki0$, and 0001 , directions and planes superimpose. These are shown as open circles for clarity

The indices of the plane normal to $\mathbf{B}$ are given by

$$\left. \begin{aligned} h &= u \\ k &= v \\ l &= \frac{2}{3}(c/a)^2 w \\ i &= -(h+k) \end{aligned} \right\} \quad (\text{A2.9})$$

and

The zone of reflecting planes may be represented on the stereogram by a great circle passing through the poles corresponding to the planes giving rise to the reflections. A typical example is shown as the great circle AB in figure A2.5. The zone axis is then the direction which lies in all the planes, the pole of the great circle. In this case, the zone axis is $[1\bar{1}01]$, and the foil plane is the plane normal to this. As can be seen, this foil plane has non-rational indices: it is fairly close to $(4\bar{4}07)$; using relation (A2.9) the indices may be shown to be

(1, $\bar{1}$, 0, 1.679). The use of the double stereogram will be demonstrated in section A2.3.4 by using this foil plane as an example.

A2.3.2 Planes Containing a Given Direction

A contrast experiment performed on a single dislocation gives a Burgers vector, but no slip plane. It is frequently instructive to know which planes contain this direction. For example, dislocations having a Burgers vector parallel to the prism face diagonals have occasionally been reported: these directions have indices $\langle 11\bar{2}3 \rangle$. For the particular case of directions $[2\bar{1}13]$, the expression (A2.5) indicates that planes $(hkil)$ contain this direction, where

$$h + l = 0 \quad (\text{A2.10})$$

Since $[2\bar{1}\bar{1}3]$ lies in all those planes whose poles

are normal to it, the locus of these planes is the great circle drawn with $[2\bar{1}\bar{1}3]$ as a pole. This is shown as CD in figure A2.5, and it can be seen that in fact the indices of the planes satisfy relation (A2.10). As an extension of this, the direction of the intersection of two planes is the pole of the great circle drawn through the two planes.

A2.3.3 Contrast Experiments

It is well known, see section 3.4.7, that the Burgers vector $\mathbf{b}$ of dislocations can be determined by tilting the foil to produce images under two-beam conditions with various values of $\mathbf{g} \cdot \mathbf{b}$, where $\mathbf{g}$ is the operative reflection vector, see section 2.4. If isotropic elasticity can be applied, when $\mathbf{g} \cdot \mathbf{b} = 0$ contrast disappears. However, if the material is elastically anisotropic, contrast will not disappear and, as described in section 3, image matching techniques must be used to determine the Burgers vector of dislocations. Nevertheless it is often useful to estimate quickly from a stereogram under which reflecting conditions $\mathbf{g} \cdot \mathbf{b} = 0$ to assess what tilting and contrast experiments are worthwhile on a particular specimen. Geometrically, $\mathbf{g} \cdot \mathbf{b} = 0$ is equivalent to the statement that the direction of the Burgers vector must lie in the plane responsible for the operating reflection. Consider the case of a foil giving the diffraction pattern represented by the great circle AB (figure A2.5). Plainly, the $(\bar{1}011)$ plane contains the $[2\bar{1}\bar{1}3]$ direction, and thus any dislocations having a Burgers vector parallel to this will go out of contrast when the foil is tilted so that this reflection is the only one operating. Similarly, the $(\bar{1}102)$ plane contains the $[11\bar{2}0]$ direction, and dislocations for which $\mathbf{b} = (a/3)[11\bar{2}0]$ will be out of contrast for this operating reflection. Conversely, if a set of dislocations are observed to go out of contrast for a particular reflection, the Burgers vector must lie parallel to a direction lying on the great circle drawn with the reflecting plane as a pole.

A2.3.4 Dislocation Geometry—Projection of Directions

In some cases, even if contrast experiments cannot be performed, some indication of the Burgers vector can be obtained from the geometry of the dislocation arrangement. In many instances, dislocations tend to be either edge or screw and thus lie normal to or parallel to the Burgers vector in the slip plane. Consider, for example, slip on the $(\bar{1}101)$ pyramidal plane. This plane contains three possible Burgers' vectors, $(a/3)[11\bar{2}0]$,

$\{(c^2 + a^2)^{1/2}/3\}[2\bar{1}\bar{1}3]$ and $\{(c^2 + a^2)^{1/2}/3\}[\bar{1}213]$, as may be determined by drawing the great circle with $(\bar{1}101)$ as a pole. Geometrically, the projection of a direction into the foil plane is the trace of a plane normal to the foil plane containing the required direction: thus, the projection of $[2\bar{1}\bar{1}3]$ in $(4\bar{4}07)$ is the trace of $(\bar{1}011)$. The direction of this trace is the pole of the great circle passing through $(\bar{1}101)$ and $(4\bar{4}07)$, shown as X in figure A2.5. The angle between the direction and its projection (which gives its apparent length in the foil plane) is the complement of the angle between the directions and the normal to the foil plane, in this case $[1\bar{1}01]$. For $[2\bar{1}\bar{1}3]$ this angle is $90 - 24 = 66^\circ$. The direction in $(\bar{1}101)$ normal to $[2\bar{1}\bar{1}3]$ is the pole of the great circle drawn through these two points, labelled Y in figure A2.5. This may be projected into the foil plane in exactly the same way. It is thus easy to prepare a map of projected edge and screw directions and projected lengths for the possible slip systems, which may then be compared with the dislocation images. In general, this is rather easier than attempting to determine the direction of the dislocation line directly, although this can be done by exactly the same method.

A2.4 Crystallographic Data for the Hexagonal Lattice

To plot a double stereogram for a hexagonal material it is necessary to know both the angles between planes and the angles between directions for the given c/a ratio. These values have been tabulated (Rarey *et al.*, 1966) and computer programmes are available which produce values (Johari and Thomas, 1970) and plot stereograms (Metzbower, 1969). Formulae for crystallographic relationships have also been given by Otte and Crocker (1965, 1966).

Appendix 2: References

- Johari, O., and Thomas, G. (1970). The stereographic projection and its applications. *Techniques in Metals Research* (ed. R. F. Bunsah), vol. 2C, Wiley, New York.
- Metzbower, E. A. (1969). *Trans. A.I.M.E.*, **245**, 435.
- Otte, H. M., and Crocker, A. G. (1965). *Phys. Stat. Sol.*, **9**, 441.
- (1966). *Phys. Stat. Sol.*, **16**, K25.
- Rarey, C. R., Stringer, J., and Edington, J. W. (1966). *Trans. A.I.M.E.*, **236**, 811.

APPENDIX 4.

STANDARD SPOT PATTERNS

This appendix includes diagrams of standard spot diffraction patterns for both cubic and hexagonal crystal structures. In the cubic cases each pattern for a given zone axis (z , defined in appendix 1) has the same, six-, four-, three- or two-fold symmetry but the reflections that occur obey the rules outlined in table 2.1. The patterns for the hexagonal crystal structure are indexed using the Miller–Bravais system outlined in appendix 2, assuming exact close packing, that is an axial ratio (c/a) of 1.633. The positions of the spots will change when the c/a changes. It is recommended that standard patterns be constructed for the c/a value corresponding to the actual material studied

using the tables of Rarey, Stringer and Edington (1966). In figures A4.1–A4.4, the indexing procedure outlined in section 2.7.2.1 has been followed.

The positions of superlattice reflections are also shown in figures A4.1 and A4.2. The intensities of the spots will depend upon their structure factor as described in section 2.3.1. The reflections that occur in cubic crystal structures are shown in tables A4.1 and A4.2.

Appendix 4: Reference

Rarey, C. R., Stringer, J., and Edington, J. W. (1966). *Trans. A.I.M.E.*, **236**, 811.

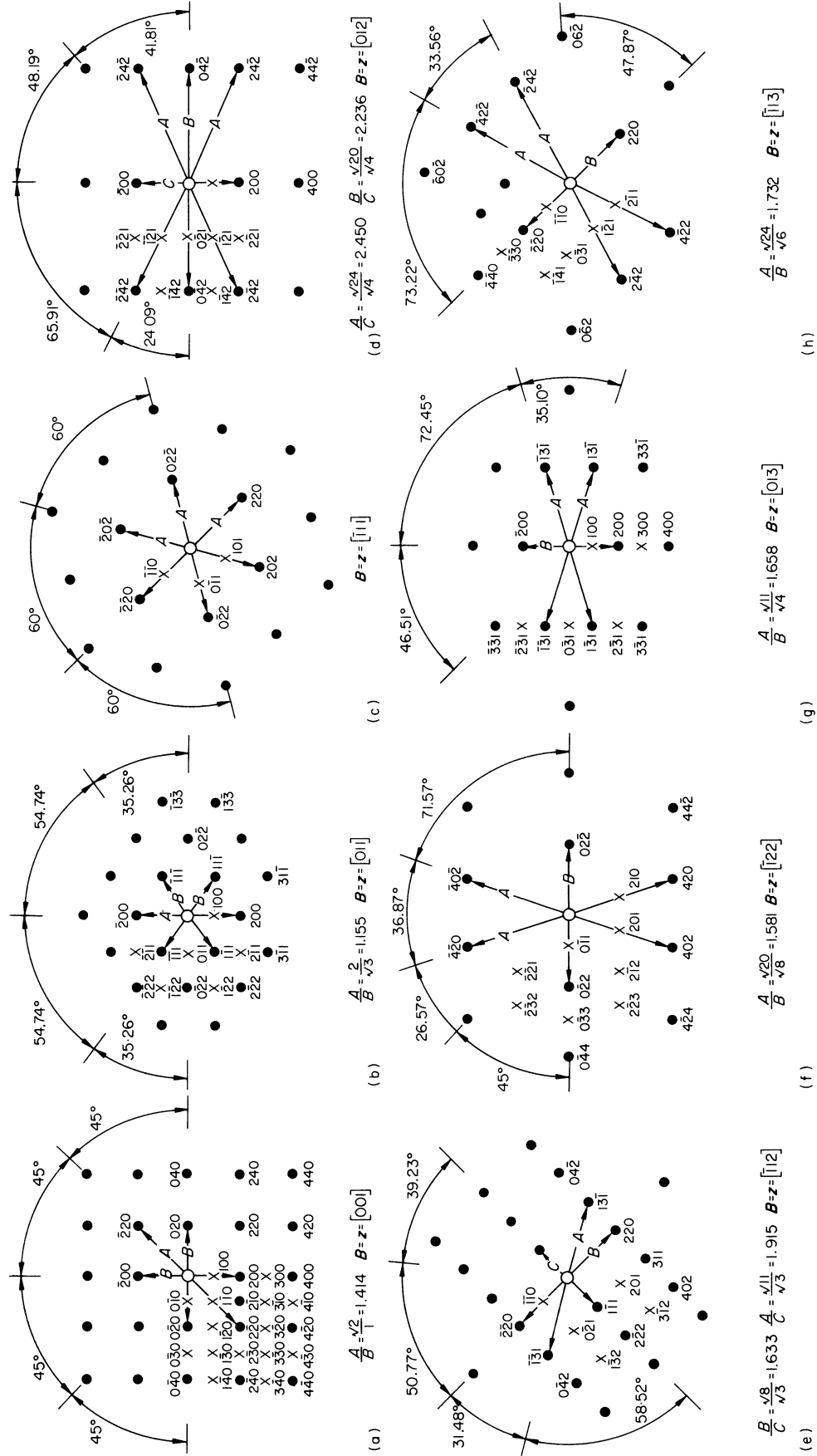


Figure A4.1 Single-crystal spot transmission electron diffraction patterns for the f.c.c. crystal structure ($u^2 + v^2 + w^2 < 22$). The zone axis z , defined in appendix 1, is the beam direction B defined in section 2.7.2, as indicated. The crosses in one quadrant of the diagram indicate the positions of the spots for the ordered f.c.c. (L1₂) unit cell. The complete pattern may be generated by repeating these spots in the remainder of the pattern and indexing using the addition of vectors, see section 2.7.2.1

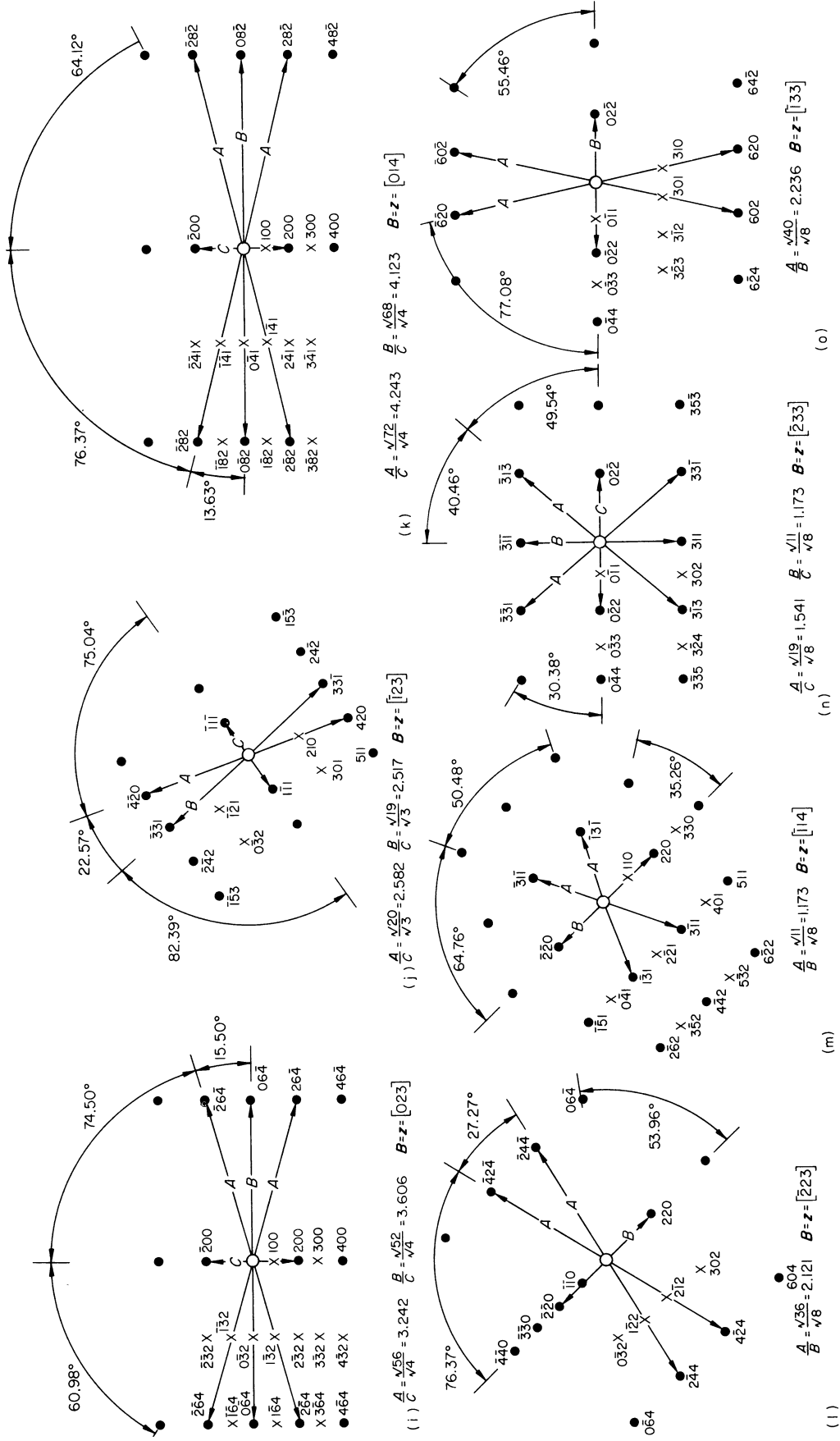


Figure A4.1 (continued)

