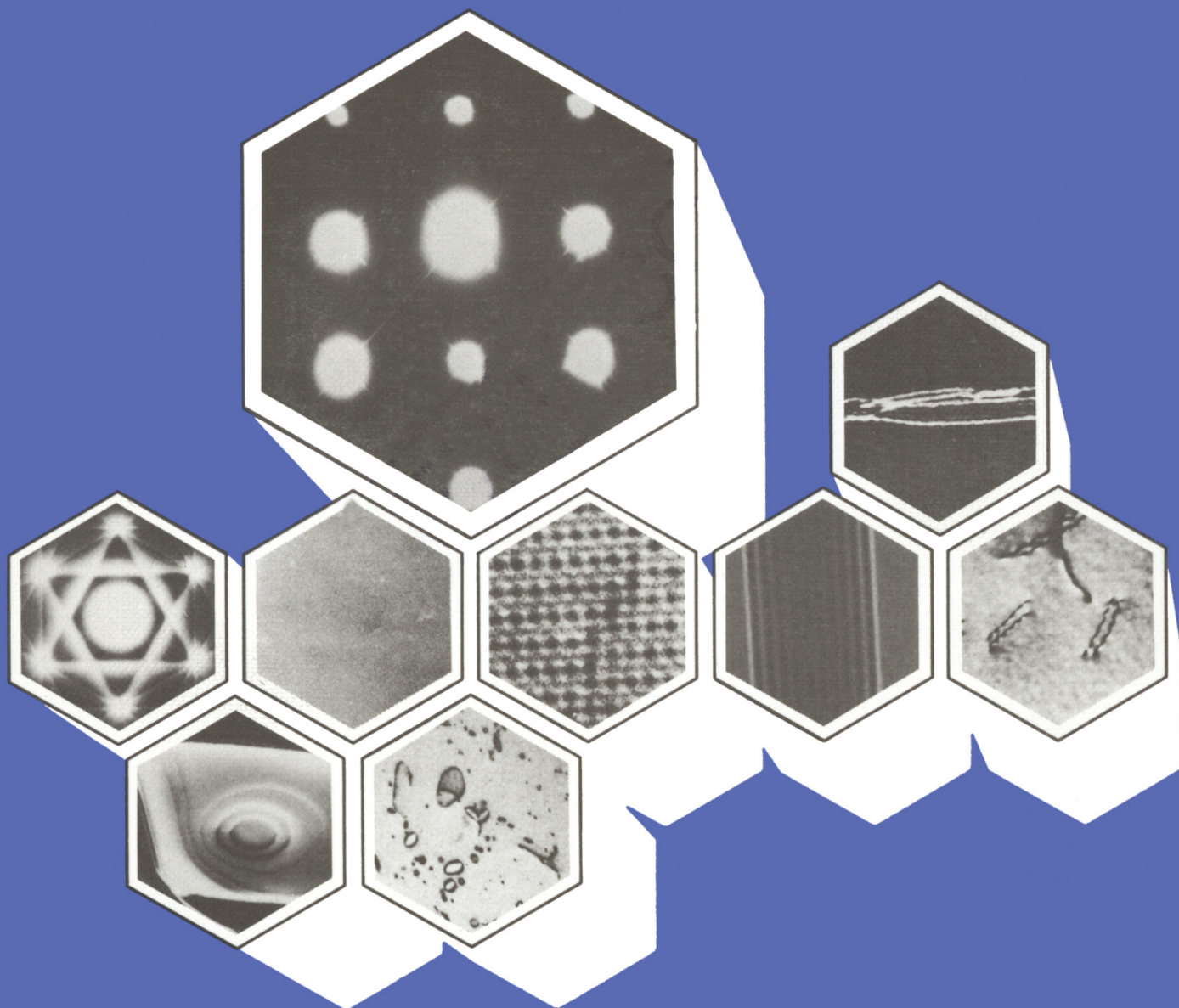


**PRACTICAL ELECTRON
MICROSCOPY
IN
MATERIALS SCIENCE**

3 **Monograph Three**
Interpretation
of Transmission
Electron
Micrographs

J W Edington



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3

INTERPRETATION OF TRANSMISSION ELECTRON MICROGRAPHS

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PREFACE

This is the third of a series of monographs on electron microscopy aimed at users of the equipment. They are written as both 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 this series. The following titles have already been published:

1. The Operation and Calibration of the Electron Microscope
2. Electron Diffraction in the Electron Microscope

and in preparation is:

4. Typical Electron Microscope Investigations

Abbreviations and Symbols Frequently Used in this Monograph

BF	Bright field (image)
DF	Dark field (image)
CDF	Centred dark field (image)
WB	Weak beam (image)
DP	Diffraction pattern
SADP	Selected area diffraction pattern
SRO	Short-range order
APB	Antiphase domain boundary
g	A vector normal to the reflecting plane
s	The deviation from the Bragg reflection position
ξ_g	The extinction distance
w	$s\xi_g$
b	The Burgers vector of an undissociated dislocation
b_p	The Burgers vector of a partial dislocation
μ	The line vector of a dislocation
FN	The upward drawn foil normal (normal to the specimen surface)
B	The upward drawn incident beam direction in the specimen, that is opposite to the direction of the electron beam
ANO	Anomalous absorption coefficient

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3. INTERPRETATION OF TRANSMISSION ELECTRON MICROGRAPHS

3.1 Image Contrast

There are two important mechanisms which produce image contrast in the electron microscope.

(1) *Diffraction contrast*. Diffracted electrons leaving the lower surface of a crystalline specimen are intercepted by the objective aperture and prevented from contributing to the image. Alternatively only one diffracted beam forms the image. See bright field (BF) and dark field (DF) images, section 1.

(2) *Phase contrast*. Some of the electrons leaving the specimen are recombined to form the image so that phase differences present at the exit surface of the specimen are converted into intensity differences in the image.

Diffraction contrast is the dominant mechanism delineating object detail $\gtrsim 15 \text{ \AA}$ in crystalline

specimens and is the most important and widely used contrast mechanism for study of crystal defects. Using this approach considerable quantitative information about the defect structure of the specimen may be obtained without operating the microscope at maximum resolution. Consequently, the technique is experimentally easy. Phase contrast is the dominant mechanism for object detail $\lesssim 10 \text{ \AA}$ and is important in lattice resolution studies and investigations of the early stages of short-range order and amorphous materials. Here each contrast mechanism is discussed in simple terms, followed by a detailed treatment of the results of its application to the analysis of crystal defects. At present most quantitative work relies primarily on diffraction contrast; consequently the emphasis lies heavily in this area.

I. DIFFRACTION CONTRAST

3.2 Summary of Theory

Quantitative analysis of crystal defects using the diffraction contrast approach usually involves detailed comparison of BF and DF images. BF images exclude diffracted, and DF images transmitted, electrons. Consequently, they are simply high-magnification maps of the intensity distribution across the transmitted or diffracted beams produced by the interaction of the specimen illuminated with the incident electron beam. Thus image contrast arises from point-to-point differences in the efficiency of the diffraction process in the region of the specimen under study. As a result, the details of the image must be interpreted in terms of a suitable diffraction theory, which has been described in detail in a number of books, for example Amelinckx (1964), Heidenreich (1964), Hirsch *et al.* (1967), Amelinckx *et al.* (1970), Murr (1971) and Valdre and Zichichi (1971), and review articles, for example Whelan (1970) and Howie (1971), to which the reader is referred for detailed theoretical background. Here we emphasise the important conclusions of the theory for images of specific types of crystal defect and describes those image features which may be used

to characterise the defects under study. Only the basic concepts of diffraction theory are included so that those factors important to the experimentalist may be defined.

Electron microscope images are normally discussed in terms of the dynamical theory of electron diffraction which incorporates as a special case the kinematical theory used in section 2 to describe electron diffraction patterns. The dynamical theory more accurately describes the diffraction process occurring in the electron microscope specimen and has been developed for the interpretation of image contrast in the electron microscope in a series of papers by Hirsch, Howie, Whelan, Amelinckx, Gevers, van Landuyt and their coworkers. The details of the theory have been summarised in the books referred to above. The simplest form of the theory only considers the case of two beams—one transmitted through, and one diffracted by, the specimen. The following factors not included in the kinematical theory of section 2 are incorporated in the dynamical theory.

(1) The intensity in the diffracted beam may be high compared with the transmitted beam.

(2) Electrons may be rediffracted back from the diffracted into the transmitted beam.

(3) Absorption of the electrons by the specimen, that is there is a limitation on penetration.

A more detailed formulation of the theory which includes several diffracted beams, the 'multi-beam' case, is summarised in the review articles by Howie (1970) and Goringe (1971). This is important for accelerating voltages $\gtrsim 300$ kV when two-beam conditions may be difficult to obtain for metallic specimens.

3.2.1 The Perfect Crystal

The simple two-beam dynamical theory describes the amplitudes ϕ_0 (transmitted) and ϕ_g (diffracted) in an element dz , depth z , in a small column of material in a thin foil thickness t , see figure 3.1. This theory assumes the following.

(1) The column is narrower than the image but wide enough to contain both the diffracted and transmitted amplitudes. This is possible because the scattering angle for 100 kV electrons is small ($2\theta \lesssim 1^\circ$).

(2) There is no contribution to the waves in the column from the rest of the specimen and conversely no loss from the column to the specimen.

(3) If the crystal is distorted, that is when a crystal defect is present (see section 3.2.4), the distortion occurs down but not across the column.

(4) Only plane waves exist in the column.

(5) Only one diffracted beam exists, that is *two-beam conditions*, see figure 2.35.

Points (1)–(3) are known as the column approximation and have been justified in the case of single dislocations by Jouffrey and Taupin (1967) and Howie and Basinski (1968) and for narrow dipoles near the foil surface by Colliex *et al.* (1969). Point (5) is more important because it specifies that images to be analysed quantitatively must *always* be produced under two-beam conditions.

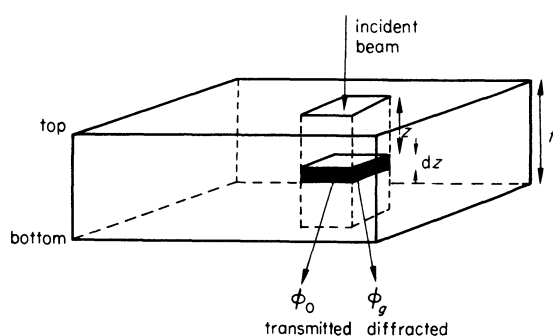


Figure 3.1 The column approximation for a perfect crystal

The theory allows for contribution back to the transmitted beam from the diffracted beam. Thus, as the transmitted wave propagates into the crystal, its amplitude ϕ_0 will be depleted by diffraction while the amplitude of ϕ_g is increased correspondingly, that is there is coupling between ϕ_0 and ϕ_g . The coupling is described by a pair of differential equations known as the Darwin–Howie–Whelan equations:

$$\begin{aligned} \frac{d\phi_0}{dz} &= \frac{i\pi}{\xi_0} \phi_0 + \frac{i\pi}{\xi_g} \phi_g \exp(2\pi isz) \\ \frac{d\phi_g}{dz} &= \frac{i\pi}{\xi_0} \phi_0 \exp(-2\pi isz) + \frac{i\pi}{\xi_g} \phi_g \end{aligned} \quad (3.1)$$

where s is the deviation from the exact Bragg angle, see figure 2.16, and ξ_0 and ξ_g are constants. The term ξ_g describes a critical distance in the perfect crystal at which the transmitted intensity falls to zero before increasing again and is known as the extinction distance, see section 3.2.3.1 for more details. The term $\exp(2\pi isz)$ represents a phase factor arising from the scattering process. The first of these equations states that the change in ϕ_0 in depth dz is partly due to forward scattering by the atoms in the element dz and partly due to scattering from the diffracted beam. Furthermore, there is a phase change of $\pi/2$, represented by the factor i , which arises from the scattering process.

3.2.2 Absorption

Two types of absorption need to be considered: (a) uniform absorption which leads to an overall decrease in intensity with increasing depth and (b) anomalous absorption which involves selective absorption of some electrons within the crystal and is responsible for some of the important depth asymmetry of many of the images of crystal defects discussed in subsequent sections.

Anomalous and uniform absorption are allowed for mathematically by introducing an imaginary component into the parameters $1/\xi_g \rightarrow 1/\xi_g + i/\xi'_g$, and $1/\xi_0 \rightarrow 1/\xi_0 + i/\xi'_0$, respectively. The term ξ_0/ξ'_0 is the uniform absorption parameter which influences the overall background intensity of the image. The term ξ_g/ξ'_g is the anomalous absorption parameter (ANO) which is responsible for many of the detailed features of diffraction contrast images. The value of ANO has been generally taken as 0.1 but Humphreys and Hirsch (1968) have pointed out that it is a function of both atomic weight and operative reflection and that this should be taken into account in some cases where accurate computation of images is necessary, for example sections 3.4–3.6.

3.2.3 Characteristic Images of Perfect Crystals

There are four types of image that are not related to the defect content of thin foils and are therefore found in almost all electron microscope specimens. These are (a) thickness fringes, (b) bend extinction contours, (c) bend contours and (d) bend centres, the details of which are most easily seen in annealed single-phase materials with a low defect density.

3.2.3.1 Thickness fringes

The depth variation of the transmitted intensity in a perfect crystal under dynamical diffracting conditions may be obtained from equations (3.1) and it can be shown that the diffracted intensity is

$$|\phi_g|^2 = \frac{\pi^2 \sin^2(\pi t s_{\text{eff}})}{\xi_g^2 (\pi s_{\text{eff}})^2} \quad (3.2)$$

and the transmitted intensity is

$$|\phi_0|^2 = (1 - |\phi_g|^2)$$

where $s_{\text{eff}} = (s^2 + \xi_g^{-2})^{1/2}$. The intensity oscillates with depth as shown in figure 3.2(a) with a periodicity of ξ_g which is known as the extinction distance. It is clear from figure 3.2(a) that the transmitted intensity is zero for specimen thickness $(n + \frac{1}{2})\xi_g$, where n is an integer. Consequently the edge of a typical wedge-shaped electropolished foil exhibits dark fringes connecting regions of the same thickness $\frac{1}{2}\xi_g$, $1\frac{1}{2}\xi_g$, $2\frac{1}{2}\xi_g$, etc., see figure 3.2(b), (c). However, when absorption is allowed for, the fringes are damped with increasing thickness and disappear before the transmitted intensity is zero, see figure 3.2(d). This often occurs above ~ 5 fringes. Clear high-contrast images of crystal defects are generally obtained at thicknesses where the thickness fringes just damp out, see figure 3.2(d). The thickness fringes show opposite contrast in dark field, compare figures 3.2(d), (e). Under kinematical conditions these thickness fringes almost disappear, as described later in this section.

The extinction distance ξ_g depends on both the operative reflection and the deviation from the exact Bragg position. The value of ξ_g at $s = 0$ may be calculated from the relationship

$$\xi_g = \frac{\pi \Omega \cos \theta}{\lambda f(\theta)} \quad (3.3)$$

where $f(\theta)$ is the atomic scattering amplitude obtained from the tables in appendix 8, θ is the Bragg angle, Ω is the volume of the unit cell and λ is the electron wavelength. Appendix 10 gives typical values of ξ_g for various metals to an accuracy $\pm 10\%$ because of inaccuracies in $f(\theta)$. Note that ξ_g increases with increasing order of reflection because $f(\theta)$ decreases as θ increases. If $s \neq 0$ then we have an effective extinction

distance $\xi_{g \text{ eff}} = \xi_g / (1 + s^2 \xi_g^2)^{1/2}$, that is ξ_g decreases with deviation from the Bragg position. Thus the spacing of the extinction fringes in an electron microscope image change if the operative reflection is changed by tilting the specimen or if s varies from point to point in the specimen because of bending of the foil.

Under kinematical conditions $s \gg 1/\xi_g$ and $s_{\text{eff}} \approx s$. Thus equation (3.2) reduces to

$$|\phi_g|^2 = \frac{\pi^2 \sin^2(\pi t s)}{\xi_g^2 (\pi s)^2} \quad (3.4)$$

which has the form shown in figure 3.2(f). In this case there are weak thickness fringes in the bright field image, figure 3.2(g), but stronger thickness fringes occur in the dark field image, see figure 3.2(h). However, in this case the extinction distance is $\ll \xi_g$ calculated for $s = 0$, and the fringes are closer together.

3.2.3.2 Bend extinction contours

This phenomenon occurs if the foil is elastically bent about a large radius like the top of a cylindrical pipe, as shown in figure 3.3(a). The $\{hkl\}$ family of reflecting planes on the right will be tilted away from the exact Bragg position by an amount depending on the distance from the point where $s = 0$ at position O. Figures 3.3(b), (c) show schematically the intensity distribution in both the transmitted beam (bright field image) and diffracted beam (dark field image) as a function of distance from O.

In this case distance is described more conveniently in terms of the deviation from the Bragg position by the parameter $w = s \xi_g$ and the graph is termed a 'rocking curve'.

The following conclusions may be drawn, figure 3.3(b).

(1) The bright field intensity depends on the sign of s (rocking curve asymmetrical about O) whereas the dark field image does not (rocking curve symmetrical about O).

(2) When s is small and positive there is a region of high transmitted intensity in bright field (termed anomalous transmission).

(3) When s is negative, overall transmission is very low in bright field.

(4) In dark field best transmission occurs at $s \approx 0$.

(5) On the left of figure 3.3(a), the same planes reflect but from their other side, that is they are now $\{\bar{h}\bar{k}\bar{l}\}$. Conclusions (1)–(4) above apply, see figure 3.3(c).

A typical example of a bend extinction contour is shown in figure 3.3(d). Note that diffraction patterns taken from points X and Y, figure 3.3(e), show (hkl) and $(\bar{h}\bar{k}\bar{l})$ reflections as indicated in

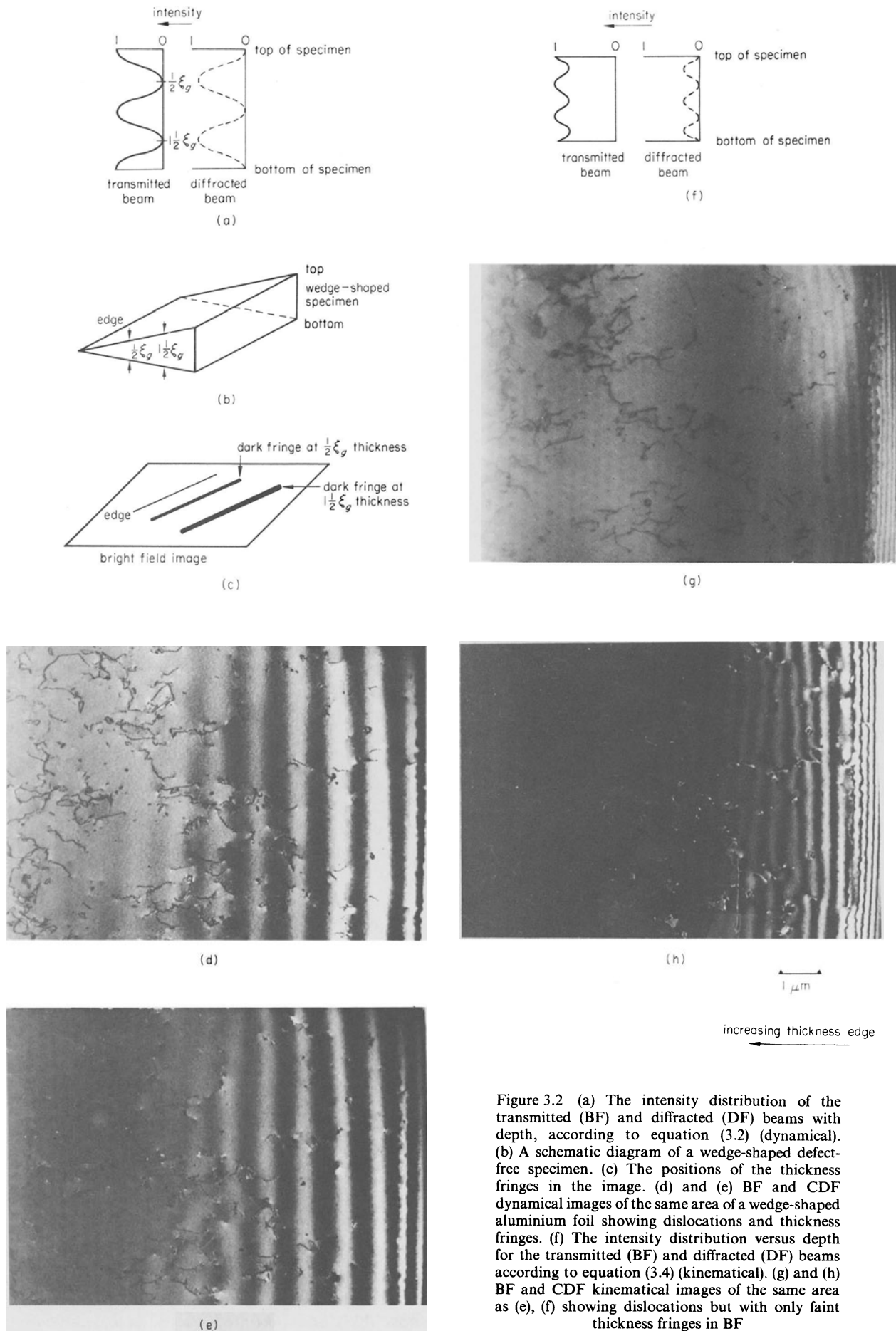


Figure 3.2 (a) The intensity distribution of the transmitted (BF) and diffracted (DF) beams with depth, according to equation (3.2) (dynamical). (b) A schematic diagram of a wedge-shaped defect-free specimen. (c) The positions of the thickness fringes in the image. (d) and (e) BF and CDF dynamical images of the same area of a wedge-shaped aluminium foil showing dislocations and thickness fringes. (f) The intensity distribution versus depth for the transmitted (BF) and diffracted (DF) beams according to equation (3.4) (kinematical). (g) and (h) BF and CDF kinematical images of the same area as (e), (f) showing dislocations but with only faint thickness fringes in BF

figure 3.3(b), (c). The edge of the specimen is visible and thickness increases on moving into the specimen. The line AOA' in figure 3.3(d) corresponds approximately to the intensity graph in figure 3.3(b), and fringes are clearly visible on travelling from O $\rightarrow$ A'. However, for the thicker regions in figure 3.3(d) these fringes are weaker because of increased absorption. The region of s small and positive corresponds to the normal region in which bright field photographs are taken because of the good transmission. In addition when s is negative the background intensity in bright field is low, see centre of bend extinction contour in figure 3.3(d). When the specimen is tilted, the bend extinction contour will move slowly across the field of view at low magnifications ($\lesssim 15\ 000$).

3.2.3.3 Bend contours

In very thin regions of the foil where the kinematical theory applies and absorption can be ignored, bending often occurs over relatively short distances producing diffuse dark lines in the bright field image, see lines AA' in figure 3.4(a). These fringes are usually $\sim 500\ \text{\AA}$ wide and may be recognised because they move rapidly across the field of view when the specimen is tilted through a small angle.

The sign of s changes from side to side of a bend contour.

3.2.3.4 Bend centres

For beam directions exactly parallel to simple crystallographic directions, characteristic bend extinction contour images occur in thin regions of bent foils. Examples are shown in figure 3.4(b)–(g) for different beam directions in the f.c.c. crystal structure. Each long arm of the image corresponds to an individual bend extinction contour and the point at the centre of all the arms is the exact orientation, B parallel to the specific crystal directions with all spots in the diffraction pattern equally bright. In order to produce such an image the specimen is elastically bent into an approximately spherical shape.

Such bend centres are used to compare the influence of operating reflections on the form of the image of crystal defects such as precipitates or for tilting specific B for comparison with a set of standard diffraction patterns.

3.2.4 Crystal Containing a Defect with a Strain Field

In general, thin electron microscope specimens contain a number of defects such as dislocations, stacking faults, precipitates, etc. Using the case

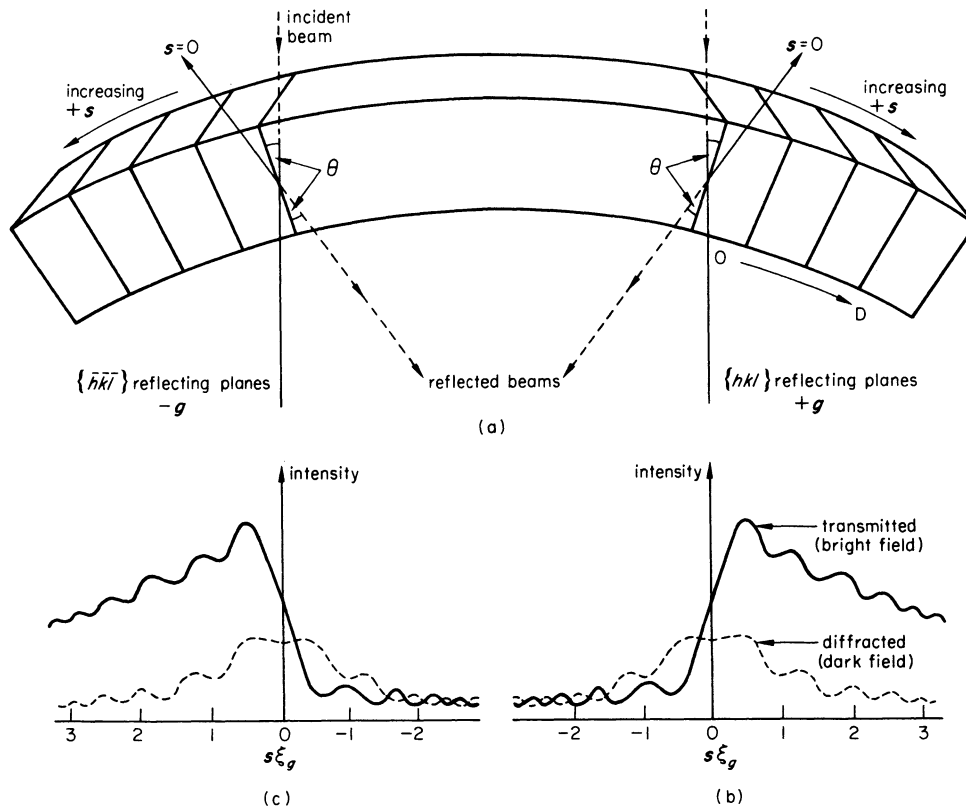
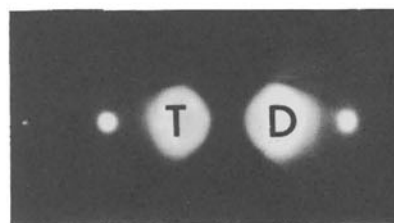
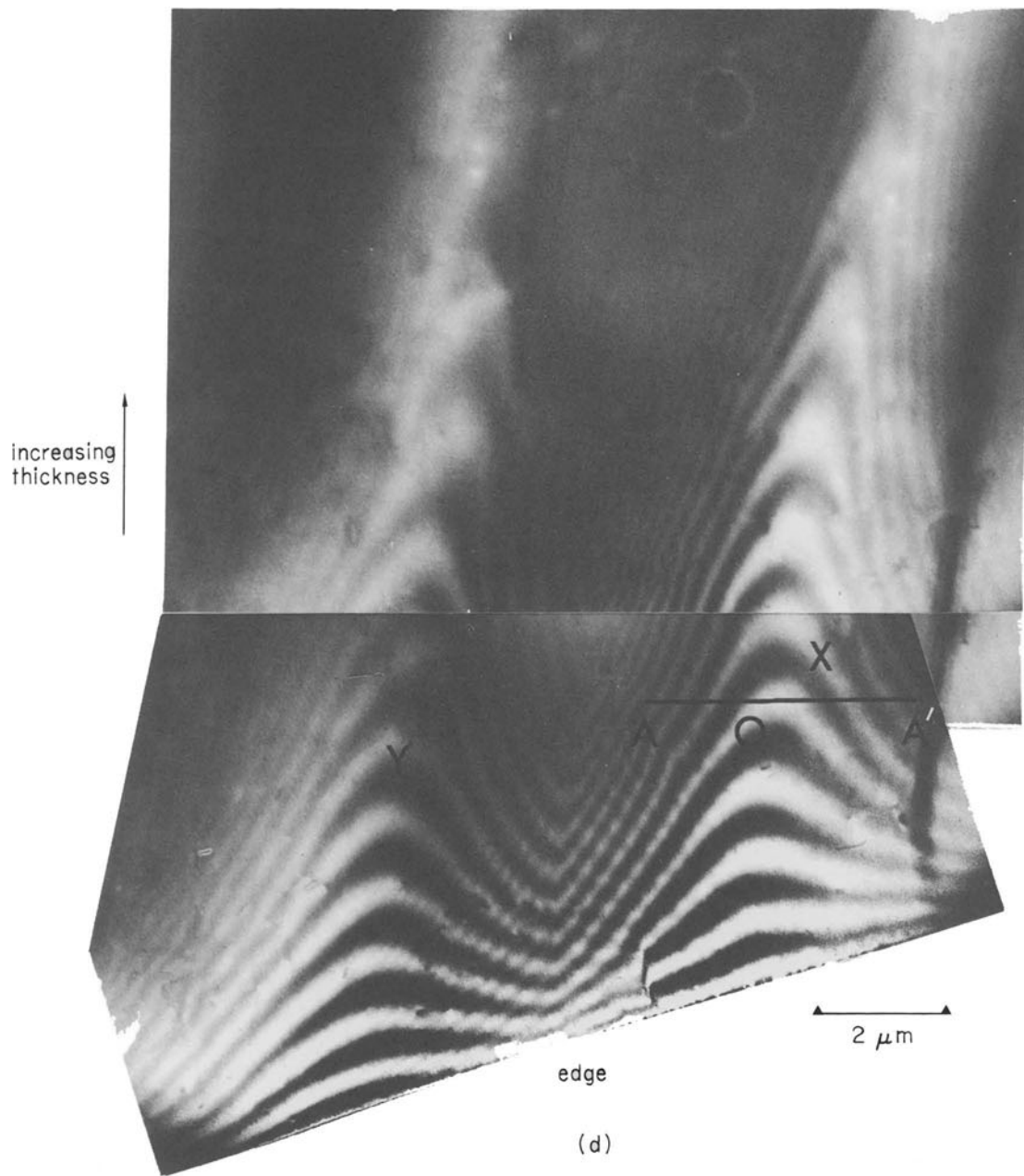
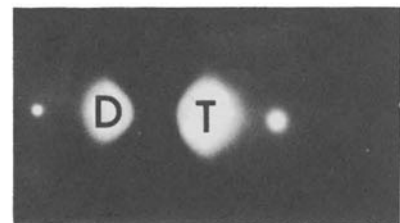


Figure 3.3 (a) A thin crystal bent into a large radius showing reflection from the opposite sides of the same set of (hkl) crystal planes. (b), (c) The intensity distribution versus position for BF and DF (rocking curves). (d) The BF image corresponding to (a). (e), (f) are SADPs showing opposite two-beam diffracting conditions at Y and X in (d)



($\bar{1}\bar{1}\bar{1}$)
beam

(e)



(111)
beam

(f)

Figure 3.3 (continued)

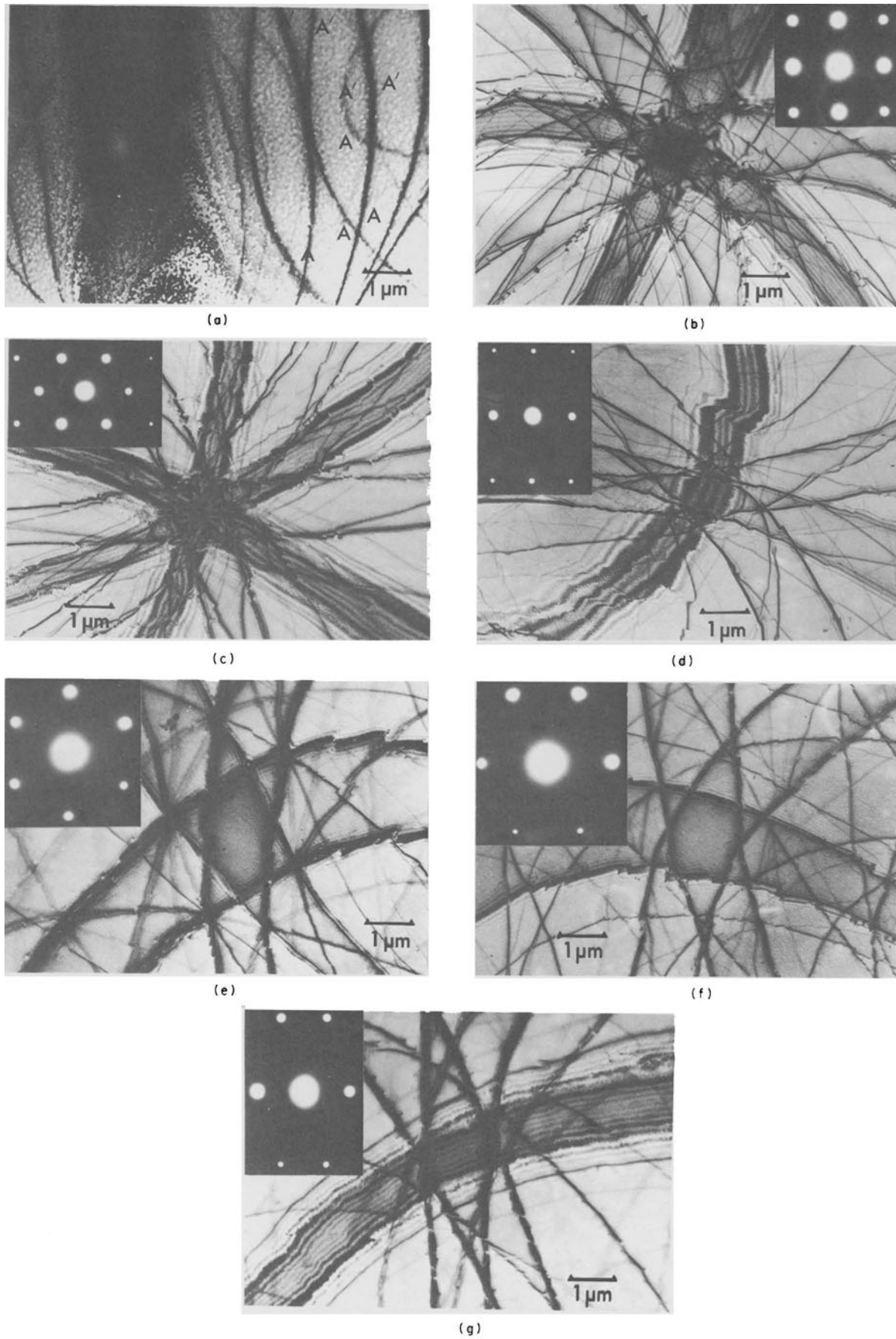


Figure 3.4 (a) Bend contours AA' and bend centres in aluminium with associated SADP inset, (b) [100], (c) [110], (d) [112], (e) [111], (f) [114], (g) [013]

of a dislocation as an example, see figure 3.5(a), the equations corresponding to equations (3.1) are

$$\frac{d\phi_0}{dz} = \frac{i\pi}{\xi_0} \phi_0 + \frac{i\pi}{\xi_g} \phi_g \exp(2\pi i s z + 2\pi i \mathbf{g} \cdot \mathbf{R}) \quad (3.5a)$$

$$\frac{d\phi_g}{dz} = \frac{i\pi}{\xi_g} \phi_0 \exp(-2\pi i s z - 2\pi i \mathbf{g} \cdot \mathbf{R}) + \frac{i\pi}{\xi_g} \phi_g \quad (3.5b)$$

if the displacement in the column arising from, for example, the strain field of the dislocation at DD in figure 3.5(a) is $\mathbf{R}(z)$. In these equations all symbols have the same meaning as in equations (3.1) and $\mathbf{g}$ is the reciprocal lattice vector for the operative reflecting plane, see figure 2.8. Absorption is included by making the same substitution as in section 3.2.2. Effectively these equations mean that the distortion of the lattice causes a phase factor $\exp(-i\alpha)$, where $\alpha = 2\pi \mathbf{g} \cdot \mathbf{R}$, to be superimposed on the normal scattering process for the perfect crystal. If the atom is displaced by $\mathbf{R}$, the phase of the scattered wave is changed by $\exp(-2\pi \mathbf{g} \cdot \mathbf{R})$. Since this phase factor in equation (3.5a) describes scattering from diffracted to transmitted beam as $\exp(+2\pi \mathbf{g} \cdot \mathbf{R})$ for the reverse process it is $\exp(-2\pi \mathbf{g} \cdot \mathbf{R})$ in equation (3.5b). Thus, when we form a BF image of a distorted crystal in the electron microscope by excluding the diffracted electrons with the objective aperture, section 1.5.2, we obtain a bright image with some dark regions arising from the enhanced diffraction by the strain field of the crystal defect.

The physical significance of equations (3.5) may be made clear as follows. Substituting $\phi'_0 = \phi_0(z) \exp(-\pi i z / \xi_0)$ and $\phi'_g = \phi_g(z) \exp(2\pi i s z - \pi i z / \xi_0 + 2\pi i \mathbf{g} \cdot \mathbf{R})$ in equations (3.5) leads to

$$\frac{d\phi'_0}{dz} = \frac{\pi i}{\xi_g} \phi'_g \quad (3.6a)$$

$$\frac{d\phi'_g}{dz} = \frac{\pi i}{\xi_g} \phi'_0 + \left(2\pi i s + 2\pi i \mathbf{g} \cdot \frac{d\mathbf{R}}{dz} \right) \phi'_g \quad (3.6b)$$

Thus the strain field of the dislocation enters the equation as $\mathbf{g} \cdot (d\mathbf{R}/dz)$ and acts like a local rotation of the reflecting planes which changes the value of s . Consequently the local diffracted intensity can be considered to increase because the local Ewald sphere construction passes nearer the $s = 0$ position in reciprocal space, see figure 2.16.

Of course the above approach also enables strain-free situations to be described in which the phase factor α arises from the simple displacement of one part of the crystal relative to another, by a vector $\mathbf{R}$, such as occurs in the case of stacking faults, see section 3.6.

Note that the characteristic features of a perfect crystal (section 3.2.3) will also be present but the details may be destroyed if a high density of crystal defects is present.

3.3 Relevance of Diffraction Theory to Studies of Crystal Defects

The differential equations described in the previous sections form the basis for the calculation of image contrast for diffraction images of crystal defects. Details of the calculations may be found in the books and review papers listed in section 3.2.

In practice several physically equivalent formulations of the Darwin–Howie–Whelan equations exist. The scattering may be described either in terms of plane waves (Howie and Whelan, 1961; Takagi, 1962; Taupin, 1964), as for equations (3.1), (3.5), or Bloch waves (Howie, 1963; Wilkens, 1964) or modified Bloch waves (Wilkens, 1966). These different mathematical expressions are all used for computing images to be expected from certain crystal defects of which four general classes may be distinguished.

- (1) A locally varying strain field (dislocation, misfitting precipitate, etc.).
- (2) A planar defect like a stacking fault at the junction of two undistorted regions of the foil.
- (3) Voids or gas bubbles in the foil.
- (4) Local changes in the extinction distance (section 3.2.3.1), for example coherent precipitates.

All of these situations are within the scope of the ideas expressed in sections 3.2.1, 3.2.2. Contrast calculations for strain field defects of type 1 generally require the numerical integration of the Darwin–Howie–Whelan equations in some form often using the Bloch wave concept because the time for computation is much reduced. However, the differential equations based on modified Bloch waves are particularly useful for slowly varying strain fields (Wilkens *et al.*, 1967; Chik *et al.*, 1967). Defects of the type (2) and (4) are not accompanied by lattice strains under ideal conditions and the best approach involves the solution of the Darwin–Howie–Whelan equations for the appropriate boundary conditions at the internal surface, that is the defect. This type of calculation is most easily performed using the scattering matrix approach (Howie and Whelan, 1961; Amelinckx, 1970).

Despite these different approaches equations (3.1) and (3.5) are useful because they contain a number of parameters that are important to the experimentalist.

- (1) The reflecting plane vector $\mathbf{g}$.
- (2) The deviation from the Bragg position s . Both (1) and (2) are controlled by tilting the specimen.

(3) The extinction distance ξ_g (see section 3.2.3.1) which increases with increasing g and decreases with increasing $|s|$; ξ_g is often used in conjunction with s to describe the deviation from the Bragg position as $w = \xi_g s$.

(4) The anomalous absorption coefficient $\text{ANO} = \xi_g/\xi'_g$ which is a function of the operative reflection, the atomic number and structure of the material. The value of ANO for cubic materials has been calculated by Humphreys and Hirsch (1968). In general fringe images such as stacking faults are sensitive to the value of ANO and may even be used to determine it (Clareborough, 1971). However, dislocation images are relatively insensitive to small variations of ANO and a value of 0.1 is normally used in computation of contrast.

It is by careful control of (1) and (2) with the full knowledge of (3) and (4) that crystal defects may be quantitatively analysed.

3.3.1 Experimental Conditions for Quantitative Analysis

The following conditions must be satisfied experimentally for quantitative analysis as described in sections 3.4–3.14, 3.21 except where specifically stated otherwise. Different experimental conditions are described for the remaining sections and the reader is referred to them for details.

(1) Images should always be produced under *two-beam dynamical conditions* so that the preceding theory may be quantitatively applied.

(2) In BF images deviation from the Bragg position, s should be small and positive (bright Kikuchi line just outside diffraction spot, see figure 2.37(c)). Under these conditions both better transmission and sharper clearer contrast are obtained than at $s = 0$ or $-ve$, see figure 3.2. Indeed for $s - ve$ the background intensity is very low. Values of $w = \xi_g s \sim 0.2$ – 1.0 may be

used as an approximate guide. (See section 2.10.2 for methods of measuring s .)

(3) In dark field the deviation from the Bragg position may be up to ± 1 but, if comparison is to be made with bright field images, specific values of w may be required depending on the case under consideration (see later sections).

(4) Thin areas < 2 – $3\xi_g$ should be avoided because significant rearrangement of mobile crystal defects occurs (Hirsch *et al.*, 1967) particularly if the friction stress on dislocations is low (metals and alloys with f.c.c. or c.p.h. crystal structure).

(5) Regions of rapidly changing thickness should be avoided if the detailed image contrast is to be studied because of the marked changes in background intensity produced by thickness fringes (see section 3.2.3.1).

(6) The most suitable specimen thickness for observation at 100 kV under all reflections are those 5 – $8\xi_g$ thick, where ξ_g is the extinction distance of a low-index reflection.

(7) Where maximum penetration is important, careful selection of the operative reflection is essential because penetration depends upon the operative reflection even in pure metals. For example in hexagonal metals $\{2\bar{2}00\}$ -type reflections are more intense and give better penetration than $\{1\bar{1}00\}$ -type reflections. Furthermore, systematically absent reflections which are present because of double diffraction, for example (0001) and $\{\bar{1}2\bar{1}2\}$ in hexagonal metals, also give poor penetration. Finally, in ordered materials superlattice reflections may also be weak leading to poor penetration and diffuse images because ξ_g is large and w is therefore usually large.

3.3.2 Recognition of Dynamical and Kinematical Diffracting Conditions in the Electron Microscope

Although the dynamical theory of electron diffraction describes most images from crystalline specimens in the electron microscope, kinematical

Table 3.1 Recognisable features of SADP and images for dynamical and kinematical diffracting conditions

SADP		BF image	
Dynamical	Kinematical	Dynamical	Kinematical
bright Kikuchi lines very close to strong diffracted spot such that $w = -1 \rightarrow +1.0$, see figure 2.37(c)	bright Kikuchi lines some distance from strong diffracted beam $w \gtrsim \pm 1.0$	$s + ve$ well defined; thickness fringes following the contours of the edge of the specimen, section 3.2.3.1, figure 3.2(d) a high-contrast image is obtained, figure 3.2(d) a slight overall graininess of the background is visible in many materials, figure 3.2(d)	few thickness fringes BF, except 1–2 very near edge of specimen, figure 3.2(g) a low-contrast image is obtained, figure 3.2(g) the overall nature of the image is diffuse, figure 3.2(g)

conditions can frequently arise. These occur for the following.

- (1) Very thin specimens, that is $< \frac{1}{2}\xi_g$, $w \gtrsim 0.3$.
- (2) Two-beam diffracting conditions far removed from the exact Bragg reflecting position, that is $w \gg 1$.

It is important to be able to recognise dynamical and kinematical diffracting conditions rapidly both in the diffraction pattern and in the image. Therefore the features of the two conditions are summarised in table 3.1. It is important to realise that there is a smooth transition from dynamical to kinematical conditions which depends on $w (= s\xi_g)$. Consequently, kinematical conditions occur as s increases, that is as the distance between a bright Kikuchi line and its associated diffraction spot increases, see section 2.10.2. Furthermore, kinematical conditions arise for smaller displacements of the bright Kikuchi line for second- and third-order reflections because ξ_g is larger (see section 3.2.3.1).

II. QUANTITATIVE ANALYSIS OF CRYSTAL DEFECTS USING DIFFRACTION CONTRAST

In part II the important image characteristics of a number of individual crystal defects are summarised. Procedures for identifying and obtaining quantitative information about each type of defect are outlined, assuming dynamical ($w = s\xi_g 0-1$ bright field and $w = 0-\pm 1$ dark field) conditions except where specified.

In many cases images of similar types of defect such as stacking faults and antiphase domain boundaries, differ only in detail. Methods for differentiation between such similar images are summarised in section 4.

3.4 Dislocations

A study of dislocation images is useful because it enables the direction of $\mathbf{b}$ to be determined. In addition, using computer matching techniques, see section 3.4.3.2, both the magnitude and sense of $\mathbf{b}$ may be obtained. In this section some features of dislocation images are discussed, emphasising those that are useful in obtaining quantitative information.

3.4.1 A Simple Treatment of Dislocation Images

The image may be calculated using equations (3.5) and depends upon the operative reflection $\mathbf{g}$, the deviation s from the Bragg position and the displacement field $\mathbf{R}$ of the dislocation. The latter depends upon the dislocation line direction $\mathbf{u}$, the Burgers vector $\mathbf{b}$, orientation within the thin foil and the elastic anisotropy of the material. However, initially the case of elastically isotropic material is used to describe qualitatively the useful features of the image.

Consider the simple case in figure 3.5(a) in which a straight screw dislocation Burgers vector $\mathbf{b}$ lies parallel to the surface of a thin foil at a depth y . The displacement $\mathbf{R}$ of the atoms in the element dz of the column defined relative to the dislocation core by the coordinate r and the angle β is given,

for an infinite elastically isotropic material, as

$$\mathbf{R} = \frac{\mathbf{b}\beta}{2\pi} \quad (3.7a)$$

Thus α to be inserted in equation (3.5) is

$$\alpha = \mathbf{g} \cdot \mathbf{b}\beta$$

Clearly the form of the dislocation image depends directly upon the term $\mathbf{g} \cdot \mathbf{b}$ which may be either zero or an integer for a perfect dislocation, because it is a scalar product of a real lattice vector with that of a reciprocal lattice. However, for a partial dislocation for which $\mathbf{b}$ is a fractional lattice vector, $\mathbf{g} \cdot \mathbf{b}$ may be zero, a fraction or an integer, see section 3.4.4.

For perfect or partial dislocations with $\mathbf{g} \cdot \mathbf{b} = 1, 2, \text{etc.}$, the image is a dark line in BF arising

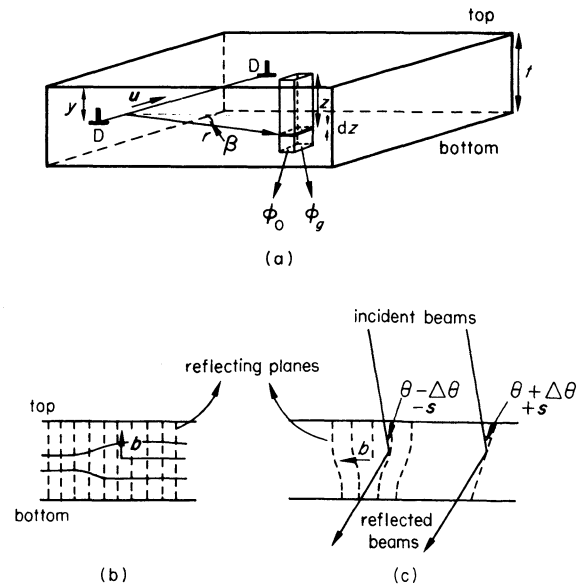


Figure 3.5 (a) The column approximation for a thin foil containing a screw dislocation DD, (b) an edge dislocation, $\mathbf{b}$ parallel to the foil normal and (c) perpendicular to it. The dashed lines are the traces of the reflecting planes

Table 3.2 Known cases of invisibility of dislocations in elastically isotropic crystals based on image calculation

Dislocation	Configuration in the foil	Reference
screw, $\mathbf{g} \cdot \mathbf{b} = 0$	$\mathbf{u}$ parallel to foil surface	Howie and Whelan (1962)
screw, $\mathbf{g} \cdot \mathbf{b} = 0$	$\mathbf{u}$ inclined to foil surface	Howie and Whelan (1962)
mixed $\mathbf{g} \cdot \mathbf{b} = 0$, and $\mathbf{g} \cdot \mathbf{b}_e = 0$	$\mathbf{u}$ parallel to foil surface	Howie and Whelan (1962)
edge $\mathbf{g} \cdot \mathbf{b}_e = 0$, and $m = \frac{1}{3}\mathbf{g} \cdot \mathbf{b}_e \wedge \mathbf{u} < 0.08$	$\mathbf{u}$ parallel to foil surface and $\mathbf{b}$ parallel to foil normal	Howie and Whelan (1962)
edge $\mathbf{g} \cdot \mathbf{b}_e = 0$	$\mathbf{u}$ parallel to foil surface, that is $\mathbf{g} \cdot \mathbf{b}_e \wedge \mathbf{u} = 0$	Hirsch <i>et al.</i> (1967)

from the finite value of α in equation (3.5a). For partial dislocations with fractional values of $\mathbf{g} \cdot \mathbf{b}$, the contrast is complicated and all cases have not been studied (see section 3.4.4). *However, the single most useful feature of dislocation images is the invisibility of dislocations in BF and DF images when $\mathbf{g} \cdot \mathbf{b} = 0$.* In this situation equation (3.5a) is identical with equation (3.1a), that is the crystal behaves as if the dislocation were not present. It is this property of the electron microscope image of dislocations, known as the ' $\mathbf{g} \cdot \mathbf{b} = 0$ invisibility criterion', which enables the direction of the Burgers vector of a dislocation to be obtained as described in section 3.4.3.1.

As pointed out in section 3.2.4 with equations (3.6), the dislocation image may be considered in terms of the distortion of the reflecting planes. The $\mathbf{g} \cdot \mathbf{b} = 0$ situation corresponds to the case when $\mathbf{b}$ lies in the reflecting plane, figure 3.5(b). Here an edge dislocation lies parallel to the surface of a thin foil with $\mathbf{b}$ parallel to the foil normal. The solid lines correspond to a set of distorted planes but the dashed lines are, to a first approximation, undistorted planes because they contain $\mathbf{b}$. Thus, as far as the diffracted and transmitted electrons are concerned, the crystal is not distorted, that is it behaves as if the dislocation were not present. However, if the reflecting plane does not contain $\mathbf{b}$, as shown in figure 3.5(c), the reflecting planes shown as dashed lines are distorted. Thus if the angle of incidence of the electrons is $\theta + \Delta\theta$ in the undistorted specimen far from the dislocation (that is everywhere else in the specimen) s will be positive and there will be a high intensity everywhere in the BF image because s positive corresponds to a low diffracted intensity, figure 2.16, that is high transmitted intensity. However, near the dislocation the reflecting planes are distorted towards $s = 0$ when the diffracted intensity increases, that is transmitted intensity will be reduced (see figure 2.16) and the dislocation is visible as a dark line on a bright background in BF.

3.4.2 Images of Perfect Dislocations

3.4.2.1 Elastically isotropic materials

This section is applicable to materials with anisotropy factor* $A \approx 1$, that is W, Al, Zn, Mg,

Ti, some MC carbides. If equation (3.7a) is rewritten for a mixed dislocation

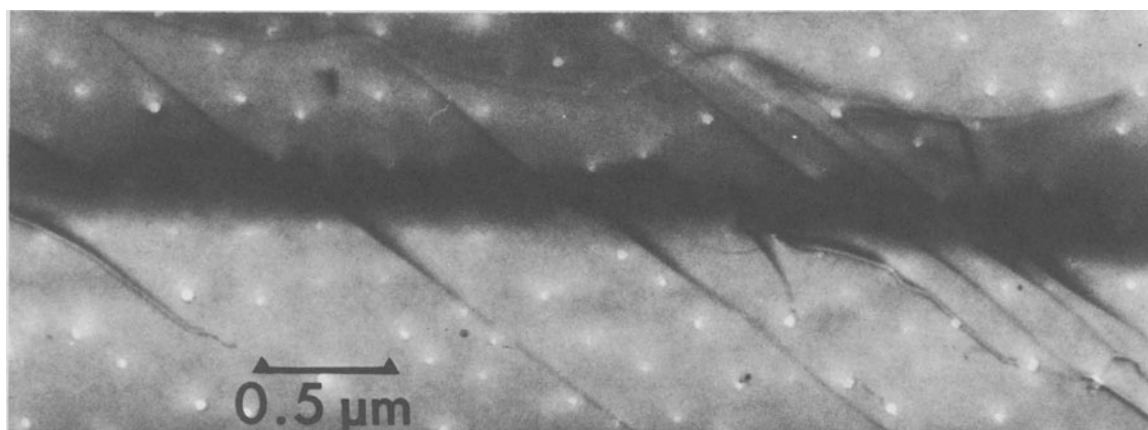
$$R = \frac{1}{2\pi} \left\{ \mathbf{b}\beta + \frac{\mathbf{b}_e \sin 2\beta}{4(1-\nu)} \right\} + \mathbf{b} \wedge \mathbf{u} \left[\left\{ 1 - \frac{2\nu}{2(1-\nu)} \right\} \ln r + \frac{\cos 2\beta}{4(1-\nu)} \right] \quad (3.7b)$$

where $\mathbf{b}_e$ is the edge component of the Burgers vector and $\mathbf{u}$ is a unit vector along the dislocation line. Thus, for a mixed dislocation, image contrast depends upon $\mathbf{g} \cdot \mathbf{b}$, $\mathbf{g} \cdot \mathbf{b}_e$ and $\mathbf{g} \cdot \mathbf{b} \wedge \mathbf{u}$. For edge dislocations $\mathbf{b} = \mathbf{b}_e$ and contrast depends on $\mathbf{g} \cdot \mathbf{b}_e$ and $\mathbf{g} \cdot \mathbf{b}_e \wedge \mathbf{u}$. It is an important consequence of this discussion that *a dislocation need not be invisible* if $\mathbf{g} \cdot \mathbf{b} = 0$ because $\mathbf{g} \cdot \mathbf{b}_e$ or $\mathbf{g} \cdot \mathbf{b} \wedge \mathbf{u}$ may not be zero.

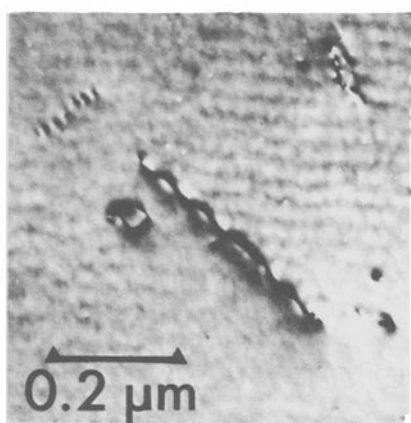
Thus, $\mathbf{g} \cdot \mathbf{b}$, $\mathbf{g} \cdot \mathbf{b}_e$ and $\mathbf{g} \cdot \mathbf{b} \wedge \mathbf{u}$ must all be zero for complete invisibility, which is a very restricted criterion compared with the $\mathbf{g} \cdot \mathbf{b} = 0$ criterion discussed above. However, in practice only faint residual contrast generally occurs when $\mathbf{g} \cdot \mathbf{b} = 0$, but $\mathbf{g} \cdot \mathbf{b}_e$ and $\mathbf{g} \cdot \mathbf{b} \wedge \mathbf{u}$ are $\neq 0$ and this has been interpreted as indicating dislocation invisibility. Consequently, the ' $\mathbf{g} \cdot \mathbf{b} = 0$ effective invisibility' criterion has been used successfully in a large number of cases to obtain the dislocation Burgers vectors. Although these rules require some experience in recognising 'effective invisibility' they are simple to use and, provided complete self-consistency, that is three cases of invisibility, is demanded, are reasonably satisfactory in determining the direction of $\mathbf{b}$ in materials that are almost elastically isotropic. The known cases of invisibility based on image contrast calculations are listed in table 3.2. Detailed calculations for all combinations of $\mathbf{B}$, $\mathbf{FN}$, $\mathbf{u}$, $\mathbf{b}$, $\mathbf{g}$, s have not been performed, although a limited number have been reported by Howie and Whelan (1962). The most important general features of $\mathbf{g} \cdot \mathbf{b} \neq 0$ dislocation images are described below.

(1) The BF image is a black line slightly displaced from the core and shifts as the sign of $\mathbf{g} \cdot \mathbf{b} \wedge \mathbf{s}$ changes. Figure 3.6(a) shows an example of this effect when a bend contour runs across a dislocation. Note displacement of the dislocation image because on crossing the contour s changes

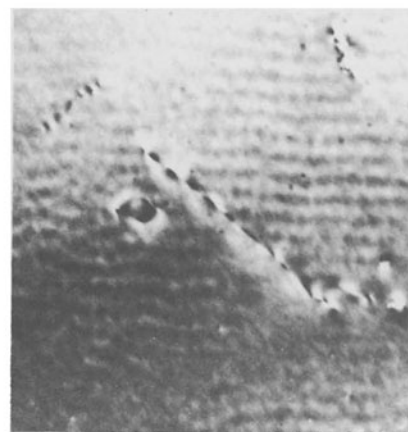
* The elastic anisotropy factor A is defined for cubic and hexagonal crystal structures in appendix 3.



(a)



(b)



(c)

Figure 3.6 (a) Dislocations parallel to the foil surface and crossing a bend contour. Note the image moves from one side of the core to the other. (b) BF and (c) CDF images of an inclined dislocation in aluminium

sign. This image displacement can be given a simple physical description using figure 3.5(c). Here the rotation of the reflecting planes on approaching the core enables the Bragg law to be exactly satisfied before the core is reached. Since maximum overall diffracted intensity occurs when the Bragg law is satisfied ($s = 0$), see section 2.4, and these diffracted electrons are excluded from the BF image by the objective aperture (see section 1.5.2); the dislocation is visible in BF as a black line displaced from the core.

(2) The BF and DF images are not necessarily complimentary at the top of the foil, because of anomalous absorption, see section 3.3.2, compare figure 3.6(b), (c).

(3) Zig-zag contrast may occur near the surface of the foil, see figure 3.7(a), or for steeply inclined dislocations in this region $w \sim 0.1-1.0$, see figure 3.7(b).

(4) Double ($w \approx 0.1$) or single ($w \sim 0.1-1.0$)

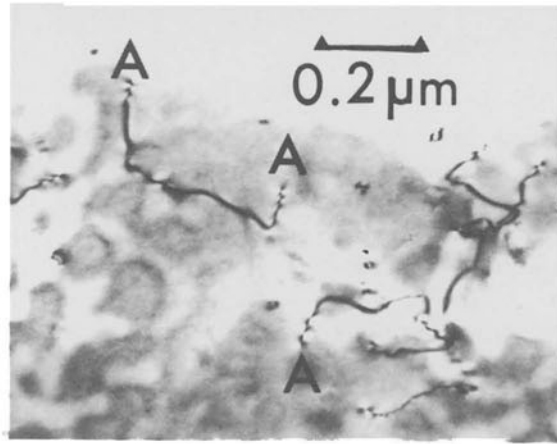
images may occur for the same value of $g \cdot b = 2$, compare figures 3.7(c), (d).

(5) Double images can occur if two-beam conditions are not strictly adhered to, see figure 3.7(e), (f).

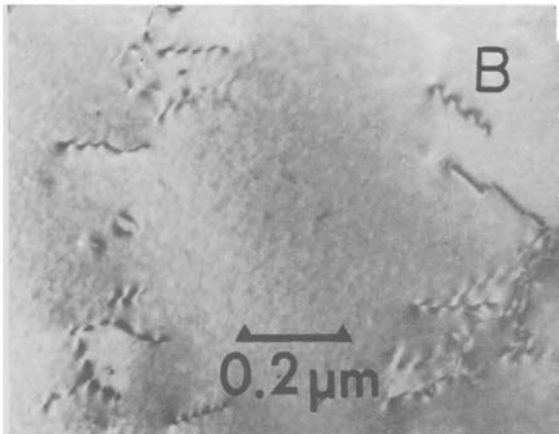
These image details are not generally used to obtain quantitative information about the dislocations. Consequently, it is only necessary to be aware of them to avoid errors in interpretation, for example the dislocation in B in figure 3.7(b) is not a helix.

3.4.2.2 Elastically anisotropic materials

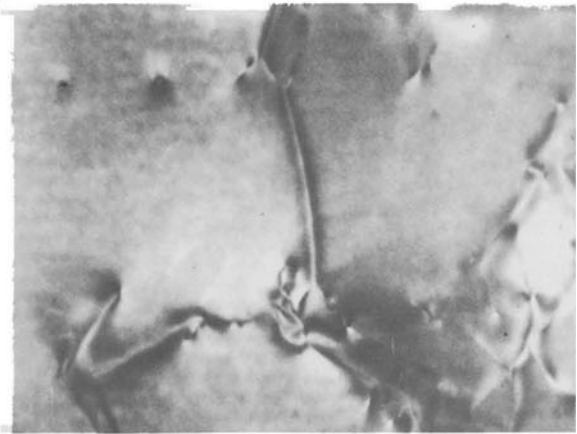
This section is applicable to most materials not listed in the previous section. Extensive calculations by a number of workers (Head *et al.*, 1967a, b, 1973; Humble, 1970) have shown that elastic anisotropy considerably modifies the above discussion, in that the reflecting planes are in general



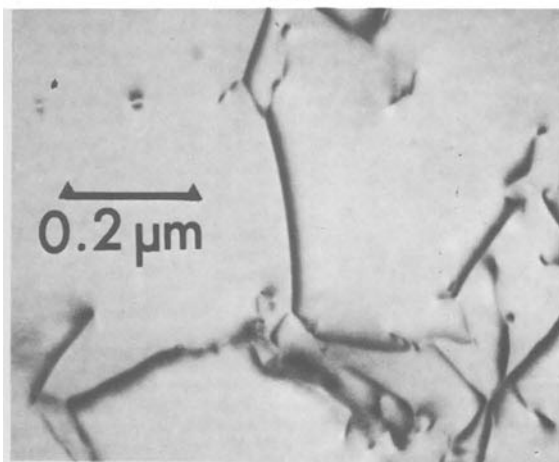
(a)



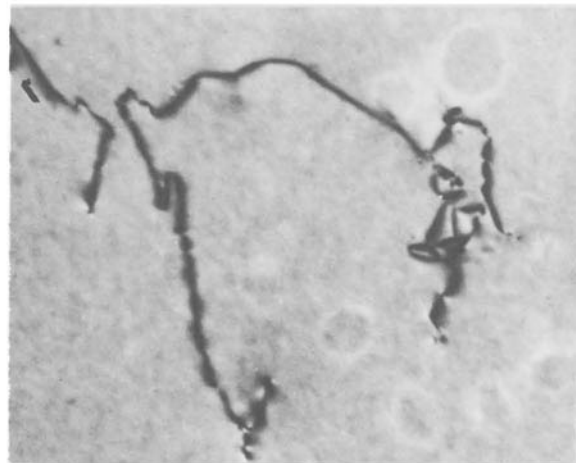
(b)



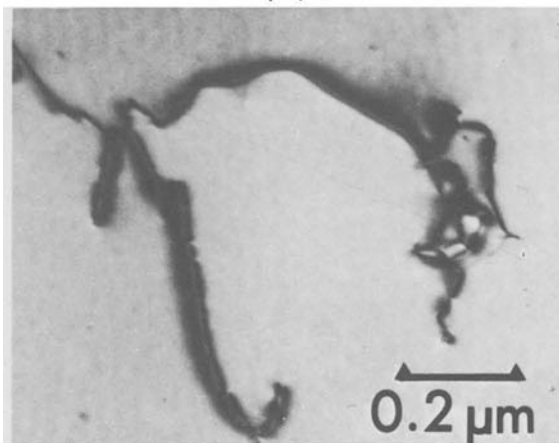
(c)



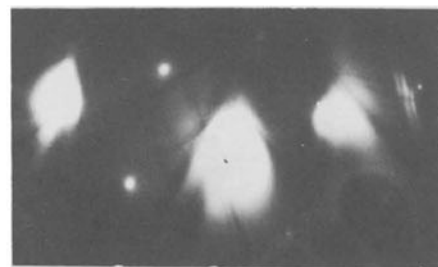
(d)



(e)



(f)



(g)

Figure 3.7 BF images of dislocations in aluminium showing (a) dotted contrast at A near the foil surface, (b) oscillating contrast at the steeply inclined dislocation B, (c) a double image for a dislocation with $g \cdot b = 2$, $s = 0$, (d) a single image for the same dislocation $g \cdot b = 2$, $s \neq 0$, (e) a single dislocation image with two-beam conditions and (f) a double image of the same dislocation with three-beam conditions shown in (g)

distorted because of the different form of the dislocation displacement field. Consequently, it is sometimes impossible to obtain well-defined conditions of invisibility. This considerably complicates the determination of the Burgers vectors as described in the next section.

There are a few particular cases when elastically anisotropic material behaves pseudo-isotropically (Nye, 1957). This behaviour occurs in cubic materials for pure edge or screw dislocations running normal to the elastic symmetry planes $\{110\}$ and $\{100\}$. In hexagonal crystal structures the basal plane and all planes perpendicular to it are elastic symmetry planes. Consequently, all dislocations lying in the basal plane and perpendicular to it exhibit pseudo-elastic isotropy.

3.4.3 Determination of the Burgers Vector of Perfect Dislocations

3.4.3.1 Elastically isotropic materials

For the situations outlined in table 3.2, the ' $\mathbf{g} \cdot \mathbf{b} = 0$ ' criterion may be applied to determine the direction of $\mathbf{b}$. Thus the dislocation will be invisible when $\mathbf{b}$ lies in the reflecting plane. Forming a series of images with two-beam conditions $w \approx 1.0$ for a series of individual reflections enables two such conditions to be obtained and, because $\mathbf{b}$ is common to both reflections, $\mathbf{b}$ must be the zone axis of the two planes.

An example of such an analysis is shown in figure 3.8 for a dislocation in aluminium. Here,

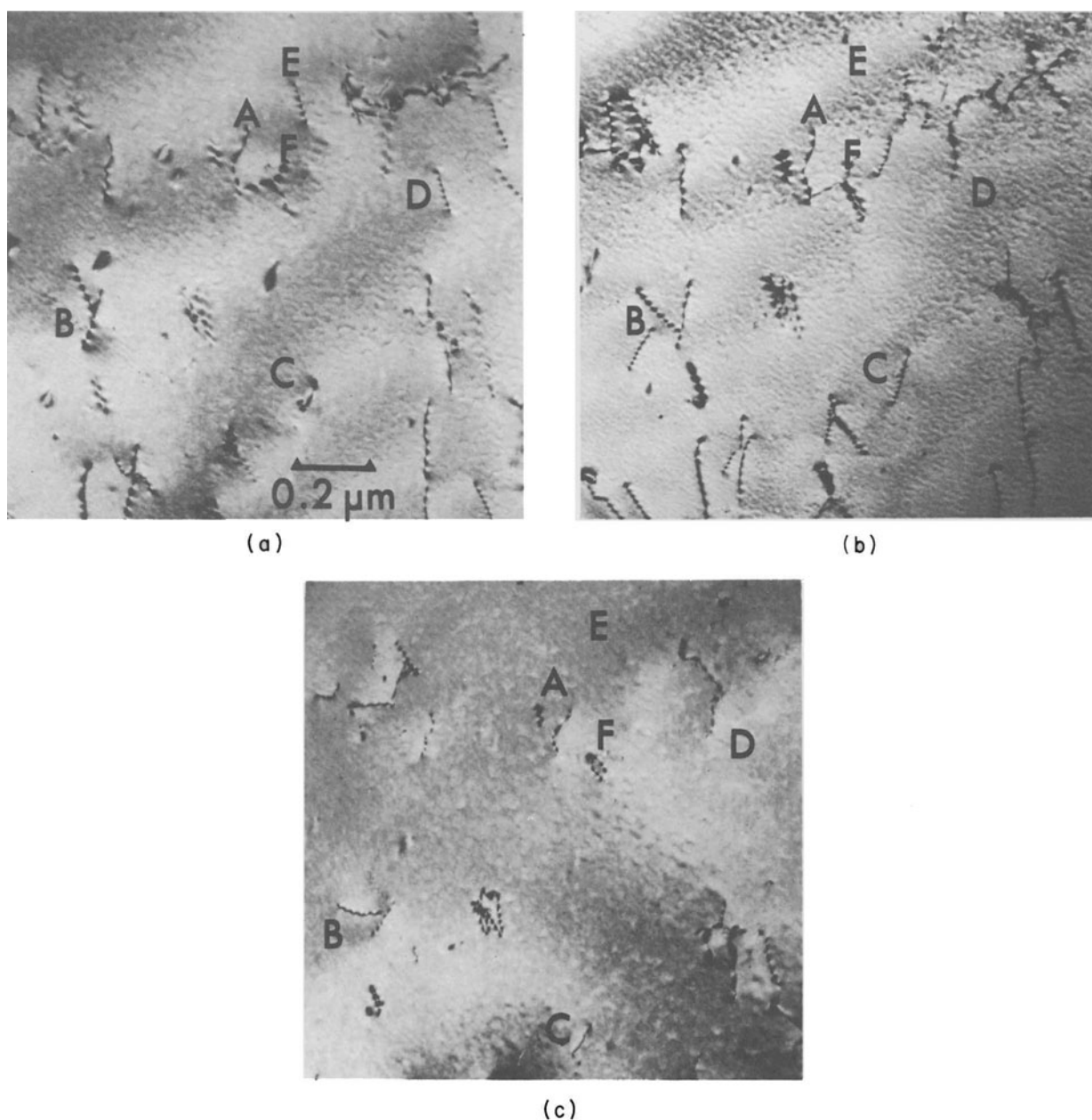


Figure 3.8 The same region of an aluminium foil with different operative reflections (a) (020), (b) (200), (c) (11 $\bar{1}$)

Table 3.3 Possible values of $g \cdot b$ for the dislocations under the three reflections in figure 3.8

Figure No.	g	b						Dislocation contrast D, E
		$\frac{1}{2}[110]$	$\frac{1}{2}[101]$	$\frac{1}{2}[011]$	$\frac{1}{2}[110]$	$\frac{1}{2}[101]$	$\frac{1}{2}[011]$	
3.8(a)	(020)	1	0	1	1	0	-1	visible
3.8(b)	(200)	1	1	0	-1	-1	0	invisible
3.8(c)	(11 $\bar{1}$)	1	0	0	0	-1	-1	invisible

the same area is imaged under a series of reflections, and the dislocations marked D, E, figure 3.8(a), with line directions $[011]$ may be analysed. Both dislocations are visible with the (020) reflection, figure 3.8(a), but invisible with (200) and (11 $\bar{1}$) reflections. Table 3.3 summarises the values of $g \cdot b$ for all possible b assuming $\frac{1}{2}\langle 110 \rangle$ type and calculated using the approach outlined in appendix 11. Using the $g \cdot b = 0$ criterion, $b = \frac{1}{2}[011]$, which has $g \cdot b = 0$ for (200) and (11 $\bar{1}$) for which both dislocations are invisible. Complete invisibility is obtained under these two reflections because the dislocations are screw orientation and both $g \cdot b_e$ and $g \cdot b \wedge u = 0$.

Note that dislocation C in figure 3.8(a) exhibits residual contrast typical of a dislocation inclined to the foil surface when $g \cdot b = 0$ but $g \cdot b_e$ and $g \cdot b \wedge u \neq 0$. Figure 3.9 shows similar contrast for a large edge dislocation loop in the plane of the foil with $b \frac{1}{2}[011]$, that is $g \cdot b = 0$. Those segments of the loop parallel to the operative g have $g \cdot b \wedge u = 0$ and are invisible. However, the remaining segments have $g \cdot b \wedge u \gtrsim 0.64$ and show characteristic faint double images.

It was pointed out earlier in section 3.4.2.1 that the ' $g \cdot b = 0$ effective invisibility' criterion has been successfully used by a large number of workers to determine the direction of b for cases not listed in table 3.2. It is recommended that this technique only be applied if at least *three consistent cases* of effective invisibility are found with $w < 1.0$ and using low-index reflections. Under such circumstances the correct determination of b is almost certain but not guaranteed. The image computation techniques described in the following section may be used if there is any doubt.

In this context it is important to realise that $g \cdot b = 1$ images can be confused with those with $g \cdot b = 0$ if w is allowed to become too large $\gtrsim 1.5$ (Loretto and France, 1969a, b). Frequently for a given reflection, $g \cdot b$ may be 1 for some dislocations and 2 for others with different b . As w increases dislocations with $g \cdot b = 1$ become invisible before those with $g \cdot b = 2$. Consequently, it must be realised that the operator may inadvertently select a value of w when observing the image which causes $g \cdot b = 2$ dislocations to be

sharp and in good contrast whereas $g \cdot b = 1$ dislocations are almost invisible and are erroneously concluded to be $g \cdot b = 0$ dislocations. Therefore, it is recommended that at least three cases of invisibility are obtained for values of $w < 1.0$. Thus the position of the bright Kikuchi line in the SADP should always be close to the strongly diffracting spot, figure 2.37(c). This effect is important and can result in erroneous determinations of b , particularly when high-index reflections are used, $\{310\}$, $\{222\}$, $\{321\}$, $\{311\}$, $\{331\}$ and $\{420\}$, see France and Loretto (1968) and Loretto and France (1969a, b). With care this problem can be avoided using currently available high-tilt specimen holders because high-index reflections are unnecessary as enough low-index reflections should be obtainable by extensive specimen tilting.

3.4.3.2 Elastically anisotropic materials

Most materials are elastically anisotropic and the $g \cdot b = 0$ invisibility criterion is frequently inapplicable. For example, for screw dislocations $b = \frac{1}{2}\langle 111 \rangle$ in α -iron and niobium considerable contrast can occur when $g \cdot b = 0$ (Humble, 1967). Under these circumstances the very beautiful technique of image matching (Head, 1967; Humble, 1968a, 1970) must be used. A detailed review of this approach has been given by Head *et al.* (1973) and extensive coverage would be out of place here. The technique is based upon computer simulation of a series of dislocation images obtained with different operative reflections for the exact reflecting conditions. For each image g, s, u, B, FN and foil thickness are known and b is varied until a match is obtained. In practice b is usually 'guessed' on the basis of the ' $g \cdot b = 0$ effective invisibility' rule and confirmed using computer image matching unless the material is so strongly anisotropic that strong images are always obtained for all reflections with $w \lesssim 1.0$.

As an illustration of the usefulness of computer matching figures 3.10–3.14 show the determination of b for a dislocation in β -brass (anisotropy factor $A^* = 7.8$) taken from the work of Head *et al.* (1967a, b) and Humble (1970). The crystallographic information included in the figure captions has

* For a definition of A see appendix 3.

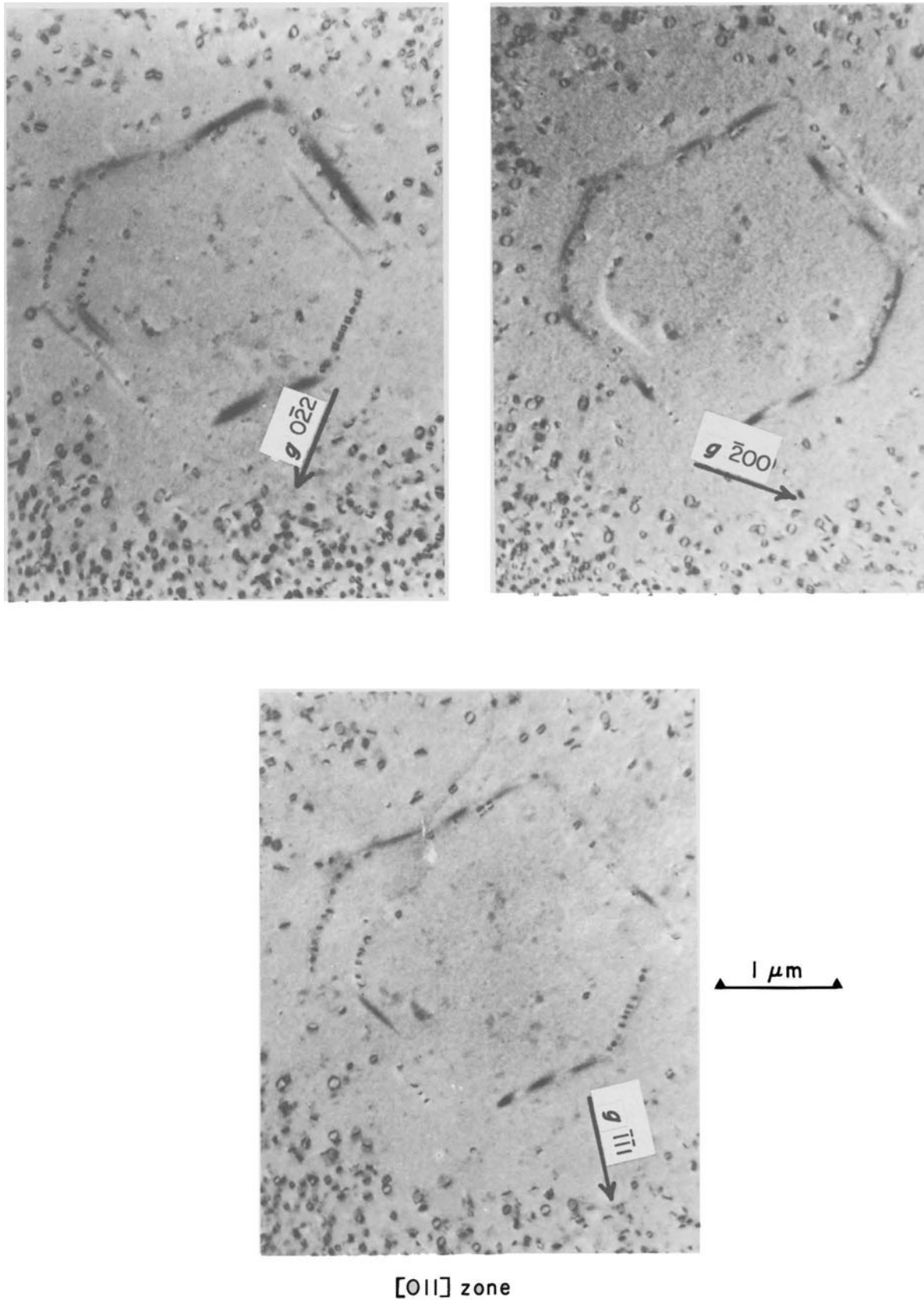


Figure 3.9 A quenched-in climb source diamond shaped loop $g \cdot b = 0$ for all three reflections, that is b is parallel $[011]$ the foil normal. Note that the invisible regions $g \cdot b \wedge u \lesssim 0.64$ are different for each reflection. Each image shows only 'residual' contrast [Courtesy of *The Philosophical Magazine*]

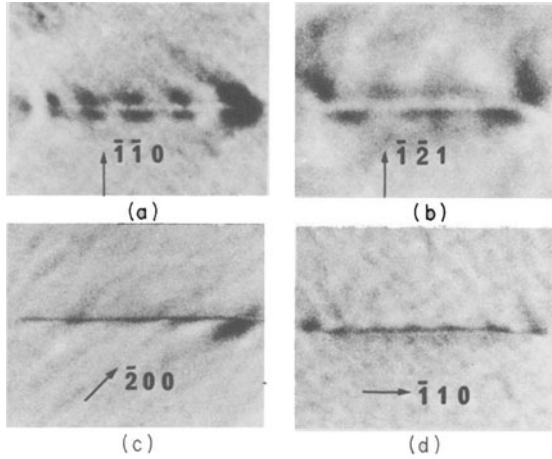


Figure 3.10 Experimental micrographs showing the images of an unknown dislocation in β -brass taken under four different reflecting vectors which are marked. The dislocation line direction, u , is close to $[\bar{1}11]$ and FN is close to $[001]$. B is close to $[001]$ for (a), (c) and (d) and to $[113]$ for (b). w is about 0.5 for (a), (c) and (d) and 0.7 for (b). The foil thickness is about $5.0\xi_{110}$ [From Humble (1970), courtesy of North-Holland Publishing Co.]

been obtained using techniques similar to those outlined in section 2.8.1 to remove the 180° ambiguity.

Figure 3.11 contains computed images for all the possible Burgers vectors in β -brass for the experimental conditions of figure 3.10(a). Figure 3.11(a), (c), (e), (g) and (i) are possible matches to the experimental image. Some are obviously better matches than others, but it may be agreed that all the images rejected are gross mismatches. Figure 3.12 shows the micrographs computed for the b of the dislocations depicted in figure 3.11(a), (c), (e), (g) and (i) but with diffracting conditions appropriate to figure 3.10(b). The image in figure 3.10(b) may be characterised as being double and dotted, the dots being much larger on one side of the line than the other. It also has a region at background intensity along the centre. Only the theoretical micrographs in figures 3.12(a) and (c) match this description; the others are mismatches. Distinction between the possible b represented by these two micrographs may be made by comparing figure 3.10(c) with figure 3.13(a) and (b). The image in figure 3.13(a) is not sufficiently one-sided and it is therefore concluded that figure 3.13(b) is the match in this case and that $b = [\bar{1}11]$. Further confirmation may be obtained by computing the theoretical micrograph for a dislocation with $b = [\bar{1}11]$ for the diffracting conditions of figure 3.10(d). This is shown in figure 3.14 which is in good agreement with the experimental image.