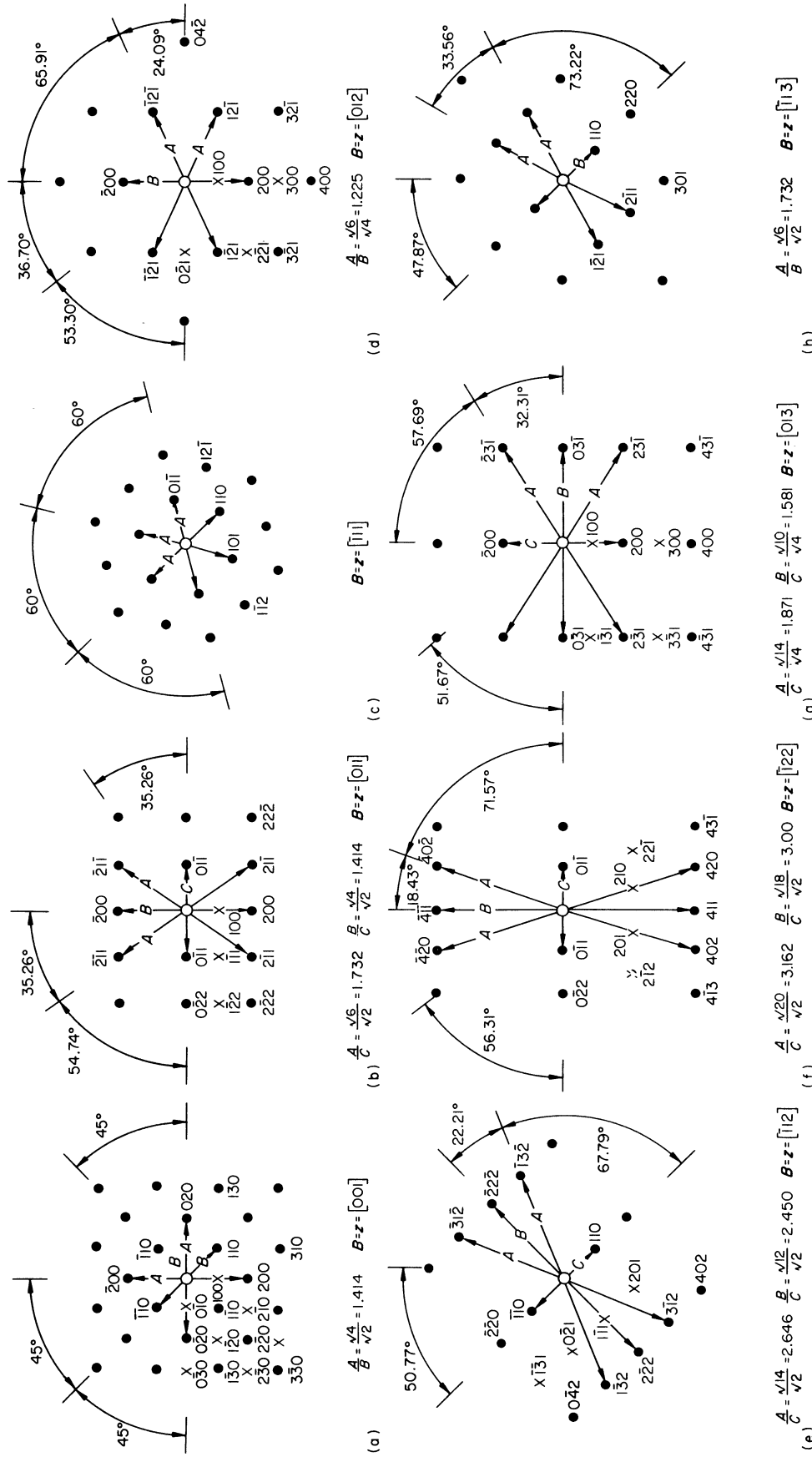


Figure A4.2 Single-crystal spot transmission electron diffraction patterns for the b.c.c. crystal structure ($u^2 + v^2 + w^2 \leq 22$). The zone axis z , defined in appendix 1, is the beam direction B , defined in section 2.7.2, as indicated. The crosses in one quadrant of the diagram indicate the positions of the spots for the ordered b.c.c. (B_2) unit cell. The complete pattern may be generated by repeating these spots in the remainder of the pattern and indexing using the addition of vectors, see section 2.7.2.1

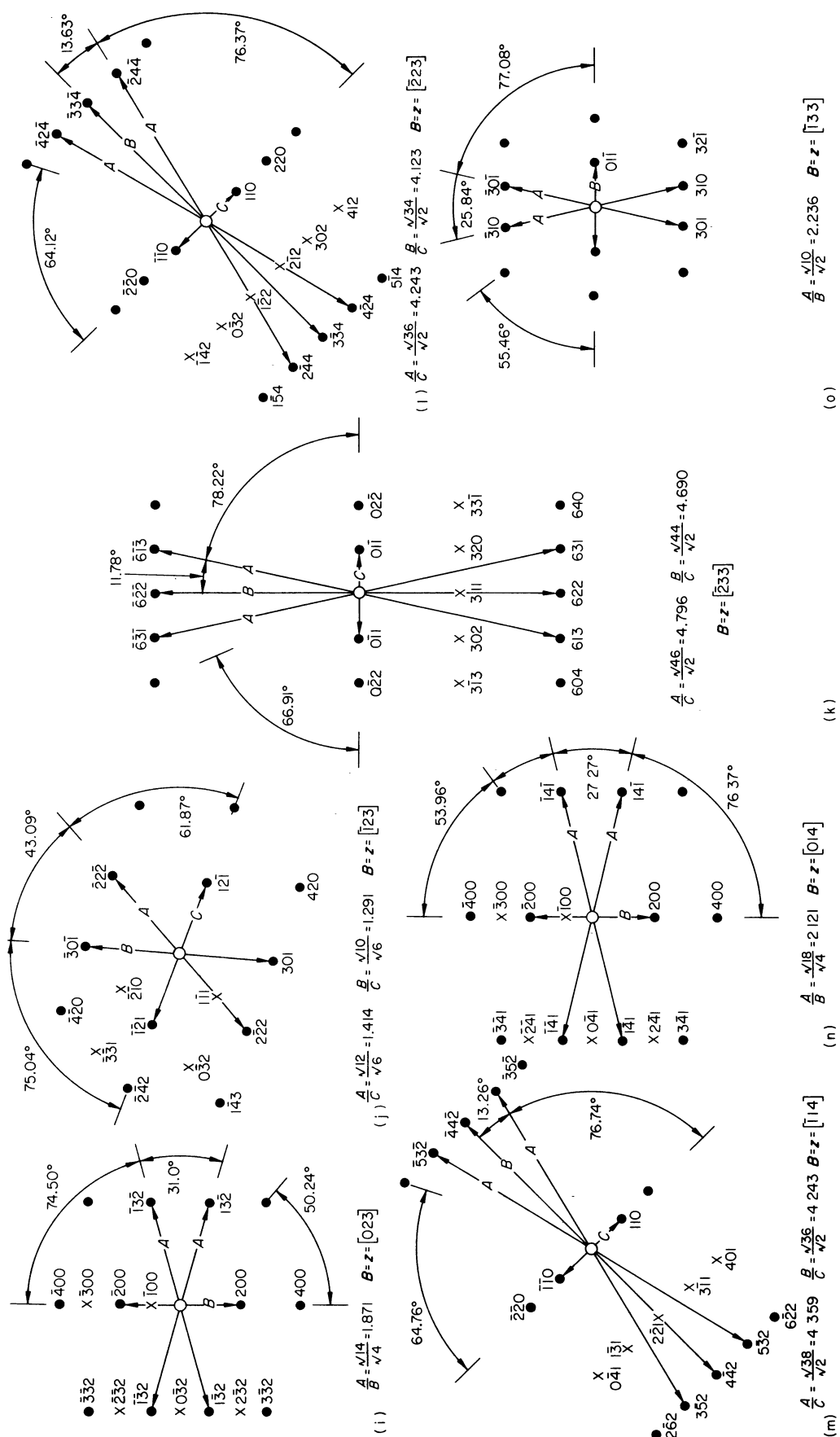


Figure A4.2 (continued)

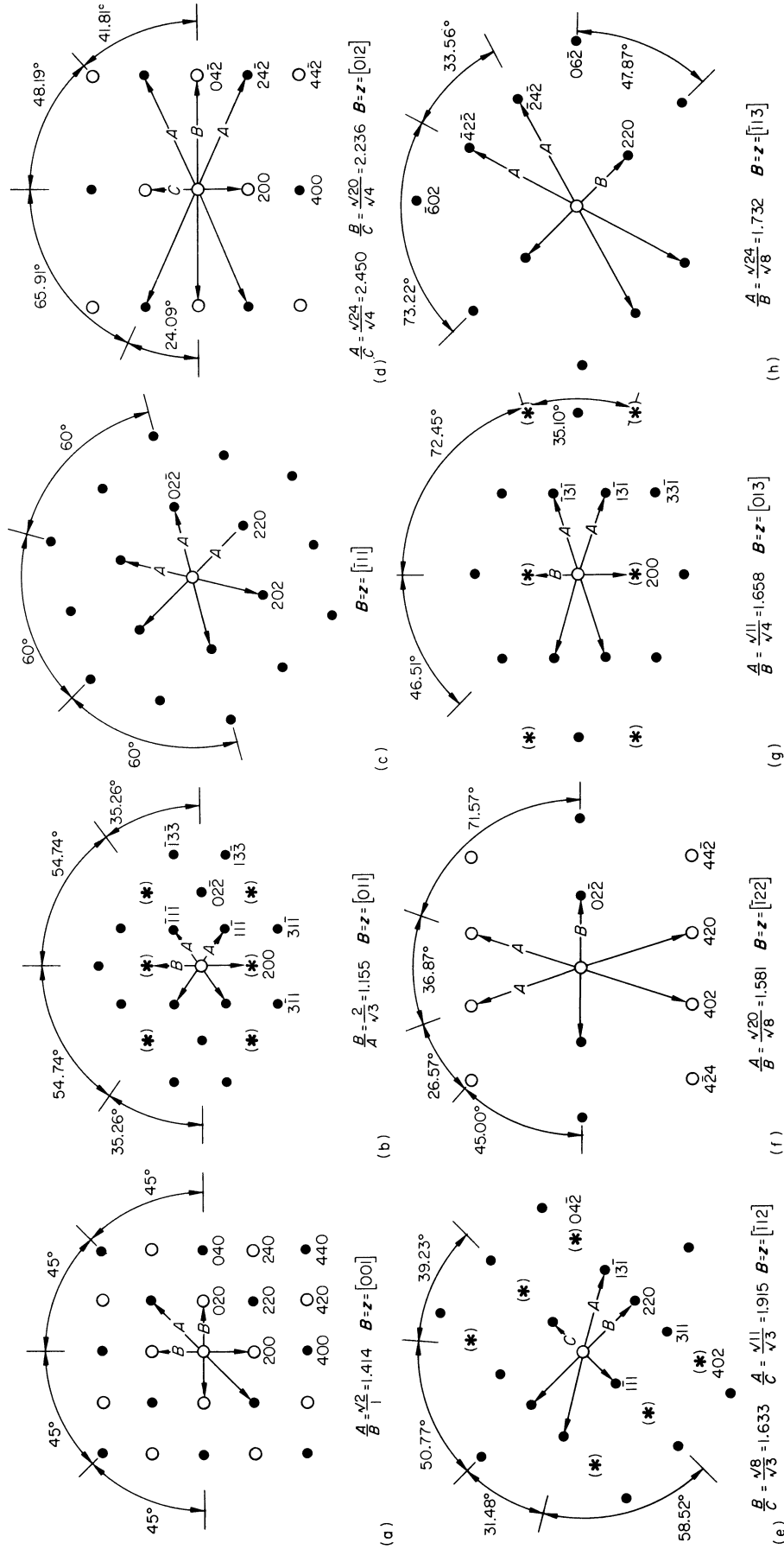


Figure A4.3 Single-crystal spot transmission electron diffraction patterns for diamond structures ($u^2 + v^2 \leq 22$): O additional weaker spots; * spots that could arise from double diffraction. The zone axis z , defined in appendix 1, is the beam direction B , defined in section 2.7.2, as indicated [After Andrews *et al.* (1971)]