In summary, the theoretical micrographs that match the experimental images in figures 3.10(a)–(d)

are figures 3.11(e), 3.12(c), 3.13(b) and 3.14 respectively. The Burgers vector of the dislocation is $[\bar{1}11]$ and since the dislocation line is also along $[\bar{1}11]$ the dislocation in question is identified as being a pure screw superlattice dislocation. Yet it shows very strong contrast when $g \cdot b$ and $g \cdot b \wedge u = 0$, entirely owing to the anisotropy of elastic constants.

The above example involves a particularly high anisotropy factor when considerable image matching is involved. However, in most materials, for example those in which $A \approx 0.5$ – 3.0 , only a limited amount of image matching is necessary because the ' $g \cdot b = 0$ effective invisibility' criterion may be used to estimate the direction of b using a minimum of three cases of 'effective invisibility'. Such a case has been reported by Lloyd and Loretto (1970) and Loretto and Wasilewski (1971).

It is important to realise that image matching enables not only the direction but also the magnitude together with the sign of b to be obtained (Head *et al.*, 1973). Furthermore, detailed studies of small dissociation of dislocations are possible (Clarebrough and Morton, 1969a, b) and this technique is therefore a very powerful tool in elucidating the detailed dislocation configuration.

3.4.4 Images of Partial Dislocations ($b_p < \text{Lattice Translation Vector}$)

Partial dislocations occur in a number of crystal structures. However, they have received most attention in f.c.c. materials for which $g \cdot b_p$ can be $0 \pm \frac{1}{3}, \pm \frac{2}{3}, \pm 1$, for both Shockley ($b_p = \frac{1}{6}\langle 112 \rangle$) and Frank ($b_p = \frac{1}{3}\langle 111 \rangle$) partials depending on the reflecting plane.

The geometrical situation to be considered in f.c.c. materials is shown in figure 3.29(a) with the stacking fault lying on an inclined plane and to the left of the partial dislocation Burgers vector b_p and positive line direction u . It is shown later in section 3.6.1.1 that the stacking fault produces a strong fringe image, see figure 3.30(a), (b) when $g \cdot R_F = \pm \frac{1}{3}$ or $\pm \frac{2}{3}$, where R_F is the fault vector. However, the fault is invisible for reflections with $g \cdot R_F = 0, 1$, etc., see figure 3.16(c)–(e). In practice it is often difficult to decide whether a partial dislocation is visible or invisible in the presence of these fringes. Consequently, the most useful situations experimentally are those described above in which the fault itself is invisible.

3.4.4.1 Fault invisible

Under these conditions $g \cdot b_p$ may be 0 or 1^* .

$g \cdot b_p = 0$ (Howie and Whelan, 1961; Silcock and Tunstall, 1964). The situation occurs for both

* Because of the geometrical relationship between the fault vector R_F and the partial b_p , integral values of $g \cdot R_F = 0, 1$, etc., correspond to integral values of $g \cdot b_p$. Furthermore, fractional values of $g \cdot R_F$ correspond to fractional values of $g \cdot b_p$.

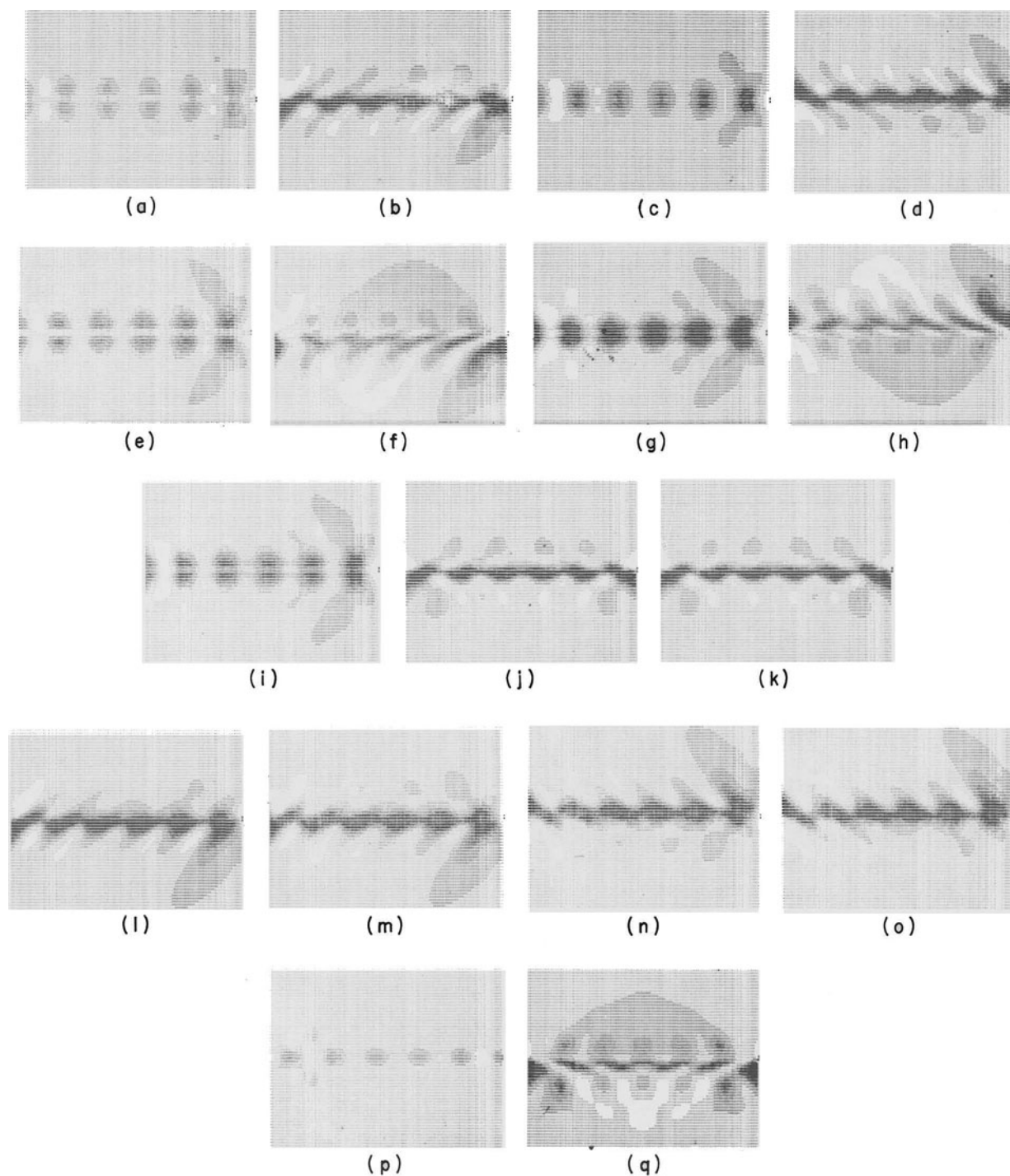


Figure 3.11 Theoretical micrographs computed for the diffraction conditions of figure 3.10(a) for dislocations of all the Burgers vectors probable in β -brass. Reading from (a) to (q) the Burgers vectors are $\frac{1}{2}[\bar{1}11]$, $\frac{1}{2}[111]$, $\frac{1}{2}[\bar{1}\bar{1}1]$, $\frac{1}{2}[\bar{1}\bar{1}\bar{1}]$, $[\bar{1}11]$, $[111]$, $[\bar{1}\bar{1}1]$, $[\bar{1}\bar{1}\bar{1}]$, $[001]$, $[010]$, $[100]$, $[011]$, $[101]$, $[0\bar{1}1]$, $[\bar{1}01]$, $[\bar{1}10]$ and $[110]$. [From Humble (1970), courtesy of North-Holland Publishing Co.]

$\{220\}$ and $\{113\}$ reflections, for which contrast calculations have been performed assuming isotropic elasticity. However, a large number of possible combinations of Shockley–Frank edge–screw nature with B , g , s and elastic anisotropy have not been considered. Since these factors have been shown to be important for perfect b the situation for partial dislocations could be complex.

With this proviso, the following conclusions may be drawn from the work of the above authors.

(1) Screw Shockley partials $g \cdot b_p = 0$ are invisible.

(2) Edge Frank partials $g \cdot b_p = 0$; $m = \frac{1}{8}g \cdot b_p \wedge u = 0.2024$ are visible showing intense double images; $w \lesssim 1.0$.

However, these cannot be regarded as rules to

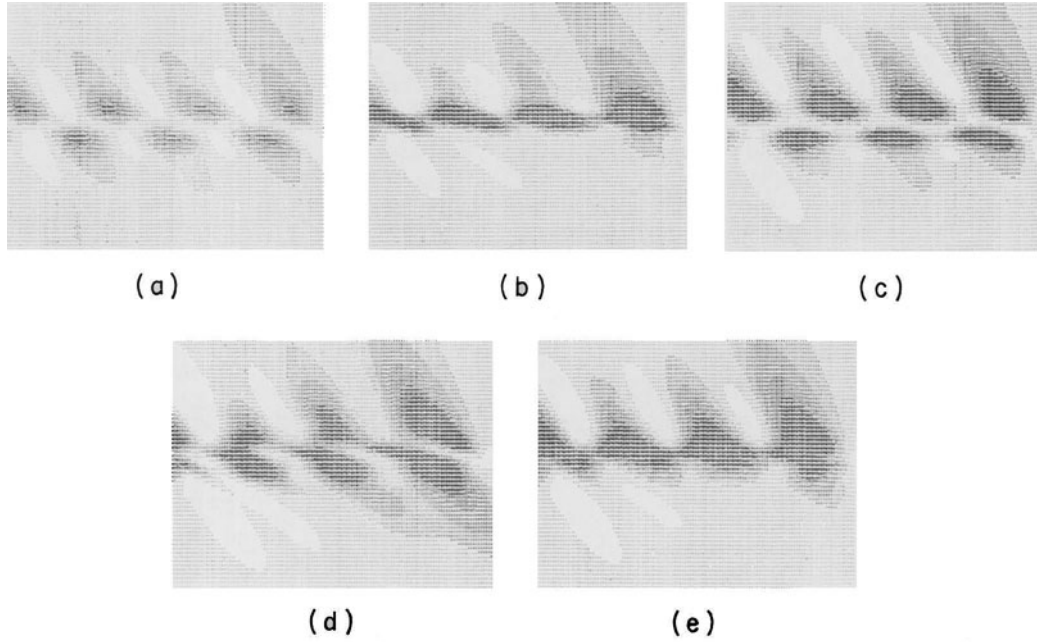


Figure 3.12 Theoretical micrographs computed for the diffraction conditions of figure 3.10(b) for the remaining possible Burgers vectors after comparing figure 3.10 with figure 3.11. Reading from (a) to (e) the Burgers vectors are $\frac{1}{2}[\bar{1}11]$, $\frac{1}{2}[\bar{1}\bar{1}1]$, $[\bar{1}11]$, $[1\bar{1}1]$ and $[001]$ [From Humble (1970), courtesy of North-Holland Publishing Co.]

cover all cases of partial dislocation images, and when inconsistencies are obtained in a series of images with different operating reflections image computation will be necessary.

$\mathbf{g} \cdot \mathbf{b}_p \pm 1$ (Howie and Whelan, 1962). This situation occurs for both $\{220\}$ and $\{113\}$ reflections and partial dislocations are visible for $w \lesssim 1.0$.

3.4.4.2 Fault visible

In this situation $\mathbf{g} \cdot \mathbf{b}_p$ may be $\pm\frac{1}{3} \pm \frac{2}{3}$.

$\mathbf{g} \cdot \mathbf{b} = \pm\frac{1}{3}$ (Howie and Whelan, 1962; Clarebrough and Morton, 1969a, b; Clarebrough, 1971). Both Frank and Shockley partials are invisible for $w = 0-1.0$, where the $\mathbf{g} \cdot \mathbf{b}_p \wedge \mathbf{u}$ term is unimportant. However, there is an exception to this rule. Clarebrough and Morton (1969a, b) showed that strong contrast can arise for Frank dis-

locations with $\mathbf{g} \cdot \mathbf{b} = \pm\frac{1}{3}$ for particular beam directions. It is clearly possible that further exceptions may exist.

$\mathbf{g} \cdot \mathbf{b} = \pm\frac{2}{3}$ (Silcock and Tunstall, 1964; Clarebrough and Morton, 1969a, b; Clarebrough, 1971). This situation occurs for $\{111\}$ and $\{200\}$ reflections and images behave in a complicated way. When the fault lies to the left of the partial as depicted in figure 3.29(a) the image is as follows.

(1) $\mathbf{g} \cdot \mathbf{b}_p = +\frac{2}{3}$ may be visible or invisible at value of w near zero, depending upon foil thickness. It may be visible independent of foil thickness at intermediate values of w (≈ 0.5) and may be weak or invisible at large values of w (≈ 1) depending upon foil thickness.

(2) $\mathbf{g} \cdot \mathbf{b}_p = -\frac{2}{3}$ may be visible or invisible at values of w near zero depending on foil thickness.

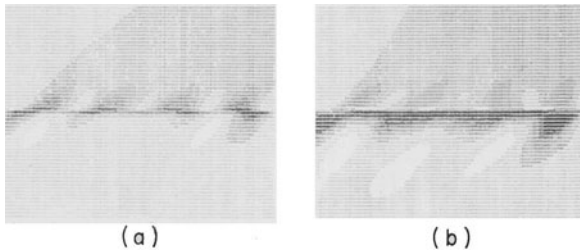


Figure 3.13 Theoretical micrographs computed for the diffraction conditions of figure 3.10(c) for the remaining possible Burgers vectors after comparing figures 3.10 and 3.11 and figures 3.10(b) and 3.12. The Burgers vector for (a) is $\frac{1}{2}[\bar{1}11]$ and for (b) is $[\bar{1}11]$ [From Humble (1970), courtesy of North-Holland Publishing Co.]

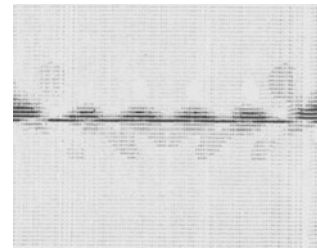


Figure 3.14 A theoretical micrograph computed for the diffraction conditions of figure 3.10 with a Burgers vector of $[\bar{1}11]$ [From Humble (1970), courtesy of North-Holland Publishing Co.]

It may be invisible if $w \gtrsim 0.7$.

The rule given by Silcock and Tunstall (1964) that if $\mathbf{g} \cdot \mathbf{b}_p = \pm \frac{2}{3}$ and $w \lesssim 0.7$ partials are visible and if $w \gtrsim 0.7$, $\mathbf{g} \cdot \mathbf{b} = -\frac{2}{3}$ is invisible but $\mathbf{g} \cdot \mathbf{b}_p = +\frac{2}{3}$ is visible is not correct for all cases.

$\mathbf{g} \cdot \mathbf{b}_p = \pm \frac{4}{3}$ (Oblak and Kear, 1972). This situation occurs for $\{111\}$ and $\{200\}$ reflections but the situation has not received much study. However, Oblak and Kear (1972) quote the rule for image visibility. If $w \gtrsim 0.7$, $\mathbf{g} \cdot \mathbf{b}_p = +\frac{4}{3}$ invisible, $\mathbf{g} \cdot \mathbf{b}_p = -\frac{4}{3}$ visible.

Nevertheless, extensive computations have not been performed for this value of $\mathbf{g} \cdot \mathbf{b}_p$ and the above rule cannot definitely be stated to apply to all $\mathbf{g} \cdot \mathbf{b}_p \pm \frac{4}{3}$ cases.

Image contrast for $\mathbf{g} \cdot \mathbf{b}_p = \pm \frac{2}{3} \pm \frac{4}{3}$ is reversed if $\mathbf{u}$ is reversed and the fault lies on the right of the partial.

3.4.4.3 A partial dislocation between extrinsic and intrinsic faults

The contrast arising from partial dislocations under these circumstances has been calculated by Amelinckx (1964) and by Tunstall and Goodhew (1966). When allowance is made for the displacement vectors of the fault and the Burgers vector of the partial dislocation the phase factor determining the contrast is $\alpha = 3\pi \mathbf{g} \cdot \mathbf{b}_p$ rather than the usual $\alpha = 2\pi \mathbf{g} \cdot \mathbf{b}_p$. Under these circumstances the partial shows up as a dark line for $\{111\}$ reflections and as a bright line for $\{200\}$ reflections. In both cases the fault fringes are visible. This effect is shown in figure 3.15(a) for a double faulted loop B. The triangular-shaped loop is a Frank dislocation; within the triangular loop is an

extrinsic stacking fault, whereas between it and the hexagonal loop is an intrinsic stacking fault. The contrast at the triangular loop is dark under (111), and bright under (200), reflections as predicted.

The above image characteristics have also been observed by Gallagher *et al.* (1966) for a Shockley partial dark under (002) but bright under $(\bar{1}11)$ reflections and separating an extrinsic and an intrinsic stacking fault, see figure 3.16(a), (b).

3.4.5 Determination of the Burgers Vector of Partial Dislocations

In attempting to determine the $\mathbf{b}_p$ for partial dislocations the image characteristics outlined in the previous section may be used. However, these cannot be described as rules because exceptions occur, as pointed out for the case of $\mathbf{g} \cdot \mathbf{b} = \pm \frac{1}{3}$ (Clarebrough and Morton, 1969a, b). Furthermore, the contrast for all combinations of $\mathbf{g} \cdot \mathbf{b}_p$, $\mathbf{u}$, $\mathbf{s}$ and elastic anisotropy has not been investigated theoretically. Nevertheless, the image characteristics outlined in the previous section have been successfully used to obtain partial dislocation Burgers vectors by Silcock and Tunstall (1964), Tunstall and Goodhew (1966), Gallagher (1966), Booker and Tunstall (1966) and Oblak and Kear (1972).

The following approach is suggested.

- (1) Attempt to determine $\mathbf{b}_p$ using reflections for which the fault is invisible and partial contrast is easier to observe. Use $w \lesssim 1.0$ and obtain at least three cases of invisibility, that is $\mathbf{g} \cdot \mathbf{b}_p = 0$.
- (2) Avoid $\mathbf{g} \cdot \mathbf{b}_p = \pm \frac{1}{3}$ or $\pm \frac{2}{3}$ situations because

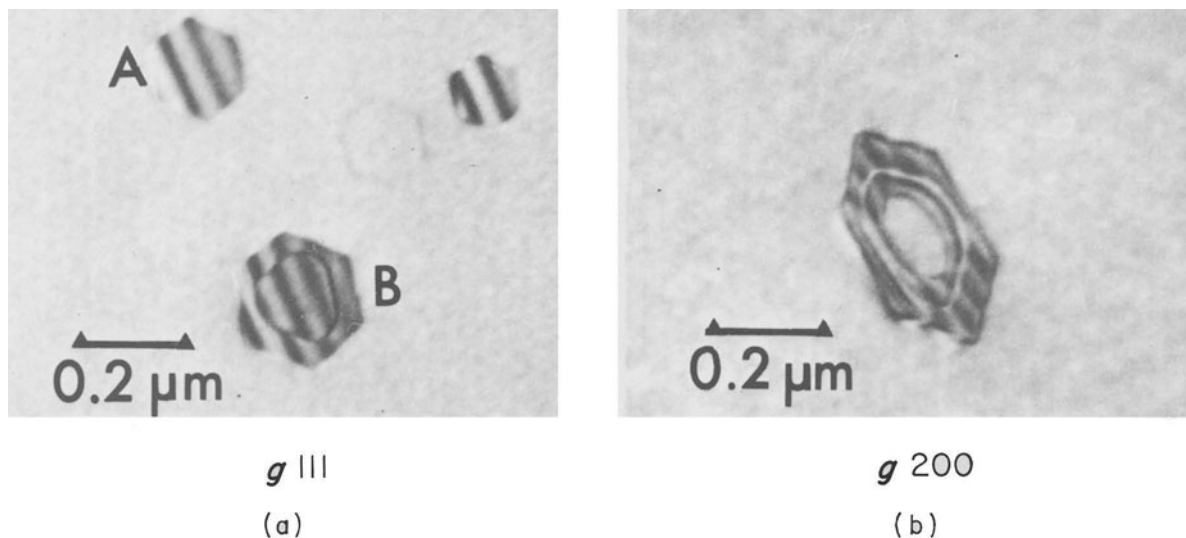


Figure 3.15 (a) A single A and double B faulted Frank loop and (b) a triple faulted loop quenched

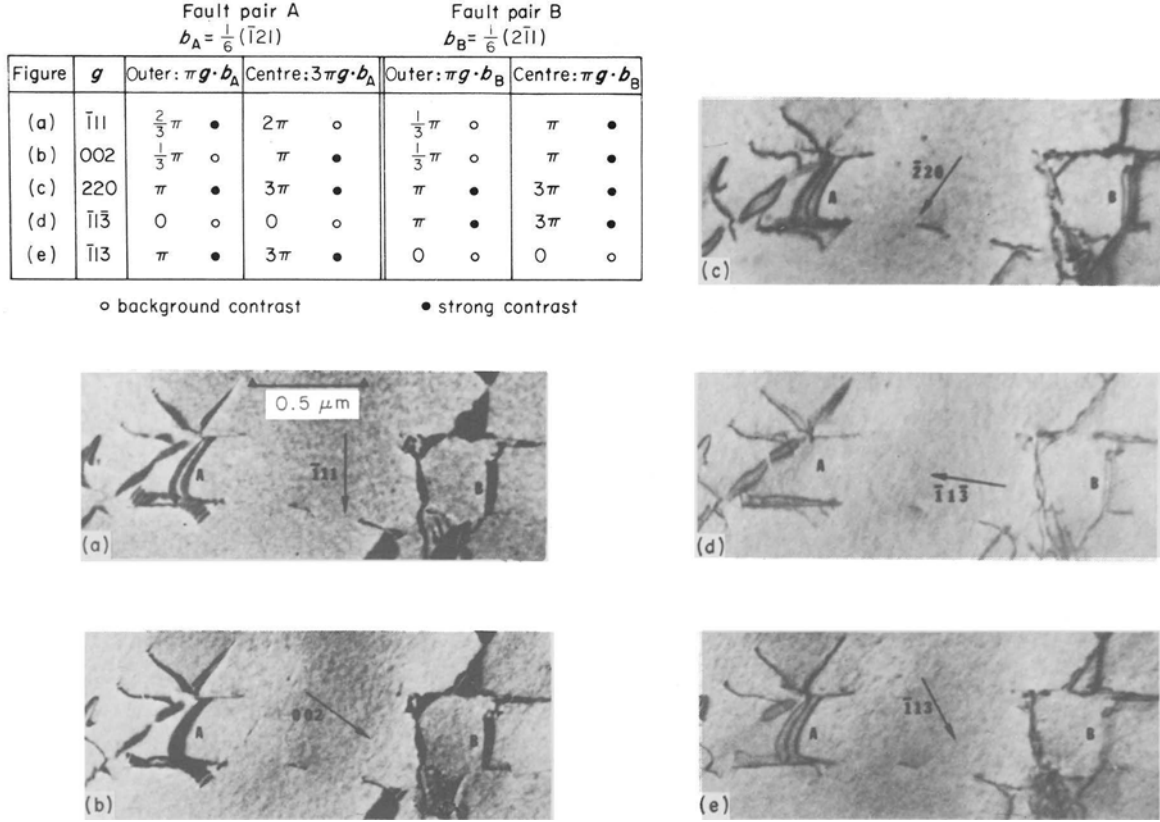


Figure 3.16 Intrinsic-extrinsic fault pairs AB in a deformed Cu-15 wt % Sn alloy, observed under five different reflections
[Courtesy of *Physica Status Solidi*]

of the difficulty of observing contrast from partials in the presence of the fringe stacking fault image.

(3) If $g \cdot b_p = \pm \frac{1}{3}$ or $\pm \frac{2}{3}$ situations cannot be avoided use computer image matching techniques described in detail by Head *et al.* (1973).

An example of the determination of the Burgers vectors of partial dislocations in an Ag-12.5 wt % In alloy from Gallagher (1966) is shown in figure 3.16 using point (1) above. A further example for Ni-base alloys and for Si is shown in section 4.11.4. In figure 3.16 two of the configurations, that is A and B, are of interest. Comparison of the images obtained with different reflections shows that the arrays consist of three dislocations with stacking fault between them and other work by Gallagher (1966) has shown that one fault is extrinsic while the other is intrinsic. Both dislocation arrays lie on the $(\bar{1}\bar{1}1)$ plane and the possible Burgers vectors of the partials are therefore $\pm \frac{1}{6}[\bar{1}21]$, $\pm \frac{1}{6}[2\bar{1}1]$, $\pm \frac{1}{6}[\bar{1}1\bar{2}]$ which lie in $(\bar{1}\bar{1}1)$, and $\pm \frac{1}{3}[\bar{1}\bar{1}1]$ perpendicular to the fault plane. The Frank partials and Shockley partial $b_p = \frac{1}{6}[\bar{1}1\bar{2}]$ can be eliminated on the basis of the invisibility of each array with $\{113\}$ reflections because $g \cdot b_p$ would be an integer for both reflections, see table 3.4. However, both the remaining b_p , that is

$\pm \frac{1}{6}[\bar{1}21]$, $\pm \frac{1}{6}[2\bar{1}1]$ have $g \cdot b_p = 0$ for these reflections. However, on the basis of the image contrast for $(\bar{1}\bar{1}3)$ and $(\bar{1}1\bar{3})$ reflections all three dislocations in array A have $b_p \pm \frac{1}{6}[\bar{1}21]$ whereas, for array B, b_p is $\pm \frac{1}{6}[2\bar{1}1]$ because $g \cdot b_p = 0$ for these cases, see table 3.4.

It was shown in section 3.4.4.3 that the contrast at the centre partial is white for $\alpha = 3\pi g \cdot b_p = 2\pi$, whereas it is dark for $\alpha = 3\pi g \cdot b_p = \pi$, see Tunstall and Goodhew (1966). Table 3.4 shows this is consistent with the b_p for the centre dislocation $\pm \frac{1}{6}[\bar{1}21]$ array A, $\pm \frac{1}{6}[2\bar{1}1]$ array B, that is both centre partials should be dark $\alpha = \pm \pi$

Table 3.4 Values of $g \cdot b_p$ and $\alpha = 3\pi g \cdot b_p$ for the possible Burgers vectors of the partial dislocations in figure 3.16 arrays A, B

Burgers vectors	Values of $g \cdot b_p$ for various operating reflections				
	$(\bar{1}\bar{1}3)$	$(\bar{1}1\bar{3})$	$(\bar{1}\bar{1}1)$	(002)	$(\bar{2}20)$
$\pm \frac{1}{6}[\bar{1}\bar{1}1]$	± 1	± 1	$\pm \frac{1}{3}$	$\pm \frac{2}{3}$	0
$\pm \frac{1}{6}[\bar{1}1\bar{2}]$	± 2	± 2	$\pm \frac{1}{3}$	$\pm \frac{2}{3}$	0
$\pm \frac{1}{6}[2\bar{1}1]$	± 1	0	$\pm \frac{1}{3}$	$\pm \frac{1}{3}$	± 1
$\pm \frac{1}{6}[\bar{1}21]$	0	± 1	$\pm \frac{2}{3}$	$\pm \frac{1}{3}$	± 1
Values of $\alpha = 3\pi g \cdot b_p$					
$\pm \frac{1}{6}[2\bar{1}1]$	± 3	0	$\pm \pi$	$\pm \pi$	$\pm \pi$
$\pm \frac{1}{6}[\bar{1}21]$	0	± 3	$\pm 2\pi$	$\pm \pi$	$\pm \pi$

for all reflections except $(\bar{1}11)$ for which that at array A is white $\alpha = \pm 2\pi$.

This result is consistent with the fault pair reported by Gallagher (1966).

3.4.6 Image Characteristics of Slightly Dissociated Partial Dislocations

The contrast from a Frank partial dislocation dissociated into a Shockley partial and a stair rod dislocation has been investigated by Clarebrough and Morton (1969a, b). These workers showed that the characteristics of the image were the following.

(1) Strong contrast always occurs for $\{111\}$ reflections if the reflecting plane g vector is not normal to the line direction of the dislocation. However, undissociated Frank partials are frequently invisible, see sections 3.4.4.1 and 3.4.4.2.

(2) Images are single and continuous with strong contrast on one side only for $\{220\}$ reflections. In contrast, undissociated Frank partials show strong dotted double images.

(3) For $\{020\}$ reflections a bright line occurs between the edge of the stacking fault and the dislocation line. However, the details of the contrast are sensitive to the operative reflection, the separation of the partials and the value of w .

Clarebrough and Morton (1969a, b) were able to show that these differences between the dissociated and undissociated cases were readily detectable at dissociations of 40 Å and that the results, which included elastic anisotropy, should be applicable to f.c.c. metals and alloys with similar elastic constants.

Dissociation of Frank partials into Shockley partials and stair rod dislocations should be readily detectable using the image characterisation summarised above. By careful use of image-matching techniques described by Head *et al.* (1973) it should be possible to measure the dissociation and cross-check with weak-beam images, section 3.17.

3.4.7 Overall Procedure for Determining the Burgers Vector of Dislocations

The following procedure may be used for perfect and partial dislocations.

(1) Choose a straight dislocation running from top to bottom of the foil in an area of constant thickness 3–8 ξ_g for a low-order reflection, for example $\{111\}$, $\{110\}$. The area should be free from bend contours to prevent variations of s across the area.

(2) Tilt the specimen as described in section 2.10.1, to establish in which type of unit triangle the foil normal FN is located, that is remove the 180° ambiguity, see section 2.8.1.

(3) Determine FN from a diffraction pattern taken at zero tilt.

(4) Determine the thickness of the foil from thickness fringes at $w = 0$, see section 3.2.3.1, using crystallographic analysis of a known defect, stereo pairs or other approaches.

(5) Obtain the unique line direction u from tilting experiments using the approach outlined in section 2.14.2.

(6) Obtain a series of photographs with different reflecting planes and known B (all two-beam conditions, with $w \lesssim 1.0$ to avoid $g \cdot b = 1$ effective invisibility, until two or three cases of near-invisibility are obtained).

(7) Either (a) analyse the data on the basis of $g \cdot b = 0$ criterion and confirm the result by a limited amount of computation allowing for elastic anisotropy or (b) if no cases of extinction are recognisable solve by extensive computer matching using the most likely b for the crystal structure involved. The technique of computer matching has been covered in the recent book by Head *et al.* (1973).

(8) If small dissociations are suspected the technique of weak beam imaging (section 3.17) should be used to check whether it is detectable and the computer programs of Head *et al.* (1973) may be used to obtain an accurate description of the dissociation.

Diffraction patterns should be taken from each area for each photograph without touching the tilt controls between recording image and diffraction pattern. Use the smallest selected area aperture and double expose the image to record the region from which the diffraction pattern is taken. Plate magnifications in the regions 20–30 000 are usually satisfactory for work of this type.

3.4.8 Image Characteristics of the Special Case of End-on Dislocations

The case in which dislocations lie normal to the thin crystal surface and parallel to the electron beam has been examined by Tunstall *et al.* (1964). Surface relaxation of the strain field was included in image calculations using isotropic elasticity theory and the following was shown.

(1) The contrast from edge dislocations viewed exactly end-on is weak and is difficult to observe. Deviation from the exact end-on position can produce complex images that cannot be analysed simply. Figure 3.17(a) shows an experimental

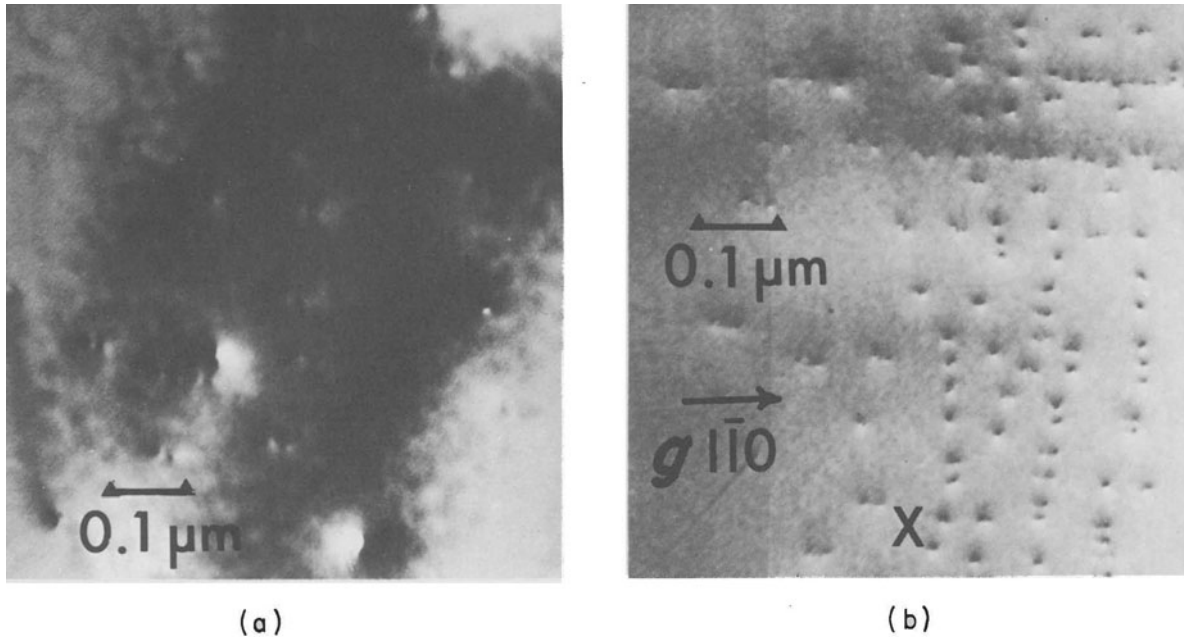


Figure 3.17 End-on edge dislocation dipoles and (b) end-on screw dislocations in aluminium

image of edge dislocation dipoles of the type shown in figure 3.18(f) viewed along u .

(2) Strong contrast occurs for screw dislocations viewed exactly end-on and images consist of a bright and a dark circular lobe oppositely disposed about the operative g vector, see figure 3.17(b). Since $g \cdot b = 0$ all of the contrast arises from surface relaxation effects. Small deviations from the exact end-on position does not produce a significant change in the form of the image and the two lobes may be seen, for example figure 3.17(b) at X.

3.4.9 Determination of the Sense of the Burgers Vector of End-on Dislocations

It is possible to determine the sense of the Burgers vector of a screw dislocation, that is b up or down using a simple rule devised by Tunstall *et al.* (1964). These workers showed that, on a positive print, if the dark lobe lies to the left of the g vector when viewed down the g vector the dislocation is a right-handed screw when looking down on the specimen, that is from the electron gun. If the dark lobe lies to the right the dislocation is a left-handed screw. Using the FS/RH criterion summarised by Hirth and Lothe (1968) b will be down in the first case and up for the second.

3.4.10 Image Characteristics of Dislocation Pairs

Dislocation pairs occur in a number of short-range ordered alloys and precipitation hardened materials. They consist of closely spaced mobile

dislocations with the same b and separated by 20–1000 Å, see figure 3.18(a). The following image characteristics (Bell *et al.*, 1964) have been reported for spacings ≥ 200 Å.

(1) The image consists of two single dislocation images.

(2) The separation does not change on reversing g or s , see dislocation pairs A, B in figure 3.18(b), (c) for $\pm g$ case.

(3) The same invisibility criteria should apply as for single dislocations independent of the separation because the coalescence of the two dislocations will simply change the magnitude not the direction of b .

These defects may be identified using the image characteristics outlined above using primarily the behaviour under $\pm g$. The Burgers vectors may be determined using the conventional approach described in section 3.4.3 for perfect dislocations. The separation of dislocation pairs may be measured most accurately using weak-beam images, see section 3.17.

A closely spaced inclined dislocation pair is shown in figure 3.18(d), (e). In this case the image spacing does not change on reversing g .

3.4.11 Image Characteristics of Dislocation Pairs

All dipoles consist of a pair of dislocations with opposite b held in a position similar to that in figure 3.18(f) by their own elastic interaction. The

electron microscope image has the following characteristics (Bell *et al.*, 1964; Forwood and Humble, 1970).

(1) For image spacing $\geq 300 \text{ \AA}$ the image is similar to that of two closely spaced dislocations, see figure 3.18(g).

(2) The spacing of the images changes on reversing g or s . Compare figures 3.18(g), (h).

(3) For steeply inclined closely spaced dipoles, that is running from the top to the bottom of the foil, the image has a characteristic centre of inversion independent of the spacing of the dislocations, see figure 3.18(i) and compare with the

similar case for a closely spaced dislocation pair, figure 3.18(d), (e).

Invisibility of dislocation dipoles is governed by the same conditions as for single dislocations in elastically anisotropic media. Complete invisibility will only be obtained for edge dislocation dipoles (not mixed) under the following conditions.

(1) The dipole lies normal to an elastic symmetry plane in the crystal.

(2) The reflecting vector g lies parallel to the dislocation line vector u .

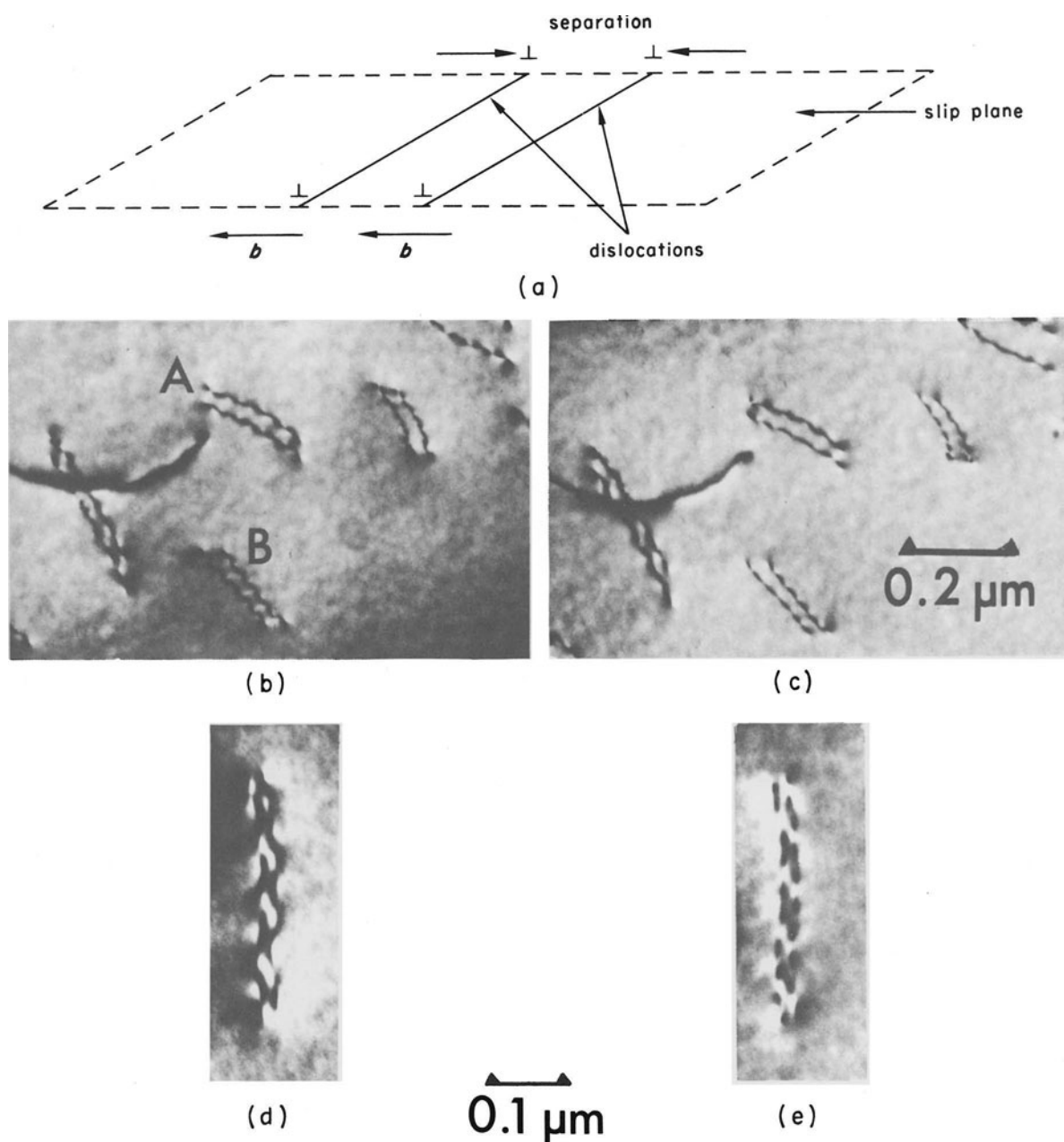
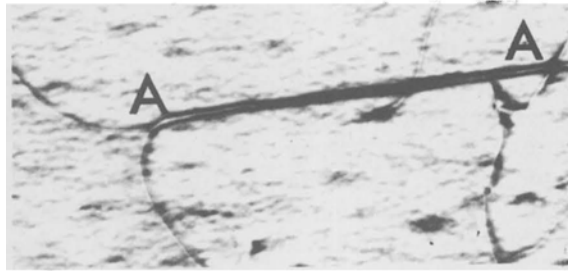
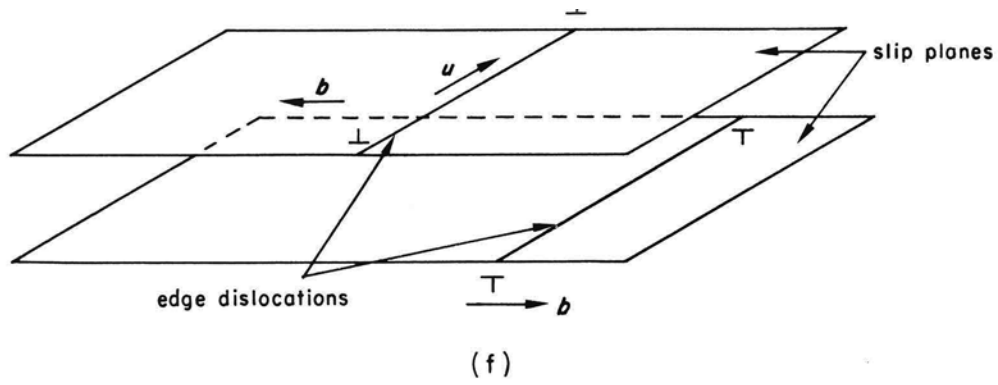
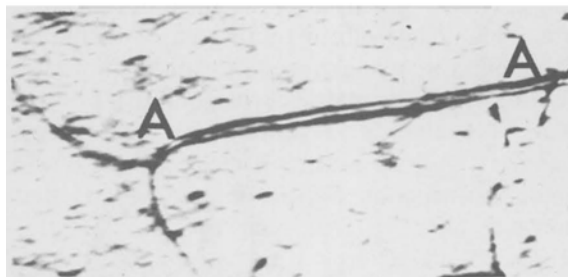


Figure 3.18 (a) A schematic diagram illustrating a dislocation pair (b), (c) $\pm g$ BF images of well-separated inclined dislocation pairs A, B, (d), (e) $\pm g$ BF images of a dislocation dipole AA, (f) A schematic diagram illustrating a dislocation dipole, (g), (h) $\pm g$ BF images of a dislocation dipole AA, (i) a BF image of an inclined dipole, (j) Frank dipoles XX in deformed copper [(g)–(i) Courtesy of *The Philosophical Magazine*]



(g)

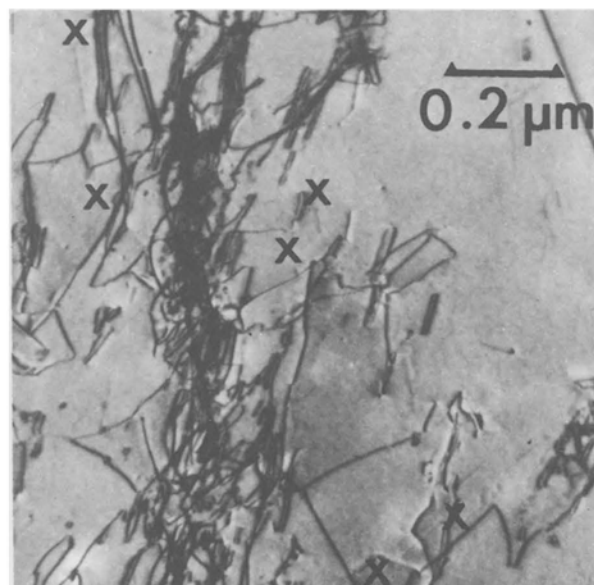
 $+g$ 

(h)

-g

0.5 μm

(i)



(i)

Figure 3.18 (continued)

However, there may be some cases of very weak contrast producing 'effective invisibility' but it is not possible to predict when these will occur.

Dipoles may be identified either from the change in image spacing on reversing g or, if the components are too close ($\lesssim 300 \text{ \AA}$), using the centre of inversion symmetry for those dipoles inclined to the foil surface. Note that the images of lines of dilatation also have centres of inversion (Humble, 1969), which could confuse this analysis if these defects were present.

The approach used to determine the Burgers vectors of dipoles can be the same as for single dislocations, provided their separation is large enough $\gtrsim 300 \text{ \AA}$ for the strain fields to be almost independent. For smaller separations when the strain fields overlap, computation techniques, as outlined by Head *et al.* (1973) may be necessary.

3.4.12 Faulted Dipoles

Long faulted dipoles in deformed f.c.c. metals have been studied by Steeds (1966, 1967) and by von Häussermann and Wilkens (1966). The exact imaging behaviour for identification purposes has not been described accurately using image matching techniques. However, faulted dipoles may be recognised in sections parallel to the slip plane $\{111\}$ as straight narrow images along $\langle 110 \rangle$ with faint contrast under $\{111\}$ and $\{220\}$ reflections, see XX, figure 3.18(j). Frequently the lines zig-zag along different $\langle 110 \rangle$. The images disappear for reflections with g parallel to the long axis of the dipole. The exact details of the image are determined by the type of dissociation of the dipole. Steeds (1966, 1967) has performed detailed contrast calculations allowing for elastic anisotropy that enable the stacking fault energy to be determined. On this basis ($\bar{1}\bar{1}1$) reflections should be used to obtain the best measure of stacking fault energy for dipoles lying along $[101]$ if $B = [\bar{1}11]$. Then the fault energy may be determined using the separation of the long components of the dipole.

3.5 Dislocation Loops

In studying dislocation loops we are primarily interested in determining whether they are vacancy or interstitial and the direction of the Burgers vector. For convenience, three classes of loops will be considered, separated on the basis of their different image behaviour into (a) large loops, diameter $\gtrsim 500 \text{ \AA}$, (b) medium loops, diameter $\sim 100\text{--}400 \text{ \AA}$, and (c) small loops, diameter $< 100 \text{ \AA}$. It should be noted that if the 'formal' method (section 3.5.2) is followed the sense of large loops is not difficult to determine. However, for small

loops considerable difficulty may be encountered and experimentation must be very precise.

3.5.1 Large Loops (Diameter $\gtrsim 500 \text{ \AA}$)

Images of such loops have the same general image characteristics described for straight perfect dislocations in section 3.4.1. Loops are invisible when $g \cdot b = 0$ and $g \cdot b_e$ and $g \cdot b \wedge u$ are unimportant, but residual images occur when these latter factors are non-zero. Again $g \cdot b = 1$ images can be mistaken for $g \cdot b = 0$ invisibility in the presence of $g \cdot b = 2$ images, because w may be selected to be large to form sharp $g \cdot b = 2$ images, as described for dislocations in section 3.4.3.1.

Since dislocation loops generally lie on inclined planes they frequently appear to be elliptical, see figure 3.19(a). Furthermore, because perfect edge loops may be either interstitial (extra plane) or vacancy (one plane missing) while there are two possible senses of inclination of the loop, an edge dislocation loop may have four possible configurations, see figure 3.19(b). It is often of interest to determine the vacancy/interstitial nature of a loop as described in the following section 3.5.2. The method depends upon whether or not the image of the loop is inside or outside the dislocation core. This is determined by the sense of rotation of the reflecting planes around the dislocation. Let the situation in the matrix correspond to a positive value of s , that is maximum transmission (see section 3.2.3.2 and figure 3.3(b)) so that the Ewald sphere construction for the matrix will be that shown in figure 3.19(d), refer to figure 2.16 for further details. Consider the strain field of an edge dislocation, figure 3.19(e). The reflecting plane XX is rotated clockwise by the strain field, that is towards $s = 0$, on the right-hand side of figure 3.19(e), whereas plane YY is rotated anticlockwise, that is away from $s = 0$. Applying this argument to figure 3.19(b), positions A correspond to rotation towards $s = 0$ but positions B to rotations away from $s = 0$. Images therefore occur approximately at A relative to the core. Thus images are formed as shown in figure 3.19(c) where the dashed line indicates the actual dislocation core. For loops 2 and 4 the image is outside the core (outside contrast) but for 1 and 3 it is inside (inside contrast). In practice this means that the sign of $g \cdot b$ is the same for loops 1 and 3 opposite from that of 2 and 4.

The sign of $g \cdot b$ may be determined as follows.

Using the SF/RH criterion* to define b and defining the positive direction around the loops as clockwise looking down from the electron gun the Burgers vectors and signs of $g \cdot b$ are those shown in figure 3.19(f). This definition requires

* This criterion is summarised by Hirth and Lothe (1968).

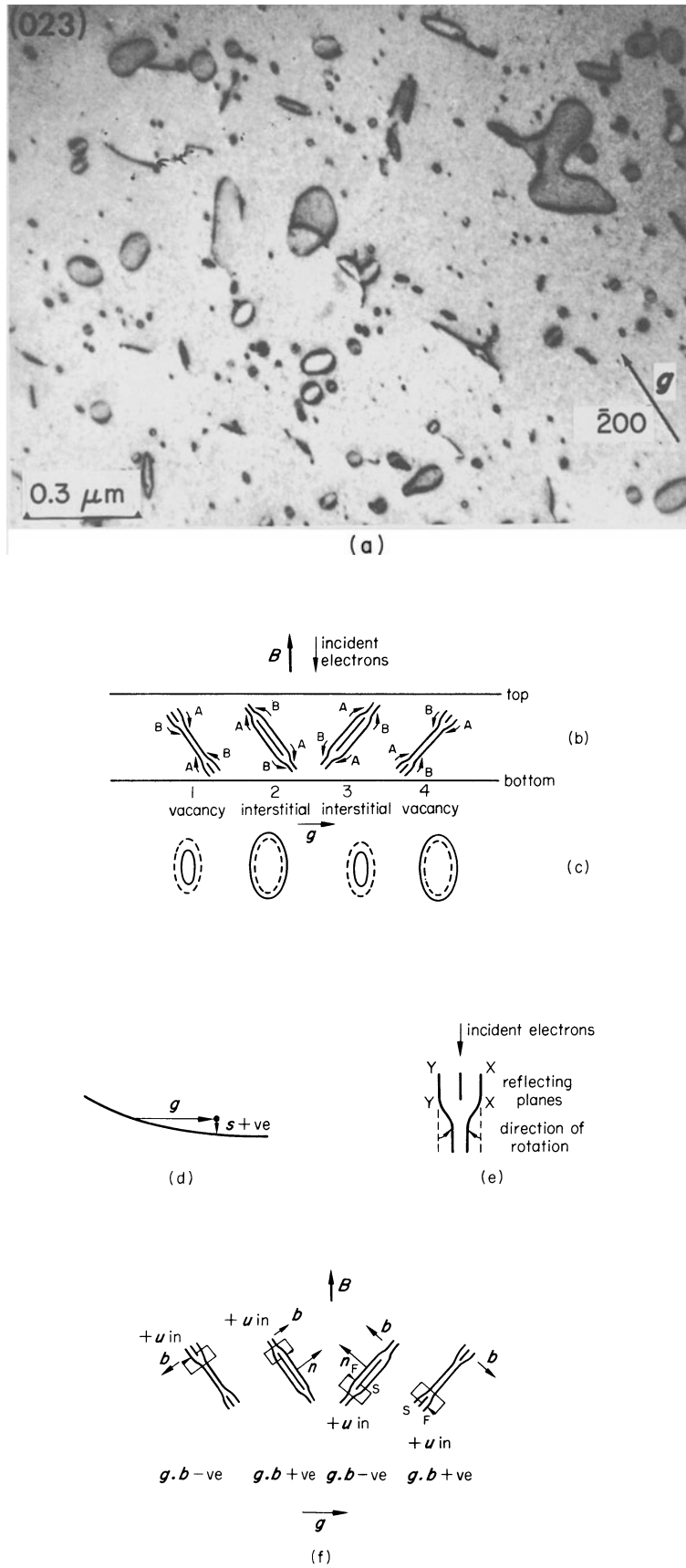


Figure 3.19 (a) Prismatic dislocation loops typical of neutron irradiated and annealed molybdenum. (b) The four possible configurations of inclined vacancy or interstitial loops. (c) The position of the image (heavy line) relative to the core (dashed line). (d) s positive for the operative reflection. (e) The rotation of the reflecting planes at an edge dislocation. (f) The direction of b for each loop, the upward drawn plane normal n , and the sign of $g \cdot b$ are shown in each case [(a) Courtesy of B. L. Eyre]

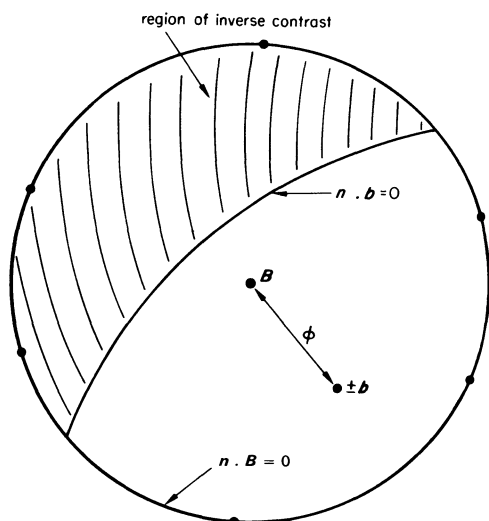


Figure 3.20 An arbitrary stereographic projection that defines two families of loop orientations, in terms of $\mathbf{n}$, figure 3.19(f), and with which the image shift of a non-edge loop is the same as the edge configuration in figure 3.19 [Courtesy of B. L. Eyre]

positive prints to be made from negatives, emulsion side up, so that the prints correspond exactly to the image on the screen without any inversions arising from printing. This is contrary to conventional photographic procedure.

By defining $\mathbf{b}$ in this way the following conclusions may be drawn.

- (1) $\mathbf{g} \cdot \mathbf{b} > 0$ (positive)* for s positive images; the image lies outside the real position of the loop.
- (2) $\mathbf{n} \cdot \mathbf{b} < 0$ vacancy loops and > 0 interstitial loops, where $\mathbf{n}$ is the upward drawn normal to the habit plane.

Thus by knowing the sign of $\mathbf{g} \cdot \mathbf{b}$ and the sense of inclination of the loop its vacancy/interstitial character may be determined.

3.5.2 Quantitative Information

The direction of the Burgers vector of a large loop may be determined using the same approach as outlined for dislocation lines in section 3.4.3 using either the $\mathbf{g} \cdot \mathbf{b} = 0$ criterion or image computation techniques described by Bullough *et al.* (1971).

By determining the size of the loop for $\pm \mathbf{g}$ images and using points (1), (2) above, section 3.5.1, it is possible to determine the interstitial or vacancy character of a loop provided the sign of $\mathbf{g} \cdot \mathbf{b}$ is known, together with loop inclination, for images taken (as normal) with s positive. A number of

approaches have been proposed including the so-called formal method of Mazey *et al.* (1962), Maher and Eyre (1971) and several geometrical methods (Groves and Kelly, 1961, 1962; Bell and Thomas, 1966; Edmonson and Williamson, 1964; Kelly and Blake, 1973a). For perfect edge loops these methods all work but Maher and Eyre have pointed out that loops with a shear component only behave like perfect edge loops provided $\mathbf{b}$ lies within the boundary conditions $\mathbf{n} \cdot \mathbf{b} = 0$ and $\mathbf{n} \cdot \mathbf{B} = 0$. Outside this geometrical situation the image positions reverse from those depicted in figure 3.19(c). In essence the above criterion states that a loop shows contrast equivalent to a perfect edge loop until the shear component reaches a certain value. Consequently, it is only safe to perform the vacancy/interstitial analysis of Mazey *et al.* within this 'safe region' outlined by $\mathbf{n} \cdot \mathbf{b} = 0$, $\mathbf{n} \cdot \mathbf{B} = 0$. This is shown in figure 3.20 for $\mathbf{b} = \frac{1}{2}[111]$, that is b.c.c. crystal structure.

The experimental procedure to obtain the vacancy/interstitial nature of a loop is as follows.

- (1) Obtain an area containing loops on inclined planes and with loop diameters $\gtrsim 500 \text{ \AA}$ at a magnification of 20–30 000.
- (2) Tilt the specimen to determine which unit triangle of the stereogram FN lies in using a Kikuchi map, see section 2.8.1, to remove the 180° ambiguity.
- (3) Tilt the specimen in a known direction recording the sense of tilt to a simple $\mathbf{B}$, for example $[110]$, $[100]$, $[111]$, and obtain a number of photographs with different operative close-in reflections and small values of w (< 1.0) to determine $\mathbf{b}$ for the loops of interest, using $\mathbf{g} \cdot \mathbf{b} = 0$ or image matching.
- (4) If necessary, to determine $\mathbf{b}$, tilt to more than one simple $\mathbf{B}$ to obtain as many operative close-in reflections as possible, see section 2.10.1.
- (5) Obtain some cases of $\pm \mathbf{g}$ with several (at least two) of these reflections close to a simple $\mathbf{B}$.
- (6) Tilt the specimen through $\sim 20^\circ$ so that $\mathbf{B}$ remains within the stereographic triangle outlined previously using the same operative reflection and obtain at least two photographs for $\pm \mathbf{g}$ for new $\mathbf{B}$.
- (7) Analyse the Burgers vector of as many loops as possible that exhibit obvious size changes for $+\mathbf{g}$ (use the data obtained under (3) and (4) using $\mathbf{g} \cdot \mathbf{b} = 0$ or image matching).
- (8) Determine the sense of inclination of the loops to be analysed either from point (6) or from a separate tilting experiment involving a $20\text{--}30^\circ$

* The actual sign of $\mathbf{g} \cdot \mathbf{b}$ depends on both the direction of viewing and the method used to define $\mathbf{b}$, for example if the Burgers vector is defined looking up from the microscope screen at the bottom of the specimen all the signs of $\mathbf{g} \cdot \mathbf{b}$ in figure 3.19(f) would be reversed. However, the final result of any loop sense determination will not be affected provided that the indexing of diffraction patterns is self-consistent.

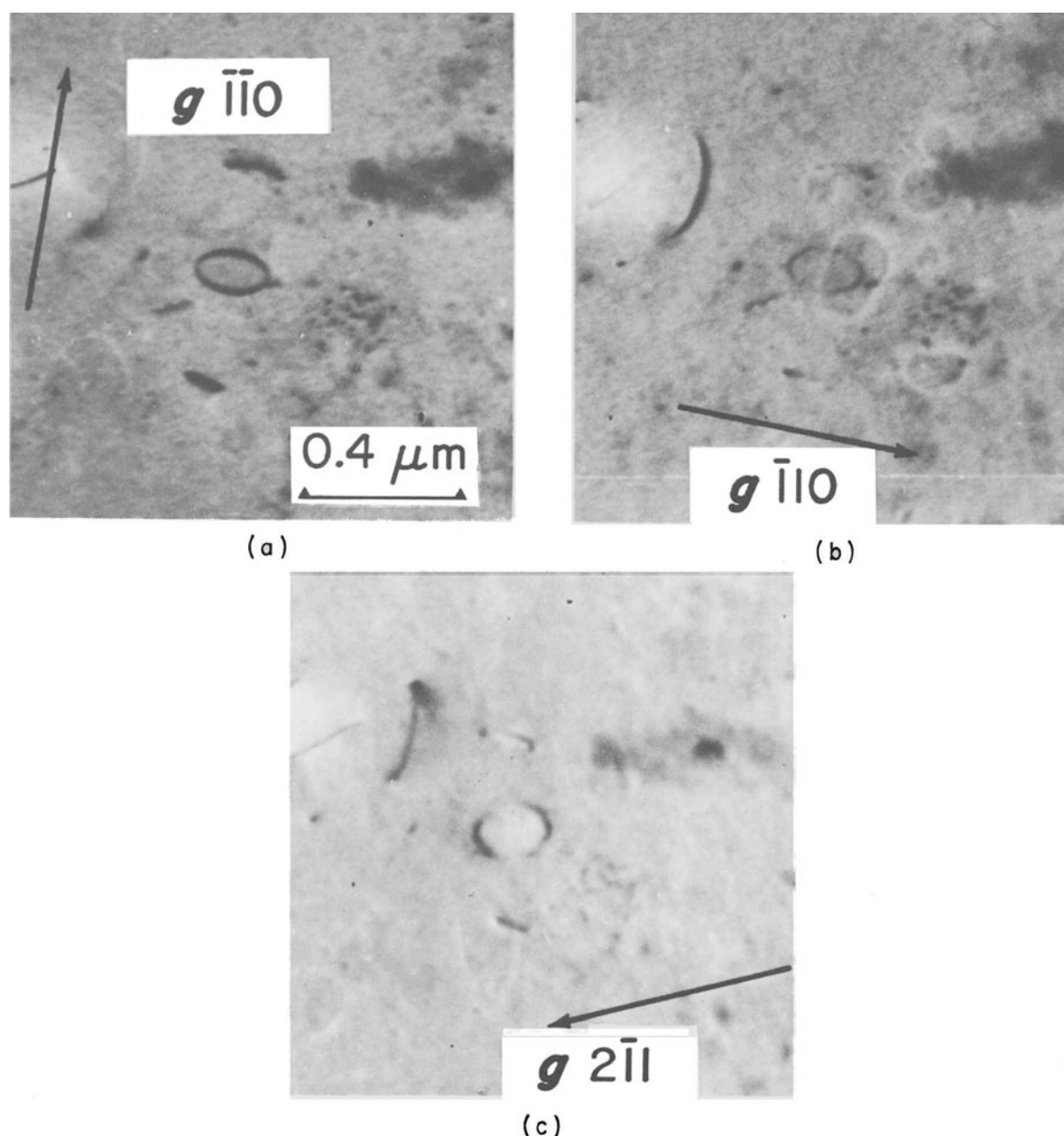


Figure 3.21 A large loop in irradiated Mo observed under three different reflections: (a) $B [001]$, $g \cdot b = 1$; (b) $B [001]$, $g \cdot b = 0$; (c) $B [\bar{1}02]$, $g \cdot b = 0$, that is $b = \frac{1}{2}[\bar{1}\bar{1}1]$ [Courtesy of B. L. Eyre]

tilt in a known sense relative to the image about a suitable operative g , for example $[010]$, $[110]$, etc.

(9) Determine the sense of specific loops using the sign of $g \cdot b$ for all $\pm g$ situations and the sense of rotation to obtain complete consistency for all observations. Ignore all loops for which consistent $\pm g$ reversal of contrast is not obtained for every $\pm g$.

A vacancy/interstitial determination using the above approach in irradiated molybdenum is shown in figures 3.21–3.25. Two beam directions B are shown together in figure 3.21 with two $g \cdot b = 0$ cases exhibiting residual contrast. Thus b is $+\frac{1}{2}[\bar{1}\bar{1}1]$. Figure 3.22(a), (e) shows the determination of the sense of b from $\pm g$ determinations

$B [001]$. Measurement shows outside contrast (a), (c) and (e), that is $g \cdot b$ is positive and $b = \frac{1}{2}[\bar{1}\bar{1}1]$. Figure 3.23 shows a similar determination $B [\bar{1}02]$ for the same loop showing outside contrast (a) and (c), that is $g \cdot b$ is positive and $b = \frac{1}{2}[\bar{1}\bar{1}1]$. Figure 3.24 shows stereographic projections summarising the results obtained from figures 3.21–3.23 for $[001]$ and $[\bar{1}02]$ stereographic projections. Shaded areas indicate the region of 'anomalous contrast' for loops with $b = \frac{1}{2}[\bar{1}\bar{1}1]$. Figure 3.25 shows a series of photographs obtained with the same operative $g = [020]$ for various angles of tilt about $g[020]$. In tilting from $B[\bar{1}02]$ to $B[107]$ the minor axis of the elliptical loop becomes smaller, that is it behaves as if its habit plane were $(\bar{1}\bar{1}1)$ as opposed to $(11\bar{1})$. Thus the

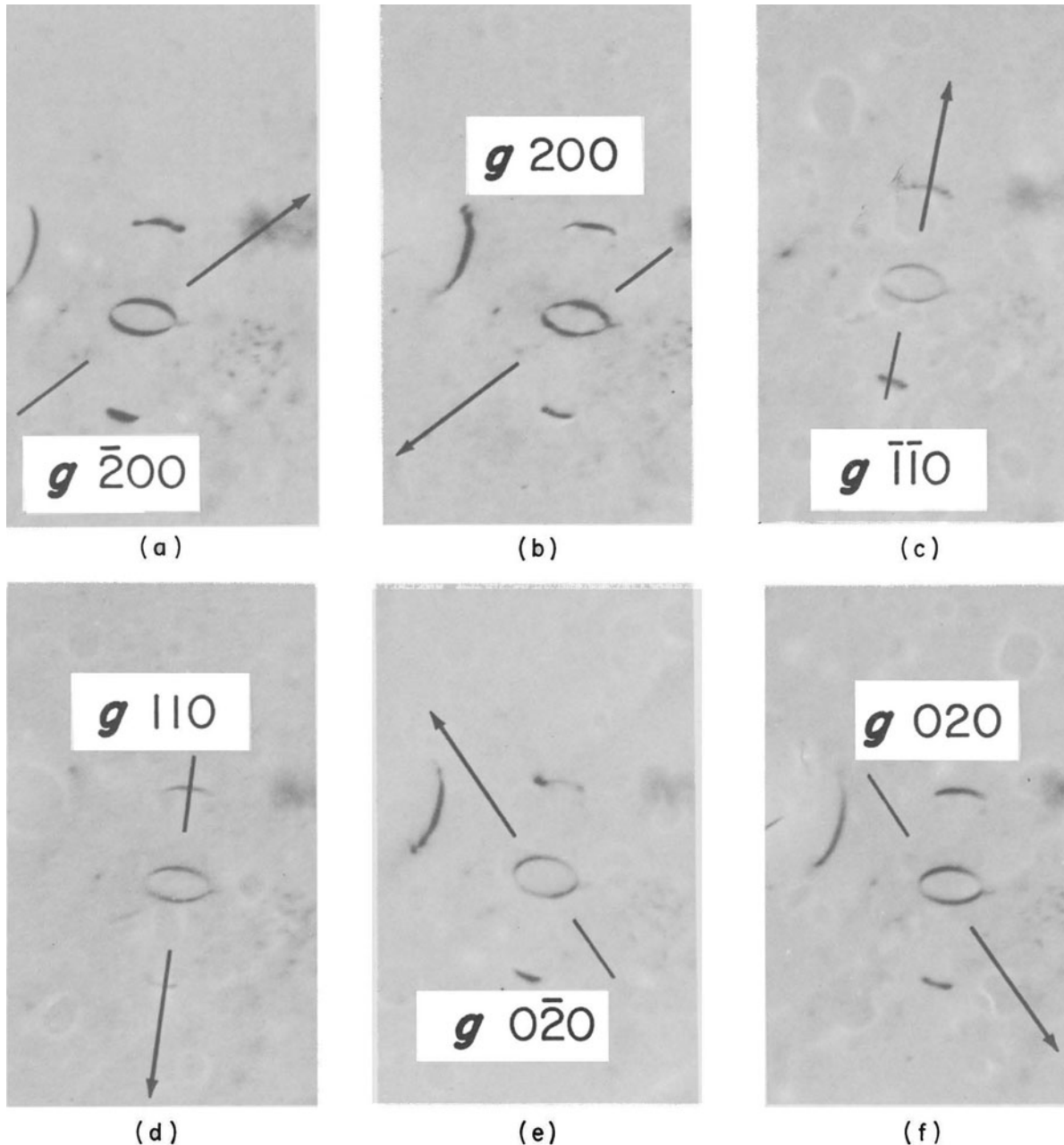


Figure 3.22 Assessment of the sense of b from $\pm g$ images for the loop in figure 3.21. In all cases $B = [001]$. The loop has outside contrast in (a), (c), (e) [Courtesy of B. L. Eyre]

loop is interstitial $b = \frac{1}{2}[\bar{1}\bar{1}1]n[\bar{1}\bar{1}1]$ and corresponds to loop 2 of figure 3.19(b).

Tunstall (1969) has shown that the above 'formal' approach using inside/outside contrast behaviour may also be used to determine the sign of large Frank loops. Using reflections for which $g \cdot b_p = \pm \frac{4}{3}$ he showed that with positive value of s a loop $g \cdot b_p = -\frac{4}{3}$ is larger than one with $g \cdot b_p = +\frac{4}{3}$ particularly when $w \approx 1.5$. However, if reflections are chosen such that $g \cdot b_p = \pm \frac{1}{3}$, this approach cannot be used. Tunstall also pointed out that, if $g \cdot b_p = \pm \frac{2}{3}$, the nature of the large Frank loop may be determined if the sign of g is known.

3.5.3 Image Characteristics of Medium-sized Loops (Diameter 100–500 Å)

Dislocation loops in this size range may be visible either as two arcs or as a single dot, depending upon the size of the loop and whether it is exhibiting 'inside' or 'outside' contrast defined in section 3.5.1. Most of these loops change size on reversing g and this behaviour may be used to obtain the vacancy/interstitial nature of the loops.

3.5.4 Quantitative Information

The Burgers vector of perfect loops may be determined using the general approach outlined

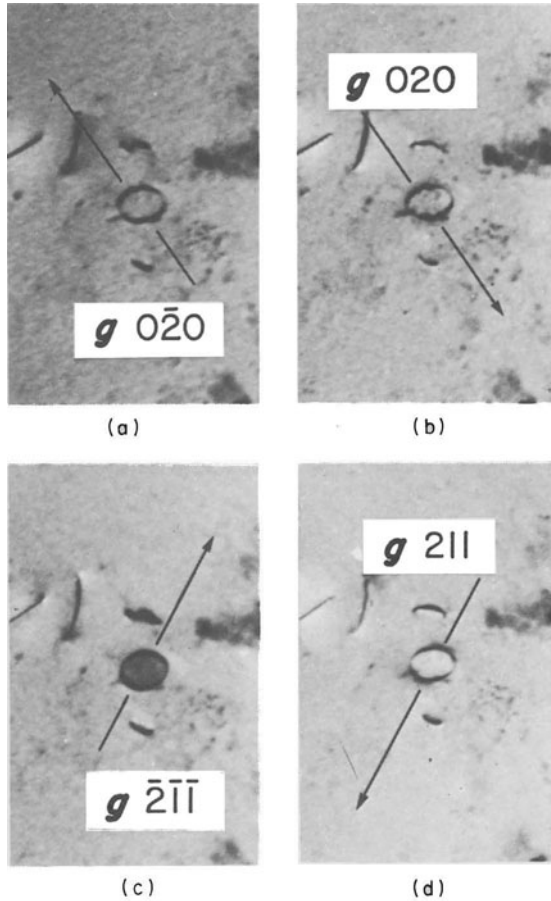


Figure 3.23 Assessment of the sense of $\mathbf{b}$ from $\pm \mathbf{g}$ images with $\mathbf{B} [\bar{1}02]$ for all the same loops as figures 3.21, 3.22. The loop has outside contrast for (a), (c) [Courtesy of B. L. Eyre]

for perfect dislocations, section 3.4.2. There are difficulties arising from elastic anisotropy. Nevertheless, residual contrast for $\{110\}$, $\{200\}$, $\{112\}$ reflections may be used to obtain a consistent $\mathbf{b}$ for Cu ($A = 3.2$) and Mo ($A = 0.8$) provided that more than one $\mathbf{g} \cdot \mathbf{b} = 0$ situation is obtained together with at least one $\mathbf{g} \cdot \mathbf{b} \neq 0$ situation. Furthermore, $\mathbf{g} \cdot \mathbf{b} = 0$ and $\mathbf{g} \cdot \mathbf{b} = 1$ invisibility situations may be confused in the presence of loops for which $\mathbf{g} \cdot \mathbf{b} = 2$ as described in section 3.4.3 for straight dislocations. However, $\mathbf{g} \cdot \mathbf{b} = 0$ is invariant for $\pm \mathbf{g}$, whereas $\mathbf{g} \cdot \mathbf{b} = 1$ is not ($w \gtrsim 0.6$) which is a useful property that enables this confusion to be minimised. An example of this latter effect is shown in figure 3.26. Here $\mathbf{g} \cdot \mathbf{b} = 0$ for defect F in (b), (c), (d) and (e) and the image is the same $\pm \mathbf{g} [\bar{1}\bar{2}1]$ and $[21\bar{1}]$ for w equal and > 0.6 . Although $\mathbf{g} \cdot \mathbf{b} = 0$ for loop E in figure 3.26(d) and (e), $\mathbf{g} \cdot \mathbf{b} \neq 0$ for (b) and (c), despite the 'residual' contrast in (b) because the image in (c) (opposite $\mathbf{g}$) is very different. In fact, $\mathbf{g} \cdot \mathbf{b} = 1$ for defect E in figure 3.26(b) and (c). Clearly it is important to provide complete self-consistency over a number of $\pm$ reflections to obtain reliable results.

Maher and Eyre (1971) have shown that the vacancy/interstitial nature of medium-sized loops may be determined using the approach outlined for large loops in section 3.5.2. However, the analysis is more difficult than for large loops because of inconsistencies that arise experimentally. Indeed, for loops $\lesssim 300$ Å in diameter, the number of loops that it is impossible to assign unambiguously vacancy or interstitial characteristics increases rapidly, even with micrographs obtained with a clean flat specimen and under the best diffracting conditions. The procedure to be followed is essentially the same as that outlined for the larger loops in the previous section. However, additional important points must be considered.

- (1) w must be $\gtrsim 0.6$ for the inside/outside contrast effect to be independent of depth in the foil.
- (2) High-order reflections, for example $\{310\}$, $\{222\}$, $\{321\}$, $w > 0.6$ must be used for some of the $\pm \mathbf{g}$ experiments because these give the best cases of inside/outside contrast.
- (3) Considerable difficulty is likely in determining the sense of inclination of the loop, particularly for loop diameters $\lesssim 200$ Å. In this case the following techniques may be used.

(a) Note the change in shape of the projected loop image during a large angle tilt and thus obtain the sense of loop inclination relative to the electron beam direction.

(b) Tilt the loop into the edge-on configuration and evaluate $\mathbf{n}$ from its trace.

(c) Note the sense of rotation of the major axis or some other image feature which reflects uniquely changes in $\mathbf{n}$ during a large angle tilt experiment and as in (a) obtain the relative loop inclination.

(d) Extend method (c) to a trace analysis by imaging the defect at three or more exact or nearly exact orientations (in principle two orientations will give an answer); define a major axis by either measurement or interpretation of the image; assign a crystallographic direction to this vector which is then assumed to lie in the plane normal to $\mathbf{B}$ and the loop plane; obtain a graphical solution by normalising the families of all possible $\mathbf{n}$ for each $\mathbf{B}$ considered to a convenient orientation; and their intersections are therefore solutions to equations of the form $\mathbf{t}_i \cdot \mathbf{n} = 0$ where $\mathbf{t}_i$ is the estimated trace of the minor axis in the foil.

No single method (a)–(d) is satisfactory in all cases, but when combined can produce consistent results, perhaps only on a fraction of the total number of loop diameters, $\lesssim 200$ Å.

At least four cases of $\pm \mathbf{g}$ must be obtained that are completely consistent and six are ideal. At

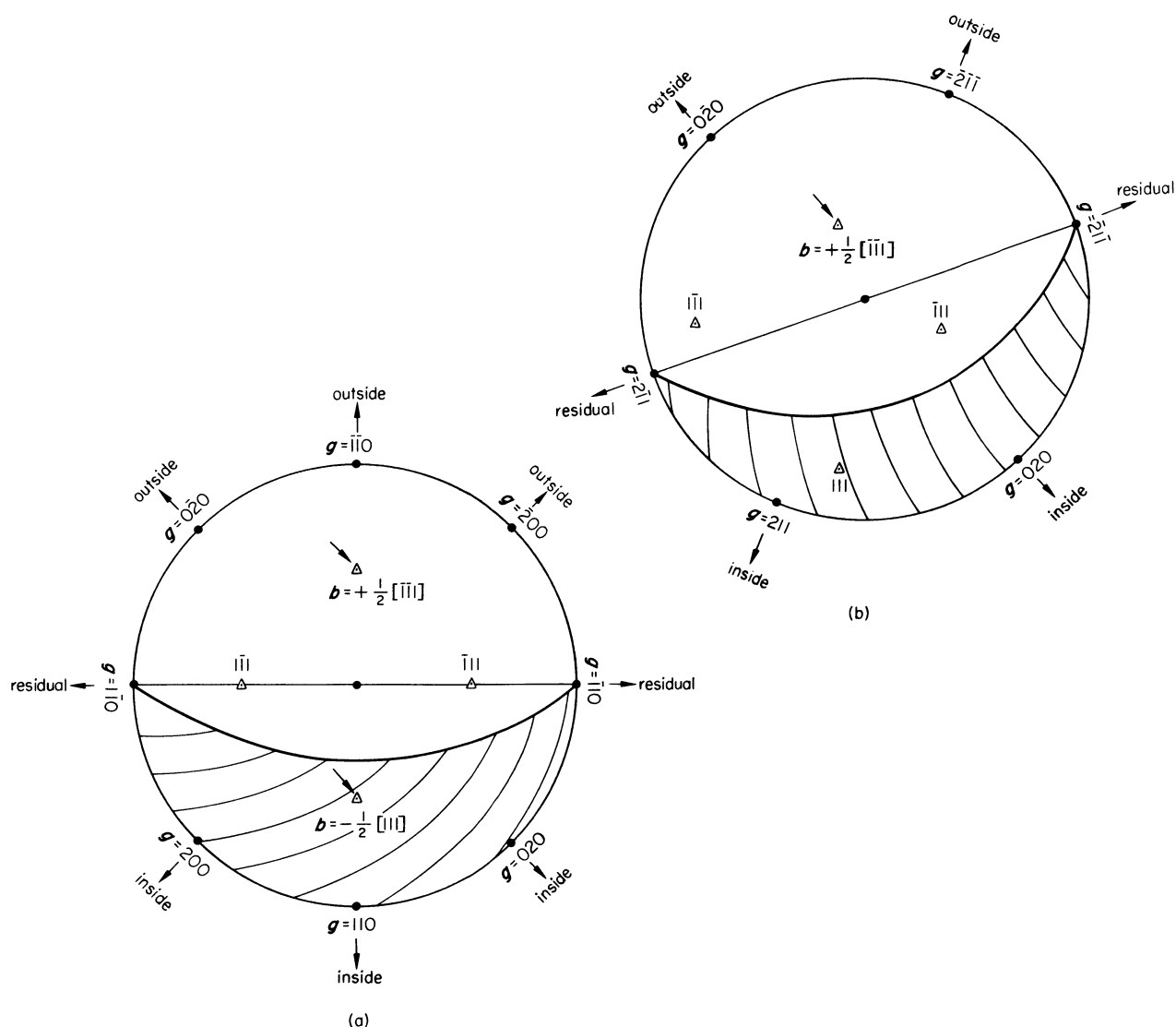


Figure 3.24 Stereographic projections summarising the results deduced from figures 3.21, 3.23. Shaded areas indicate the region of reversed edge behaviour for a loop with $b = \pm\frac{1}{2}[\bar{1}\bar{1}1]$. (a) (001) and (b) ($\bar{1}02$) projections [Courtesy of B. L. Eyre]

least two different B (~ 20 – 30° apart) must be used for the same $\pm g$ pair and different $\pm g$ pairs obtained for the two B .

It should be emphasised that considerable difficulty will be encountered for loop diameters $\lesssim 200$ Å. Nevertheless, if the method described in the previous section is used, bearing in mind points (1)–(3) above, and provided complete self-consistency is demanded quite reliable evaluations have been reported for loop diameters, ~ 100 Å in copper and molybdenum (Barry and Eyre, 1970; Barry and Maher, 1970; Maher and Eyre, 1971).

In summary Maher and Eyre (1971) have reported that for molybdenum the direction of b (that is $\pm\langle 111 \rangle$) can be readily defined by a two extinction criterion based on residual image properties of $g = \pm\langle 110 \rangle$ and $\pm\langle 211 \rangle$ and by an in-contrast $g = \pm\langle 200 \rangle$ image. It has been shown that the sense of b may be evaluated from a number of diffracting conditions of the type $\pm g$,

where the vectors selected included one or more of the following: $\langle 310 \rangle$, $\langle 222 \rangle$ and $\langle 321 \rangle$ if $w > 0.6$. Using 'safe' foil orientations and demanding internal self-consistency within the data a significant fraction of perfect loops in the size range $100 \text{ Å} < \text{diameter} < 500 \text{ Å}$ as well as diameter $> 500 \text{ Å}$ may be unambiguously analysed.

The same technique may be used in copper (Barry and Eyre, 1970; Barry and Maher, 1970) using $\pm g\langle 111 \rangle$ and $\langle 200 \rangle$ to determine the direction of b and $\pm g\langle 220 \rangle$ and $\langle 331 \rangle$ to determine inside/outside contrast and the sense of b .

3.5.5 Image Characteristics of Small Loops (Diameter $\lesssim 100$ Å)

Only exactly edge loops in elastically isotropic material will be considered here because these have received most attention. Work on loops with

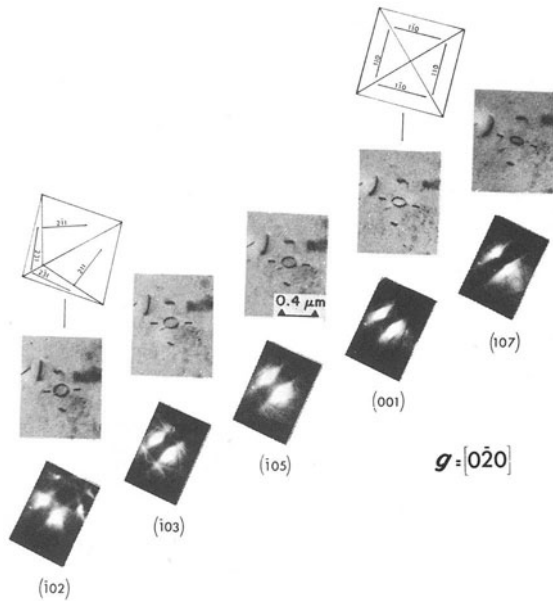


Figure 3.25 The large loop in figure 3.21 observed during a tilting sequence from B $[102]$ to $[107]$ using the same (020) reflection. The projected width of the loop is reduced, which is consistent with a sense of inclination for $n \approx [\bar{1}\bar{1}1]$ or $[\bar{1}1\bar{1}]$. However, the image rotates clockwise as would the major axis of a circle on $(\bar{1}\bar{1}1)$; compare the geometry of the $\langle 211 \rangle$ traces in (102) with $[\bar{1}\bar{1}0]$ and $[\bar{1}10]$ traces in (001) [Courtesy of B. L. Eyre]

a shear component is still in process (von Haüssermann, 1972) but the results are complex and only limited calculations for the elastically anisotropic case have been reported (McIntyre *et al.*, 1970).