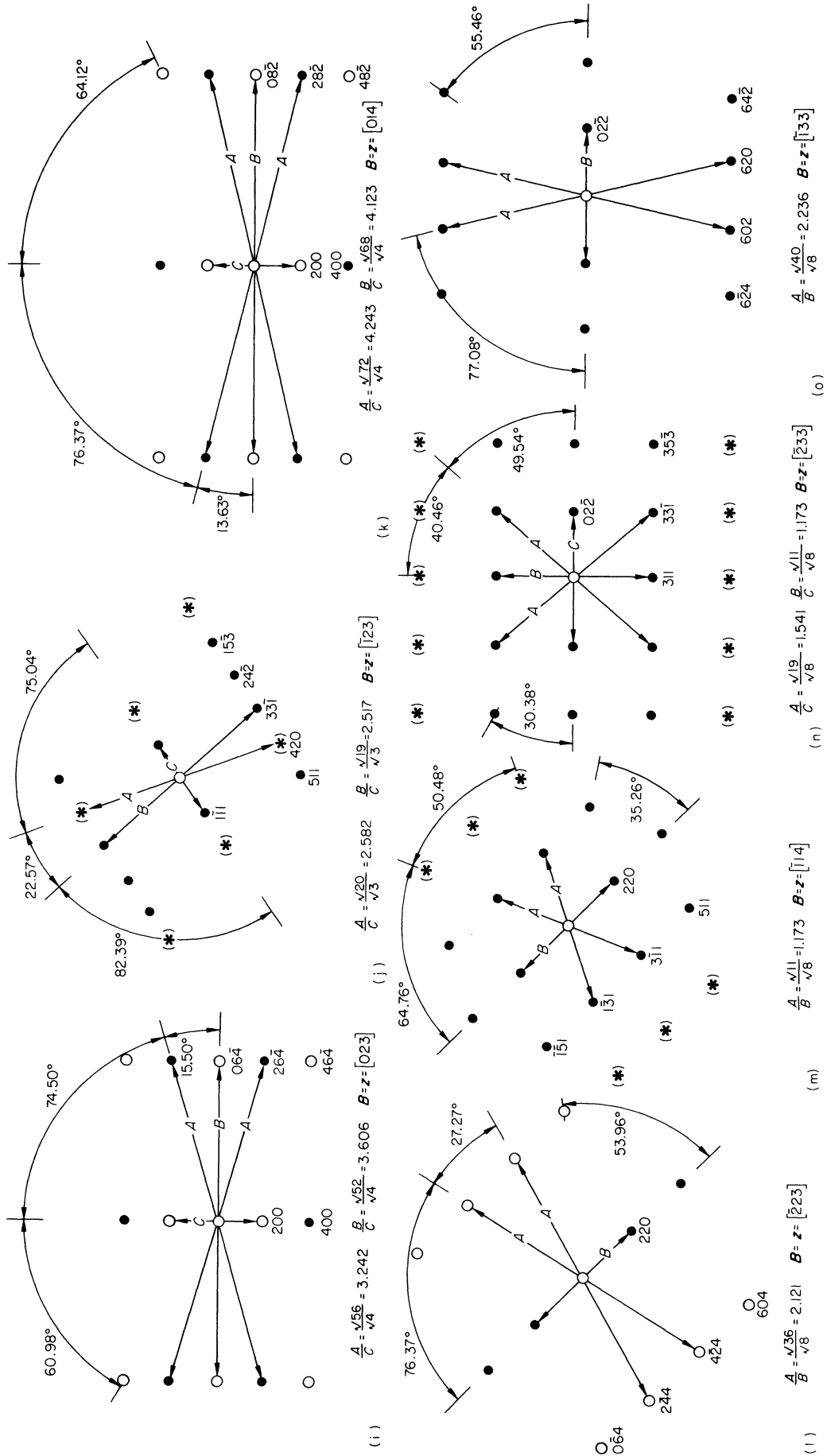


Figure A4.3 (continued)

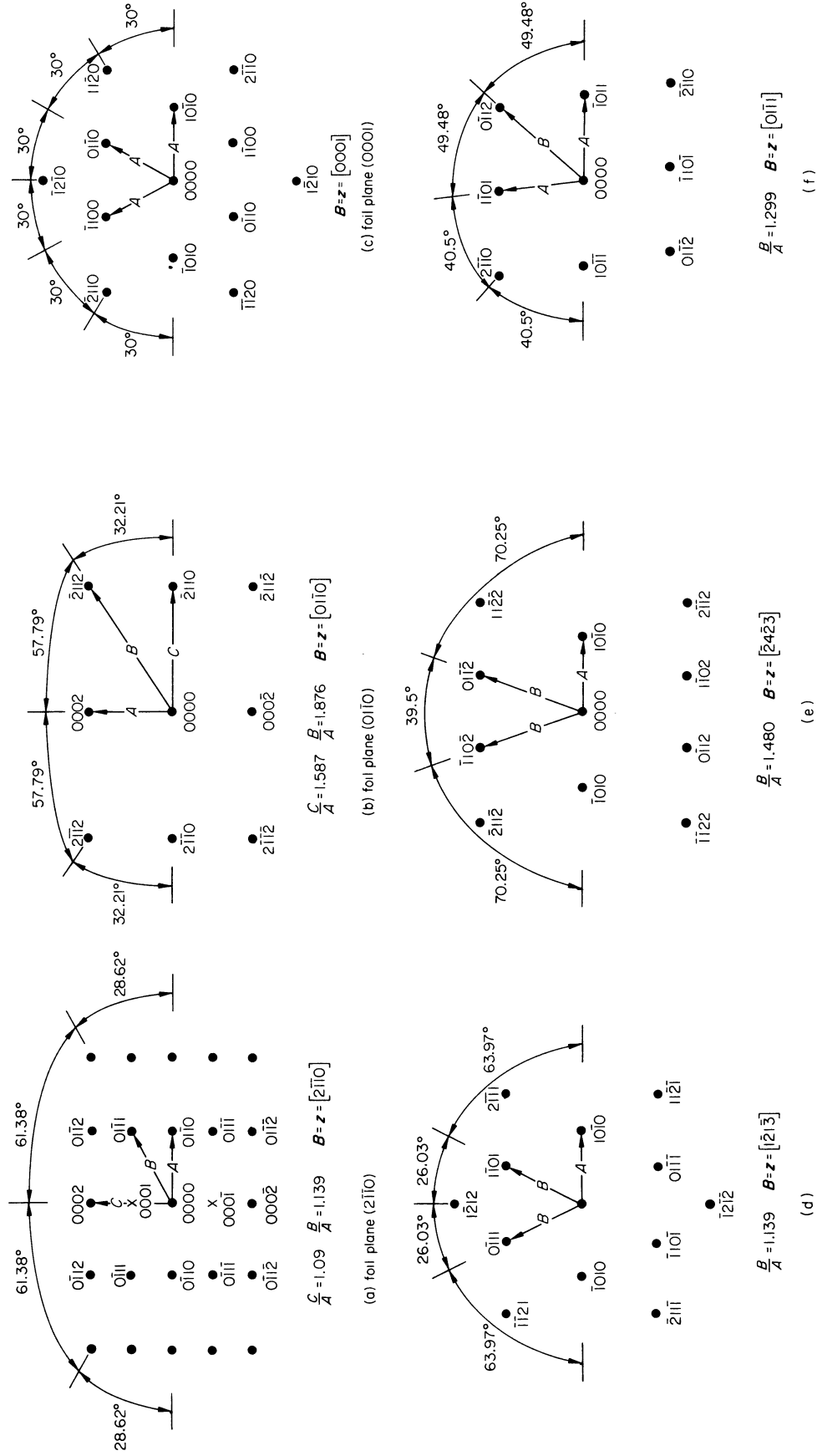


Figure A4.4 Single-crystal spot transmission electron diffraction patterns for the c.p.h. crystal structure. The zone axis, z , defined in appendix 1, is the beam direction B , defined in section 2.7.2. The crosses indicate the positions of reflections forbidden by the structure factor but occurring in the pattern by double diffraction

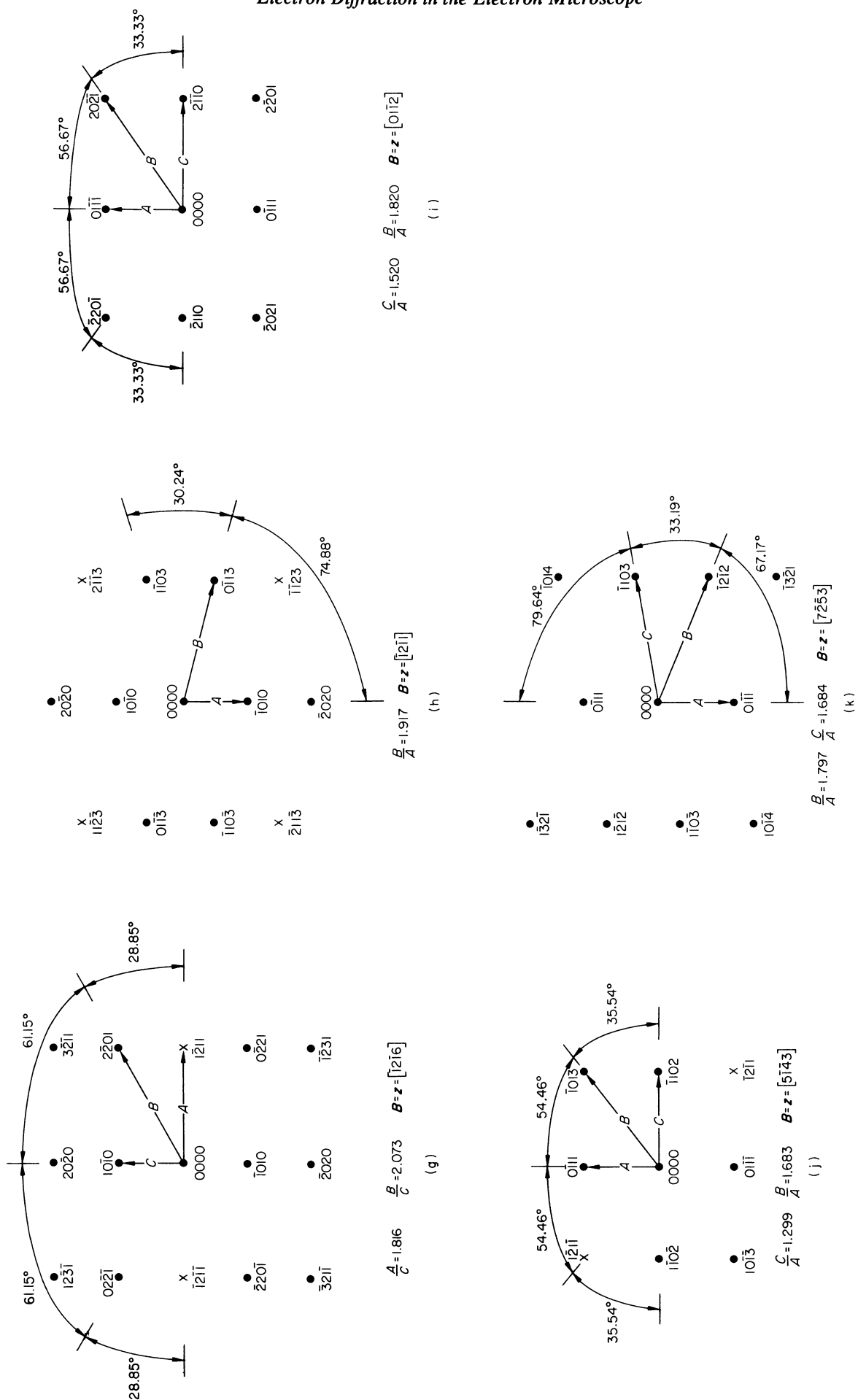


Figure A4.4 (continued)

Table A4.1 Occurrence of reflections for the cubic crystal structures

Line no. $N =$ $h^2 + k^2 + l^2$	hkl indices	$N^{1/2} =$ $(h^2 + k^2 + l^2)^{1/2}$	f.c.c. diamond			Line no. $N =$ $h^2 + k^2 + l^2$	hkl indices	$N^{1/2} =$ $(h^2 + k^2 + l^2)^{1/2}$	b.c.c.	f.c.c.	diamond
1	100	1.00				33	522, 441	5.745			
2	110	1.414	×			34	530, 433	5.831	×		
3	111	1.732		×	×	35	531	5.916		×	×
4	200	2.00	×	×	×	36	600, 442	6.00	×	×	×
5	210	2.236				37	610	6.083			
6	211	2.450	×			38	611, 532	6.164	×		
7	—	—				39	—	—			
8	220	2.828	×	×	×	40	620	6.325	×	×	×
9	300, 221	3.00				41	621, 540, 443	6.403			
10	310	3.162	×			42	541	6.481	×		
11	311	3.317		×	×	43	533	6.557		×	×
12	222	3.464	×	×		44	622	6.633	×	×	×
13	320	3.606				45	630, 542	6.708			
14	321	3.742	×			46	631	6.782	×		
15	—	—				47	—	—			
16	400	4.00	×	×	×	48	444	6.928	×	×	
17	410, 322	4.123				49	700, 632	7.00			
18	411, 330	4.243	×			50	710, 550, 543	7.071	×		
19	331	4.359		×	×	51	711, 551	7.141		×	×
20	420	4.472	×	×	×	52	640	7.211	×	×	
21	421	4.583				53	720, 641	7.280			
22	332	4.690	×			54	721, 633, 552	7.349	×		
23	—	—				55	—	—			
24	422	4.899	×	×	×	56	642	7.483	×	×	×
25	500, 430	5.00				57	722, 544	7.550			
26	510, 431	5.099	×			58	730	7.616	×		
27	511, 333	5.196		×	×	59	731, 553	7.681		×	×
28	—	—				60	—	—			
29	520, 432	5.385				61	650, 643	7.810			
30	521	5.477	×			62	732, 651	7.874	×		
31	—	—				63	—	—			
32	440	5.657	×	×	×	64	800	8.00	×	×	×