3.5.5.1 $g \cdot b \neq 0$

Small edge Frank loops exhibit black/white contrast lobes in both bright and dark field images if the loops are within $\sim 1.25\xi_g$ of the surface and are imaged under dynamical two-beam conditions ($w \lesssim 1$), see figure 3.28.

The contrast behaviour of such edge loop images has been studied theoretically by a number of workers using the infinitesimal loop approximation to describe the strain field of the loop (Rühle *et al.*, 1965; Rühle, 1967a, b; McIntyre and Brown, 1966).

The main features of the contrast behaviour of small Frank loops (diameter $\ll \xi_g$, that is $0.2\xi_g$) have been summarised by Rühle (1972) and Wilkens (1969, 1970) and are described below.

(1) If $w \neq 0$ both vacancy and interstitial loops exhibit lobes of black/white contrast if they lie within $\sim 1.25\xi_g^*$ of the top and bottom of the foil.

(2) If a vector l is defined as joining the black to the white lobe, the direction of l is usually approximately within $\pm 10^\circ$ parallel to the projected $\langle 111 \rangle$ habit plane normals, independent

of the operative reflection. Various directions of l occur in figure 3.28.

(3) For a given type of loop, that is vacancy or interstitial, the sense of black/white contrast in relation to g depends on depth. This is shown schematically in figure 3.27(a), (b) without allowing for the spatial relationship between g and l . The foil may be divided into three layers, L_1 , L_2 , L_3 at the top and bottom surfaces corresponding to 0.025 , 0.35 to $0.7\xi_g$ and 0.8 to $1.25\xi_g$ with intermediate regions $\sim 0.1\xi_g$ thick in which black dot images form.

(4) Referring to figure 3.27(a), for a vacancy loop; g and l point in the same approximate direction (allowing for (2) above) in BF layers 1, 3, 5 but in the opposite approximate direction in layers 2, 4, 6, that is $g \cdot l$ positive layers 1, 3, 5 and negative layers 2, 4, 6. This situation is reversed for an interstitial loop, see figure 3.27(b).

(5) BF and DF images are identical at the top of the foil (layers 1–3, figures 3.27(a), (b)) but opposite at the bottom (layers 4–6, figures 3.27(a), (b)).

(6) Loops well within the foil, that is further than $1.25\xi_g$ from the surface, exhibit 'black dot' contrast due to anomalous absorption assuming $ANO = 0.1$. For smaller values of ANO the contrast reversal extends further into the foil.

(7) Wilkens (1970) has reported that for kinematical conditions $|s| \gg 1/\xi_g$ all loops show 'black dot' contrast for which the 20% image width shown in figure 3.47 is within 10% of projected loop width.

(8) According to Wilkens (1970), for dynamical conditions at $s = 0$ the 20% image width measured perpendicular to l is $\sim 1.2 \times$ the projected loop diameter.

(9) The presence or absence of the above behaviour is influenced by the magnitude of the parameter $P = g \cdot b R_L^2 / \xi_g^2$ for a Frank loop where R_L is the radius of the loop. It has been calculated by McIntyre and Brown (1966) and Chik *et al.* (1967) that if $P \ll 1$ black/white contrast has the characteristics of points (1)–(6) and, more importantly, oscillates rapidly with depth (point (2)). However, if $P \gg 1$ the depth oscillations are suppressed and all loops at the top and bottom behave like layers 1 and 6 in figure 3.27(a), (b). However, for unresolved loops $R_L \ll \xi_g$ the parameter $P \ll 1$ and depth oscillations of the type in figure 3.27 would always be expected for loops with $g \cdot b = 1$ (low-order reflection). The very large Burgers vectors necessary to produce large enough values of P to suppress depth oscillations are unlikely. Furthermore, increasing R_L would have the effect of producing resolvable loops. For

* See section 3.2.3.1 for definition of ξ_g .

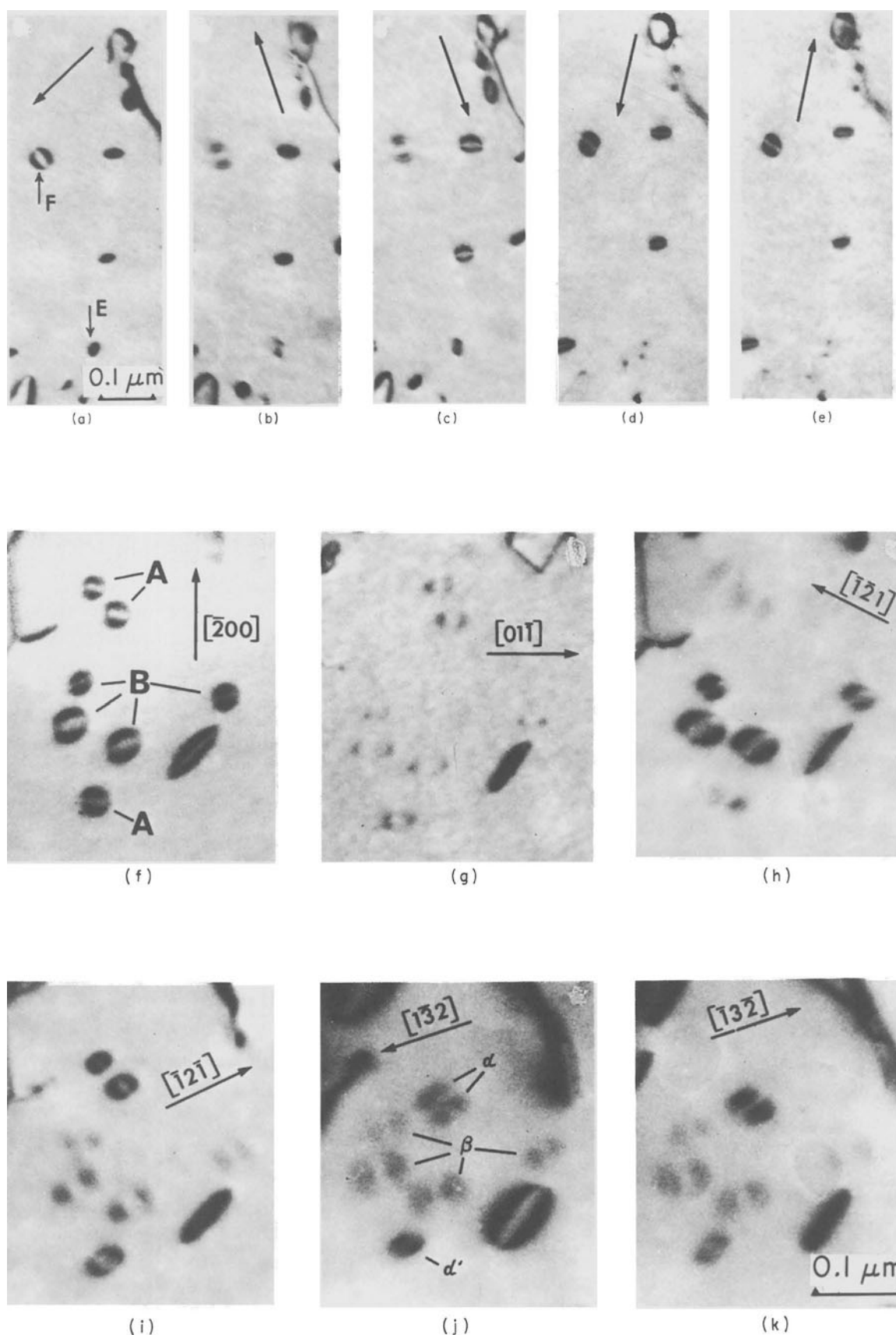


Figure 3.26 (a)–(e) Two medium-sized loops in neutron-irradiated and annealed molybdenum. Loop F lies perpendicular to the electron beam while E is parallel to it: (a) $B [012]$, $g [200]$; (b) and (c) $B [012]$, $g \pm [\bar{1}21]$; (d) and (e) $B [135]$ and $g \pm [21\bar{1}]$. (f)–(k) shows the Burgers vector analyses based on images with the (a) (200) , (b) $(01\bar{1})$, (c) $(\bar{1}2\bar{1})$, (d) $(\bar{1}2\bar{1})$, (e) $(\bar{1}3\bar{2})$, (f) $(\bar{1}3\bar{2})$ reflections [Courtesy of B. L. Eyre]

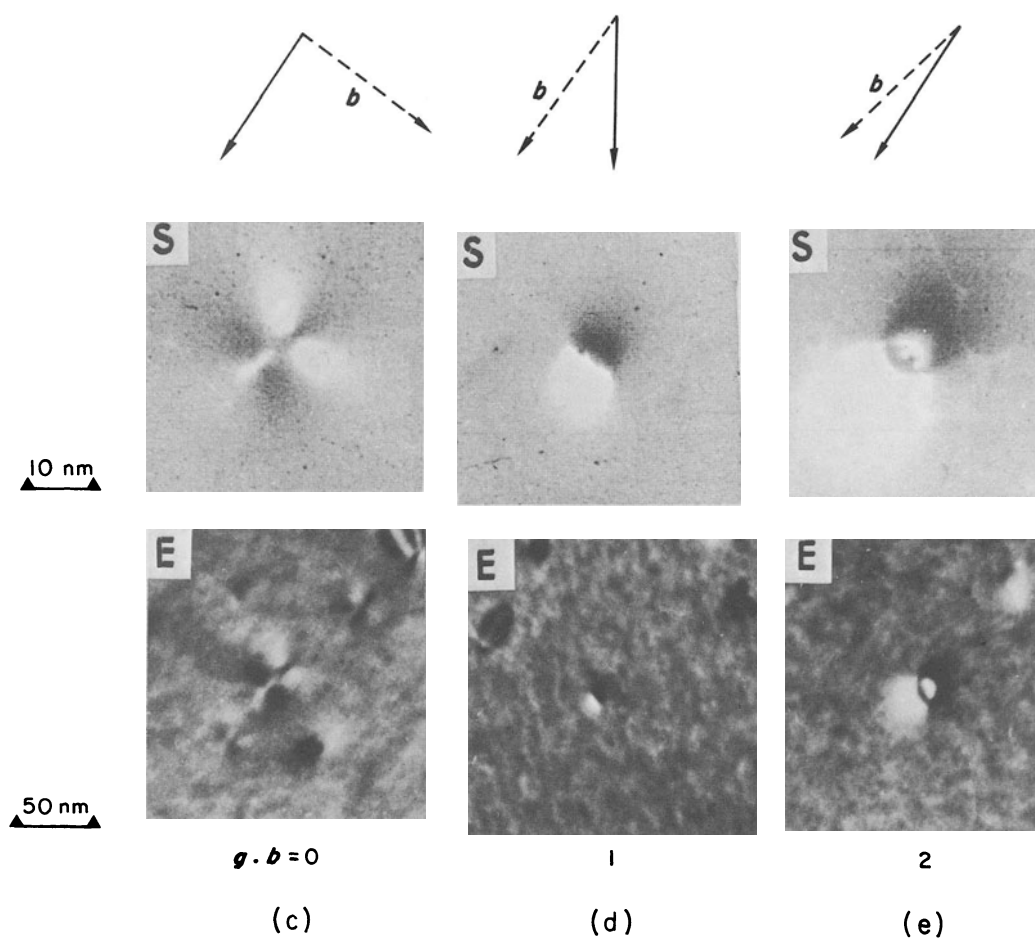
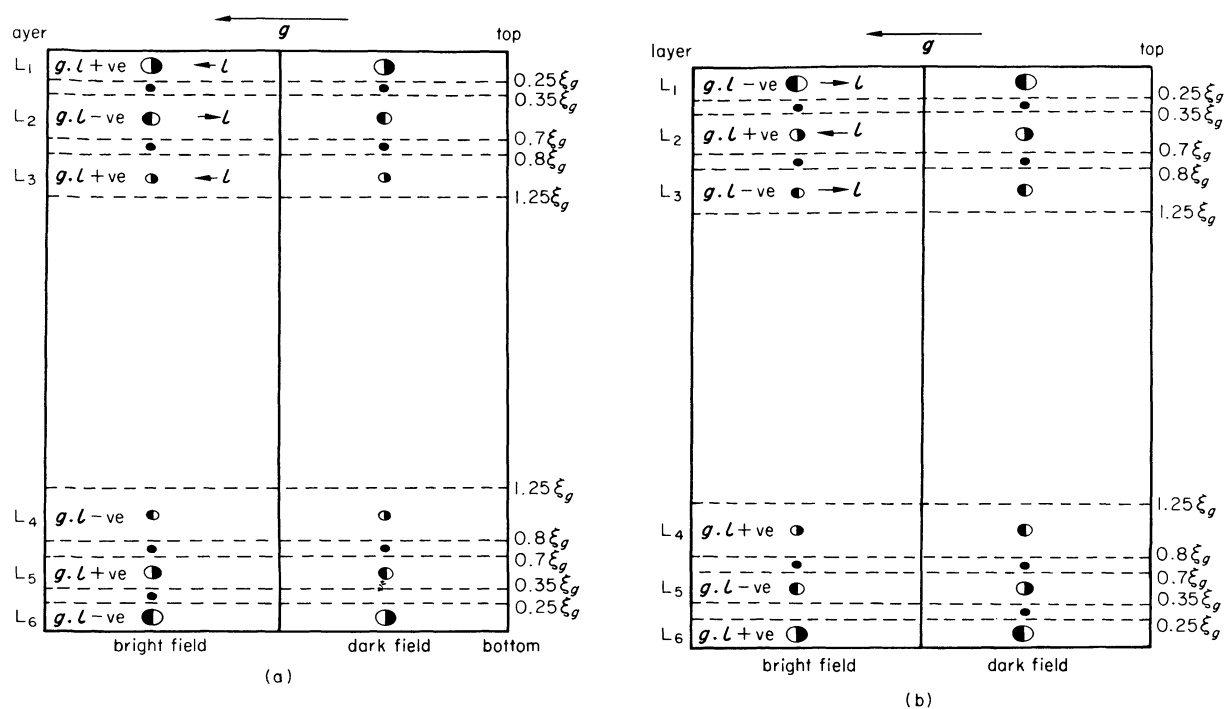


Figure 3.27 A schematic diagram illustrating the reversal of BF and DF image contrast with depth for a Frank (a) vacancy, (b) interstitial loop. (c)–(e) These show real (E) and computer-simulated (S) images of edge loops 46 Å in diameter and located in layer 6, (a), (b). In all cases $\mathbf{B} [011] \mathbf{g} [21\bar{1}]$ and $w = 0$. (c) $\mathbf{b} = \frac{1}{2}[1\bar{1}1]$ and $\mathbf{g} \cdot \mathbf{b} = 0$, (d) $\mathbf{b} = \frac{1}{2}[01\bar{1}]$ $\mathbf{g} \cdot \mathbf{b} = 1$ and (e) $\mathbf{b} [100]$ $\mathbf{g} \cdot \mathbf{b} = 2$ [(c)–(e) Courtesy of B. L. Eyre]

loops with a misfit parameter $P \approx 1$ further contrast calculations are necessary to determine image behaviour, preferably not using the infinitesimal loop approximation.

(10) Wilson and Hirsch (1972) have shown that dissociated small Frank loops produce 'notched' black/white contrast under some reflections.

3.5.5.2 $g \cdot b = 0$

Wilkens (1970) has reported that Frank loops show very weak contrast for $g \cdot b = 0$ conditions. However, Eyre *et al.* (1971) using computer simulation techniques have shown that the situation is more complicated. The form of the image of a Frank loop $b = \frac{1}{2}\langle 110 \rangle$ or $\frac{1}{2}\langle 111 \rangle$ or $\langle 100 \rangle$ in b.c.c. metals depends on the inclination of the loop within $\pm 10^\circ$ of edge-on geometry in the foil (n orthogonal to B , figure 3.19(f)) and consists of three black lobes and three white lobes, see figure 3.27(c), whereas loops inclined at 45° and 90° to B show faint simple black/white contrast. Consequently, a $g \cdot b = 0$ criterion cannot be used for determination of the direction of b . Computer matching techniques must be used.

3.5.6 Quantitative Information

Quantitative studies of very small loops require very precise experimentation and are one of the most difficult to perform satisfactorily. For example, it is only recently that it has been agreed that both vacancy and interstitial loops occur in irradiated copper (Rühle *et al.*, 1968; Ipohorski and Brown, 1970). Consequently, it must be emphasised *considerable* care must be exercised.

Two types of experiment are possible, namely the determination of the Burgers vector and vacancy/interstitial sense of the loop. In these terms, the important and useful features of image contrast are (3)–(5), (7), (9) in section 3.5.5.1.

3.5.6.1 Determination of the Burgers vector

First it is necessary to ascertain that the defect under observation is indeed a Frank loop. It is assumed that the image has been shown to be a dislocation loop and not another defect such as a precipitate, void, gas bubble, etc., using the approach in section 3.15. It is here only necessary to determine if the image is characteristic of a Frank loop and does not have a shear component. If the images have the properties (1), (2) and (6), section 3.5.5.1, for a number of different reflections and complex images are not obtained it can be concluded that the defect probably is a Frank loop. Note however, that in high stacking fault energy materials Frank loops arising from irradiation damage are unlikely since the fault has probably

been removed to form a loop with a shear component.

The determination of the direction of b for a Frank loop simply involves crystallographic analysis of the projection of b , that is l into the diffraction pattern, bearing in mind point (6), section 3.5.5.1. A large number of loops > 200 should be analysed to obtain a good statistical survey.

3.5.6.2 Determination of the vacancy/interstitial sense of the loop

Diepers and Diehl (1966), Diepers (1967a, b) and Rühle (1967) have developed an experimental approach designed to differentiate between vacancy and interstitial edge loops. The results of this approach are not valid if loops have a significant shear component. Only preliminary information is available for loops with a shear component (von Haüsserman, 1972) and definite conclusions cannot be drawn at present. Details of the theoretical and experimental studies have been summarised by Wilkens (1969, 1970), Rühle (1972) and Eyre (1972).

The procedure rests on the determination of the sign of $g \cdot l$ for two-beam conditions with $g \cdot b \neq 0$ and $w < 1.0$ for a defect of known depth in the foil. Determination of the depth of the defect in the foil presents some difficulty because it must be known accurately to determine in which layer the defect is situated. Indirect techniques have been used by several authors (Brimhall *et al.*, 1966; Bell and Thomas, 1966; Rühle, 1967b) involving change of sign of $g \cdot l$ on changing g (that is ξ_g). However, the best method involves stereomicroscopy (Diepers and Diehl, 1966; Diepers, 1967a, b; Rühle, 1967a), see section 1.6.

The procedure for determining the sense of the loop is as follows.

(1) Prove the defects behave as edge Frank loops as above.

(2) Evaporate gold onto the top surface of the specimen to form islands $\sim 50 \text{ \AA}$ diameter and $10 \text{ \AA}$ thick.

(3) Obtain images of a given area with two-beam conditions ($w \lesssim 1.0$) for second-order reflections. These give better defined lobe contrast than the first-order reflections and require less accuracy of depth measurement since ξ_g increases with increasing order of reflection.

(4) Obtain stereo pairs separated by a tilt of $20\text{--}30^\circ$ with the same operative reflection, sign and value of w under kinematical conditions. Only in this case does the centre of the black spot correspond to the real position of the defect in the foil.

(5) Measure the depth of the defects from the top surface of the foil using stereo viewing techniques (see section 1.6). The accuracy of depth measurement can be as good as $\pm 20 \text{ \AA}$ under

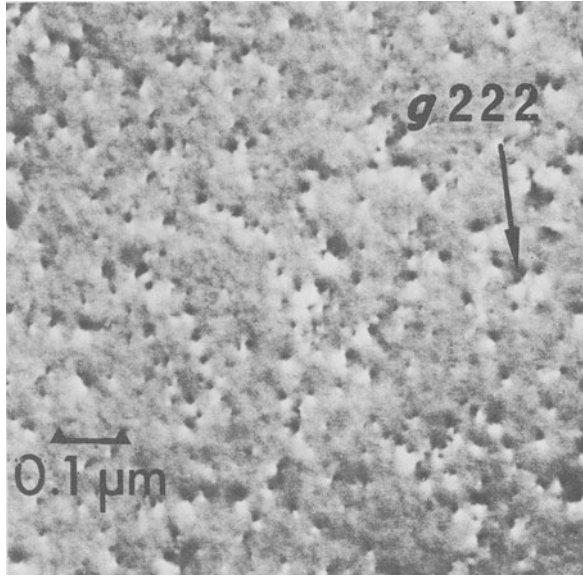


Figure 3.28 Ion damage in a Ni-4.5 wt % Ti-1.96 wt % C alloy [Courtesy of R. B. Scarlin]

favourable conditions provided the surface oxide film has negligible thickness and carbon contamination has been effectively eliminated by an anti-contaminator at the specimen. Note that surface films can lead to the wrong sign of a defect because depth measurements are wrong (Wilson, 1968).

(6) Use the sign of $\mathbf{g} \cdot \mathbf{l}$ to determine the vacancy/interstitial nature of the loop, knowing the depth and ξ_g , that is in which layer of figure 3.27 the loop lies. Thus for layer 1 in BF, $\mathbf{g} \cdot \mathbf{l}$ is positive for a vacancy loop but negative for an interstitial loop. Note that, since $\mathbf{l}$ is approximately parallel to the projection of $\langle 111 \rangle$, $\mathbf{g}$ is in general not parallel to $\mathbf{l}$. Thus the sign of $\mathbf{g} \cdot \mathbf{l}$ follows the behaviour in figure 3.27, whereas its magnitude does not.

An example of the determination of the vacancy/interstitial nature of ion damage in a nickel alloy is shown in figure 3.28. A high density of black/white strain field images is visible with the vector $\mathbf{l}$ running in the same sense relative to $\mathbf{g}$ for most of the loops. Experiments showed that for most loops $\mathbf{l}$ was parallel within $\pm 10^\circ$ of the projected $\langle 111 \rangle$ for different $\mathbf{g}$ and $\mathbf{B}$. Thus the behaviour of the defects is characteristic of Frank loops and the analysis described above may be applied. Stereo electron microscopy showed that almost all the defects lay within $\sim 100 \text{ \AA}$ of the surface. The value of $\mathbf{g} \cdot \mathbf{l}$ was positive for most of the defects, see figure 3.28, which since they lie in layer 1 of the BF image, figure 3.27, are vacancy in character.

3.6 α Boundaries, Stacking Faults, Planar Precipitates

The α -boundary type of image is usually produced in the electron microscope by either a stacking

fault or a planar precipitate. In studying such images we are interested in proving that the defect is an α boundary. In addition it may be necessary to determine the direction and magnitude of its displacement vector, for example extrinsic/intrinsic nature of a stacking fault. In effect the term α boundary describes an interface that produces a phase factor $\alpha = 2\pi \mathbf{g} \cdot \mathbf{R}_n$ in equation (3.5a), where $\mathbf{R}_n$ is a displacement vector associated with the boundary. Thus α can have a range of values from $0-2\pi$ depending on $\mathbf{R}_n$. However, the specific case of $\alpha = \pi$ is described separately in section 3.7 because of its special image characteristics.

Most studies of α boundaries have been confined to the case of stacking faults in the f.c.c. crystal structure although a limited amount of work has been performed on other crystal structures. The type of image obtained from stacking faults can be similar to that of planar coherent precipitates consequently these are treated in the same section.

3.6.1 Image Characteristics of Stacking Faults

3.6.1.1 Single stacking faults $\alpha = 0, \pm 2\pi/3, \pm 4\pi/3, \pm 2\pi$

The contrast arising from stacking faults has been studied theoretically for f.c.c. metals by Whelan and Hirsch (1957a, b), Hirsch *et al.* (1960) and Hashimoto *et al.* (1960, 1962). A typical experimental situation is shown schematically in figure 3.29(a) in which a stacking fault lies in an inclined plane in a thin foil. The displacement associated with such a fault on $\{111\}$ in an f.c.c. crystal structure is shown schematically in figure 3.29(c), (d). An intrinsic stacking fault may be created by removing the central C (111) plane and re-welding the crystal together, that is a collapse of crystal I on to crystal II in the direction $\mathbf{R}_F$, figure 3.29(d), of magnitude $\frac{1}{3}[111]$ the (111) interplanar spacing. The intrinsic fault created has the (111) stacking sequence ABCABABC. An extrinsic fault would have a plane inserted ABCABACABC and $\mathbf{R}_F$ would be reversed. The stacking fault introduces a phase factor into the transmitted and diffracted beams $\alpha = 2\pi \mathbf{g} \cdot \mathbf{R}_F$, equations (3.5). In the f.c.c. case for a fault on (111) $\mathbf{R}_F = \pm \frac{1}{3}[111]$ and $\alpha = 0, \pm 2\pi/3, \pm 4\pi/3$ etc. The important conclusions of the theory are as follows.

(1) The bright field image consists of a series of alternate dark and bright fringes running parallel to the intersection of the fault plane and the foil surface; this is $[uvw]$ in figure 3.29(a). Furthermore, the outer fringes in BF are both dark or light, that is image symmetric. The geometric situation and a BF image of a stacking fault are shown in figure 3.29(a), (b). Absorption damps out fringe contrast in the centre of the foil, figure 3.30(a).

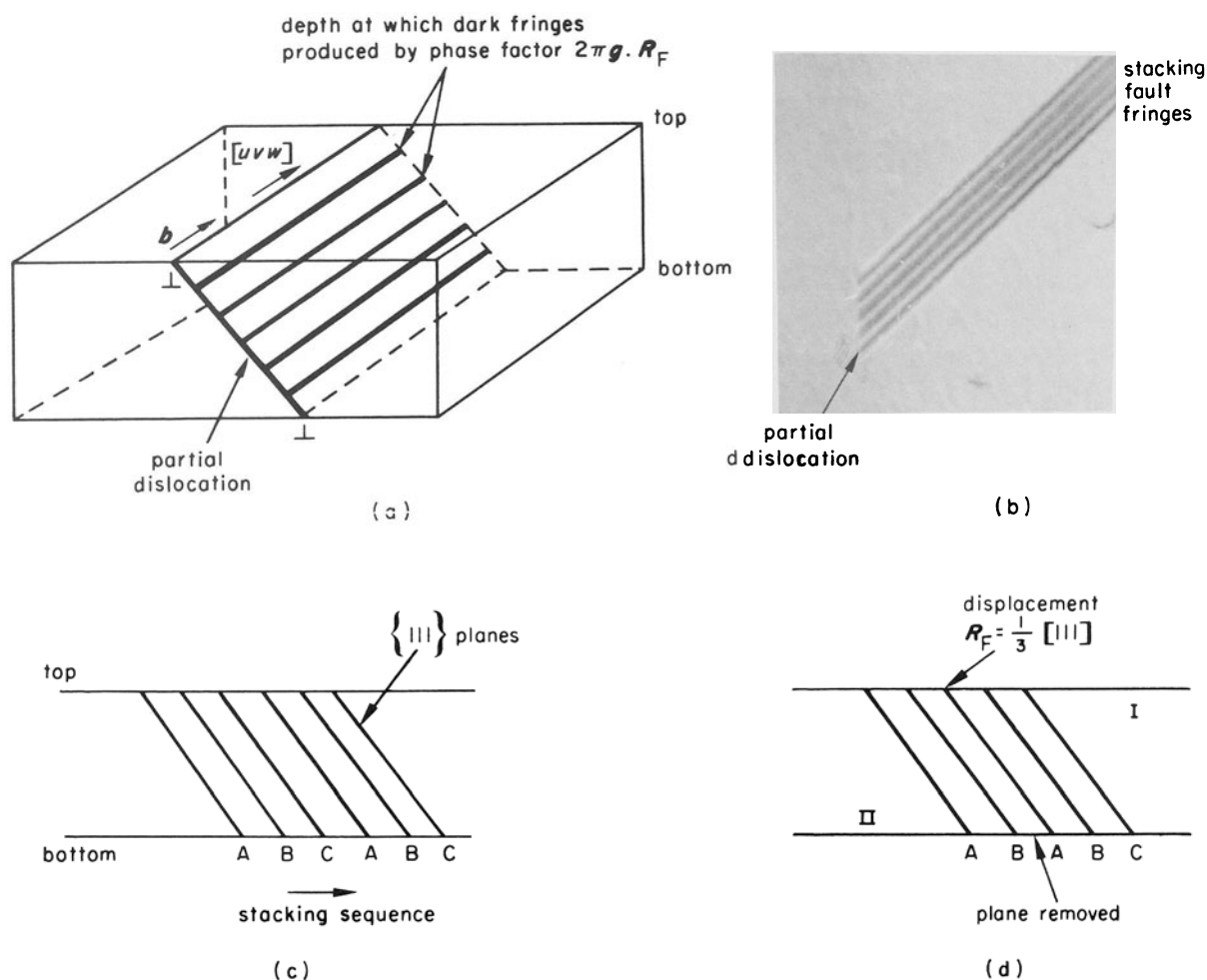


Figure 3.29 A schematic illustration of a stacking fault lying on an inclined plane showing the depths of the fringes and the position of the bounding partial dislocation. (b) The BF image in the microscope. (c) The perfect stacking sequence for inclined $\{111\}$ planes. (d) The sequence when an intrinsic (f.c.c.) stacking fault is present

(2) The dark field image is also a series of fringes but the outer fringes are not the same, figure 3.30(b), that is the image is asymmetric. If CDF is performed using the same reflection as was operative in the BF image the first fringe (that at the top of the foil) is the same in BF and DF but the last fringe shows opposite contrast. However, normally CDF is performed using opposite g to the BF (see section 1.5.2) and the top fringes are then opposite contrast, compare figure 3.30(a), (b). This behaviour enables the sense of inclination of the fault plane to be determined.

(3) On a positive print the first fringe at the top of the foil in BF is bright for $g \cdot R_F$ positive and dark for $g \cdot R_F$ negative which is the basis of methods for determination of the extrinsic/intrinsic nature of a fault.

(4) Stacking faults are invisible when $g \cdot R_F = 0$, 1, etc., see figure 3.16(c)–(e).

(5) As the thickness of the specimen changes, fringes add from the centre by branching of existing fringes; the additional fringe occurs at the centre

of the fringe pattern and the outer fringes are unaffected, see figure 4.1 at A.

(6) At $s = 0$ the number of dark fringes is $(n - 1)\xi_g =$ the foil thickness t . However, $s \neq 0$, the number of fringes is not simply related to t .

(7) The minimum value of $g \cdot R_F$ for which observable fringe contrast occurs for the general fault is 0.02 (Humphreys *et al.*, 1967).

3.6.1.2 Overlapping stacking faults

These have the following characteristics described by Gevers *et al.* (1964a) and Humble (1968a, b).

(1) Closely spaced overlapping faults on parallel (111) planes cause fringe shifts because the phase angle changes by $\pm \frac{2}{3}$ to $\pm \frac{4}{3}$, see figure 3.31 at 2. Every third set of fringes should show zero contrast since $\alpha = 3 \times \pm 2\pi/3 = 2\pi$, see figure 3.31 at 3. Very faint fringes may, however, be formed as shown in figure 3.31 at 6, which may be used to determine the separation of the overlapping fault planes by image matching techniques (Humble, 1968a).

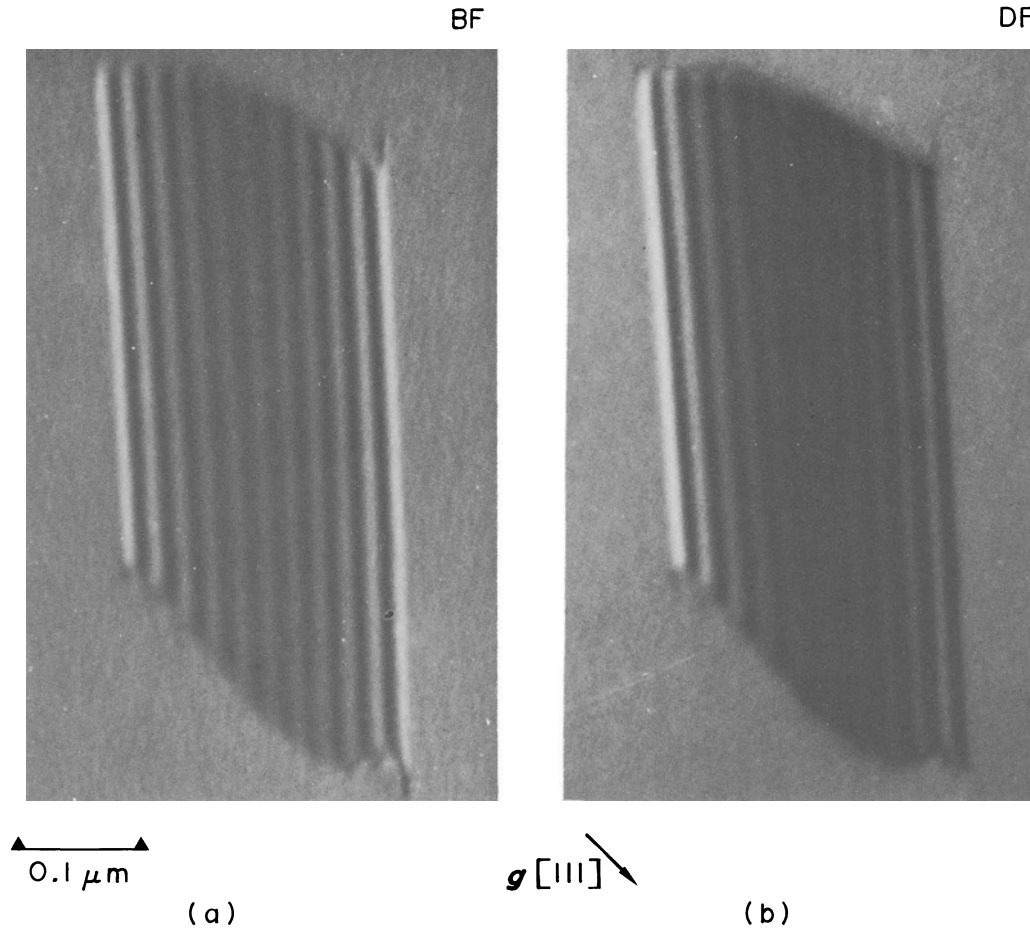


Figure 3.30 A stacking fault in a Co-7 at % Ti alloy: (a) BF; (b) CDF

(2) Overlapping faults separated by significant distances can cause damping or extinction of fringes in the region of overlap, see figure 4.1 at X. For a detailed discussion, see Gevers *et al.* (1964a).

(3) Crossing faults produce complicated characteristic of overlapping fringes parallel to the

main fault pattern with secondary fringes parallel to the line of intersection YY in figure 4.1 which shows intersecting not crossing faults.

3.6.2 Quantitative Information on Stacking Faults

Stacking faults may be identified and their extrinsic/intrinsic nature determined. To maximise contrast $s = 0$ should always be used (Sheinin and Botros, 1970).

3.6.2.1 Identification

α boundaries may be identified by comparison of BF and CDF images and by the addition of fringes by branching in the centre of the fringe pattern (points (2) and (8), section 3.6.1.1). Both are characteristic only of α boundaries.

3.6.2.2 Determination of the direction of the displacement R_n

Since the fault is invisible when $g \cdot R_n = 0$, the $g \cdot R_n = 0$ invisibility criterion may be used to determine R_n by obtaining cases of invisibility with two different operative reflections. Then R_n will be the common direction to the two reflecting planes.

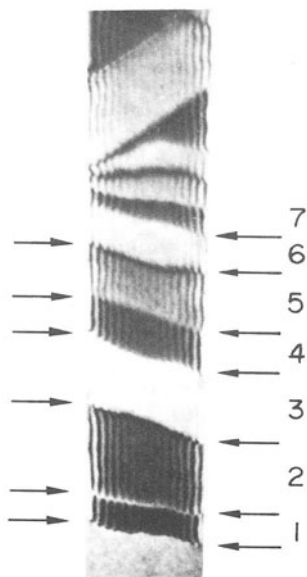


Figure 3.31 Overlapping stacking faults in a Co-7 at %Ti alloy. The partial dislocations are arrowed and the numbers indicate the number of overlapping faults

The magnitude of R_n may be determined from the number of overlapping fringes required to produce invisibility for stacking faults in f.c.c. metals, see section 3.6.1.1. In principle the same approach should be applicable when $\alpha \neq 2\pi/3$ but considerable care should be taken because faint fringes may occur when $\alpha \approx 0$ or 2π . Under these circumstances computer matching may be used to determine the magnitude of R_n (Hollox *et al.*, 1972).

3.6.2.3 Determination of the extrinsic/intrinsic nature

(a) *f.c.c. crystal structures.* A number of methods for determination of the intrinsic/extrinsic sense of stacking faults have been given (Howie, 1961; Hashimoto *et al.*, 1962; Art *et al.*, 1963; Gevers *et al.*, 1963; van Landuyt *et al.*, 1966b). In principle the method rests on point (3), section 3.6.1.1, since the nature of the top fringe depends upon whether $g \cdot R_F$ is positive or negative. Rules have been developed involving comparison of BF and DF images (Art *et al.*, 1963) from a DF image only (Gevers *et al.*, 1963) or from a BF image (van Landuyt *et al.*, 1966b).

It is important to note that the fringe contrast is sensitive to slight alterations of s which can make identification of the nature of the first fringe difficult. Consequently, determinations should always be performed at $w = 0$ when using the method of Art *et al.* (1963). Using the method of Gevers *et al.* w may be 0 or positive while for that of van Landuyt *et al.* w must be 0 or negative, see Head (1969).

The simplest analysis is shown here, that is that involving a DF micrograph. The rule is as follows.

If the origin of the g vector is placed at the centre of the fault in a CDF image, g points away from the light outer fringe if the fault is extrinsic and towards it if it is intrinsic for all $\{200\}$, $\{222\}$ and $\{440\}$ (type A) reflections regardless of the sense of inclination of the fault. If the operative reflection is of the type B $\{400\}$, $\{111\}$, $\{220\}$ the reverse is true. An intrinsic fault is shown in figure 3.30(b).

The procedure to determine the nature of the fault in this way is the following.

- (1) Obtain both a BF and CDF* image at $w = 0$ and verify that the defect has fault ($\alpha = \pm \frac{2}{3}\pi$) characteristics, that is points (1) and (2), section 3.6.1.1. (BF image symmetric, DF image asymmetric.)
- (2) Take a diffraction pattern in the CDF mode.

Table 3.5 The nature of the fringes at the top (T) and bottom (B) of the foil for stacking faults of types I and II in wurtzite

g vector	Type I				Type II			
	(1)		(2)		(1)		(2)	
	T	B	T	B	T	B	T	B
$h - k = 3n + 1$	$l = \text{even}$							
	B	D	D	B	D	B	B	D
$h - k = 3n - 1$	$l = \text{odd}$							
	D	B	B	D	D	B	B	D
$h - k = 3n$	$l = \text{even}$							
	D	B	B	D	B	D	D	B
$h - k = 3n + 1$	$l = \text{odd}$							
	B	D	D	B	B	D	D	B

D, dark; B, bright. (1), (2) fault starts on A or B plane.

(3) Draw g on the negative from the transmitted spot to the diffraction spot used to form the image.

(4) Transfer g to the positive print making allowance for the lens rotation and the 180° rotation (see section 1.5.1) setting the origin of g to the fault centre.

(5) Use the above rule to determine the sense of the fault.

(b) *Hexagonal crystal structures.* The determination of the sense of stacking faults in c.p.h. metals has not been studied in detail. However, Blank *et al.* (1964) have shown that it is possible to differentiate between basal plane faults produced by deformation or vacancy/interstitial condensation. The method relies on the difference in faulting sequence for the two following cases.

(1) Type I condensation faults ABABCBCBC[†] formed by the collapse of either a vacancy or interstitial faulted loop following a Shockley partial shear to the low-energy fault above, that is $\frac{1}{2}[0001] + \frac{1}{3}\langle 10\bar{1}0 \rangle$.

(2) Type II deformation faults ABABCACA[†] formed by the dissociation of two Shockley partials $b \frac{1}{3}\langle 10\bar{1}0 \rangle$.

Blank *et al.* showed that both types of fault are invisible for reflections with $h = k = 0$ or $3n$, where n is an integer, for example (0002) reflection. However, both faults are visible for reflections with $h - k = 3n + 1$ and table 3.5 shows the nature of the dark field image fringes for the cases of type I and II faults starting on the A(1) and B(2) plane. Using two DF images, for l even and odd for the same value of $h - k$ type I and type II faults may be differentiated. The rule is as follows.

If the outer fringes have the same contrast for l even and odd the fault is a deformation fault type II. If the outer fringes have different

* If an analysis is used in which it is necessary to compare BF and DF images it is very important to remember that the conventional CDF technique (see section 1.5.2) reverses g . Thus the top fringes of the stacking fault show the opposite not the same contrast.

† These faults are described by Hirth and Lothe (1968).

contrast the fault is a vacancy or interstitial condensation fault of type I.

The procedure is the following.

- (1) Obtain two CDF images with $h - k = 3n + 1$ and $w = 0$.
- (2) Use the above rule to determine the nature of the fault.

3.6.3 Image Characteristics of Plate Precipitates

Some coherent and semi-coherent precipitate plates lying on inclined planes exhibit fringe images that arise from the displacement associated with the precipitate misfit. Figure 3.32 shows schematically the misfit R_n normal to the habit plane of a thin coherent precipitate inserted between the C and A (111) matrix planes with perfect atomic matching in the (111) plane. Provided the precipitate does not contribute to the diffraction process (that is is thin) then α fringes ($\alpha = 2\pi g \cdot R_n$) will occur with symmetric BF and asymmetric DF images like stacking faults.* Clearly, depending upon the magnitude of R_n , α may have any value and therefore fringes may not disappear for an integral number of overlapping precipitates.

The depth periodicity and intensity of displacement fringes used need not be the same as for a stacking fault. When $w = 0$ the depth periodicity is $\xi_g/2$ whereas if $w \gtrsim 1$ it is equal to the effective extinction distance $\xi'_g = \xi_g/(1 + w^2)^{1/2}$. Furthermore, the intensity of the fringe contrast depends upon the misfit and decreases when $g \cdot R_n$ approaches 0 or an integer. Finally, it is important to realise that there has been no detailed theoretical analysis of boundaries with a misfit component in the boundary plane, that is R not normal to the habit plane. The above discussion may not necessarily apply to this case.

3.6.4 Quantitative Information on Plate Precipitates

Displacement fringes do not normally give a great deal of information about a precipitate because the periodicity is only a function of the matrix structure and the intensity is subject to many variables. However, the direction and sense of displacement, that is inwards or outwards relative to the precipitate, has been reported for simple cases with R_n normal to the habit plane for 'effectively' non-diffracting precipitates, for example stainless steel (Silcock and Tunstall, 1964), silicon (Booker and Tunstall, 1966), GaAs (Laister and Jenkins, 1968, 1969). If the precipitate diffracts significantly, the fringes may develop a δ component, section 3.8, figure 4.2, and neither the direction nor magnitude of R_n can be obtained.

* If $\alpha = \pi$ then the fringes will be characteristic of π boundaries described in section 3.7.

The procedure to analyse the sense of misfit from a precipitate plate is as follows.

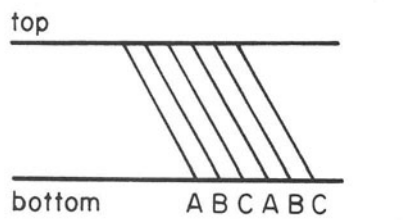
- (1) Obtain BF and CDF images under two-beam conditions at $w = 0$. Show that the BF image is symmetric and the DF asymmetric, that is the image consists of displacement fringes and can be treated as if it were a stacking fault.
- (2) Obtain at least two cases of invisibility to check that the misfit is perpendicular to the habit plane, that is there is no complication from mismatch strain in the habit plane.
- (3) Use the rules described in section 3.6.2.3 to determine the sense of the strain field.

It is important to realise that, unless (1) and (2) are fulfilled, the analysis may not necessarily be applicable. Section 4.2.1.2 describes such a case of precipitates in niobium that do not exhibit pure α fringes.

A typical analysis of displacement fringes is shown in figure 3.32(c)–(f) which show boride precipitates in vanadium carbide. The material is single-crystal $VC_{0.84}$ diffusion doped with boron at 1800 °C. Trace analysis and stereo microscopy showed that the defect A lay on $(11\bar{1})$, $B = [110]$. Under the (220) and (202) reflections defect A was invisible, see figure 3.32(e), (f). Consequently, the displacement vector is parallel to $[11\bar{1}]$. Detailed consideration of fringe images (Hollox *et al.*, 1972) showed that the displacement vector was $\sim \frac{1}{3}[11\bar{1}]$. Comparison of BF and CDF images, figure 3.32(c), (d), shows that the defects produce α -type fringes like stacking faults, that is symmetric BF and asymmetric DF images. Furthermore, the sense of the displacement can be determined from the DF image, using the method of Gevers *et al.* (1963), section 3.6.2.3. The reflecting plane is $\{111\}$ and g points from the centre of the fault to the dark outermost fringe for defect A. Thus the sense of the displacement vector is equivalent to an intrinsic fault for A. Sections C and D of the long defect on $(11\bar{1})$ can be analysed as extrinsic while E and F are intrinsic. In the case of this long defect the above behaviour is consistent with overlapping defects, similar to stacking faults in f.c.c. metals, section 3.6.1.2.

3.7 π Boundaries: Antiphase Domain Boundaries

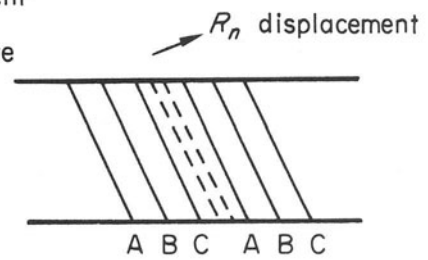
π boundaries are a special case with $\alpha = \pi$ and have specific image characteristics. In studying π boundaries we are interested in proving that they are π not α boundaries and in identifying the defect responsible for the image. π boundaries usually occur for antiphase domain boundaries (APB) in ordered materials. This defect occurs



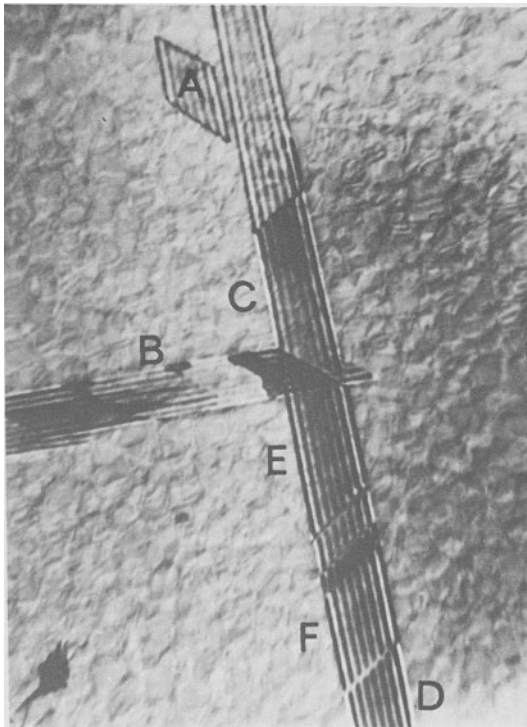
(111) stacking sequence

(a)

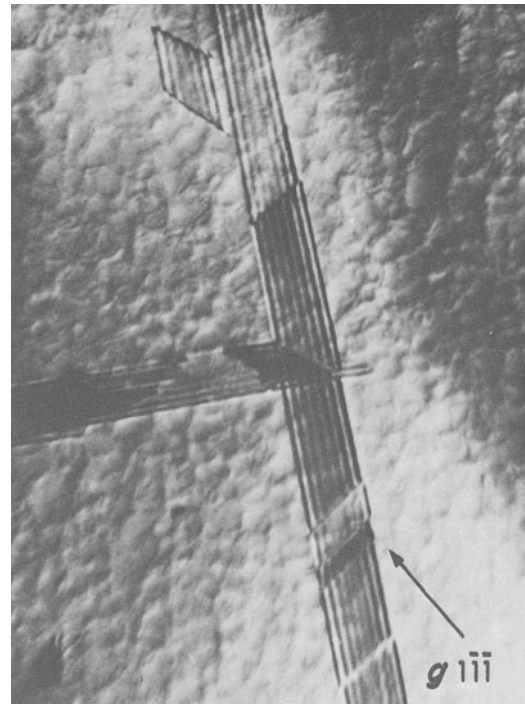
extra planes
of coherent
planar
precipitate



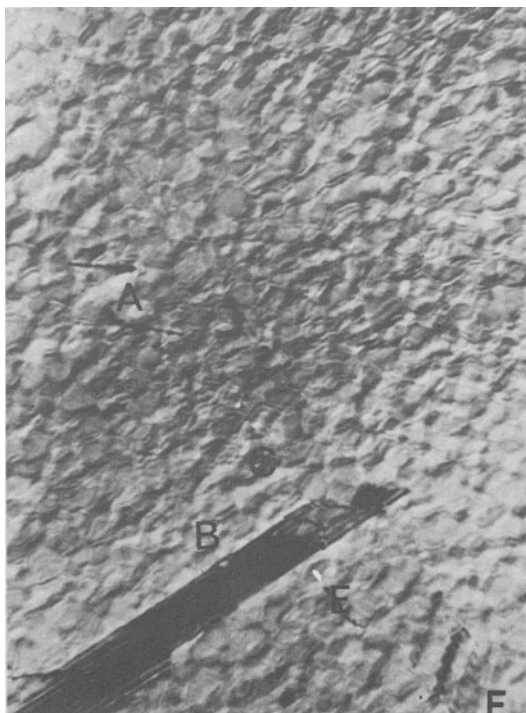
(b)



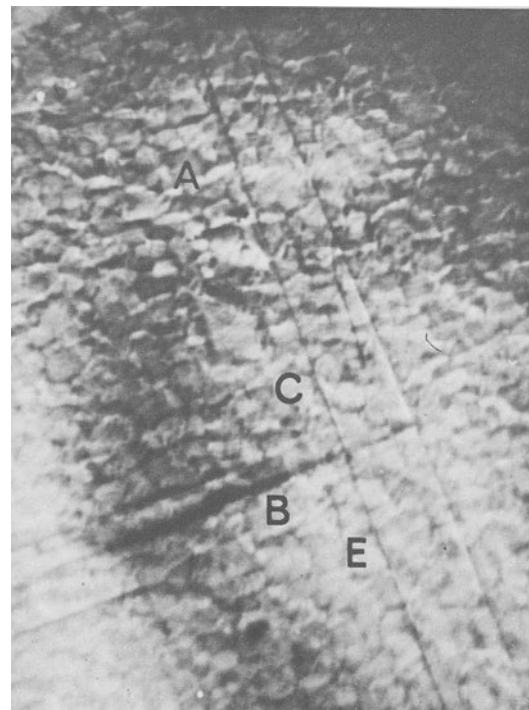
(c)



(d)



(e)



(f)

Figure 3.32 The stacking sequence of (111) planes in vanadium carbide (a) without, (b) with a planar precipitate on (111), (c) BF and (d) DF images of the planar precipitate. g points towards A, C-F fringe in defect A (intrinsic) and the bright fringe in defect B (extrinsic). (e) and (f) fringes invisible for the same area with $(2\bar{2}0)$ and (202) reflections operating respectively

when there is a change in the identity of the atom at a given lattice point but there is no atomic stacking change like that at a stacking fault. A schematic example is shown in figure 3.33(a), (b) in which there is a change in atom identity such that like atoms become nearest neighbours at the boundary XY.

3.7.1 Image Characteristics

3.7.1.1 Single boundaries

Electron microscope contrast has been treated theoretically in terms of the kinematical theory by Marcinkowski (1962), and the dynamical theory by Fisher and Marcinkowski (1961) and van Landuyt *et al.* (1964). The results have been used to determine the APB vector in TiO_2 (van Landuyt, 1966). The APB may be characterised by a displacement P , see figure 3.33(b), and a phase angle $\alpha = 2\pi g \cdot P$ is introduced in equations (3.5). However, because P is an interatomic distance $g \cdot P = 0, 1, 2$, etc., for fundamental reflections and $\alpha = 0, 2\pi, 4\pi$, etc., see table 3.6. Thus the APB is invisible. For superlattice reflections $g \cdot P$ can have values $0, \frac{1}{2}$, etc., that is $\alpha = 0, \pi$, etc., and a fringe image occurs for $\alpha = \pi$.

In essence the phase angle α arises because of the difference in atomic scattering factor across the APB because of the change in ordering sequence.

The image of a π boundary or APB consists of alternate black/white fringes running parallel to the intersection $[uvw]$ of the habit plane with the foil surface. When absorption is included the detailed characteristics of the image obtained for two-beam conditions with $w = 0$ are as follows.

(1) The APB image only occurs for excitation of reflections for which $\alpha = \pi$, that is in general

for superlattice, not fundamental, reflections, compare figures 3.33(c), (e).

(2) Both bright and dark field images are symmetric about a foil centre, that is the outside fringes of a given image are both black or both white, compare figures 3.33(c), (d).

(3) Over the whole depth of the foil the bright and dark field images are exactly opposite, compare figures 3.33(c), (d).

(4) The fringes are parallel to the centre line of the habit plane. Thus the nature of the first and last fringes for a given g is determined by the foil thickness and two fringes add to every thickness fringe, see figure 3.33(f).

(5) The fringe spacing is $\frac{1}{2}\xi_g$, where ξ_g is the extinction distance given by the formula

$$\xi_g = \frac{\pi a_0^3 \cos \theta}{\lambda F} \quad (3.8)$$

for cubic materials where a_0 is the lattice parameter of the ordered unit cell, θ is the Bragg angle, λ is the electron wavelength and F is the structure factor, see table 3.7.

(6) The fringes are continuous along a closed loop of boundary, see figure 3.33(g).

van Landuyt *et al.* have reported detailed calculations of fringe profiles for positive and negative values of w . The details of the changes in the form of the image are complicated but the following important facts emerge.

(1) Dark field images are no longer symmetrical.

(2) In bright field images with positive w the depth fringe spacing can increase to ξ_g . In certain cases for negative w the fringe spacing can decrease $< \frac{1}{2}\xi_g$.

Clearly then, studies of APB should be performed

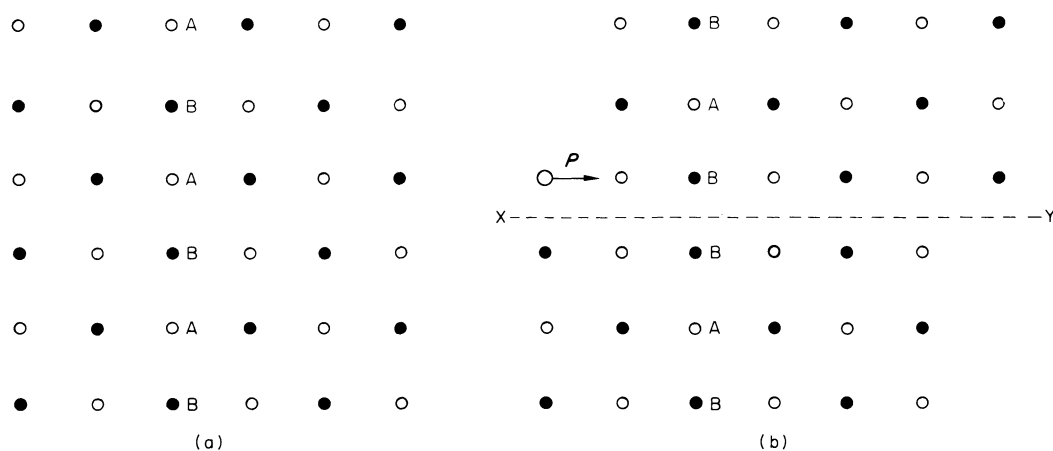


Figure 3.33 An array of A and B atoms (a) perfect, (b) sheared through P to create an antiphase domain boundary (APB) XY. (c) BF and (d) CDF images of growth APB in ordered Fe_3Al using a (100) superlattice reflection. (e) An image of the same area with a (200) fundamental reflection. (f) An APB in TiO_2 showing the influence on the image of a change in thickness along XY. (g) A closed loop of APB in TiO_2 . (h) Overlapping APB in TiO_2

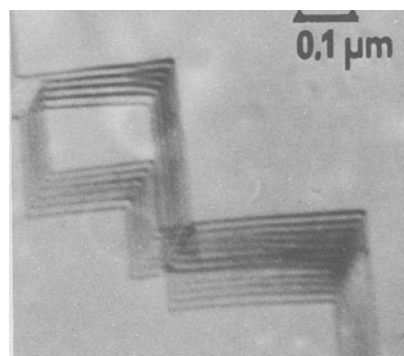
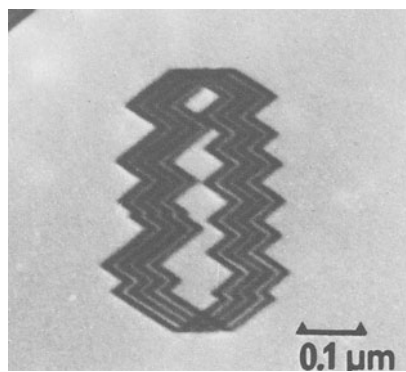
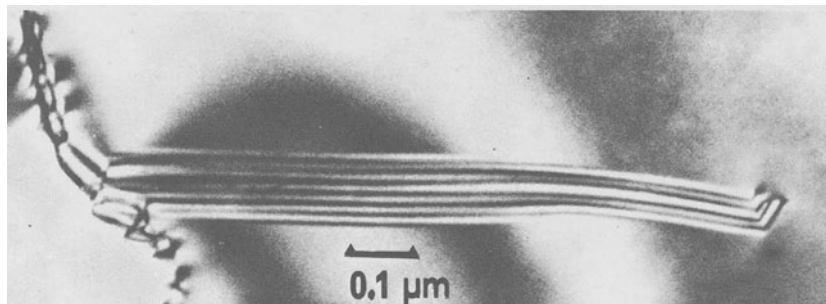
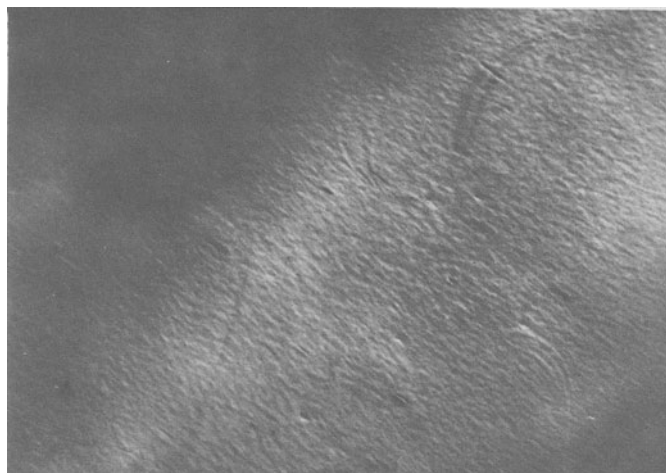
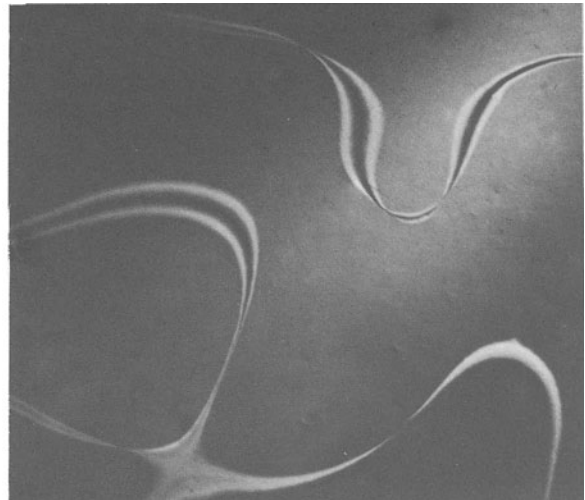
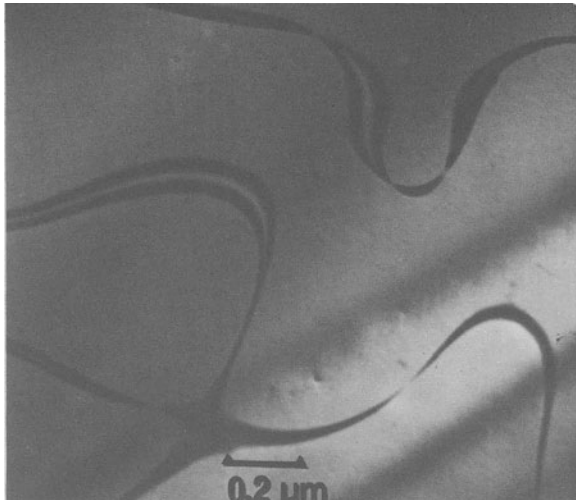


Figure 3.33 (continued)

Table 3.6 Values of $\alpha = 2\pi \mathbf{g} \cdot \mathbf{P}$ for APB $\mathbf{P} = \frac{1}{2}\langle 110 \rangle$ for $L1_2$ structure (Ni_3Al)

$\mathbf{g}$ α	$\langle 100 \rangle$ type S $\pm \pi$ or 0	$\langle 110 \rangle$ type S $\pm \pi$ or 0	$\langle 111 \rangle$ type F 0	$\langle 200 \rangle$ type F 0	$\langle 210 \rangle$ type S $\pm \pi$ or 0
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at $w = 0$, that is bright Kikuchi line running through associated diffraction spot.

3.7.1.2 Overlapping boundaries

Overlapping inclined APB have also been treated theoretically by van Landuyt *et al.* (1964). The most important conclusions are the following.

(1) *For intersecting domains when* (a) $w = 0$ both bright field and dark field images are symmetrical, show opposite contrast and the fringe spacing is $\frac{1}{2}t_g$. This has the consequence of continuous fringes for an overlapping intersection, see figure 3.33(g). When (b) $w \neq 0$ the fringes are still continuous but are modulated in intensity along their length.

(2) *For overlapping parallel domains when* (a) $w = 0$ the contrast is uniform dark or light depending upon the depth separation of the APB. When (b) $w \neq 0$ weak fringes parallel to the line of intersection occur, see figure 3.33(h).

3.7.2 Quantitative Information

Using the image properties outlined above for $w = 0$, APB (π boundaries) may be easily recognised, for example see Drum and Whelan (1965). The most important properties are (1)–(4) and (6) above, the simplest tests being (1) that they are only visible for superlattice reflections, and (2) symmetric and complementary images in BF, DF with superlattice reflections.

It is also possible to determine the direction of displacement of an APB using the $\mathbf{g} \cdot \mathbf{P} = 0$ invisibility criterion because α can be zero for the certain combinations of $\mathbf{g}$ and $\mathbf{P}$, see tables, appendix 13. The procedure is essentially the same as that for a $\mathbf{g} \cdot \mathbf{R}_F = 0$ fault vector determination, see section 3.6.2.2, that is obtain two different superlattice reflections under which the APB is invisible so that the zone axis of the reflecting planes defines $\mathbf{P}$.

3.8 δ Boundaries

These are defects separating two regions of a crystal with slightly different values of s or $\mathbf{g}$ for the same operative reflection. In such cases we are interested both in identifying these defects from the possible alternatives of α or π boundaries and in obtaining a detailed crystallographic description of the boundary. δ boundaries have been reported in a number of materials such as niobium suboxide (van Landuyt, 1964; van Landuyt *et al.*, 1966a), tantalum carbide (Villagrana and Thomas, 1965), nickel oxide (Remaut *et al.*, 1964), cobalt oxide (Gevers *et al.*, 1964c) and barium titanate (Gevers *et al.*, 1964c), cubic–hexagonal interface (Fourdeaux *et al.*, 1967) and coherent precipitate–matrix interfaces in nickel alloys (Ardell, 1967) and cobalt–titanium alloys (Thompson and Edington, 1971). A δ boundary generally arises as a result of an ordering process that differently distorts two parts of the crystal.

3.8.1 Image Characteristics

3.8.1.1 Single boundaries

The geometry of a typical inclined δ boundary in a thin foil is shown in figure 3.34(a) for the case of barium titanate in which the images arise from a difference in s . Crystals 1 and 2 are two marginally differently oriented regions of the specimen that have been produced as a result of the tetragonal distortion arising from the ordering process occurring below the ferroelectric transition temperature. In crystals 1 and 2 the tetragonal distortion has arisen along two different $\langle 100 \rangle$ directions of the original cubic lattice. Since the distortion between the two regions is small ($c/a = 1.0098$) the same planes can contribute to diffraction with different deviations, s_1, s_2 from the Bragg position; as shown in figure 3.34(b) in which the two reciprocal lattices are designated by triangles and circles.

Table 3.7 Structure factors F for simple ordered crystal structures containing atoms A and B

Superlattice type	F superlattice reflections	F fundamental reflections
B_2	$F = S(f_A - f_B)$, that is $h + k + l$ odd	$F = (f_A + f_B)$, that is $h + k + l$ even
$L2_1$ and $D0_3$	$F = 4(f_B - f_C)$, hkl all odd $F = 4(f_B + f_C - 2f_A)$, hkl all even, $(h + k + l)/2$ odd	$F = 4(f_B + f_C + 2f_A)$, hkl even, $(h + k + l)/2$ even
$L1_2$	$F = (f_A - f_B)$, hkl mixed	$F = (3f_A + f_B)$, hkl unmixed
$L1_0$	$F = 2(f_A - f_B)$, hkl mixed	$F = 2(f_A + f_B)$, hkl unmixed
$D0_{19}$	$\{10\bar{1}1\}$ $F = (3f_B^2 - 6f_B f_A + 3f_A^2)^{1/2}$ $\{11\bar{2}0\}$ $F = 2(f_B - f_A)$	$\{20\bar{2}0\}$ $F = (f_B^2 + 5f_B f_A + 7f_A^2)^{1/2}$ (0002) $F = (2f_B + 3f_A)$

S is the Bragg–Williams order parameter $S = (p - r)/(1 - r)$ if p is the probability that the right site for A atom is indeed filled by an A atom and r is the fraction of A atoms in the alloy.

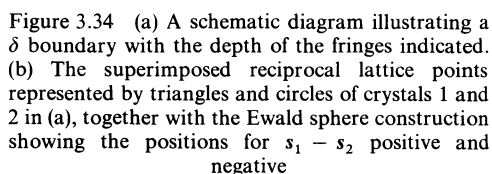


Figure 3.34 (a) A schematic diagram illustrating a δ boundary with the depth of the fringes indicated. (b) The superimposed reciprocal lattice points represented by triangles and circles of crystals 1 and 2 in (a), together with the Ewald sphere construction showing the positions for $s_1 - s_2$ positive and negative

$s_1 = -s_2$ and ξ_g the same for crystals 1 and 2 serves to delineate the most important features.

- (1) The image of the boundary consists of alternate black and white fringes running parallel to the intersection of the boundary with the surface, XY, shown schematically in figure 3.34(a) and in practice, figure 3.35(a).
- (2) The BF image is asymmetric (outer fringes show the opposite contrast) while the DF image is symmetric, see figure 3.35(a), (b).
- (3) The nature (that is bright or dark) of the fringe at the top of the foil depends only on the sign of $\Delta s = s_1 - s_2$ as shown in table 3.8.
- (4) The image contrast is only a function of Δs not the absolute value of s . Consequently, once two-beam conditions have been established the specimen can be slightly tilted to change the absolute value of s_1 or s_2 but Δs is unchanged and the contrast is unaffected.
- (5) The fringes reverse contrast if the opposite reflecting plane is used to produce an image. This arises because the sign of Δs changes.
- (6) In a wedge-shaped foil fringes are added in the centre of the bright extinction contours in both bright and dark field images. This effect is shown schematically in figure 3.36(a). Contrast is a maximum in the dark thickness fringes in both images. In practice, the thickness contours can change direction across the boundary if either the extinction distance is different in crystals I and II or $s_1 \neq s_2$ when the extinction distances are the same, see figure 3.36(b). In this latter case, the periodicity of the thickness fringes is

$$\begin{array}{cc} \text{crystal I} & \text{crystal II} \\ \xi_{1g} = \frac{\xi_g}{\{1 + (s_1 \xi_g)^2\}^{1/2}} & \xi_{2g} = \frac{\xi_g}{\{1 + (s_2 \xi_g)^2\}^{1/2}} \end{array} \quad (3.9)$$

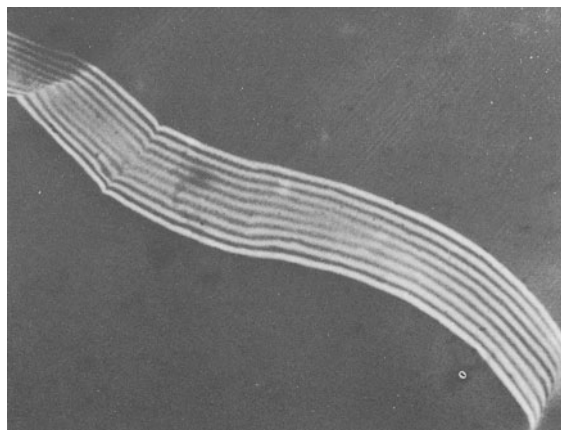


Figure 3.35 (a) BF and (b) CDF images of an inclined δ boundary in vanadium carbide

* Note that fringes will also arise if $\xi_{g_1} \neq \xi_{g_2}$, as well as for $s_1 \neq s_2$ because $\delta = w_1 - w_2$. If there is a significant contribution from a difference in ξ_g to the image, then the fringes should change drastically on tilting the specimen for $+s$ to $-s$. This effect is not included in the theory produced by Gevers *et al.* (1964b).

Table 3.8 The nature of the fringe at the top of the foil for δ boundaries

	Bright field fringe at foil surface		Dark field fringe at foil surface	
	Top	Bottom	Top	Bottom
$\Delta s > 0$	B	D	B	B
$\Delta s < 0$	D	B	D	D

B, bright fringe; D, dark fringe.

where ξ_g is the extinction distance of both crystals. In either case the periodicity of the thickness fringes in the region of the boundary is the average of periods ξ_{1g} and ξ_{2g} , that is the contours simply join up. However, in this case the fringes still add in the same way as shown in figure 3.36(b).

(7) Fringe spacing can be different at the top and bottom of the foil. This arises because the values of the effective extinction distance ξ_g , that is the fringe periodicity is determined at the top of the foil by crystal I and at the bottom by crystal II. The values of ξ_{g1} and ξ_{g2} given by equations (3.5) will be different depending on the values of s_1 and s_2 .

(8) The centre of the fringe pattern is not necessarily the geometrical centre of the specimen. For a specimen diffracting in an asymmetric case, that is $g_1 \neq g_2$ the centre of the fringe pattern is an equal number of fringes from each surface, that is $n\xi_{g1} = n\xi_{g2}$. This can be seen most clearly when the boundary runs through a region of increasing thickness. Fringes add from the 'fringe centre' not geometric centre.

3.8.1.2 Overlapping boundaries

The case of two overlapping δ boundaries* in thick crystals has been discussed in detail by Remaut *et al.* (1965, 1966). The geometrical case considered by these workers is shown in figure 3.37(a). The main conclusions for their theoretical treatment can be separated on the basis of the geometry of the situation.

(a) *Both foil thickness and separation large.* In practice this corresponds to a situation with z_2 (figure 3.37(a)) large enough for large regions of non-overlap of domain boundaries to occur at the top and bottom of the foil but with a region of overlap in the centre. Anomalous absorption is included in the calculation and the important fringe image characteristics are as follows.

(1) The nature of the first and last fringes in the region of overlap depends only on the sign of δ . These fringes are bright if δ is positive and

dark if δ is negative, see figure 3.37(b) at X and Y, δ positive.

(2) The nature of the outside fringes of the non-overlap portion of the image is independent of z_2 , that is does not change for wedge-shaped domain 2, see figure 3.37(b) at Y.

(3) The BF image is symmetric but the CDF image is asymmetric, see figure 3.37(b), (c) at X and Y.

(4) In BF the *mean intensity* of the overlapping part compared with the non-overlapping part is

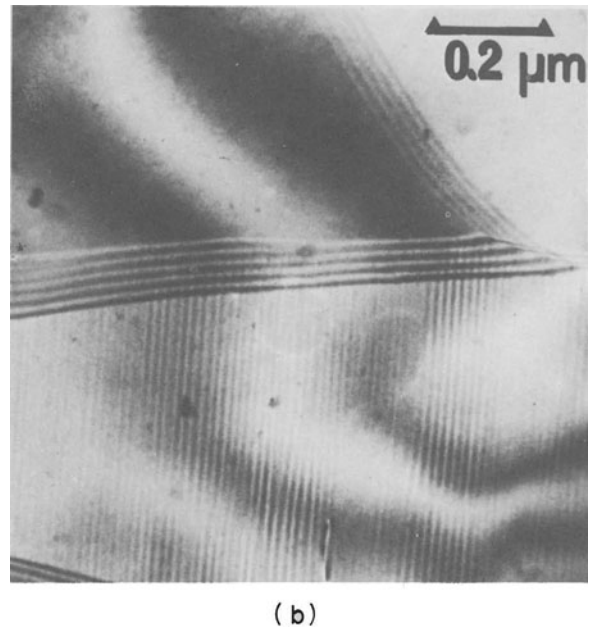
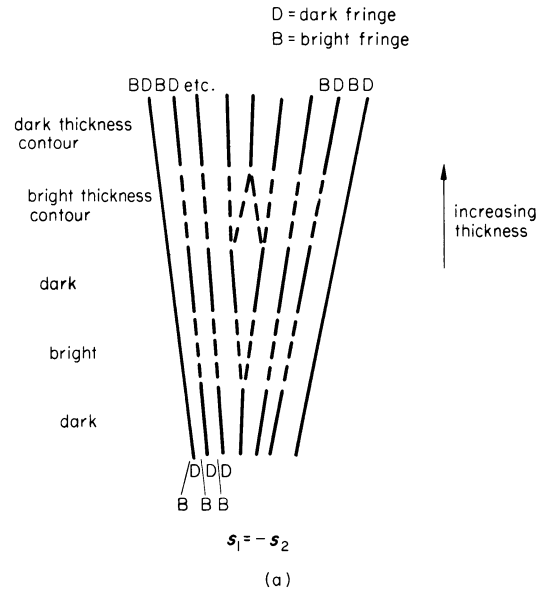


Figure 3.36 (a) A schematic diagram illustrating the influence of thickness changes on a δ boundary image and (b) a typical BF image in vanadium carbide

* Remaut *et al.* (1967) have considered in detail the special case of three overlapping boundaries in BaTiO_3 . The outer boundaries were 90° boundaries whereas the centre boundary was an 180° boundary. Wavy fringes are observed experimentally and predicted theoretically.

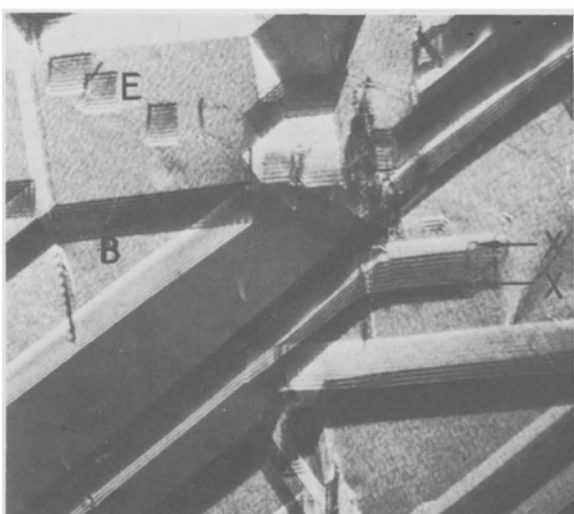
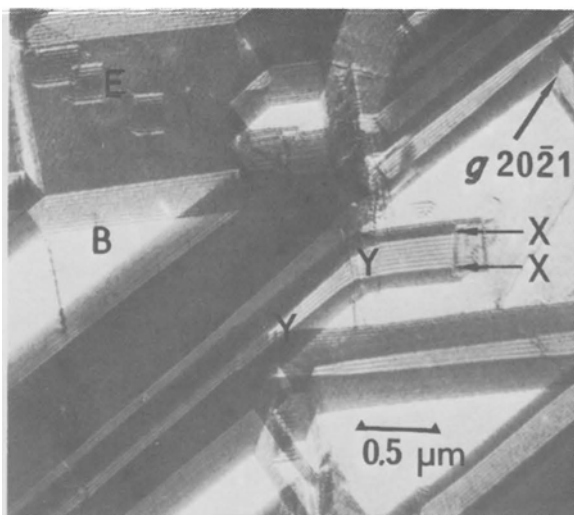
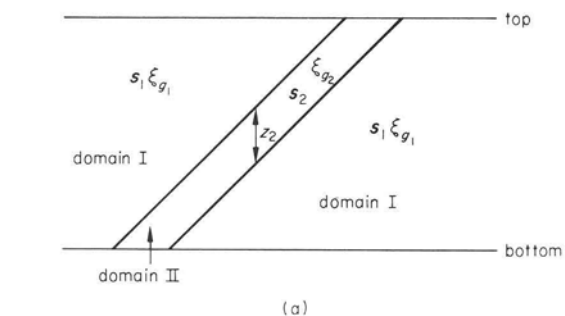


Figure 3.37 (a) A schematic diagram illustrating overlapping δ boundaries. (b) BF and (c) CDF images of δ boundaries in hydrogen contaminated Ti_3Sn [Courtesy of Metallurgical Transactions]

stronger if δ is positive and weaker if δ is negative, see figure 3.37(b) at X, δ positive.

(b) *Thickness large but domain walls closely spaced.* The calculations assume no absorption in crystal II, figure 3.37(a), that is $z_2 < 0.1\xi_g$. In effect, this corresponds to the case when there are no fringes in the non-overlapping part. The fringe patterns are similar to those of a stacking fault.

(1) Bright field images are symmetrical, whereas dark field images are asymmetrical, see E, figure 3.37. Bright and dark field images have the same contrast at the top and opposite contrast at the bottom.

(2) The fringe contrast is damped in the centre of the foil, figure 3.37(c).

(3) The depth periodicity of the fringes is $\sim \xi_g$ in the matrix.

(4) For the conditions described above the nature of the outer fringes of the bright field image depends on the sign of δ . If δ is positive, outer fringes are dark in BF whereas if δ is negative they are bright. Thus δ is negative in figure 3.37 at E.

(5) If $w_{(\text{matrix})}$ is positive, the contrast is strongest at the top of the foil but, if $w_{(\text{matrix})}$ is negative, it is strongest at the bottom of the foil.

(6) The contrast of the outer fringes relative to background depends on z_2 , being weak at z_2 small or $\approx \frac{1}{2}\xi_g$ and strong when $z_2 \approx \frac{1}{2}\xi_g$.

(c) *Foil thickness small, domain walls closely spaced.* The separation of the domain boundaries in this case is $0-0.3\xi_g$ and the foil thickness is $2-3\xi_g$. For material with small absorption lengths, the images are similar to those of stacking faults. However, for the large absorption lengths complex images can occur with ill-defined outer fringes and fringe spacings of ξ_g or $\frac{1}{2}\xi_g$. For details of fringe profiles for particular experimental cases the reader is referred to Remaut *et al.* (1966).

(d) *Variations in the spacing of domain walls—domain 2 wedge shaped.* In this case z_2 increases in magnitude along the length of domain 2 and some coherency at the boundary would be lost. If this coherency loss is ignored, the following conclusions may be drawn.

(1) The number of fringes in the overlap region will vary as z_2 increases even if the foil thickness does not change. The fringes are parallel to the nearest foil surface, see figure 3.37(b) at Y, the fringe pattern is symmetric and new fringes will be created in the centre by forking of fringes like a stacking fault, see figure 3.37(b) at Y, giving rise to pseudo-thickness fringes. Forking occurs in regions of poor contrast.

(2) The fringes in the non-overlap parts are not parallel, see figure 3.37(b), (c) at Y.

(3) The fringes at the edge of the overlapping region are not parallel, see figure 3.37(b), (c) at Y.

(4) The fringes at the right side of the overlap region are parallel to those in the left non-overlap region and vice versa, see figure 3.37(b), (c) at Y.

3.8.2 Quantitative Information

Single boundaries can be readily recognised from any of the characteristics outlined in section 3.8.1.1. The simplest method is to compare BF and DF images which are asymmetric and symmetric respectively, that is opposite of stacking fault contrast.

It is also possible to obtain detailed information about the structure of the material in some cases from a study of fringe geometry. For example, it has been shown that a knowledge of the diffraction vector and the nature of the top and bottom fringes enables the geometry of the crystals on either side of the boundary to be deduced. In the case of barium titanate the direction of the tetragonal axes can be deduced (Gevers *et al.*, 1964c). For both nickel and cobaltous oxide (Gevers *et al.*, 1964c) the direction of lattice contraction due to ferromagnetic ordering can be determined. In niobium, the distortion resulting from interstitial ordering has been shown to vary continuously within a given domain. Fourdeaux *et al.* (1967) were able to show that δ fringes arise at the f.c.c.-c.p.h. interface in Co-Cr alloys because the hexagonal phase does not have the exact c/a ratio for close packing. The sense of deviation of c/a from exactly 1.63 could be determined from the form of the images. Finally, the matrix distortion around γ' particles in a nickel-aluminium alloy can be estimated from a study of δ fringes produced at the precipitate-matrix interface (Ardell, 1967). For details of these experiments the reader is referred to the original papers.

3.9 Mixed α - δ Boundaries

Such boundaries are most likely to occur in special cases such as precipitate-matrix interfaces. The α fringes might arise from a phase interference from the two parts of the crystal whereas the δ component could arise from a slight misorientation of the reflecting planes together with a possible difference in ξ_g and ζ'_g . Such a case has been reported by van Landuyt *et al.* (1966a) in niobium and is described in detail in section 4.2.1.2.