Table A4.2 Occurrence of reflections for the c.p.h. crystal structure

Common allowed reflections			Common forbidden reflections (sometimes these occur by double diffraction, depending upon B)
10 $\bar{1}$ 0	3 $\bar{2}$ 11	20 $\bar{2}$ 3	0001
1 $\bar{2}$ 10	2 $\bar{2}$ 01	24 $\bar{2}$ 3	0003
20 $\bar{2}$ 0	211 $\bar{2}$	10 $\bar{1}$ 4	1 $\bar{2}$ 11
1 $\bar{2}$ 30	01 $\bar{1}$ 2	2114	1 $\bar{2}$ 13
30 $\bar{3}$ 0	0002	10 $\bar{1}$ 5	3033
1340	11 $\bar{2}$ 2	1 $\bar{2}$ 16	1 $\bar{2}$ 15
5140	213 $\bar{2}$		
01 $\bar{1}$ 1	10 $\bar{1}$ 3		

APPENDIX 5. KIKUCHI MAPS

As described in section 2.7.3.3, a Kikuchi map consists of the distribution of Kikuchi lines within the unit stereographic triangle. It is extremely

useful in a number of instances, sections 2.8.1, 2.10.1, 2.11.2, 2.14.2. Typical maps for f.c.c., b.c.c. and c.p.h. crystal structures are shown in

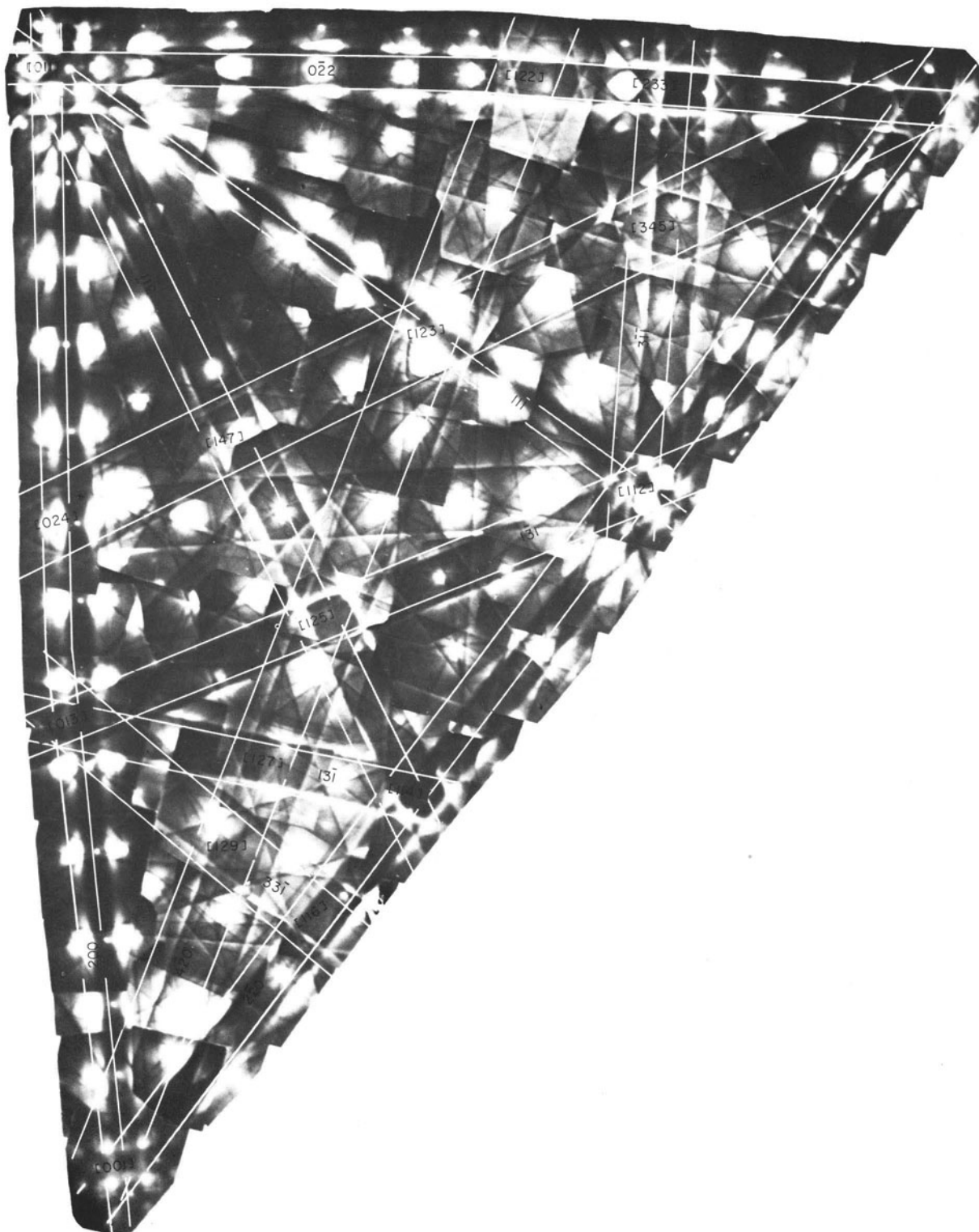


Figure A5.1 An indexed Kikuchi map for the f.c.c. crystal structure; 100 kV. The indices in square brackets are those of the beam direction B for the particular Kikuchi line pattern. The unbracketed indices are those for the Kikuchi line pair
[Courtesy of A. Samuelson]

figures A5.1 to A5.3. These may be made by tilting the specimen to sample *B*, for example $[001]$, $[111]$, $[011]$, etc., then obtaining photographs of

successive overlapping SADPs by tilting about specific reflections as outlined in section 2.10.1 so that the complete stereographic triangle is covered.

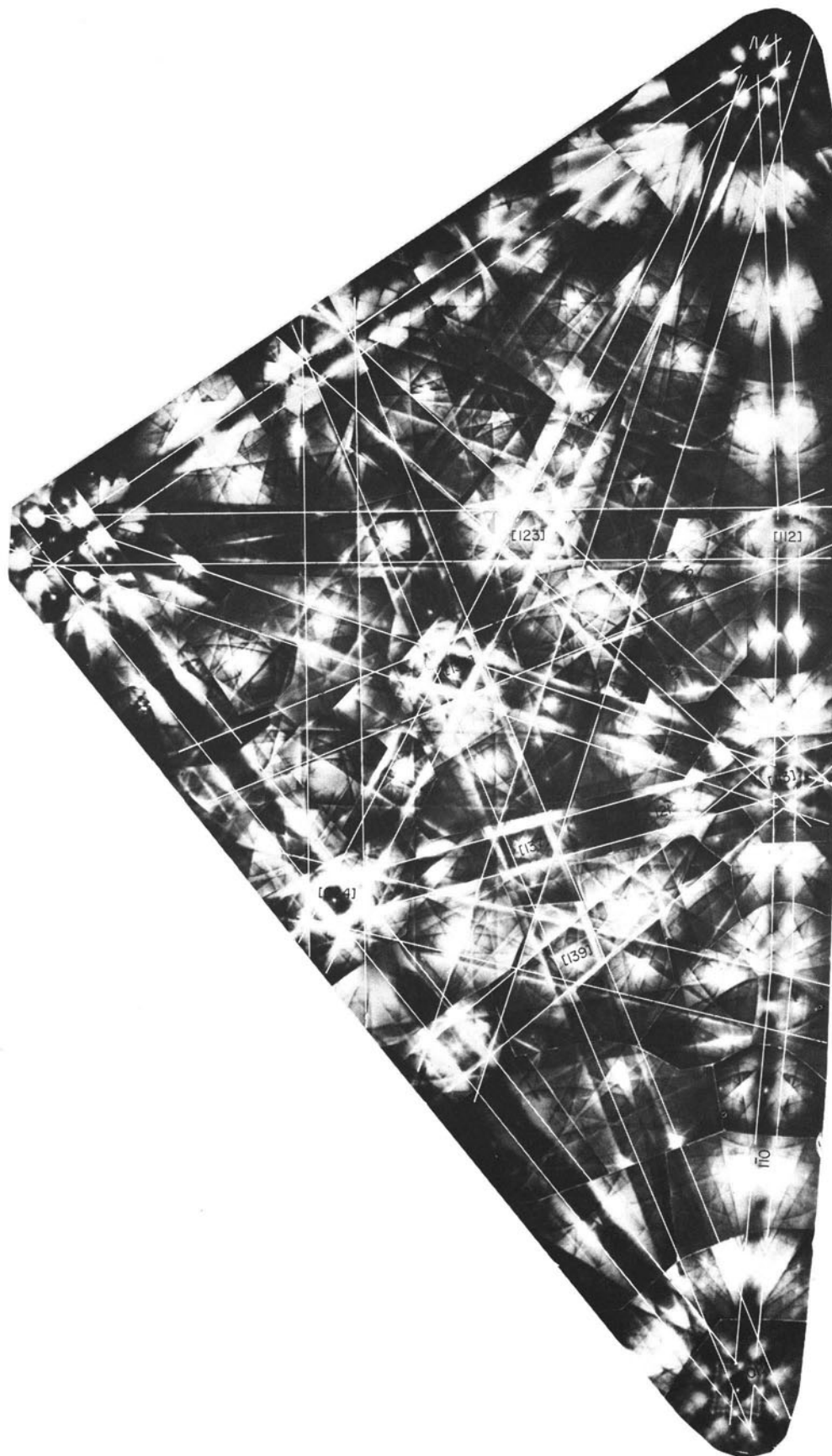
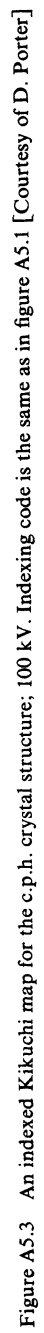


Figure A5.2 An indexed Kikuchi map for the b.c.c. crystal structure; 100 kV. Indexing code is the same as in figure A5.1
[Courtesy of G. Sawyer]



The pictures may then be fitted together over an enlarged unit triangle of the stereogram such that the Kikuchi lines join. If the map is fitted directly to the unit triangle, it will be found that matching of the Kikuchi lines will not be perfect because a constant camera length is used whereas the scale of the stereographic projection varies from point to point. Furthermore the Kikuchi lines are not straight, see section 2.6.3. However, the maximum error which occurs is $\sim \pm 2^\circ$. For maximum accuracy, it is advised that Kikuchi maps be constructed so that the Kikuchi lines join and the

map is slightly distorted, that is it does not fit the unit triangle directly. Nevertheless the position of a particular Kikuchi pattern may be determined to $\sim 1^\circ$ by locating it on the map and measuring distances from three important zone axes, for example $[110]$, $[112]$ and $[111]$. A map should be constructed by each research worker because it creates familiarity with the specimen tilting procedures.

In figures A5.1 to A5.3 prominent zone axes [] are indexed together with important Kikuchi line pairs ().

APPENDIX 6. INTERPLANAR ANGLES AND SPACINGS OF SELECTED MATERIALS

Tables A6.1–A6.6 list interplanar spacings and angles for various materials. Further information may be obtained in Andrews *et al.* (1971). For interplanar angles, the specific combinations of indices for the two planes may be obtained with the equation for $\cos \phi$, table A1.1, for the cubic crystal structure.

Appendix 6: Reference

Andrews, K. W., Dyson, D. J., and Keown, S. R. (1971). *Interpretation of Electron Diffraction Patterns*, Hilger-Watts, London.
Barrett, C. S. (1971). *Structure of Metals*, McGraw-Hill, New York.

Table A6.1 Angles between crystallographic planes (and between crystallographic directions) in crystals of the cubic system [After Barrett (1971)]

{ <i>HKL</i> }	{ <i>hkl</i> }	Values of angles between <i>HKL</i> and <i>hkl</i> planes (or directions)					
100	100	0°	90°				
	110	45°	90°				
	111	54°44'					
	210	26°34'	63°26'	90°			
	211	35°16'	65°54'				
	221	48°11'	70°32'				
	310	18°26'	71°34'	90°			
	311	25°14'	72°27'				
	320	33°41'	56°19'	90°			
	321	36°42'	57°41'	74°30'			
110	110	0°	60°	90°			
	111	35°16'	90°				
	210	18°26'	50°46'	71°34'			
	211	30°	54°44'	73°13'	90°		
	221	19°28'	45°	76°22'	90°		
	310	26°34'	47°52'	63°26'	77°5'		
	311	31°29'	64°46'	90°			
	320	11°19'	53°58'	66°54'	78°41'		
	321	19°6'	40°54'	55°28'	67°48'	79°6'	
111	111	0°	70°32'				
	210	39°14'	75°2'				
	211	19°28'	61°52'	90°			
	221	15°48'	54°44'	78°54'			
	310	43°6'	68°35'				
	311	29°30'	58°31'	79°59'			
	320	36°49'	80°47'				
	321	22°12'	51°53'	72°1'	90°		
210	210	0°	36°52'	53°8'	66°25'	78°28'	90°
	211	24°6'	43°5'	56°47'	79°29'	90°	
	221	26°34'	41°49'	53°24'	63°26'	72°39'	90°
	310	8°8'	31°57'	45°	64°54'	73°34'	81°52'
	311	19°17'	47°36'	66°8'	82°15'		
	320	7°7'	29°45'	41°55'	60°15'	68°9'	75°38'
	321	17°1'	33°13'	53°18'	61°26'	68°59'	83°8'
							90°
211	211	0°	33°33'	48°11'	60°	70°32'	80°24'
	221	17°43'	35°16'	47°7'	65°54'	74°12'	82°12'
	310	25°21'	49°48'	58°55'	75°2'	82°35'	
	311	10°1'	42°24'	60°30'	75°45'	90°	
	320	25°4'	37°37'	55°33'	63°5'	83°30'	
	321	10°54'	29°12'	40°12'	49°6'	56°56'	
		70°54'	77°24'	83°44'	90°		
221	221	0°	27°16'	38°57'	63°37'	83°37'	90°
	310	32°31'	42°27'	58°12'	65°4'	83°57'	
	311	25°14'	45°17'	59°50'	72°27'	84°14'	
	320	22°24'	42°18'	49°40'	68°18'	79°21'	84°42'
	321	11°29'	27°1'	36°42'	57°41'	63°33'	74°30'
		79°44'	84°53'				