3.9.1 Image Characteristics

The contrast from mixed α - δ boundaries has been computed by Gevers *et al.* (1965) and it was shown that the image characteristics are intermediate between those of pure α and pure δ fringes the ratio depending upon the relative strength of the two components. The general features of the images are as follows.

(1) As a small δ component is added to an α image the nature of the outer fringes in the bright field image remains the same but their intensity is

reduced. In the dark field image, the outer fringes remain opposite in contrast.

(2) As the δ component becomes greater deviations from the symmetry properties become more marked so that the outer fringes of the bright field images may become opposite in nature with one of the fringes very weak. The dark field image may become symmetric bounded by fringes of the same type. Thus in the bright field case ignoring the weak fringe at the top surface, the image has δ boundary characteristics.

3.9.2 Quantitative Information

No detailed quantitative information can be obtained from α - δ boundaries. Indeed it may be extremely difficult even to identify them. Detailed fringe profiles may be found in the paper by Gevers *et al.* (1965) and a case of α - δ fringes is shown in figure 4.2. It is clear that the faint fringes predicted by these workers must be the main identifying characteristic together with the use of reflections for which $\alpha = 0$ and the pure δ component remains.

3.10 Interfaces

For convenience, interfaces have been separated into those separating the same phase (for example grain boundaries) and different phases (for example precipitate-matrix), which are considered separately.

3.10.1 Image Characteristics of Interfaces Between Like Crystal Structures

Interest lies not only in the general features of the image, but also in the interpretation of the fine structure frequently observed in electron microscope images of these interfaces.

Two situations may be considered, namely low-angle and high-angle boundaries.

3.10.1.1 Low-angle boundaries

When the misorientation is very small, $\lesssim 1^\circ$, for example in crept specimens, the dislocations making up the inclined boundary may be readily distinguishable as a network under normal two-beam dynamical conditions, see figure 3.38(a). The same reflection is operative in both subgrains but there is a slight difference in the value of s , compare the position of the Kikuchi lines AA in the diffraction patterns in figure 3.38(b), (c). For higher misorientations $\sim 2^\circ$ a fringe image such as that shown in figure 3.39(a) may occur. Under these conditions weak beam images, section 3.17, may be used to distinguish the details of the network, see figure 3.39(b).

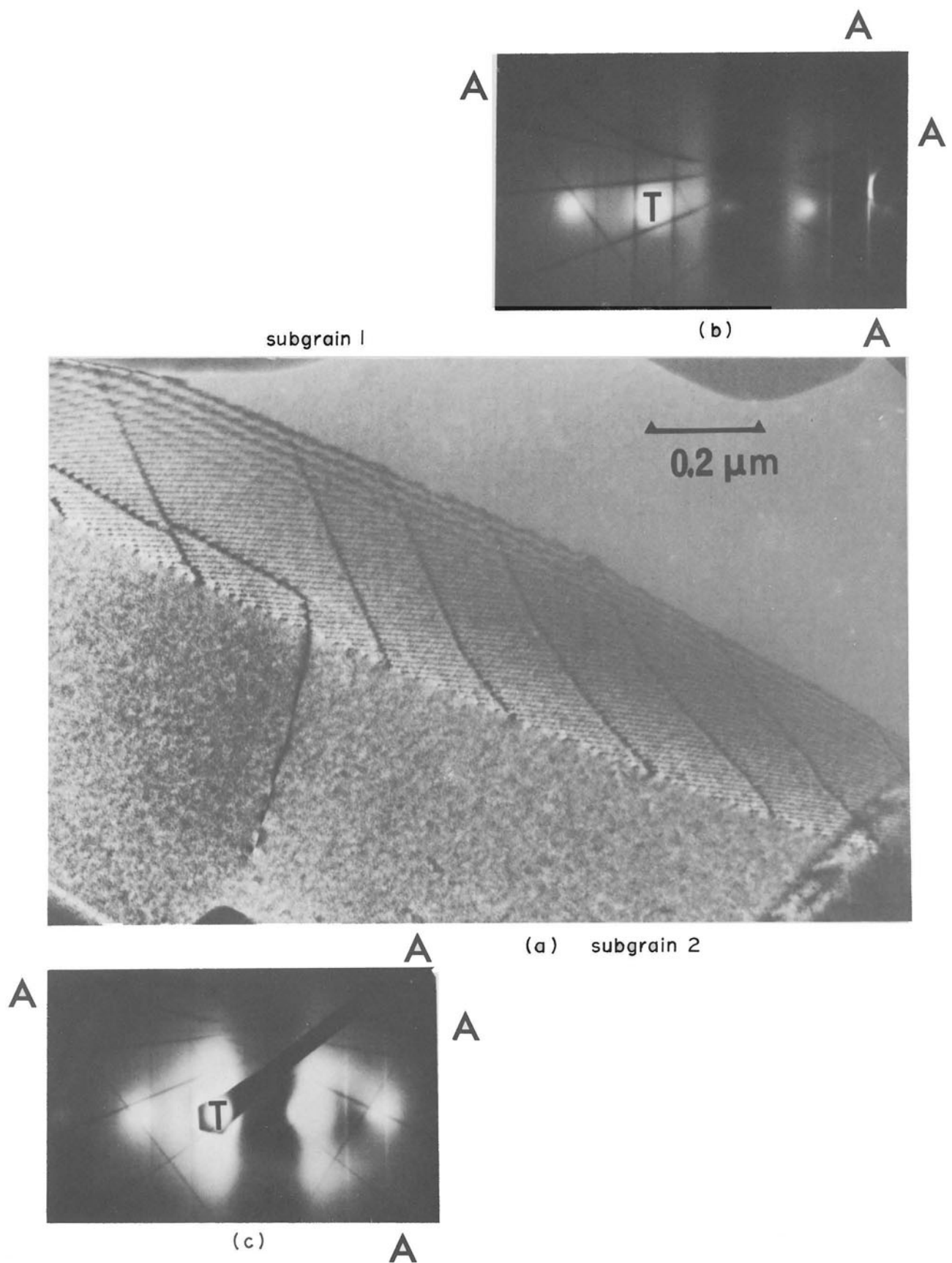
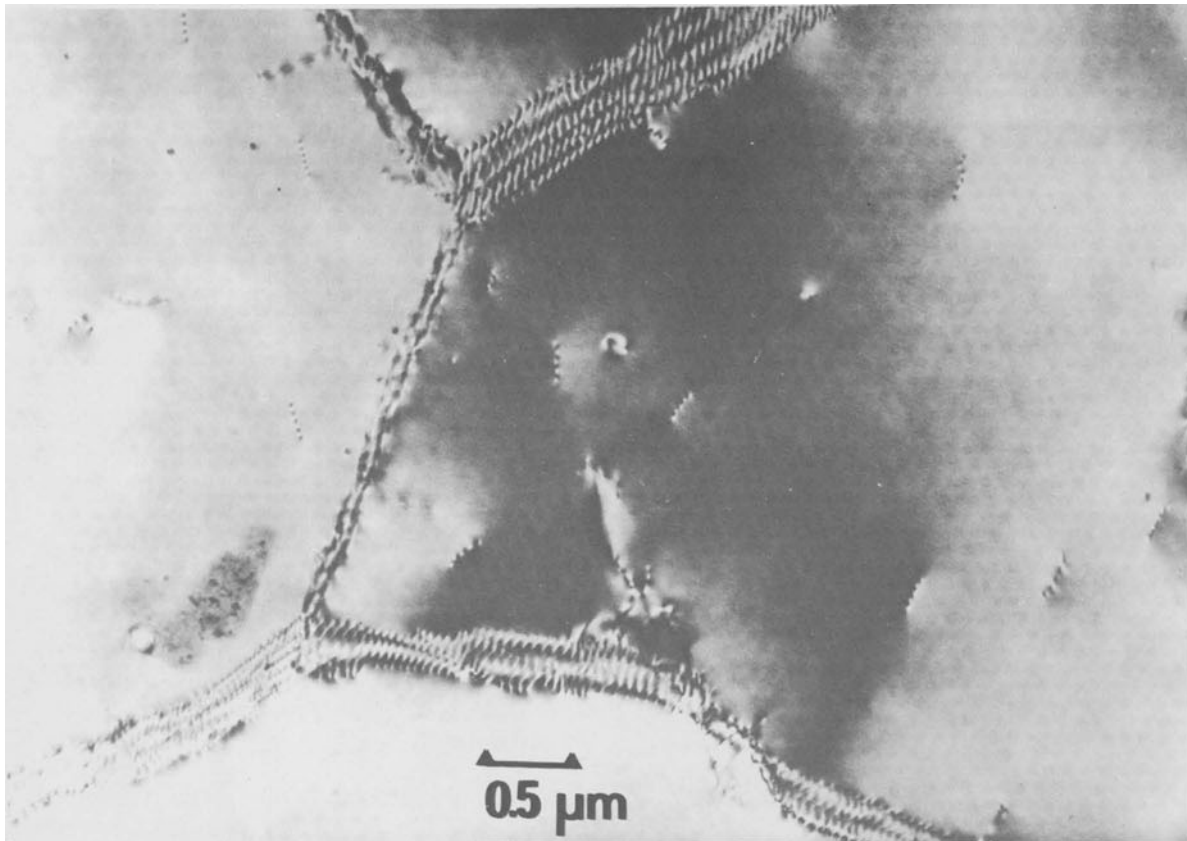
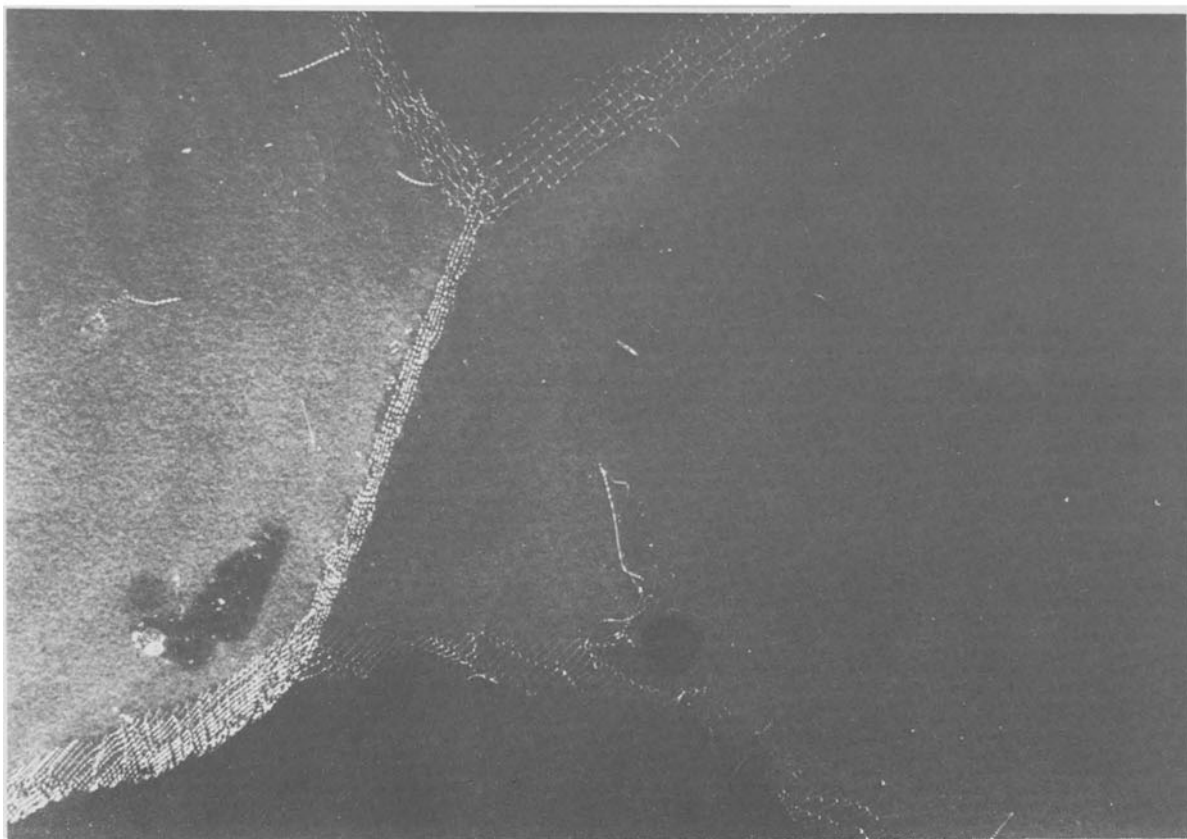


Figure 3.38 (a) A low-angle boundary in aluminium grains in a superplastically deformed Al–40 wt% Zn alloy. (b), (c) SADP from subgrains 1 and 2 respectively [Courtesy of A. Samuelsson]



(a)



(b)

Figure 3.39 (a) A BF image of a low-angle boundary array in aluminium. (b) A weak beam CDF image of the same area [From Nordlander and Thölen (1973), courtesy of *Journal of Microscopy*]

3.10.1.2 High-angle boundaries

The most common case observed experimentally is shown schematically in figure 3.40(a) in which an inclined grain boundary is imaged with two-beam dynamical conditions in one grain and kinematical conditions in the other, that is s very large on all reflections. Such a boundary is visible in BF as fringes with the same spacing as thickness fringes, that is $\xi'_g = \xi_g/(1 + w^2)^{1/2}$, see figure 3.40(b). Note that the thickness fringes join the grain boundary fringes. If dynamical conditions exist in both grains complex images can occur, see figure 3.40(c) particularly if ξ_g is different for the two operative reflections.

A number of different fine structure effects have been reported to be superimposed on this type of image. Detailed interpretation of the contrast effects in these images in terms of grain boundary structure is not complete at present. Consequently, considerable care should be taken in drawing conclusions from them. Nevertheless, examples of the different types of 'fine structure' image are described below.

(1) *Steps in the boundary.* This effect has been reported for grain boundaries by Gleiter (1969) and for coherent twin boundaries, see figure 3.41(a), which illustrates a typical observation of a coherent twin boundary in an austenitic stainless steel. Note that the boundary fringes are all equally displaced which implies that there is a small step in the interface perhaps, where the boundary lies on a different coherent twin plane. The step height may be calculated from the formula

$$h = m \sin \beta \sin \phi \quad (3.10)$$

See Gleiter (1969) where m is the fringe displacement, ϕ is the angle between the fringe and the grain boundary line, β is the angle between the boundary and the foil surface, see figure 3.40(a).

Steps that are relatively large $\gtrsim 1000$ Å long may be recognised by their shape and their planes determined by trace analysis using the approach outlined in section 2.14.1. Medium-sized steps 50–500 Å may be identified by fringe displacement particularly under two-beam imaging conditions. However, steps < 50 Å high are very difficult to interpret except in well-defined cases such as coherent twin boundaries.

(2) *Absorbed lattice dislocations.* In material deformed at low and ambient temperatures very straight strong line contrast effects are often observed in grain boundaries (see Ishida and Henderson-Brown, 1967; Ishida *et al.*, 1969; Lin and McLean, 1968; Barry and Mahajan, 1971). For small strains these often lie at the head of the active slip planes, see figure 3.41(b), and their

density increases with increasing strain. These appear to be lattice dislocations that have become lodged in, or very near to, the boundary.

Similar images but not associated with slip bands, have been reported after grain boundary sliding at elevated temperatures (Buzzichelli and Mascanzoni, 1971; Kegg *et al.*, 1973).

(3) *Structure lines.* These defects, often observed in recrystallised material, are visible as faint narrow closely spaced images, see figure 3.41(c). They are observed with two-beam dynamical conditions and have been studied extensively by Gevers *et al.* (1966), Schober (1970), Schober and Balluffi (1970, 1971) and Schober and Warrington (1971), Levy (1969) and Ishida *et al.* (1969). The details of interpretation of these defects is not clear at present, although they have been attributed to structural effects, for example Schober and Balluffi (1970, 1971), Schober and Warrington (1971) and Pumphrey (1972). The most important feature of these images is the change in spacing as the boundary plane changes, for example in curved boundaries.

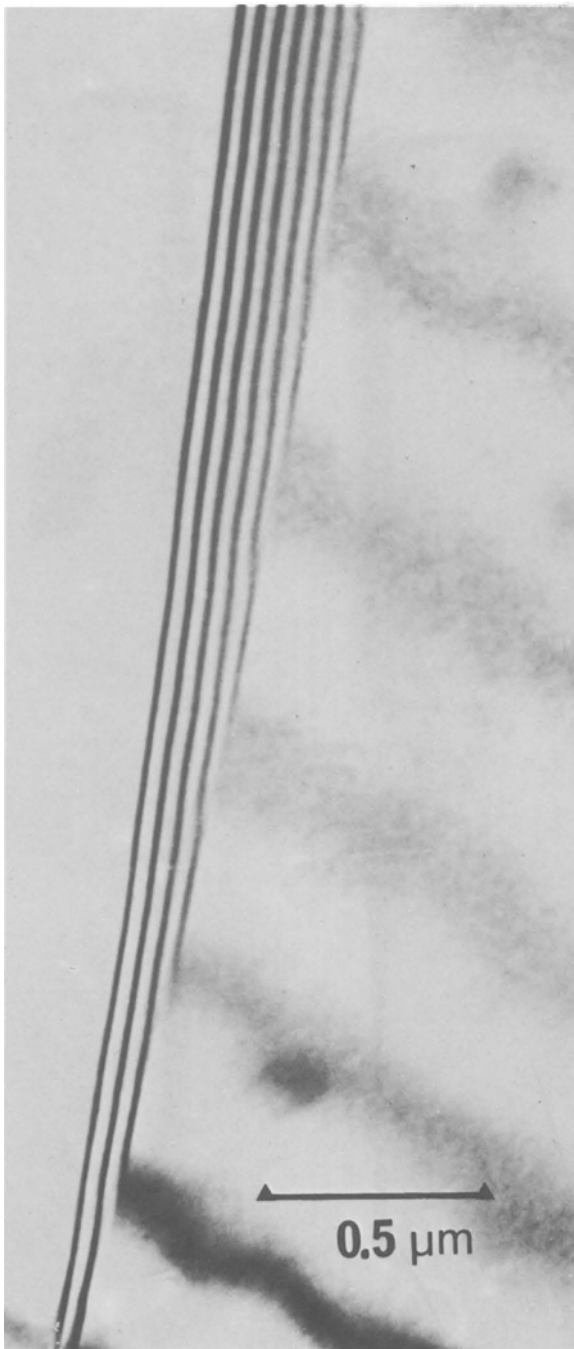
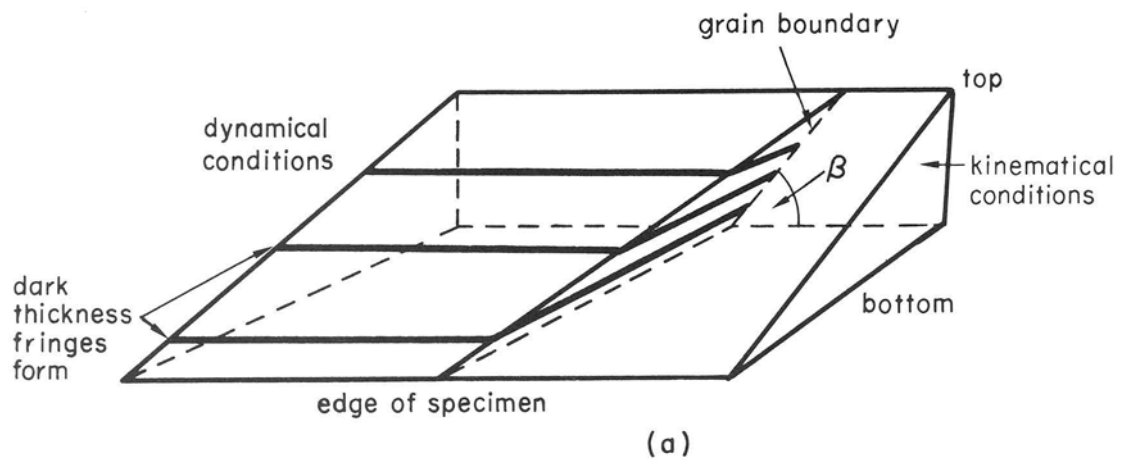
(4) *Moiré patterns.* Moiré patterns described in detail in section 3.21 can arise at grain boundaries if, for a particular reflection, there is suitable double diffraction (see section 2.16.1). An example is shown in figure 3.41(d) which is the same boundary as in figure 3.41(c) but imaged with a different reflection. The most important characteristics that enable Moiré patterns to be identified are the following.

- (i) The fringe spacing does not change when the boundary changes plane.
- (ii) The direction of the fringes depends on g .

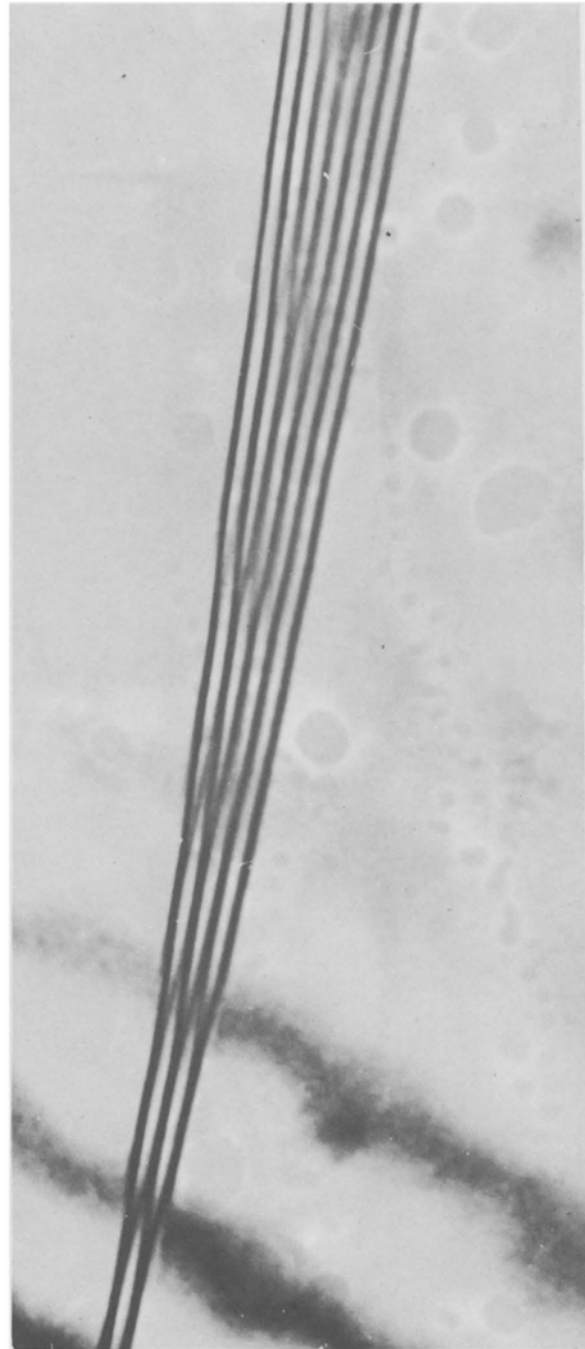
3.10.1.3 Twin boundaries

This is a particularly simple geometrical case and the image is usually obtained with dynamical two-beam conditions in the matrix or the twin and kinematical conditions in the other crystal. Under these conditions, the image of an inclined boundary simply consists of conventional thickness fringes, see figure 3.41(a), and is similar to that of a grain boundary.

Images of fine structure in coherent twin boundaries have been reported by a number of workers. Pumphrey and Bowkett (1971) have studied the Burgers vectors of dislocations in twin boundaries using invisibility criteria for common reflections when the fringes disappeared, see figure 3.42. Under these conditions there are no Moiré fringes because the reflecting plane is exactly continuous and dislocation invisibility can be readily judged, see dislocations B, figure 3.42(b), (c).



(b)



(c)

Figure 3.40 (a) A schematic diagram illustrating an inclined grain boundary in a wedge-shaped region of a thin specimen, (b) a typical grain boundary image with dynamical conditions in one grain, and (c) the same boundary with dynamical conditions operative in both grains

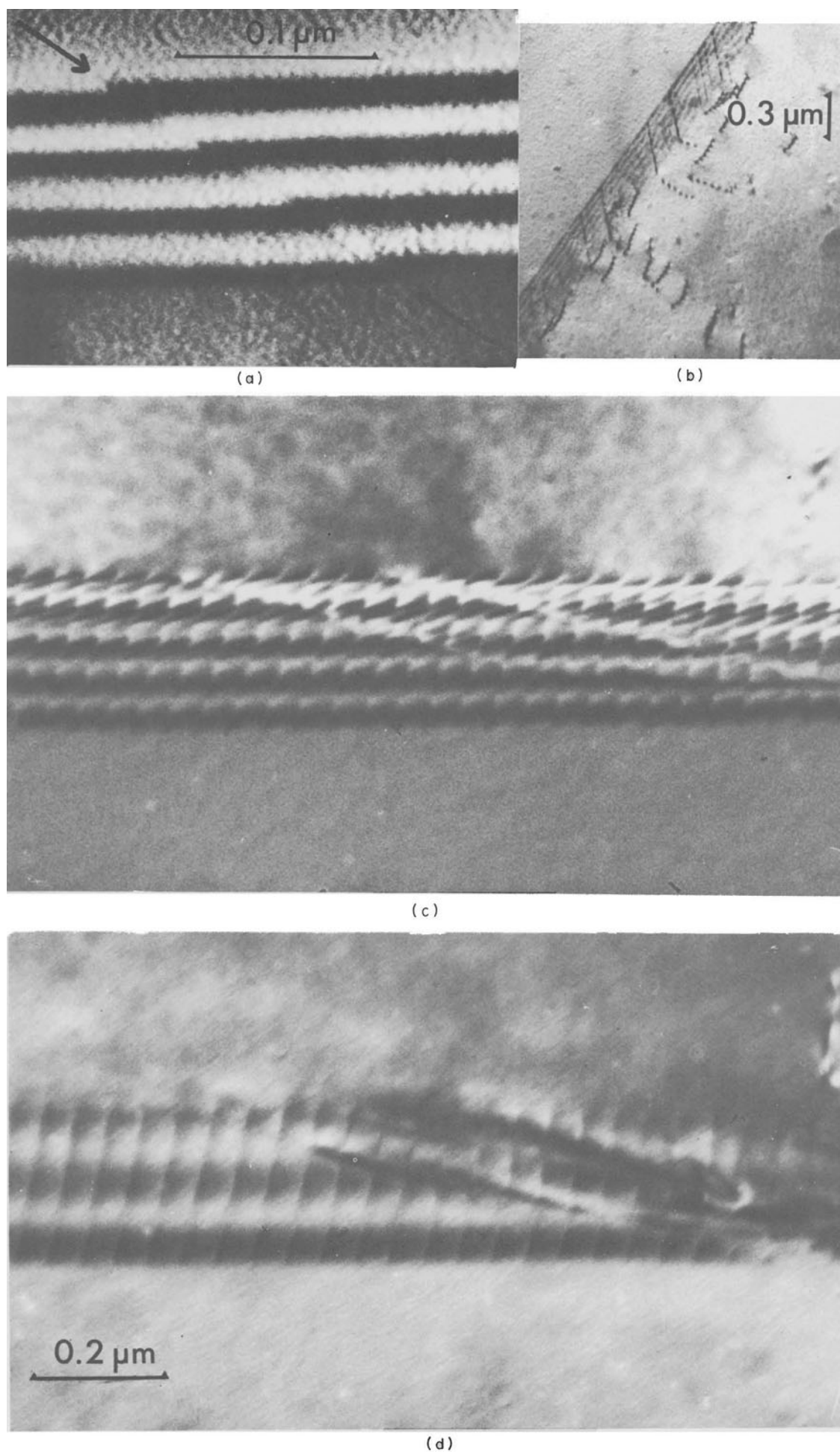


Figure 3.41 (a) A step (arrowed) in a twin boundary in austenitic stainless steel. (b) A grain boundary in lightly deformed austenitic stainless steel. (c), (d) Structure lines and Moiré fringes on the same boundary in austenitic stainless steel imaged under different conditions [Courtesy of P. H. Pumphrey]

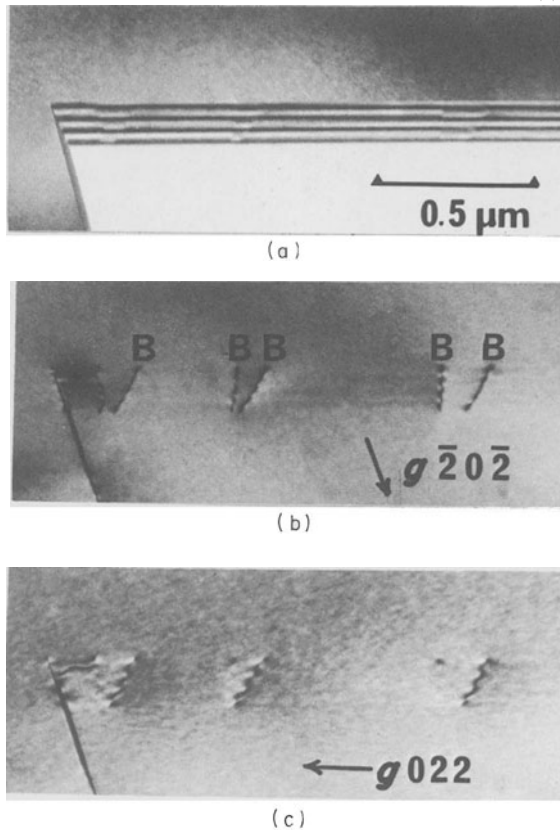


Figure 3.42 A twin in austenitic stainless steel imaged under different reflections [From Pumphrey and Bowkett (1971), courtesy of *The Philosophical Magazine*]

Overlapping fringe effects have been reported for partial dislocations in coherent twin boundaries in f.c.c. (Whelan, 1959) and b.c.c. (Hull, 1962) materials. The Burgers vector of the twinning partial may be determined from the fringe shifts at each partial. For partials $b_p = \frac{1}{6}\langle 112 \rangle$ f.c.c. or $b_p = \frac{1}{6}\langle 111 \rangle$ b.c.c. fringe contrast shifts by half a fringe separation at the partial due to the additional phase angle $\alpha = n \times 2\pi/3$, when n faults overlap, an effect similar to that in figure 3.31.

3.10.2 Quantitative Information on Interfaces Between Like Crystal Structures

3.10.2.1 Low-angle boundaries

When the misorientation is low $\lesssim 1^\circ$, it is possible to perform a Burgers vector analysis on the various segments of networks and on slip dislocations that have interacted with the network (Carrington *et al.*, 1960). The same general approach may be used as was outlined for perfect dislocations in section 3.4.3, but the same difficulties will be encountered and image computation may be required. A further problem arises because the network segment under discussion only extends a limited distance through the foil. Thus in situations where an inclined

dislocation running from top to bottom of the foil would produce a dotted image, apparent (and incorrect) invisibility may occur for dislocation segments parallel to the surface and at a depth intermediate between dots on an inclined dislocation. To avoid such problems several segments at different depths in the crystal should be observed simultaneously in networks where the network symmetry enables identification of those segments likely to have the same Burgers vector.

The remaining quantitative information that may be obtained is the network plane, from trace analysis, see section 2.14.1, and the misorientation from SADP, see section 2.11.2.

3.10.2.2 High-angle boundaries

Grain boundaries may be readily recognised from the high angle of misorientation detectable in SADP taken from either side of the boundary. Dynamical two-beam conditions may be produced in one grain and the junction of thickness fringes with the boundary fringes checked. Alternatively the general morphology, for example curvature or triple points, may be used for identification.

The misorientation across grain boundaries may be measured using SADP, see section 2.11.2. Furthermore, the line direction of defects in the boundary may be obtained using the approach outlined for dislocations in section 2.14.2. The Burgers vector of defects such as dislocations has been attempted (Fisher *et al.*, 1966; Tunstall, 1970; Dingley, 1972) using computer matching techniques. However, these experiments are extremely difficult because accurate descriptions of the strain field of the defect are not available and the interpretation can be uncertain. In general, the Burgers vector studies of this type should be avoided until much more computation has been performed.* Structure lines and Moiré patterns may be identified in terms of their behaviour, when the boundary plane changes, and absorbed dislocations if they lie at the head of a pile-up.

3.10.2.3 Twin boundaries

Twins may be identified from SADP taken across the twin boundaries, see section 2.12.2. However, little quantitative information can be determined from the form of the image. Twin plane, line direction of boundary defects and the Burgers vector of dislocations may be obtained using effectively the same approaches as for grain boundaries. In the case of twins, however, the Burgers vector of defects in the interface may be analysed using common reflecting planes (Pumphrey and Bowkett, 1971).

* See Note added in proof, p. 96.

3.10.3 Image Characteristics of Precipitate–Matrix Interfaces

Coherent, semi-coherent or incoherent precipitate–matrix interfaces can occur in precipitation hardening systems. In general fringe images from these interfaces may be observed if the particle is large enough. The type of contrast responsible for the image may be determined using the approach outlined in sections 3.6–3.9, and the interface plane may be determined from trace analysis, see section 2.14.1. In addition the defects responsible for the fine structure in images of interfaces are of interest. These defects are discussed below.

3.10.3.1 Coherent interfaces

δ fringe contrast can arise (Ardell, 1967) if there is very small misfit between precipitate and matrix, see section 3.8.1. Alternatively, displacement α fringes may arise if a greater mismatch occurs between precipitate and matrix, see figure 3.32 and section 3.6.3. Again Moiré fringes may arise if the correct double diffraction conditions occur, and these may be identified using the rules outlined in section 3.21.7. For large particles (diameter $\approx \xi_g$) displacement fringes can give rise to contrast at curved interfaces that lie perpendicular to the electron beam if the combination of depth and curvature is correct. Fine structure images in coherent interfaces have only been reported after deformation when coherency is lost. Thus interface dislocations occur in γ' strengthened Ni-base alloys (Merrick and Nicholson, 1962).

3.10.3.2 Partially coherent interfaces

If the foil is oriented such that the matrix is under two-beam dynamical diffraction conditions, while the precipitate is not, thickness fringes occur, like those described in section 3.2.3.1 for grain boundaries, and with the same periodicity as thickness fringes $\xi_{g\text{eff}} = \xi_g(1 + w^2)^{1/2}$. Displacement α fringe contrast described in section 3.6.3 may also occur with fringe shifts at dislocations which are present to alleviate the mismatch.

In this case the sense of the strain field $\pm R_n$ may be determined as described in section 3.6.4 provided that a section of the interface without dislocations between top and bottom of the foil is used, that overlapping precipitates are avoided and that the precipitate does not contribute to the diffraction process, that is remains thin. Moiré patterns may also occur under the correct diffraction conditions but the relationship between the details of the Moiré pattern structure and that of the interface has not been quantitatively studied.

Some partially coherent interfaces contain dislocations. These may be imaged, where Moiré patterns are absent. Detailed studies have been reported for some simple cases, for example θ' in aluminium–copper alloys (Pollard and Nutting, 1965; Laird and Aaronson, 1968; Weatherly and Nicholson, 1968), Ag_2Al in aluminium–silver alloys (Laird and Aaronson, 1967), Ni_3Ti in nickel–chromium–titanium alloys (Merrick and Nicholson, 1962), cobalt precipitates in copper (Phillips, 1966), S precipitates in aluminium–copper–magnesium alloys, β' precipitates in aluminium–magnesium–silicon alloys and γ' in nickel–chromium–titanium–aluminium alloys (Weatherley and Nicholson, 1968).

Weatherley and Nicholson have considered the experimental conditions under which interfacial dislocations may most easily be observed for the simple case of an interface lying parallel to the foil surface and without specimen tilt, see figure 3.43(a). In this configuration there are neither displacements, δ , nor Moiré fringes. They concluded that the conditions for observation of interfacial dislocations are more stringent than for ordinary dislocations, because of the thinness of the particles t_p and the large extinction distance ξ_g associated with many precipitate reflections. Often t_p/ξ_g will be small $\ll 1$ and Holland *et al.* (1965) have shown that images are very weak if t/ξ_g is $\lesssim 0.25$. Thus the contribution of the precipitate to a given image may be small even if $g \cdot b = 0$.

In order to form good BF* images of interfacial dislocations it is necessary to use the following.

(1) Two-beam conditions with precipitate and matrix reflections close together in reciprocal space such that w is almost the same in each crystal. If different values of w are selected in matrix and precipitate the background intensity may obliterate dislocation contrast.

(2) Reflections in both precipitate and matrix with short extinction distances.

(3) Dynamical images, $w \approx 0.3$ in both precipitate and matrix. Typical images are shown in figure 3.43, and are clearly similar to those of conventional dislocations.

Interface ledges $\sim 2 \text{ Å}$ high have also been studied in some detail in Al–Ag containing γ' Ag_2Al plates by Laird and Aaronson (1967). In the case of θ' particles in Al–Cu alloys the ledges are larger $15\text{--}65 \text{ Å}$ high (Weatherly and Sargent, 1970; Laird and Aaronson, 1968). The contrast arising from such ledges has been studied in detail by Weatherly and Sargent (1970) using the geometry shown in figure 3.44(a). The ledge is treated as a dislocation $b = \frac{1}{2}[001]\theta'$ and the following features were characterised for precipitates $\sim 150 \text{ Å}$ thick.

* If a DF image is formed using the precipitate reflection the t_p/ξ_g effect will produce weak images.

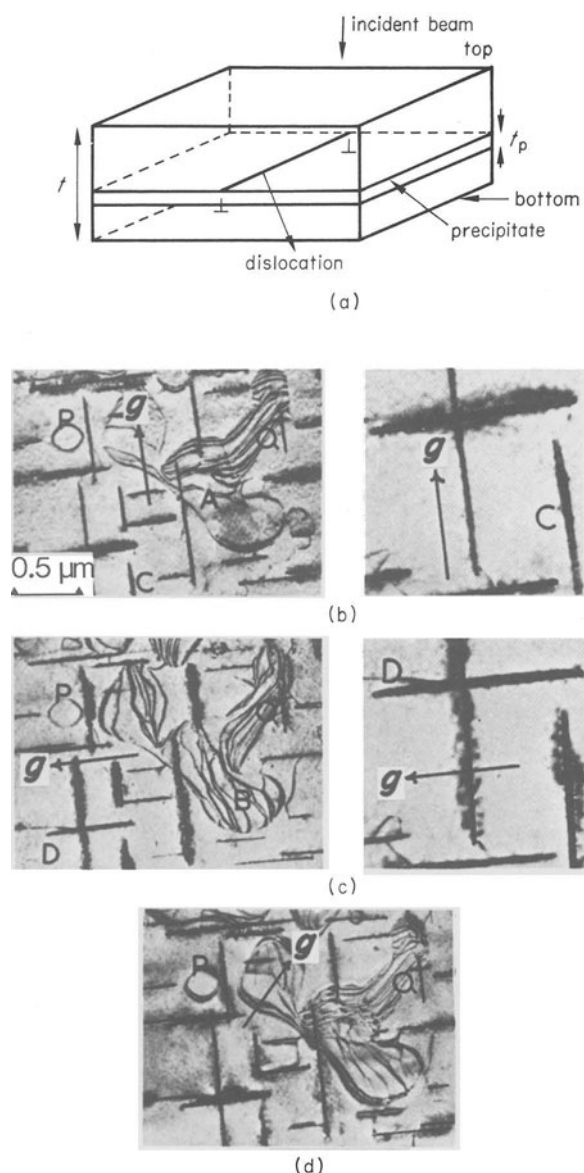


Figure 3.43 (a) A schematic diagram illustrating the geometry of a thin precipitate lying parallel to the foil surface and containing a dislocation in the interface. (b)–(d) BF images of interfacial dislocations ABCD at θ' particles in Al–4 wt % Cu aged 1 day at 275 °C. P represents dislocations around the periphery of the plates and Q complex dislocations arising from the agglomeration of two plates. (b) g [200] dislocations A visible with enlargement showing dislocations C. (c) g [020] dislocations B visible with enlargement showing dislocations D. (d) g [220] all dislocations visible [From Weatherly and Nicholson (1968), courtesy of *The Philosophical Magazine*]

(4) For precipitate DF the images are often wide (~ 300 Å), see figure 3.44(b).

(5) In two-beam precipitate DF with no significant excitation of a matrix reflection, that is only the precipitate diffracts, continuous ledges in the interface (like circular dislocation loops) have a line of no contrast approximately perpendicular to g . On one side of the line of no contrast the image of the ledge on a positive print is a black line whereas

in the other it is a white line, see figure 3.44(b), provided that $w \sim 1.0$ – 1.5 . For a given g and type of step (that is either growth or dissolution) the black and white image reverses depending upon whether or not the ledge lies on the top or bottom surface of the precipitate. Compare schematic diagrams, figure 3.44(c). For a given ledge the black/white contrast reverses across the line of no contrast when g or w reverse sign, see figure 3.44(c).

(6) When a matrix reflection is excited in BF with g in the habit plane a faint double image is observed, see figure 3.44(d), like $g \cdot b = 0$ and $g \cdot b \wedge u \gtrsim 0.64$ for a dislocation, section 3.4.2.1.

(7) When a matrix reflection with g not in the habit plane is excited displacement fringes are observed in BF at the interface, see figure 3.44(e). These fringes are displaced at the ledges and the magnitude of the displacement is related to ledge height (Gleiter, 1969), see section 3.10.1.3.