Table A6.1 (continued)

$\{HKL\}$	$\{hkl\}$	Values of angles between HKL and hkl planes (or directions)						
310	310	0°	25°51'	36°52'	53°8'	72°33'	84°16'	90°
	311	17°33'	40°17'	55°6'	67°35'	79°1'	90°	
	320	15°15'	37°52'	52°8'	58°15'	74°45'	79°54'	
	321	21°37'	32°19'	40°29'	47°28'	53°44'	59°32'	
		65°		85°9'	90°			
311	311	0°	35°6'	50°29'	62°58'	84°47'		
	320	23°6'	41°11'	54°10'	65°17'	75°28'	85°12'	
	321	14°46'	36°19'	49°52'	61°5'	71°12'	80°44'	
320	320	0°	22°37'	46°11'	62°31'	67°23'	72°5'	90°
	321	15°30'	27°11'	35°23'	48°9'	53°37'	58°45'	68°15'
		72°45'	77°9'	85°45'	90°			
321	321	0°	21°47'	31°	38°13'	44°25'	50°	60°
		64°37'	69°4'	73°24'	81°47'	85°54'		

Table A6.2 Interplanar spacings of selected materials with f.c.c. crystal structure

	γFe	Cu	Pt	Al	Au	Ag	Pb	Ni	Co (β)
Lattice parameter a_0 (Å)	3.5852	3.6150	3.9231	4.0496	4.0780	4.0862	4.9505	3.5238	3.5520
hkl	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)
111	2.070	2.087	2.265	2.338	2.355	2.359	2.858	2.0345	2.0508
002	1.793	1.808	1.962	2.025	2.039	2.044	2.475	1.7619	1.7760
022	1.268	1.278	1.387	1.432	1.442	1.445	1.750	1.2460	1.2560
113	1.081	1.090	1.183	1.221	1.230	1.231	1.493	1.0623	1.0708
222	1.035	1.044	1.133	1.169	1.177	1.180	1.429	1.0172	1.0254
004	0.896	0.904	0.981	1.012	1.020	1.022	1.238	0.8810	0.8880
133	0.823	0.829	0.900	0.929	0.936	0.938	1.136	0.8084	0.8149
024	0.802	0.808	0.877	0.906	0.912	0.914	1.107	0.7880	0.7943
224	0.732	0.738	0.801	0.827	0.832	0.834	1.011	0.7193	0.7250
333	0.690	0.696	0.755	0.779	0.785	0.786	0.953	0.6782	0.6836
115									
044									
135	0.606	0.610	0.663	0.685	0.689	0.691	0.837	0.5956	0.6004
006	0.598	0.603	0.654	0.675	0.680	0.681	0.825	0.5873	0.5920
244									
026									
335	0.547	0.551	0.598	0.618	0.622	0.623	0.755	0.5374	0.5417
226	0.541	0.545	0.591	0.611	0.615	0.616	0.746	0.5313	0.5355
444	0.518	0.522	0.566	0.585	0.589	0.590	0.715	0.5086	0.5127

Table A6.3 Interplanar spacings of selected materials with b.c.c. crystal structure

	αFe	Cr	Mo	W	Nb	Ta	V
Lattice parameter a_0 ($\text{\AA}$)	2.8661	2.8850	3.1463	3.1652	3.3007	3.3058	3.0390
hkl	d spacing ($\text{\AA}$)	d spacing ($\text{\AA}$)	d spacing ($\text{\AA}$)	d spacing ($\text{\AA}$)	d spacing ($\text{\AA}$)	d spacing ($\text{\AA}$)	d spacing ($\text{\AA}$)
011	2.027	2.040	2.225	2.238	2.334	2.338	2.149
002	1.433	1.443	1.573	1.583	1.650	1.653	1.5195
112	1.170	1.178	1.285	1.292	1.348	1.350	1.2409
022	1.013	1.020	1.113	1.119	1.167	1.169	1.0746
013	0.906	0.912	0.995	1.001	1.044	1.045	0.9611
222	0.828	0.833	0.908	0.914	0.953	0.954	0.8773
123	0.766	0.771	0.841	0.846	0.882	0.884	0.8121
004	0.717	0.721	0.787	0.791	0.825	0.826	0.7598
114 } 033 }	0.676	0.680	0.742	0.746	0.778	0.779	0.7162
024	0.641	0.645	0.704	0.708	0.738	0.739	0.6796
233	0.611	0.615	0.671	0.675	0.704	0.705	0.6480
224	0.585	0.589	0.642	0.646	0.674	0.675	0.6203
015 } 134 }	0.562	0.566	0.617	0.621	0.647	0.648	0.5960

Table A6.4 Interplanar spacings of selected materials with diamond cubic crystal structure

Lattice parameter (Å)	Si 5.4282	Ge 5.6580
(hkl)	d spacing (Å)	d spacing (Å)
111	3.1340	3.2667
220	1.9194	2.0007
311	1.6365	1.7058
400	1.3571	1.4145
331	1.2453	1.2980
422	1.1080	1.1549
511	1.0447	1.0889
440	0.9596	1.0002
531	0.9175	0.9564
620	0.8582	0.8945
533	0.8278	0.8629
444	0.7835	0.8167
711	0.7601	0.7923

Table A6.6 Interplanar spacings of graphite, $a = 2.461 \text{ Å}$, $c = 6.708 \text{ Å}$

hkil	d spacing (Å)
0002	3.354
10 $\bar{1}$ 0	2.131
10 $\bar{1}$ 1	2.031
10 $\bar{1}$ 2	1.799
0004	1.677
10 $\bar{1}$ 3	1.543
11 $\bar{2}$ 0	1.231
11 $\bar{2}$ 2	1.155
0006	1.118

Table A6.5 Interplanar spacings of selected materials with close-packed hexagonal crystal structure

		Be	Zn	Ti	Mg	Zr	Gd	Co	Cd	Re
Lattice parameters	a_0 (Å)	2.285	2.664	2.950	3.209	3.231	3.636	2.507	2.979	2.761
	c_0 (Å)	3.584	4.046	4.683	5.210	5.147	5.782	4.069	5.617	4.458
	c_0/a_0	1.568	2.856	1.587	1.593	1.593	1.590			
$hkil$	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	d spacing (Å)	
0001	3.584	4.947	4.683	5.210	5.148	5.783	4.068	5.617	4.458	
01 $\bar{1}$ 0	1.979	2.308	2.555	2.779	2.798	3.149	2.170	2.580	2.390	
0002	1.792	2.473	2.342	2.605	2.574	2.891	2.035	2.808	2.229	
01 $\bar{1}$ 1	1.733	2.092	2.243	2.452	2.439	2.765	1.915	2.344	2.107	
01 $\bar{1}$ 2	1.329	1.687	1.726	1.901	1.894	2.130	1.484	1.900	1.630	
0003	1.195	1.649	1.561	1.737	1.716	1.928	1.356	1.872	1.486	
11 $\bar{2}$ 0	1.143	1.332	1.475	1.605	1.616	1.818	1.253	1.489	1.380	
11 $\bar{2}$ 1	1.089	1.287	1.407	1.534	1.541	1.734	1.198	1.440	1.318	
01 $\bar{1}$ 3	1.023	1.342	1.332	1.473	1.463	1.644	1.150	1.515	1.262	
02 $\bar{2}$ 0	0.990	1.154	1.278	1.390	1.399	1.574	1.085	1.290	1.195	
11 $\bar{2}$ 2	0.964	1.173	1.248	1.366	1.368	1.539	1.067	1.316	1.173	
02 $\bar{2}$ 1	0.954	1.124	1.233	1.343	1.350	1.519	1.048	1.257	1.154	
0004	0.896	1.237	1.171	1.303	1.287	1.446	1.017	1.404	1.114	
02 $\bar{2}$ 2	0.866	1.046	1.122	1.226	1.229	1.383	0.957	1.172	1.053	
11 $\bar{2}$ 3	0.826	1.036	1.072	1.179	1.176	1.323	0.920	1.166	1.011	
01 $\bar{1}$ 4	0.816	1.090	1.064	1.180	1.169	1.314	0.921	1.233	1.010	
02 $\bar{2}$ 3	0.762	0.945	0.989	1.085	1.084	1.219	0.847	1.062	0.931	
12 $\bar{3}$ 0	0.748	0.872	0.966	1.051	1.058	1.190	0.820	0.975	0.903	
12 $\bar{3}$ 1	0.732	0.859	0.946	1.030	1.036	1.166	0.804	0.961	0.885	
0005	0.717	0.989	0.937	1.042	1.030	1.157	0.814	1.123	0.891	
11 $\bar{2}$ 4	0.705	0.906	0.917	1.011	1.007	1.132	0.780	1.022	0.867	
12 $\bar{3}$ 2	0.690	0.823	0.893	0.974	0.978	1.101	0.761	0.921	0.837	
01 $\bar{1}$ 5	0.674	0.909	0.879	0.976	0.966	1.086	0.761	1.030	0.835	
02 $\bar{2}$ 4	0.664	0.844	0.863	0.950	0.947	1.065	0.742	1.950	0.815	
03 $\bar{3}$ 0	0.660	0.769	0.852	0.927	0.933	1.050	0.723	0.859	0.797	
03 $\bar{3}$ 1	0.649	0.760	0.838	0.912	0.918	1.033	0.712	0.850	0.784	
12 $\bar{3}$ 3	0.634	0.771	0.821	0.899	0.900	1.013	0.701	0.865	0.772	
03 $\bar{3}$ 2	0.619	0.735	0.800	0.873	0.877	0.987	0.682	0.822	0.750	
11 $\bar{2}$ 5	0.607	0.794	0.791	0.874	0.868	0.976	0.682	0.897	0.749	
0006	0.597	0.825	0.781	0.868	0.858	0.964	0.678	0.936	0.743	

APPENDIX 7.

ELECTRON WAVELENGTH

Table A7.1 Electron wavelength λ for common accelerating voltages ≤ 200 kV (calculated using equation (2.1))

Accelerating voltage (kV)	λ (Å)	λ^{-1} (Å ⁻¹)
10	0.122	8.194
20	0.0859	11.64
30	0.0698	14.33
40	0.0602	16.62
50	0.0536	18.67
60	0.0487	20.55
70	0.0448	22.30
80	0.0418	23.95
90	0.0392	25.52
100	0.0370	27.02
200	0.0251	39.87

APPENDIX 8. ATOMIC SCATTERING AMPLITUDES

This appendix contains tables of atomic scattering amplitudes for electrons f_θ (in Å) as a function of $(\sin \theta)/\lambda$. For definitions of f_θ , see section 2.2.1. This parameter is used to calculate structure

factors, section 2.3.1, and extinction distances, section 3.2.3.1.

Appendix 8: Reference

Ibers, J. A. (1957). *Acta Cryst.*, **10**, 86.

Table A8.1 Atomic scattering amplitudes for electrons f_0 in Å: self-consistent field calculations

Element Z		$(\sin \theta)/\lambda$ (Å ⁻¹)																
		0.00	0.05	0.10	0.15	0.20	0.25	0.30	0.35	0.40	0.50	0.60	0.70	0.80	0.90	1.00	1.10	1.20
H	1	0.529	0.508	0.453	0.382	0.311	0.249	0.199	0.160	0.131	0.089	0.064	0.048	0.037	0.029	0.024	0.020	0.017
He	2	(0.445)	0.431	0.403	0.363	0.328	0.288	0.250	0.216	0.188	0.142	0.109	0.086	0.068	0.055	0.016	0.038	0.032
Li	3	3.31	2.78	1.88	1.17	0.75	0.53	0.40	0.31	0.26	0.19	0.14	0.11	0.09	0.08	0.06	0.05	0.05
Be	4	3.09	2.82	2.23	1.63	1.16	0.83	0.61	0.47	0.37	0.25	0.19	0.15	0.12	0.10	0.08	0.07	0.06
B	5	2.82	2.62	2.24	1.78	1.37	1.04	0.80	0.62	0.50	0.33	0.24	0.18	0.14	0.12	0.10	0.08	0.07
C	6	2.45	2.26	2.09	1.74	1.43	1.15	0.92	0.74	0.60	0.41	0.30	0.22	0.18	0.14	0.12	0.10	0.08
N	7	2.20	2.10	1.91	1.68	1.44	1.20	1.00	0.83	0.69	0.48	0.35	0.27	0.21	0.17	0.14	0.11	0.10
O	8	2.01	1.95	1.80	1.62	1.42	1.22	1.04	0.88	0.75	0.54	0.40	0.31	0.24	0.19	0.16	0.13	0.11
F	9	(1.84)	(1.77)	1.69	(1.53)	1.38	(1.20)	1.05	(0.91)	0.78	0.59	0.44	0.35	0.27	0.22	0.18	0.15	(0.13)
Ne	10	(1.66)	1.59	1.53	1.43	1.30	1.17	1.04	0.92	0.80	0.62	0.48	0.38	0.30	0.24	0.20	0.17	0.14
Na	11	4.89	4.21	2.97	2.11	1.59	1.29	1.09	0.95	0.83	0.64	0.51	0.40	0.33	0.27	0.22	0.18	0.16
Mg	12	5.01	4.60	3.59	2.63	1.95	1.50	1.21	1.01	0.87	0.67	0.53	0.43	0.35	0.29	0.24	0.20	0.17
Al	13	(6.1)	5.36	4.24	3.13	2.30	1.73	1.36	1.11	0.93	0.70	0.55	0.45	0.36	0.30	0.25	0.22	(0.19)
Si	14	(6.0)	5.26	4.40	3.41	2.59	1.97	1.54	1.23	1.02	0.74	0.58	0.47	0.38	0.32	0.27	0.23	(0.20)
P	15	(5.4)	5.07	4.38	3.55	2.79	2.17	1.70	1.36	1.12	0.80	0.61	0.49	0.40	0.33	0.28	0.24	0.21
S	16	(4.7)	4.40	4.00	3.46	2.87	2.32	1.86	1.50	1.22	0.86	0.64	0.51	0.42	0.35	0.30	0.25	0.22
Cl	17	(4.6)	4.31	4.00	3.53	2.99	2.47	2.01	1.63	1.34	0.93	0.69	0.54	0.44	0.37	0.31	0.26	0.23
	18	4.71	4.40	4.07	3.56	3.03	2.52	2.07	1.71	1.42	1.00	0.74	0.58	0.46	0.38	0.32	0.27	0.24
	19	(9.0)	(7.0)	5.43	(4.10)	3.15	(2.60)	2.14	(1.00)	1.49	1.07	0.79	0.61	0.49	0.40	0.34	0.29	(0.25)
Ca	20	10.46	8.71	6.40	4.54	3.40	2.69	2.20	1.84	1.55	1.12	0.84	0.65	0.52	0.42	0.35	0.30	0.26
Sc	21	(9.7)	8.35	6.30	4.63	3.50	2.75	2.29	1.92	1.62	1.18	0.89	0.69	0.54	0.44	0.37	0.32	(0.27)
Ti	22	(8.9)	7.95	6.20	4.63	3.55	2.84	2.34	(1.97)	1.67	1.23	0.93	0.72	0.57	0.47	0.69	0.33	0.29
V	23	(8.4)	7.60	6.08	4.60	3.57	2.88	2.39	(2.02)	1.72	1.28	0.97	0.76	0.60	0.49	0.41	0.35	0.30
Cr	24	(8.0)	7.26	5.86	4.55	3.56	2.89	2.42	2.06	1.76	1.32	1.01	0.80	0.63	0.51	0.43	0.36	(0.31)
Mn	25	(7.7)	7.00	5.72	4.48	3.55	2.91	2.44	(2.08)	1.79	1.36	1.04	0.83	0.66	0.54	0.45	0.38	0.32
Fe	26	(7.4)	6.70	5.55	4.41	3.54	2.91	2.45	(2.11)	1.82	1.39	1.08	0.86	0.69	0.56	0.47	0.39	0.34
Co	27	(7.1)	6.41	5.41	4.34	3.51	2.91	2.46	(2.12)	1.84	1.42	1.11	0.89	0.71	0.58	0.49	0.41	0.35
Ni	28	(6.8)	6.22	5.27	4.27	3.48	2.90	2.47	(2.13)	1.86	1.46	1.14	0.92	0.74	0.61	0.50	0.43	0.36
Cu	29	(6.5)	6.00	5.14	4.19	3.44	2.88	2.46	2.12	1.87	1.47	1.16	0.95	0.77	0.63	0.52	0.45	(0.38)
Zn	30	6.2	5.84	4.98	4.11	3.39	2.86	2.45	(2.11)	1.88	1.48	1.19	0.96	0.78	0.65	0.54	0.46	0.39
Ga	31	(7.5)	6.70	5.62	4.51	3.64	3.00	2.53	2.18	1.91	1.50	1.20	0.98	0.81	0.67	0.56	0.47	0.41
Ce	32	(7.8)	6.89	5.93	4.81	3.87	3.16	2.63	2.24	1.94	1.51	1.22	0.99	0.83	0.69	0.58	0.49	0.42
As	33	(7.8)	6.99	6.05	5.01	4.07	3.32	2.74	2.31	1.99	1.54	1.23	1.01	0.85	0.71	0.59	0.50	0.43
Se	34	(7.7)	6.99	6.15	5.18	4.24	3.47	2.86	2.40	2.05	1.57	1.23	1.02	0.86	0.72	0.61	0.52	0.44
Br	35	(7.3)	6.80	6.15	5.25	4.37	3.60	2.97	2.49	2.12	1.60	1.27	1.04	0.88	0.73	0.62	0.53	0.45
Kr	36	(7.1)	6.70	6.13	5.31	4.47	3.71	3.08	2.58	2.19	1.64	1.29	1.05	0.90	0.75	0.64	0.55	0.47
Ag	47	(8.8)	8.24	7.47	6.51	5.58	4.75	4.05	3.46	2.97	2.22	1.70	1.35	1.09	0.90	0.76	0.66	0.57
W	74	(14)	—	11.80	—	7.43	—	5.16	—	3.85	2.99	2.39	1.96	1.63	1.38	1.18	1.02	0.89
Hg	80	(13.3)	12.26	10.82	9.18	7.70	6.48	5.50	4.72	4.09	3.16	2.51	2.05	1.70	1.44	1.23	1.07	0.93

Note. () = interpolation or extrapolation. Values at $(\sin \theta)/\lambda = 0$ not in brackets are calculated. Some reasons for the large differences, particularly at low scattering angles, between data common to Tables A8.1 and A8.2 are discussed by Ibers (1957). The values are based on the rest mass of the electron. For electrons of energy E , multiply by $m/m_0 = \{1 - (v/c)^2\}^{-1/2}$.

Table A8.2 Mean atomic scattering amplitudes for electrons f (atomic number = 20–104) in Å:
Thomas–Fermi–Dirac statistical model

		(sin θ)/λ(Å ⁻¹)																			
Element	Z	0.00	0.05†	0.10	0.15	0.20	0.25	0.30	0.35	0.40	0.50	0.60	0.70	0.80	0.90	1.00	1.10	1.20	1.30	1.40	1.50
Ca	20	5.4	5.08	4.57	3.85	3.13	2.52	2.06	1.72	1.45	1.07	0.82	0.65	0.53	0.44	0.37	0.31	0.27	0.23	0.20	0.18
Sc	21	5.6	5.27	4.72	3.98	3.24	2.61	2.14	1.78	1.51	1.12	0.86	0.68	0.55	0.45	0.38	0.32	0.28	0.24	0.21	0.19
Ti	22	5.8	5.46	4.88	4.12	3.35	2.70	2.21	1.85	1.57	1.16	0.89	0.71	0.57	0.47	0.40	0.34	0.29	0.25	0.22	0.20
V	23	5.9	5.65	5.03	4.24	3.45	2.79	2.29	1.91	1.62	1.20	0.93	0.74	0.60	0.49	0.41	0.35	0.30	0.26	0.23	0.21
Cr	24	6.1	5.84	5.17	4.37	3.56	2.88	2.36	1.98	1.68	1.25	0.96	0.76	0.62	0.51	0.43	0.37	0.32	0.27	0.24	0.21
Mn	25	6.2	5.93	5.34	4.49	3.66	2.97	2.43	2.04	1.73	1.29	0.99	0.79	0.64	0.53	0.45	0.38	0.33	0.29	0.25	0.22
Fe	26	6.4	6.13	5.48	4.62	3.76	3.05	2.51	2.10	1.79	1.33	1.03	0.82	0.66	0.55	0.46	0.39	0.34	0.30	0.26	0.23
Co	27	6.5	6.32	5.62	4.73	3.87	3.14	2.58	2.16	1.84	1.37	1.06	0.84	0.69	0.57	0.48	0.41	0.35	0.31	0.27	0.24
Ni	28	6.7	6.41	5.74	4.85	3.97	3.22	2.65	2.23	1.89	1.41	1.09	0.87	0.71	0.59	0.49	0.42	0.36	0.32	0.28	0.25
Cu	29	6.8	6.61	5.89	4.97	4.06	3.30	2.72	2.29	1.95	1.45	1.13	0.90	0.73	0.60	0.51	0.43	0.38	0.33	0.29	0.25
Zn	30	7.0	6.70	6.03	5.08	4.16	3.38	2.79	2.35	2.09	1.49	1.16	0.92	0.75	0.62	0.52	0.45	0.39	0.34	0.30	0.26
Ga	31	7.2	6.89	6.15	5.20	4.25	3.46	2.86	2.41	2.05	1.53	1.19	0.95	0.77	0.64	0.54	0.46	0.40	0.35	0.31	0.27
Ge	32	7.3	7.09	6.29	5.32	4.35	3.54	2.93	2.46	2.10	1.57	1.22	0.97	0.79	0.66	0.56	0.47	0.41	0.36	0.31	0.28
As	33	7.5	7.18	6.41	5.43	4.44	3.62	2.99	2.52	2.15	1.61	1.25	1.00	0.82	0.68	0.57	0.49	0.42	0.37	0.32	0.29
Se	34	7.6	7.37	6.65	5.53	4.54	3.70	3.06	2.58	2.20	1.65	1.28	1.02	0.84	0.70	0.59	0.50	0.43	0.38	0.33	0.29
Br	35	7.8	7.47	6.68	5.63	4.63	3.78	3.13	2.64	2.25	1.69	1.32	1.05	0.86	0.71	0.60	0.51	0.44	0.39	0.34	0.30
Kr	36	7.9	7.56	6.80	5.74	4.71	3.85	3.19	2.69	2.31	1.73	1.35	1.08	0.88	0.73	0.62	0.53	0.46	0.40	0.35	0.31
Rb	37	8.0	7.75	6.92	5.85	4.80	3.93	3.26	2.75	2.35	1.77	1.38	1.10	0.90	0.75	0.63	0.54	0.47	0.41	0.36	0.32
Sr	38	8.2	7.85	7.04	5.96	4.89	4.00	3.32	2.80	2.40	1.80	1.41	1.13	0.92	0.77	0.65	0.55	0.48	0.42	0.37	0.33
Y	39	8.3	8.04	7.16	6.06	4.98	4.07	3.38	2.86	2.45	1.84	1.44	1.15	0.94	0.78	0.66	0.57	0.49	0.43	0.38	0.33
Zr	40	8.5	8.14	7.28	6.16	5.06	4.15	3.45	2.91	2.50	1.88	1.47	1.17	0.96	0.80	0.68	0.58	0.50	0.44	0.39	0.34
Nb	41	8.6	8.23	7.40	6.27	5.15	4.22	3.51	2.97	2.54	1.92	1.50	1.20	0.98	0.82	0.69	0.59	0.51	0.45	0.39	0.35
Mo	42	8.7	8.42	7.52	6.36	5.24	4.29	3.57	3.02	2.59	1.95	1.53	1.22	1.00	0.84	0.71	0.69	0.52	0.46	0.40	0.36
Tc	43	8.9	8.52	7.63	6.47	5.31	4.36	3.63	3.08	2.64	1.99	1.56	1.25	1.02	0.85	0.72	0.62	0.53	0.47	0.41	0.37
Ru	44	9.0	8.62	7.75	6.56	5.40	4.43	3.69	3.13	2.68	2.03	1.58	1.27	1.04	0.87	0.74	0.63	0.55	0.48	0.42	0.37
Rh	45	9.1	8.81	7.85	6.66	5.48	4.50	3.75	3.18	2.73	2.06	1.61	1.30	1.06	0.89	0.75	0.64	0.56	0.49	0.43	0.38
Pd	46	9.3	8.90	7.97	6.75	5.56	4.57	3.81	3.23	2.77	2.10	1.64	1.32	1.08	0.90	0.77	0.66	0.57	0.50	0.44	0.39
Ag	47	9.4	9.00	8.07	6.85	5.64	4.64	3.87	3.28	2.82	2.13	1.67	1.34	1.10	0.92	0.78	0.67	0.58	0.51	0.45	0.40
Cd	48	9.5	9.19	8.19	6.95	5.72	4.71	3.93	3.34	2.86	2.17	1.71	1.37	1.12	0.94	0.79	0.68	0.59	0.52	0.46	0.40
In	49	9.6	9.29	8.31	7.03	5.80	4.78	3.99	3.39	2.91	2.20	1.73	1.39	1.14	0.95	0.81	0.69	0.60	0.53	0.46	0.41
Sn	50	9.8	9.38	8.40	7.13	5.88	4.84	4.05	3.44	2.95	2.24	1.76	1.41	1.16	0.97	0.82	0.71	0.61	0.54	0.47	0.42
Sb	51	9.9	9.48	8.50	7.22	5.95	4.91	4.10	3.49	3.00	2.27	1.79	1.44	1.18	0.99	0.84	0.72	0.62	0.55	0.48	0.43
Te	52	10.0	9.57	8.62	7.31	6.03	4.97	4.16	3.54	3.04	2.31	1.81	1.46	1.20	1.00	0.85	0.73	0.63	0.55	0.49	0.44
P	53	10.1	9.77	8.71	7.39	6.11	5.04	4.22	3.59	3.08	2.34	1.84	1.48	1.22	1.02	0.87	0.74	0.64	0.56	0.50	0.44
Xe	54	10.2	9.86	8.81	7.49	6.19	5.10	4.27	3.64	3.13	2.38	1.87	1.51	1.24	1.04	0.88	0.76	0.66	0.57	0.51	0.45
Cs	55	10.4	9.96	8.93	7.57	6.26	5.17	4.33	3.68	3.17	2.41	1.90	1.53	1.26	1.05	0.89	0.77	0.67	0.58	0.52	0.46
Ba	56	10.5	10.05	9.02	7.66	6.34	5.23	4.39	3.73	3.21	2.45	1.93	1.55	1.28	1.07	0.91	0.78	0.68	0.59	0.52	0.47
La	57	10.6	10.15	9.12	7.75	6.40	5.30	4.44	3.78	3.26	2.48	1.95	1.57	1.30	1.09	0.92	0.79	0.69	0.60	0.53	0.47
Ce	58	10.7	10.24	9.21	7.84	6.49	5.36	4.50	3.83	3.30	2.51	1.98	1.60	1.32	1.10	0.94	0.80	0.70	0.61	0.54	0.48
Pr	59	10.8	10.44	9.31	7.92	6.56	5.42	4.55	3.88	3.34	2.55	2.01	1.62	1.33	1.12	0.95	0.82	0.71	0.62	0.55	0.49
Nd	60	10.9	10.53	9.41	8.01	6.63	5.48	4.60	3.93	3.38	2.58	2.03	1.64	1.35	1.13	0.96	0.83	0.72	0.63	0.56	0.50
Pm	61	11.0	10.63	9.53	8.10	6.70	5.55	4.66	3.97	3.43	2.61	2.06	1.66	1.37	1.15	0.98	0.84	0.73	0.64	0.57	0.50
Sm	62	11.1	10.72	9.62	8.17	6.77	5.61	4.71	4.02	3.47	2.65	2.09	1.69	1.39	1.17	0.99	0.85	0.74	0.65	0.57	0.51
Eu	63	11.2	10.82	9.72	8.25	6.85	5.67	4.77	4.07	3.51	2.68	2.11	1.71	1.41	1.18	1.00	0.86	0.75	0.66	0.58	0.52
Gd	64	11.4	10.92	9.79	8.34	6.91	5.73	4.82	4.11	3.55	2.71	2.14	1.73	1.43	1.20	1.02	0.88	0.76	0.67	0.59	0.53
Tb	65	11.5	11.01	9.88	8.42	6.98	5.79	4.87	4.16	3.59	2.74	2.17	1.75	1.45	1.21	1.03	0.89	0.77	0.68	0.60	0.53
Dy	66	11.6	11.11	9.98	8.50	7.05	5.85	4.92	4.20	3.63	2.78	2.19	1.77	1.47	1.23	1.05	0.90	0.78	0.69	0.61	0.54
Ho	67	11.7	11.20	10.08	8.58	7.12	5.91	4.98	4.25	3.67	2.81	2.22	1.80	1.48	1.25	1.06	0.91	0.79	0.70	0.61	0.55
Er	68	11.8	11.30	10.17	8.66	7.19	5.97	5.03	4.30	3.71	2.84	2.25	1.82	1.50	1.26	1.07	0.92	0.80	0.70	0.62	0.56
Tm	69	11.9	11.49	10.27	8.74	7.26	6.03	5.08	4.34	3.75	2.87	2.27	1.84	1.52	1.28	1.09	0.94	0.81	0.71	0.63	0.56
Kb	70	12.0	11.59	10.36	8.82	7.33	6.09	5.13	4.39	3.79	2.91	2.30	1.86	1.54	1.29	1.10	0.95	0.82	0.72	0.64	0.57
Lu	71	12.1	11.63	10.44	8.90	7.40	6.15	5.18	4.43	3.83	2.94	2.32	1.88								

APPENDIX 9. SUPERIMPOSED STEREOGRAMS FOR VARIOUS COMMON ORIENTATION RELATIONSHIPS

This appendix contains superimposed stereograms (figures A9.1–A9.6) for two phases with well-defined orientation relationships. These stereograms are particularly useful in interpreting diffraction patterns containing reflections from both precipitate and matrix.

For any given B in one phase the SADP should contain both its own and second-phase spots.

Their positions may be found by constructing the great circle with pole, B , see appendix 1. This contains all reflecting planes, that is those whose normals are perpendicular to B , see figure 2.20(e) and section 2.7.2. An example is shown in figure A9.7 for the Nishiyama–Wassermann relationship. B is $[110]$ in the b.c.c. phase and $[\bar{1}11]$ in the f.c.c. phase. The great circle AB with this pole is shown in

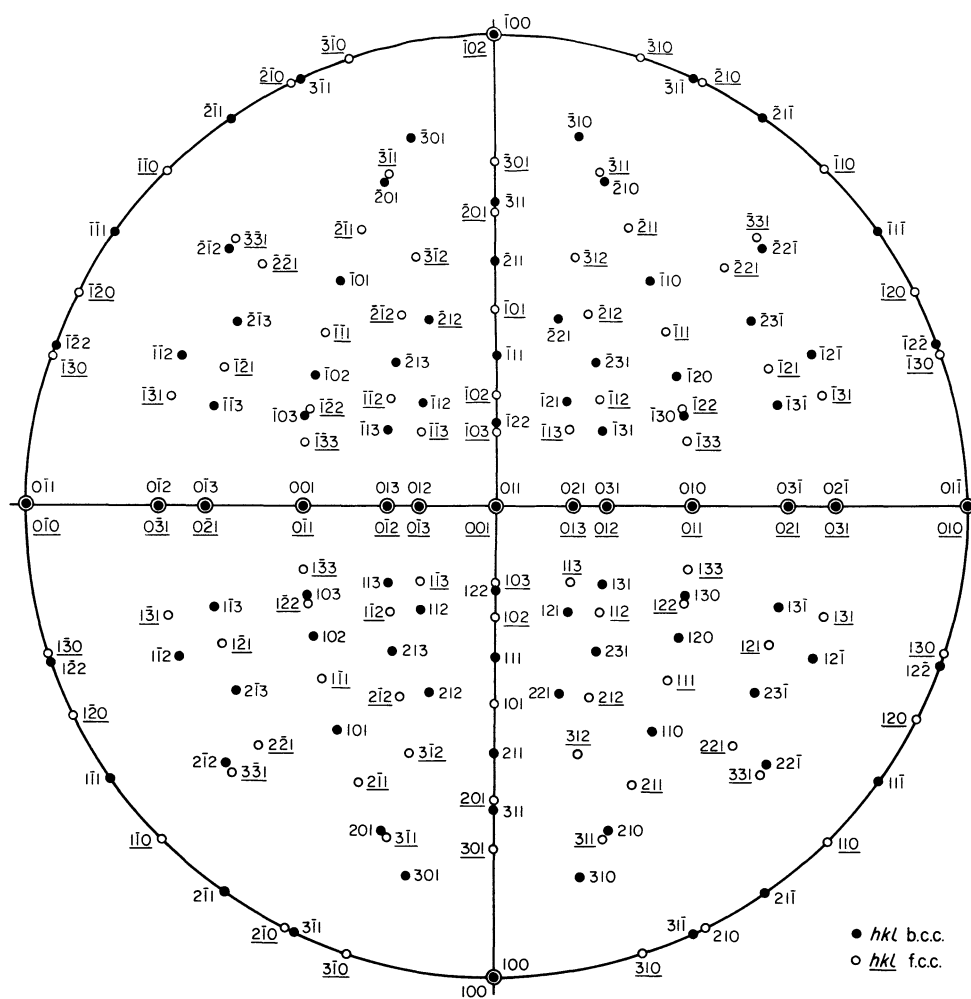


Figure A9.1 Stereographic projection representing the Bain relationship between body- and face-centered cubic materials
[After Andrews *et al.* (1971)]
(100) b.c.c. // (100) f.c.c.
(011) b.c.c. // (010) f.c.c.
(011) b.c.c. // (001) f.c.c.

figure A9.2 and the superimposed diffraction patterns in figure A9.7. All spots lie in this great circle, distance from the centre spot $\propto 1/d_{hkl}$, and with the correct angular relationships.

It is frequently convenient to prepare standard patterns showing these orientation relationships, for simple B in the matrix, for example $\langle 001 \rangle$, $\langle 111 \rangle$, $\langle 110 \rangle$, $\langle 112 \rangle$, so that these B may be obtained in a given area by tilting the specimen,

thus enabling the orientation relationship to be confirmed, see section 2.11.

Appendix 9: Reference

Andrews, K. W., Dyson, D. J., and Keown, S. R. (1971). *Interpretation of Electron Diffraction Patterns*, Hilger, London.

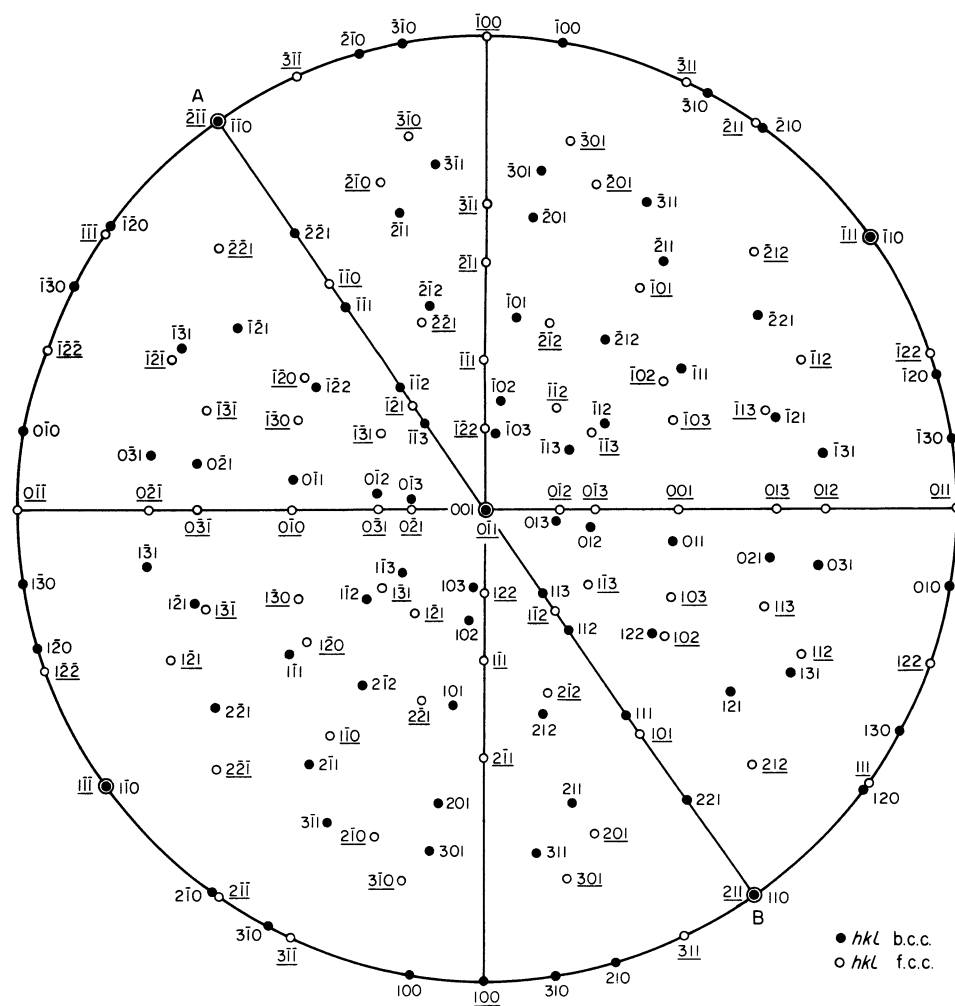


Figure A9.2 Stereographic projection representing the Nishiyama-Wassermann orientation relationship between body- and face-centred cubic materials [After Andrews *et al.* (1971)]

(001) b.c.c. // (011) f.c.c.

(110) b.c.c. // (111) f.c.c.

(110) b.c.c. // (211) f.c.c.

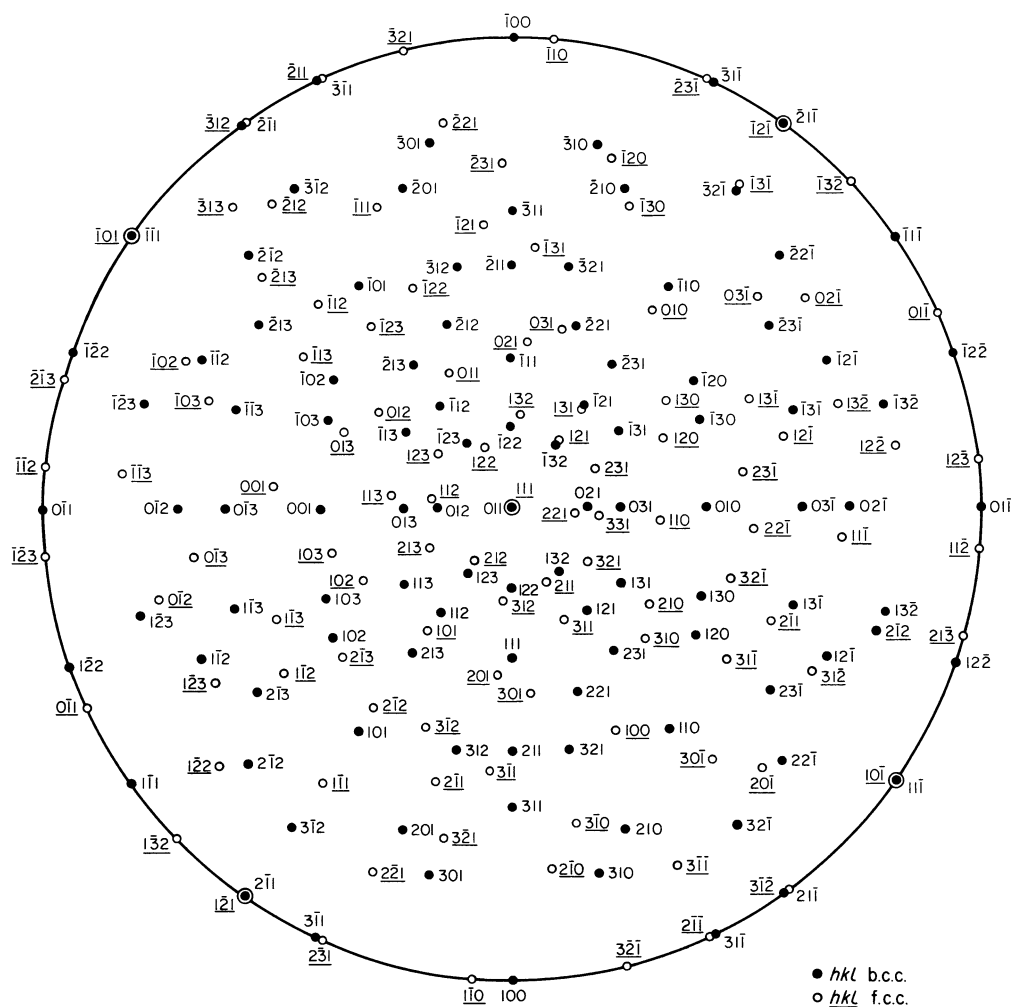
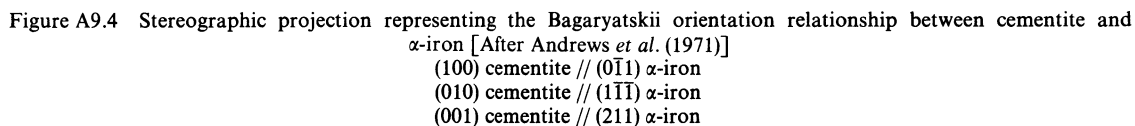


Figure A9.3 Stereographic projection representing the Kurdjumov-Sachs orientation relationship between body- and face-centred cubic materials [After Andrews *et al.* (1971)]

(011) b.c.c. // (111) f.c.c.

(11 $\bar{1}$) b.c.c. // (10 $\bar{1}$) f.c.c.

(21 $\bar{1}$) b.c.c. // ($\bar{1}$ 21) f.c.c.



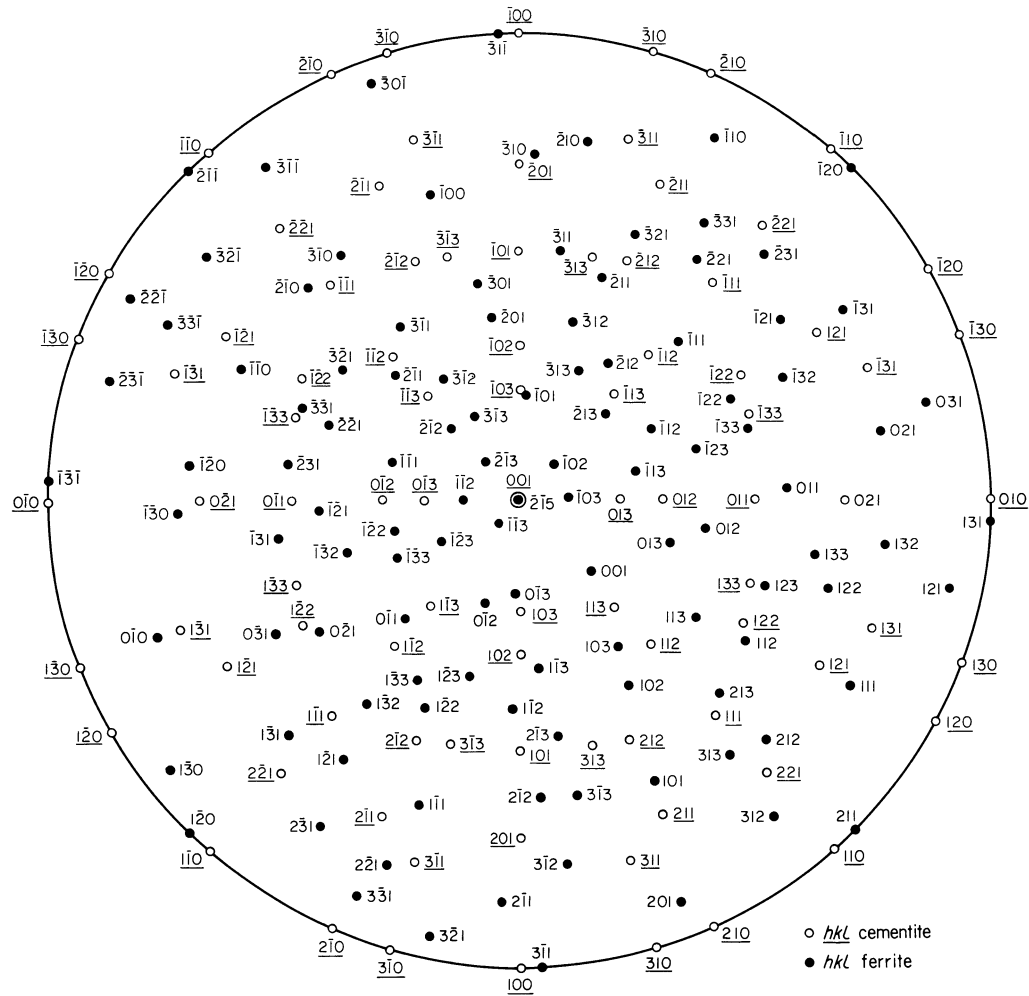


Figure A9.5 Stereographic projection representing the Pitsch orientation relationship between cementite and α -iron.

[After Andrews *et al.* (1971).]

(100) cementite // (311) α -iron

(010) cementite // (131) α -iron

(001) cementite // (215) α -iron

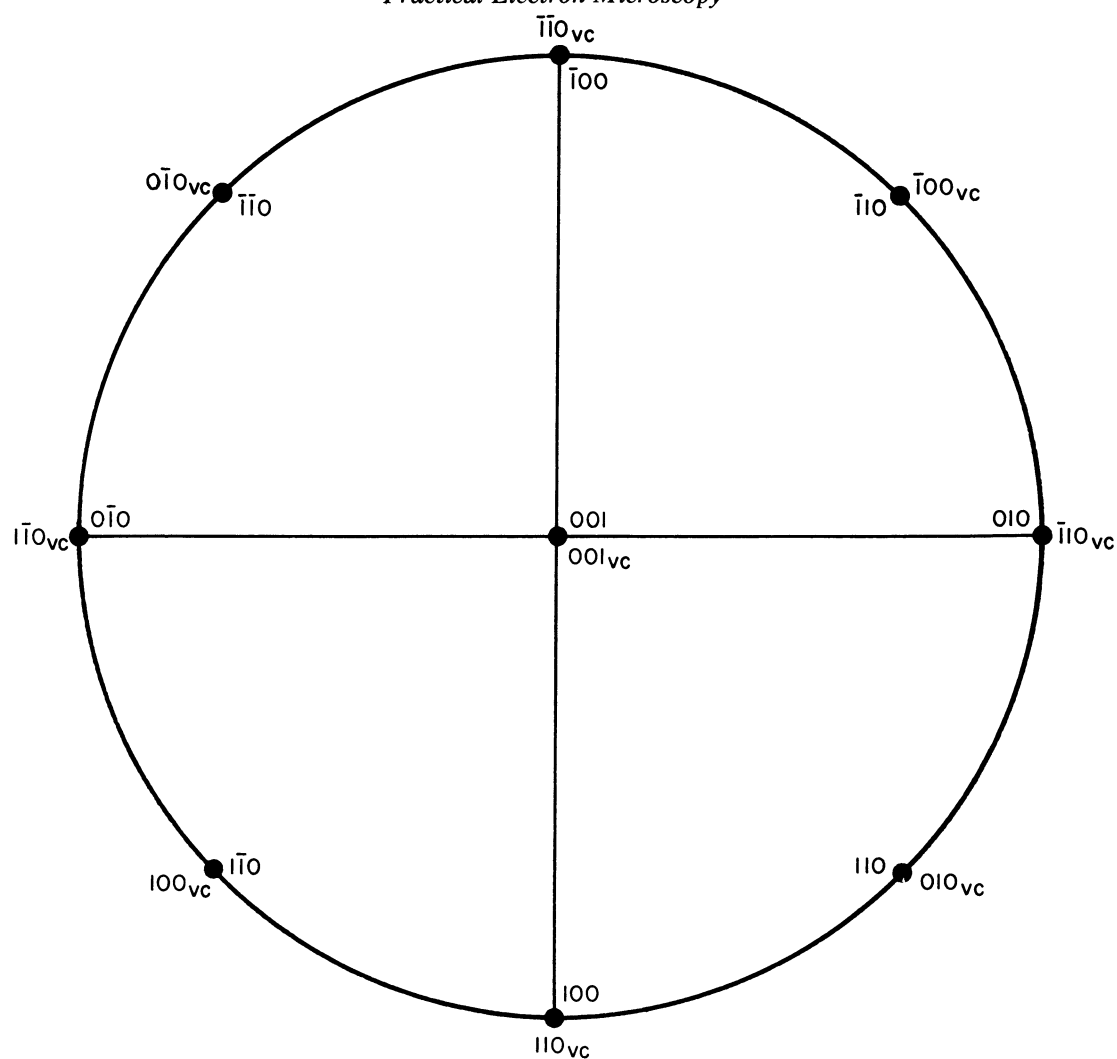


Figure A9.6 Stereographic projection representing the Baker–Nutting orientation relationship between vanadium carbide and α -iron
 $(001) \text{ VC} // (001) \alpha\text{-iron}$
 $(\bar{1}10) \text{ VC} // (010) \alpha\text{-iron}$
 $(110) \text{ VC} // (100) \alpha\text{-iron}$

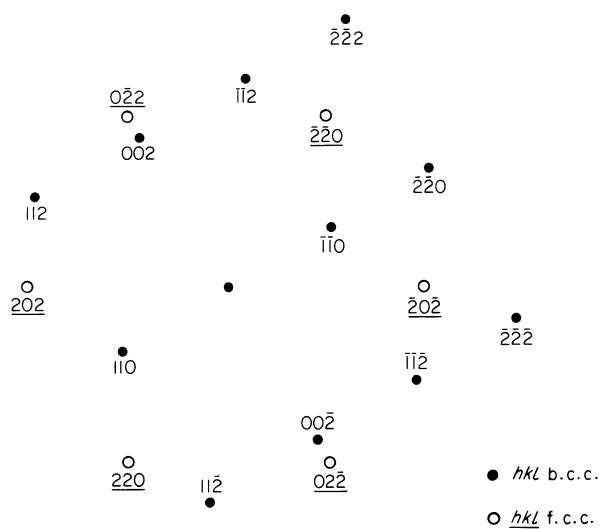


Figure A9.7 Superimposed spot diffraction patterns in which $[\bar{1}11] \text{ f.c.c.} // [\bar{1}10] \text{ b.c.c.} = B$ for the Nishiyama–Wassermann orientation relationship depicted in figure A9.2

APPENDIX 14.

ILLUSTRATION OF THE INHERENT AMBIGUITY IN THE INTER- PRETATION OF SELECTED AREA ELECTRON DIFFRACTION PATTERNS OF CEMENTITE

The assignment of indices to reflections on a selected area electron diffraction pattern is based on the correlation of interplanar spacing and interangular measurements. Owing to the large unit cell of the orthorhombic cementite structure it is often very difficult to assign the correct indices to reflections in the diffraction pattern since the differences in interplanar spacings of higher-order reflections are very small. It is usually possible to index reflecting zones of low order correctly but even in such cases some difficulty may be encountered.

In order to illustrate this difficulty the $[301]_c^*$ and $[102]_c$ zones will be considered. These two zones are drawn out in figure A14.1 on the scale of a typical photographic plate.

In table A14.1 the expected values of angles from $(010)_c$ and the distance from the centre spot for each reflection are shown for both the $[301]_c$ and $[102]_c$ zones. $\Delta\theta$ is the angular difference between corresponding reflections. For example, the angular difference between $(\bar{1}33)_c$ and $(\bar{2}31)_c$ is 1.77° . The column, spacing on plate, refers to the actual

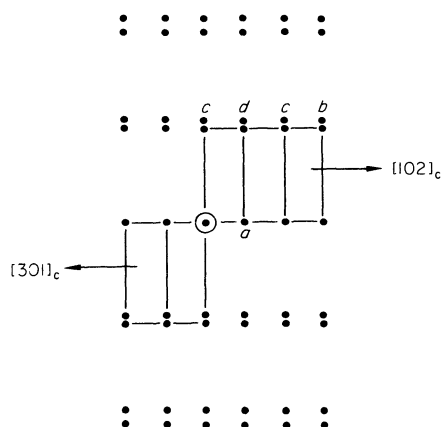


Figure A14.1 Diagram of $[301]_c$ and $[102]_c$ reflecting cementite zones. The possible indices that can be assigned to a, b , etc., are given in table A14.1

Table A14.1

	Reflection		Angle from $(010)_c$		$\Delta\theta$ (deg)	Spacing on plate R (mm)		ΔR (mm)
	$[301]_c$	$[102]_c$	$[301]_c$	$[102]_c$		$[301]_c$	$[102]_c$	
<i>a</i>	010	010	0	0	0	5.07	5.07	0
<i>b</i>	$\bar{1}33$	$\bar{2}31$	40.12	38.35	1.77	19.90	19.40	0.50
<i>c</i>	$\bar{1}23$	$\bar{2}21$	51.65	49.88	1.77	16.35	15.72	0.63
<i>d</i>	$\bar{1}13$	$\bar{2}11$	68.42	67.15	1.27	13.8	13.08	0.72
<i>e</i>	$\bar{1}03$	$\bar{2}01$	90.0	90.0	0	12.85	12.02	0.83

spacing on the plate (R) between the centre spot and the appropriate reflection. The interplanar spacing (d) is determined from the relation $d = \lambda L/R$ (where λL is the camera constant). The spacing on the plate is considered in this example in order to illustrate the experimental inaccuracy. ΔR is the difference in spacing between corresponding reflections in the two zones. For example, the difference in spacing on the plate between a $(\bar{1}33)_c$ reflection belonging to the $[301]_c$ zone and a $(\bar{2}31)_c$ reflection belonging to the $[102]_c$ zone is 0.5 mm.

From the tabulated values of table A14.1, it is clear that the maximum angular difference between corresponding reflections is less than 2° , which is very difficult to determine on a diffraction pattern. The maximum difference in distance from the centre spot is 0.83 mm. Bearing in mind that the typical diameter of a cementite reflection is 0.5 mm, it is clear that it is very difficult to distinguish between these two zones. A distinction can only be made if the camera constant is known to a high degree of accuracy, which is not normally the case. The $[301]_c$ zone is, however, 45° away from $[102]_c$ and a method has to be found to enable an unambiguous indexing of reflections. Three different approaches can be taken.

(i) The camera constant can be determined in the case of pearlite diffraction patterns by calculation from a known ferrite reflection in the pattern.

(ii) The foil can be tilted in the microscope so that

* Note. $[uvw]_c$ refers to the zone axis as a crystal direction in cementite and $(hkl)_c$ refers to reciprocal lattice vectors: $hu + kv + lw = 0$.

the Kikuchi centre was in the centre of the pattern. (The actual Kikuchi centre could normally not be seen in pearlite diffraction patterns but a tilting condition was found such that reflections were symmetrical about the centre and of even intensity.) The presence of higher-order reflections belonging to the same zone greatly facilitated in the accurate

measurement of the distance of a reflection from the centre spot.

(iii) A second diffraction pattern can be taken from the same area at a different tilting condition. When this new zone was identified and the angle of tilt known, one of the previous possibilities could be ruled out.