3.10.3.3 Incoherent interfaces

Large particles such as fibres or inclusions exhibit thickness fringes like those at high-angle grain boundaries. Fine structure effects similar to those for grain boundaries also occur, see figure 3.44(f).

3.10.4 Quantitative Information on Precipitate–Matrix Interfaces

3.10.4.1 Coherent interfaces

Clearly trace analysis and similar techniques, section 2.14.1, may be used to obtain the interface planes. Furthermore, in some cases the Burgers vector of dislocations in the interface may be determined. Examples are shown in figure 4.25, section 4.11.2, for a γ' structural commercial Ni–base alloy.

Analysis of displacement fringes, see section 3.6.4, enables the sign of the mismatch between precipitates and matrix to be determined. Analysis of δ fringes has enabled the tetragonal distortion at the interface of γ' precipitates to be measured (Ardell, 1967).

3.10.4.2 Partially coherent interfaces

The b of a dislocation in the precipitate matrix interface may be studied in elastically isotropic materials using the criteria listed in section 3.10.3.2. Weatherly and Nicholson (1968) have studied this problem in a number of aluminium alloys.

An example of the identification of the Burgers vector of dislocations lying in the interface of θ' particle in an aluminium–copper alloy is shown in figure 3.43, using different matrix reflections. Here the precipitate particle has cube–cube orientation relationship, the lattice parameter

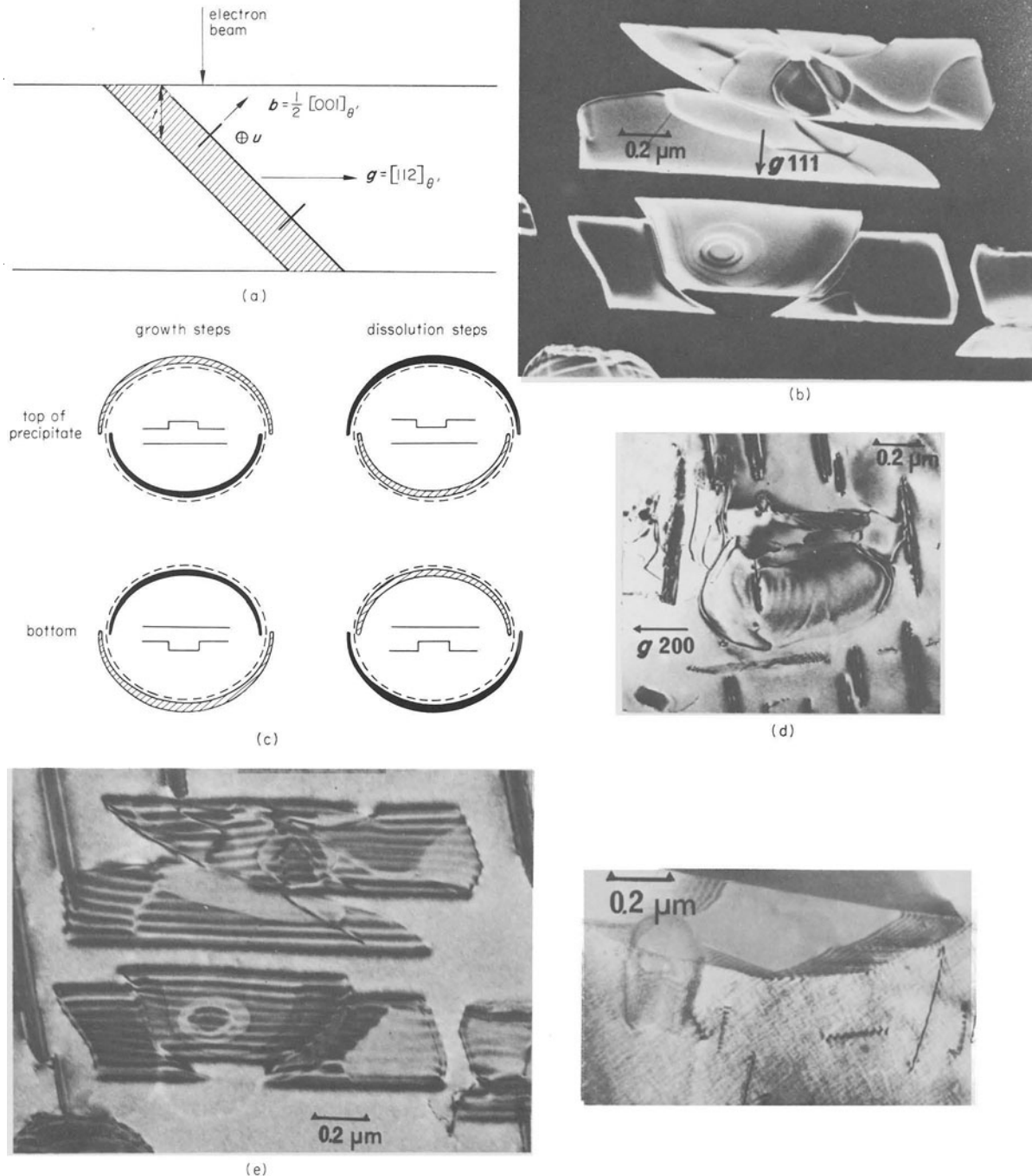


Figure 3.44 The geometry of the thin foil used by Weatherly and Sargent to compute images of ledges in θ' precipitates in Al-4 wt % Cu. (b) A CDF image obtained with a (111) matrix reflection showing θ' particle with ledges. (c) Schematic diagrams showing images of growth and dissolution steps. The position of the step is given by the dashed line, the heavy line being black contrast and the shaded region bright. (d) A BF image with a (200) reflection showing the ledges as faint double images. (e) Displacement fringes visible in BF, g [111]. (f) Defects in the interface between Ni_3Al particles and NiAl matrix. [(a)–(e) From Weatherly and Sargent (1970), courtesy of *The Philosophical Magazine*]

of aluminium being $4.049 \text{ \AA}$ compared with $a = 4.04 \text{ \AA}$, and $c = 5.80 \text{ \AA}$ for θ' . Provided reflections of the type $\{hk0\}$ $\{h00\}$ are used points (1)–(3), section 3.10.3.2, are satisfied. When $g = [\bar{2}00]$ dislocations A and C are visible; when $g = [0\bar{2}0]$ B and D are visible. Applying the $g \cdot b = 0$ criterion the array must consist of dislocations $b \pm [10l]$ and $[01l]$. The θ' plate with the dislocations D lies on (100) and by analogy with the θ' plane on (001)

must be covered by a network of dislocations $b \pm [l10]$ and $\pm [l01]$. The dislocations marked D have $b \pm [l10]$ and are visible $g[0\bar{2}0]$ and invisible $g = [\bar{2}00]$ therefore $l = 0$. Hence the network AB for a $(001)_{\text{Al}}$ is $\pm [100]$ and $\pm [010]$. This conclusion has been confirmed using (111), $(112)_{\theta'}$ and $(\bar{1}\bar{1}2)_{\theta'}$ reflections which give strong contrast for all dislocations.

In the above case the analysis is satisfactory

because the aluminium is almost elastically isotropic. However, considerable difficulty would be expected if the matrix were elastically anisotropic in view of the work described in sections 3.4.2, 3.4.3. Even in aluminium w must be $\lesssim 0.3$ or $\mathbf{g} \cdot \mathbf{b} = 1$ situations may be mistaken for $\mathbf{g} \cdot \mathbf{b} = 0$. Consequently, it is important to realise that this type of investigation is extremely difficult to perform.

In addition quantitative information has been obtained on Al–Cu alloys of the nature of ledges at θ' CuAl₂ plates. Using the information listed for ledges in section 3.10.3.2, point (6), and assuming that the strain field of the ledge could be approximated by an edge dislocation loop, figure 3.44(a) ($\mathbf{b} = \frac{1}{2}[001]\theta'$, that is perpendicular to the habit plane). Weatherly and Sargent (1970) were able to determine the growth/dissolution nature of ledges. The procedure is as follows.

(1) Obtain a dark field micrograph with $w = +1.7$ using a precipitate reflection (see section 1.5.2 for procedure) such that the precipitate thickness is $0.25\text{--}0.35\xi_g$.

(2) Determine the top and bottom on the foil (that is sense of inclination of precipitate) by comparing bright and dark field images with a matrix reflection that produces displacement fringes.*

(3) Rotate the positive print until the sense of inclination is with the top on the left of the operative $\mathbf{g}$, like figure 3.44(a).

(4) Use the rule that, for an interstitial precipitate, the step is dissolution if the white contrast lies inside the continuation of the dark contrast past the line of no contrast, see figure 3.44(c). If the dark contrast is inside the continuation of white contrast then it is a growth step. This rule is reversed for a vacancy type precipitate strain field. Figure 3.44(b) is a growth step.

Note that this analysis is restrictive in that it only applies to precipitate thicknesses of $0.25\text{--}0.35\xi_g$ and $w = +1.7$ and edge ledges for elastically isotropic materials. Considerable care will be necessary in its application to other alloy systems when the matrix or precipitate is elastically anisotropic or the ledge does not have a simple edge strain field.

3.10.4.3 Incoherent interfaces

At present the only information that may be obtained is the plane of the interface by trace analysis, section 2.14.1, and the orientation relationships between phases using SADP, section 2.11.

* Since opposite $\mathbf{g}$ dark field technique is normally used images show opposite contrast at top and same at bottom of foil.

† For definition of $\mathbf{l}$ see section 3.5.5.

3.11 Strain Field Images from Small Misfitting Precipitates

Small misfitting precipitates give rise to images consisting of lobes of contrast. In some cases these images may be used to determine the sense, that is vacancy or interstitial, of the strain field, the magnitude of the strain and the size of the particle.

3.11.1 Image Characteristics

Small precipitates with a spherical strain field arising from volume mismatch show one of two types of images under two-beam conditions (McIntyre and Brown, 1966); depending upon the magnitude of the parameter $P_s = \mathbf{g} \varepsilon r_0^3 / \xi_g^2$, where ε is the misfit strain and r_0 the constrained particle radius the following occurs.

(1) If $P_s \lesssim 0.2$ the images consist of small black/white lobes and black dots in both bright and dark field similar to the case of Frank loops. Such a typical DF image is shown in figure 3.45(a) which shows small cobalt precipitates in copper.

(2) If $P_s \gtrsim 0.2$ the images consist of large black/black lobes in BF unless the precipitate lies within $\sim \xi_g$ of the surface in which case black/white lobes occur. Typical BF images are shown in figure 3.45(d) for a spherical Al₃Li δ' precipitate imaged under a series of reflections. The particle 1 imaged above is shown by precipitate CDF in figure 3.45(d).

These two cases will be considered respectively for the case of a spherically symmetrical strain field, that is a misfitting sphere in elastically isotropic material which is the only situation investigated theoretically up to the present.

3.11.1.1 $P_s \lesssim 0.2$

The image characteristics are as follows.

(1) The vector $\mathbf{l}^\dagger$ joining the centre of the black lobe to that of the white lobe on the positive print is always parallel to $\mathbf{g}$.

(2) The sign of $\mathbf{l}$ oscillates with depth exactly like the case in figure 3.27 and layers 1, 2 and 3 of the same approximate dimensions exist. Consequently, in figure 3.45(a), in which all the precipitates have the same interstitial strain field $\mathbf{g}$ points towards some light and some dark lobes which reflects the presence of defects in different layers of figure 3.27.

(3) In the centre of the foil and between layers the bright field image is a black dot.

(4) BF and DF images are identical for layers 1–3 as in figure 3.27 but opposite for layers 4–6.

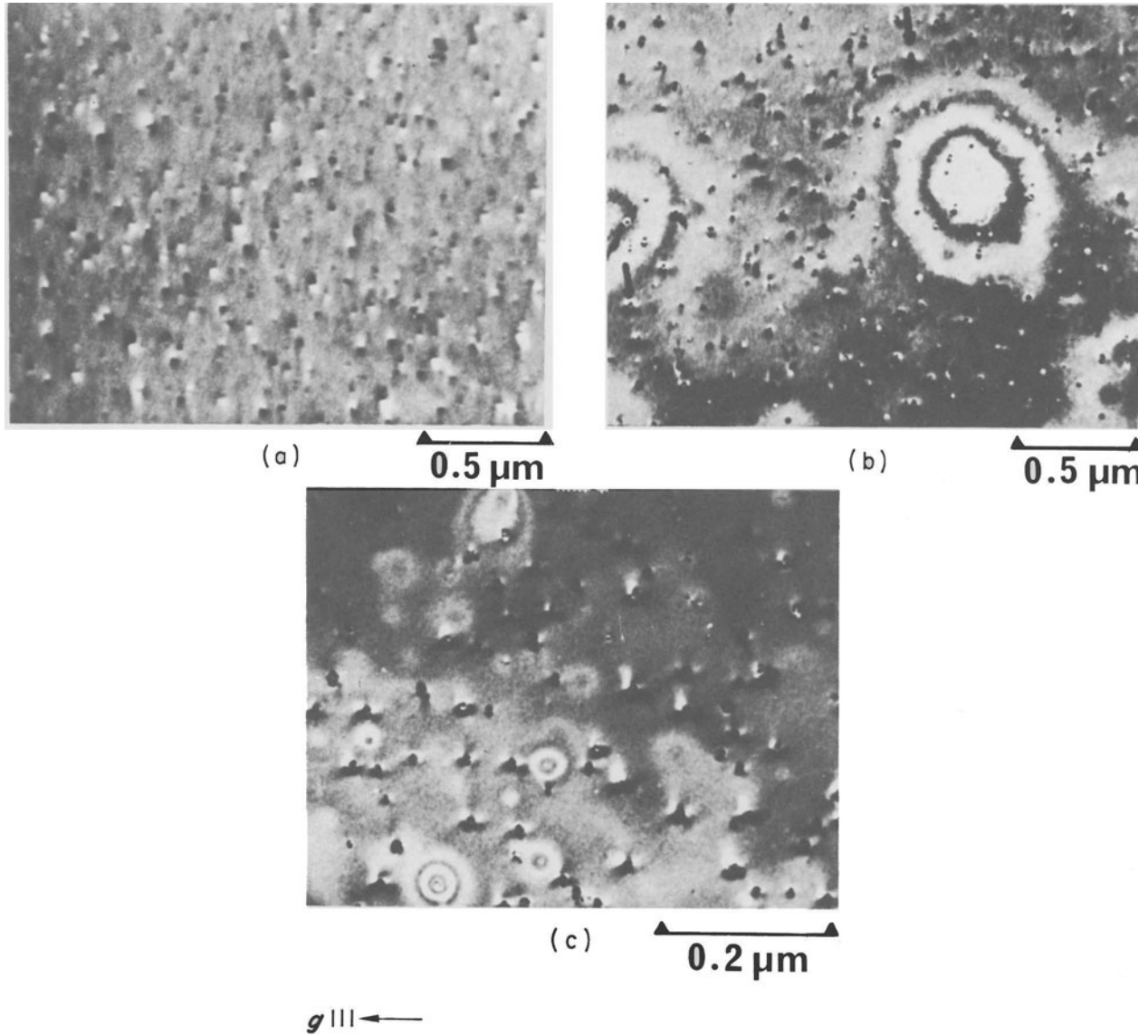


Figure 3.45 The influence of the parameter P_s on the CDF image of misfitting precipitates in Cu-Co alloy: (a) $P_s \sim 0.05$; (b) $P_s \sim 0.1$; (c) $P_s \sim 0.4$. BF images same matrix reflection operative in all cases. (d) CDF image of large δ' particle 1 and associated BF images under the reflections shown. (e) A CDF image showing bright/dark strain field lobes of particles near the surface. The material for (d), (e) is Al-10 at %Li aged for 24 hours at 250 °C [(a)–(c) From McIntyre and Brown (1966), courtesy of *Journal de Physique et Le Radium*]

(5) The sign of l reverses in each layer of figure 3.27 if the strain field is changed from interstitial to vacancy.

3.11.1.2 $P_s \gtrsim 0.2$

The image characteristics are as follows.

(1) The images are much bigger than for the case with $P_s \lesssim 0.2$ considered above. Typically the image diameter is $\sim \xi_g$ (compared with $\lesssim 0.3\xi_g$ for $P_s \lesssim 0.2$).

(2) All precipitates except those within ξ_g of the foil surface exhibit black/black lobes with a line of no contrast that is always perpendicular to g . This behaviour is clearly visible for the specific image of particle 1 in figure 3.45(d).

(3) In BF and DF precipitates within $\sim \xi_g$ of the foil surface exhibit black/white lobe images, see figure 3.45(e), at A. The black/white lobe images are identical in BF and DF for defects at the top of the foil but opposite at the bottom.

The transition between $P_s \lesssim 0.2$ and $P_s \gtrsim 0.2$ is shown for the CDF images of cobalt particles in copper, figures 3.45(a)–(c). In the first the particles are small $P_s \lesssim 0.2$ and the layer effect is observed with images present with different signs of l , that is precipitates at different levels in figure 3.27. However, in figure 3.45(c) $P_s \gtrsim 0.2$, that is the cobalt particles are larger and all near-surface precipitates exhibit images with the same sign of l , that is layers 2 and 4 of figure 3.27 have disappeared.

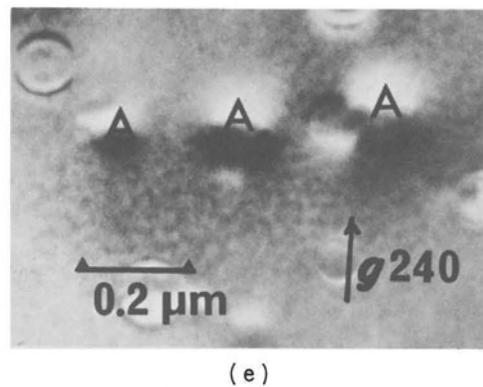
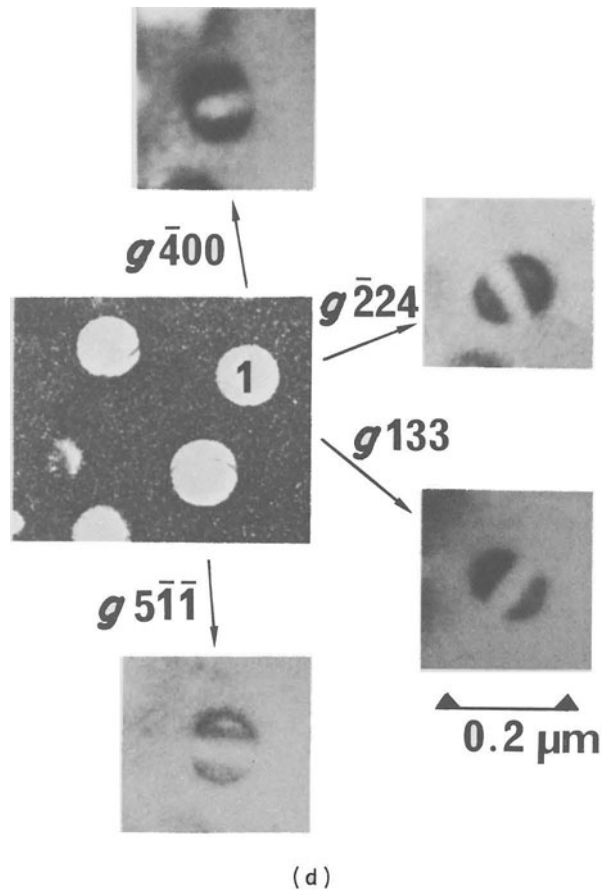


Figure 3.45 (continued)

3.11.2 Quantitative Information

3.11.2.1 Determination of the sense of the strain field

This experiment is difficult to perform. The following rules may be used.

(1) If $P_s \lesssim 0.2$ in BF, g points towards a dark lobe if the defect is interstitial and lies in layers 1, 3, 5; but it points towards the bright lobe if the interstitial defect lies in layers 2, 4, 6, figure 3.27(b). If the opposite behaviour is found the precipitate is vacancy.

(2) If $P_s \gtrsim 0.2$ a CDF image must be used. Then if g points towards a dark lobe the precipitate is

interstitial but if towards a bright lobe it is vacancy (layers 2, 4 removed in figure 3.27). This is the rule of Ashby and Brown (1963a, b).

Note that the theoretical treatment assumes a spherically symmetrical strain field, that is the line of no contrast is always perpendicular to g and l is always parallel to g . Consequently, the analysis is only strictly applicable to this case and that for a plate precipitate if the strain field corresponds to the Frank loop discussed in section 3.5.5. However, Degischer (1972) has shown that the sign of the strain field may be determined uniquely in Cu–Co alloys despite anisotropy of the strain field image, that is line of no contrast not

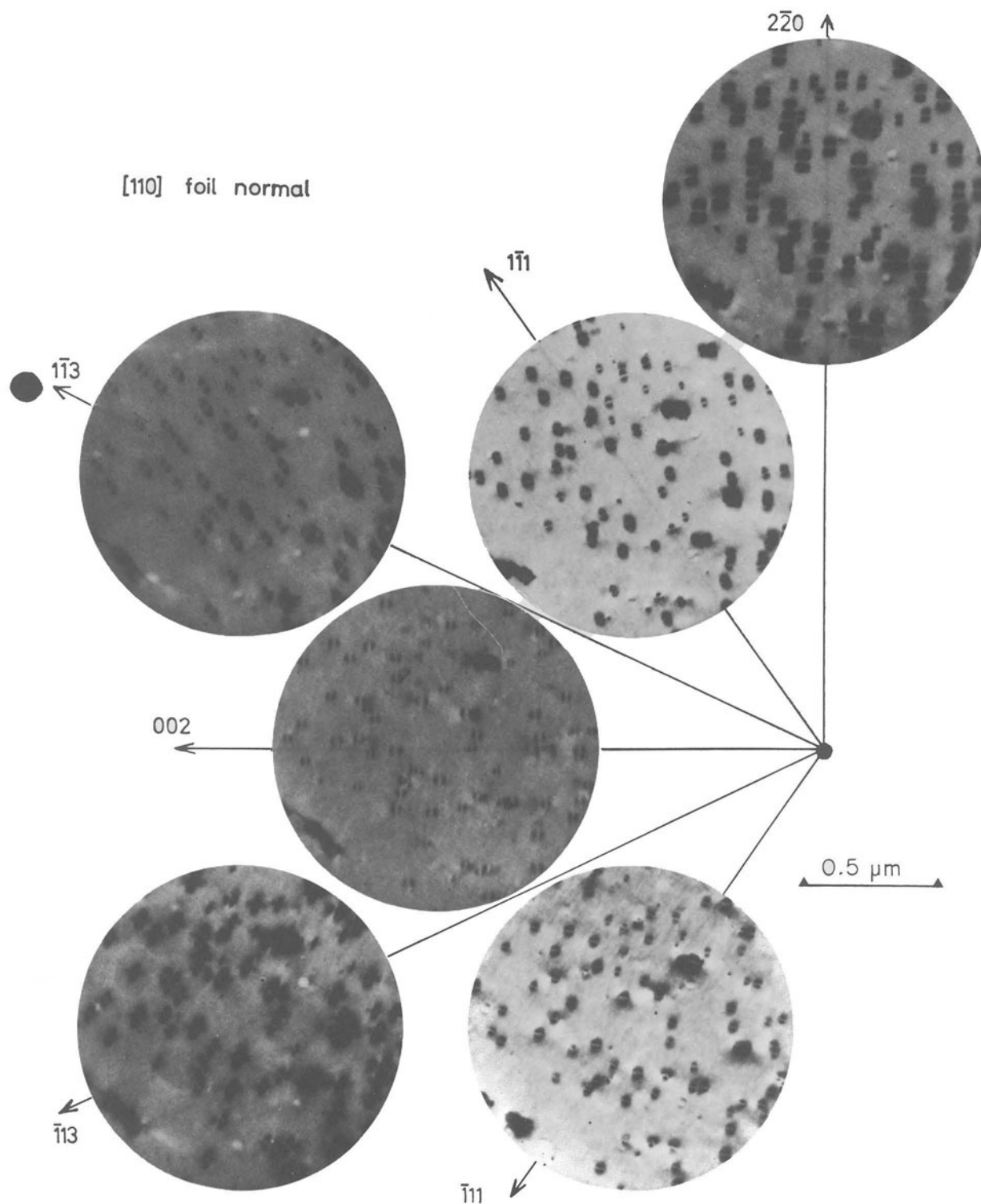


Figure 3.46 BF images of the same area showing misfitting Co precipitates in Cu–Co alloys for different reflections [From Degischer (1972), courtesy of *The Philosophical Magazine*]

perpendicular to g , figure 3.46. Consequently, this criterion of a spherical strain field need not be too rigorously applied, at least when $P_s \gtrsim 0.2$. However, there is no guarantee of a correct result until full theoretical calculations have been performed for the non-spherical strain field.

The procedure is as follows.

(1) Check for a spherically symmetrical strain field, that is $P_s \lesssim 0.2$, l always parallel to g or $P_s \gtrsim 0.2$ line of no contrast always perpendicular to g .

(2a) For the case of $P_s \lesssim 0.2$ proceed as for Frank loops, see section 3.5.6. This case may be recognised by the small images and the presence

of only black/white lobe images in BF and DF with opposite signs of l in one area for CDF images.

(2b) If $P_s \gtrsim 0.2$, that is large images, black/black lobes present with a few black/white lobes proceed as follows.

(3) Obtain a CDF image of the area of interest.

(4) Draw the direction of g in the positive print.*

(5) Determine the sense of the strain field using the black/white images of the particles near the surface and the rule g points from black to white lobe for a vacancy strain field and vice versa for an interstitial strain field.

Figure 3.45(e) shows a typical example $P_s \gtrsim 0.2$. The particles of Al_3Li are spherical, see figure 3.45(d), and the line of no contrast is always perpendicular to g . In figure 3.45(e) which is a CDF of a different area using a matrix reflection, g points towards the bright lobe of defects A which are therefore vacancies.

3.11.2.2 Estimation of the magnitude of the misfit ε

Ashby and Brown (1963b) showed that the width of the image for $P_s \gtrsim 0.2$ is predominantly controlled by the parameter P_s . Thus, if r_0 is known, an estimate of the misfit parameter can be made using the graph in figure 3.47(a) for spherically symmetrical strain fields only. These authors suggested that the 2%, 20% or 50% image widths could be used as estimates of the image size provided they were normalised to ξ_g for the operative reflection. The 2% width is the easiest to use in most cases. Ashby and Brown (1963b) also reported that the misfit may also be calculated for precipitate plates using the graph in figure 3.47(a), where r_0 is the radius of the disc and θ is the angle between the reciprocal lattice vector g and the upward drawn normal to the disc plane. However, their calculations assumed isotropic elasticity and the accuracy of their approach for the anisotropic case has not been checked experimentally.

Significant errors in any estimate of ε may easily occur ($\pm 30\%$) primarily because of errors in ξ_g and r_0 , the latter being particularly difficult to determine in the absence of precipitate CDF images. Ashby and Brown (1963a) suggest that the diameter $2r_0$ can be estimated for large particles from the image width along the line of no contrast using the contour map in figure 3.47(b). Alternatively, Woolhouse and Brown (1968) suggest that the particle size and shape may be obtained from BF images under kinematical conditions when most of the image arises from the high-strain region at the interface (the bright Kikuchi line

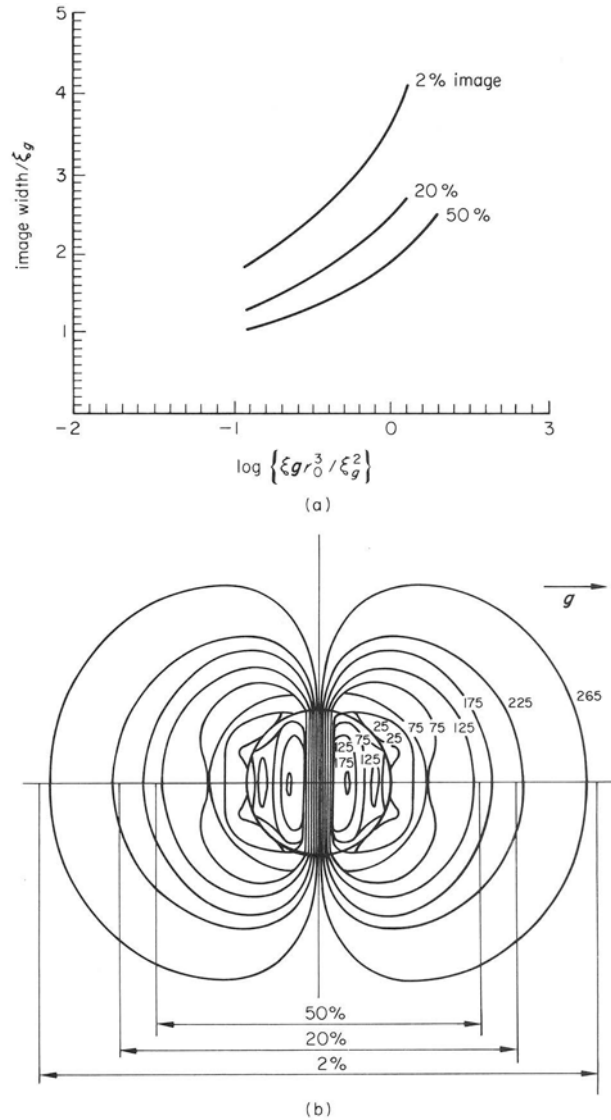


Figure 3.47 (a) The predicted variation of the 2%, 20% and 50% image width as a function of the misfit parameter $\xi_g r_0^3 / \xi_g^2$. (b) Typical predicted contrast around a spherically symmetrical misfitting particle showing the definition of the 2%, 20% and 50% image width. The numbers illustrate intensities relative to the background intensity of 272 [From Ashby and Brown (1963a), courtesy of *The Philosophical Magazine*]

must be displaced by $\frac{1}{4}g$ from the operative bright spot). A maximum radius of up to 100 Å may be estimated in this way using $\{111\}$, $\{200\}$ and $\{220\}$ reflections. (Note that these images only outline the projection of the image into the diffraction pattern.) In the particle size range 100–200 Å the $\{113\}$ reflections may be used but above this diameter the kinematical image can be displaced from the interface by 30–40 Å depending on precipitate size and operative reflection.

Since particle size measurement is one of the major sources of inaccuracy in determining the mismatch of particles, special care should be

* Note that g is drawn in the CDF diffraction pattern from the off-centred transmitted spot to the centred bright diffraction spot.

taken. The best method is CDF using precipitate reflections with different B parallel to major matrix zone axes to determine particle shape. If this cannot be done, kinematical BF or weak beam images (section 3.17) may be used, making wide use of different reflections and B to synthesise the particle shape. Extraction replica studies of particle sizes have limited usefulness because of possible dissolution of particles during extraction.

It is important to realise that, for strain measurements, the following conditions must hold.

- (1) w must be zero.
- (2) Black/white images must be avoided.
- (3) The foil thickness must be large compared with r_0 .
- (4) Only two-beam conditions may be used.
- (5) Prints must be produced with a visible background density.

For most materials this simple approach due to Ashby and Brown cannot be used because spherically symmetrical strain fields occur only infrequently. Particle shape effects and elastic anisotropy

can be very important in controlling image width. Sass *et al.* (1967) showed that asymmetric images occur for situations in which g is not perpendicular to a symmetry plane of the precipitate. Typical symmetric and asymmetric examples are shown in figure 3.48 for a Ni–Ti alloy containing misfitting cube γ' particles. These latter workers suggest that the error in the measured value of mismatch for a cube precipitate images under a $\{220\}$ reflection may still be only 25%, decreasing for smaller values of the mismatch. Similar results have been found for copper–cobalt alloys (Degischer, 1972) in which the precipitate particles are not spheres (Woolhouse and Brown, 1968).

Nevertheless, significant errors can occur when the strain field is not spherically symmetrical. Consider the Cu–Al₂O₃ system which was considered by Ashby and Brown (1963a, b) to have a large mismatch but has a non-spherical strain field. If r_0 is measured from an image chosen carefully to have a clear straight line of no contrast perpendicular to g , using the above line of no contrast approach of Ashby and Brown, the value obtained is half that determined from extraction replicas. Consequently, the actual mismatch is about ten times smaller than that estimated using r_0 determined from the line of no contrast.

In our opinion estimates of the magnitude of the misfit are unreliable except for spherically symmetrical strain fields when particle sizes are accurately estimated from precipitate CDF. Measurements of strain fields on non-spherical particles such as Co particles in Cu–Co alloys (Ashby and Brown, 1963b) must be regarded only as estimates. Similarly the complex strain fields of cuboid carbide particles in stainless steels are difficult to measure accurately (Froes *et al.*, 1967). However, in some cases, for example figure 3.45(d), gas bubbles in irradiated copper and stainless steel (Brown and Mazey, 1964) and silica particles in copper (Weatherly, 1968) relatively accurate values can be obtained.

3.11.3 Image Characteristics of Misfitting Plate Precipitates

The case of high volume fractions of GP zones with large coherency strains has been discussed in detail by Tanner (1966), Fillingham *et al.* (1972) and Phillips and Tanner (1973) for Cu–Be alloys. The precipitates lie on $\{100\}$ matrix planes but produce matrix strains in $\langle 110 \rangle$ which are primarily responsible for the strain field contrast in BF. However, the form of the strain field is such that for some matrix reflections some precipitates are invisible and streaks in diffraction patterns have a characteristic distribution, see section 2.18.2. Tanner (1966) points out that the precipitate

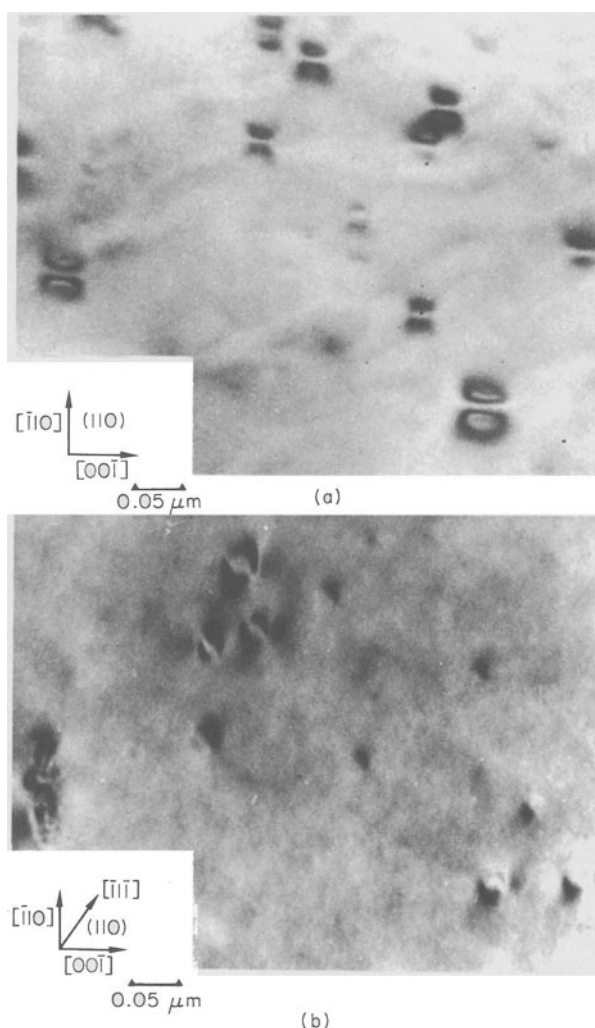


Figure 3.48 BF images of cuboid Ni₃Ti precipitates taken using different reflections [From Sass *et al.* (1967), courtesy of *The Philosophical Magazine*]

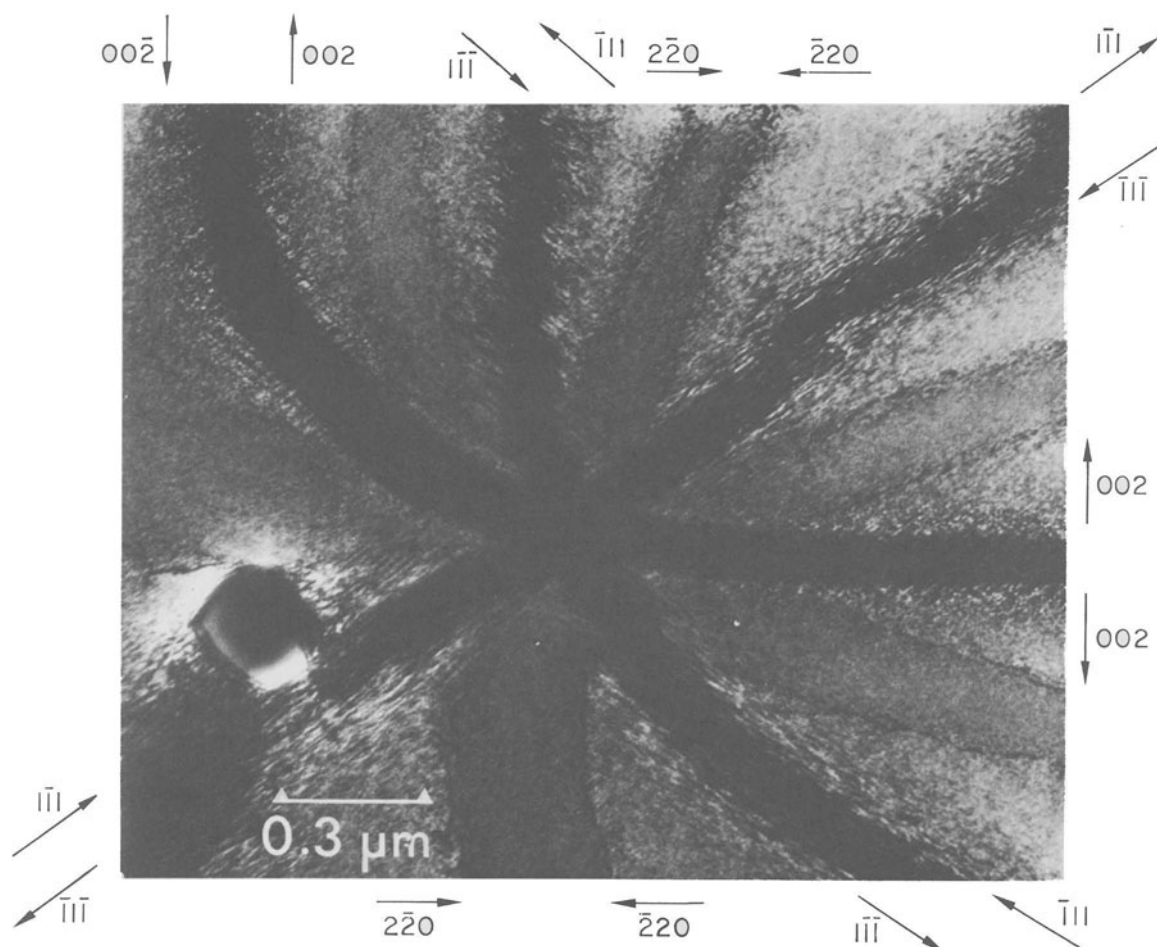


Figure 3.49 A $[110]$ bend centre image of GP zones in Cu-2 wt %Be [From Tanner (1966), courtesy of *The Philosophical Magazine*]

invisibility may be understood in terms of a ' $\mathbf{g} \cdot \mathbf{b} = 0$ invisibility criterion' similar to that described for dislocations in section 3.4.2.1. Figure 3.49 shows the characteristic form of the bright field image of these precipitates for $\mathbf{B} \langle 110 \rangle$ at the centre of a dimpled region of the foil, see section 3.2.3.4. The precipitates image as dark streaks parallel to the projection of $\langle 110 \rangle$ and for some reflections $\mathbf{g} \cdot \mathbf{b}_{110} = 0$ and images do not occur. Furthermore, some $\langle 110 \rangle$ directions superimpose on projection into the (110) plane. Thus, referring to figure 3.49 when $\mathbf{g} = [1\bar{1}\bar{1}]$, $[\bar{1}11]$, $[1\bar{1}1]$, $[\bar{1}\bar{1}\bar{1}]$, a single set of streaks is observed. However, when $\mathbf{g} = [002]$, $[00\bar{2}]$, $[2\bar{2}0]$, $[\bar{2}20]$, two sets of streaks mutually inclined at 70° are observed. This behaviour together with the direction of streaking in the diffraction pattern, see section 2.18.2, may be used to identify GP zones of this type. Tanner (1966, 1968) has shown that similar effects occur in ordering of Ni_3V , CuAu , CoPt and NiPt , while Okamoto and Thomas (1971) have observed similar effects in Ni_4Mo . Peacock (1973) has made similar observations for the initial stages of precipitation of Co in the intermetallic compound CoAl . However, it is important to realise that, for larger precipitates, more complex images can occur

because of contribution to the image of diffraction by the precipitate and/or partial loss of coherency which modifies the strain field.

3.11.4 Quantitative Information on Misfitting Plate Precipitates

On the basis of the image characteristics outlined in section 3.11.3 strain field images of plate precipitates may be recognised. However, at no stage may the misfit be measured using any of the analyses outlined previously in section 3.11.2. If the precipitate shape streak is detectable in the diffraction pattern, the habit plane may be determined, see section 2.18.1.

3.12 Cavities

In studying cavities we are primarily concerned with demonstrating that the defect is a cavity and determining its shape.

3.12.1 Image Characteristics

Theoretical calculations of the contrast from cavities have been carried out by a number of workers (Brown and Mazey, 1964; van Landuyt

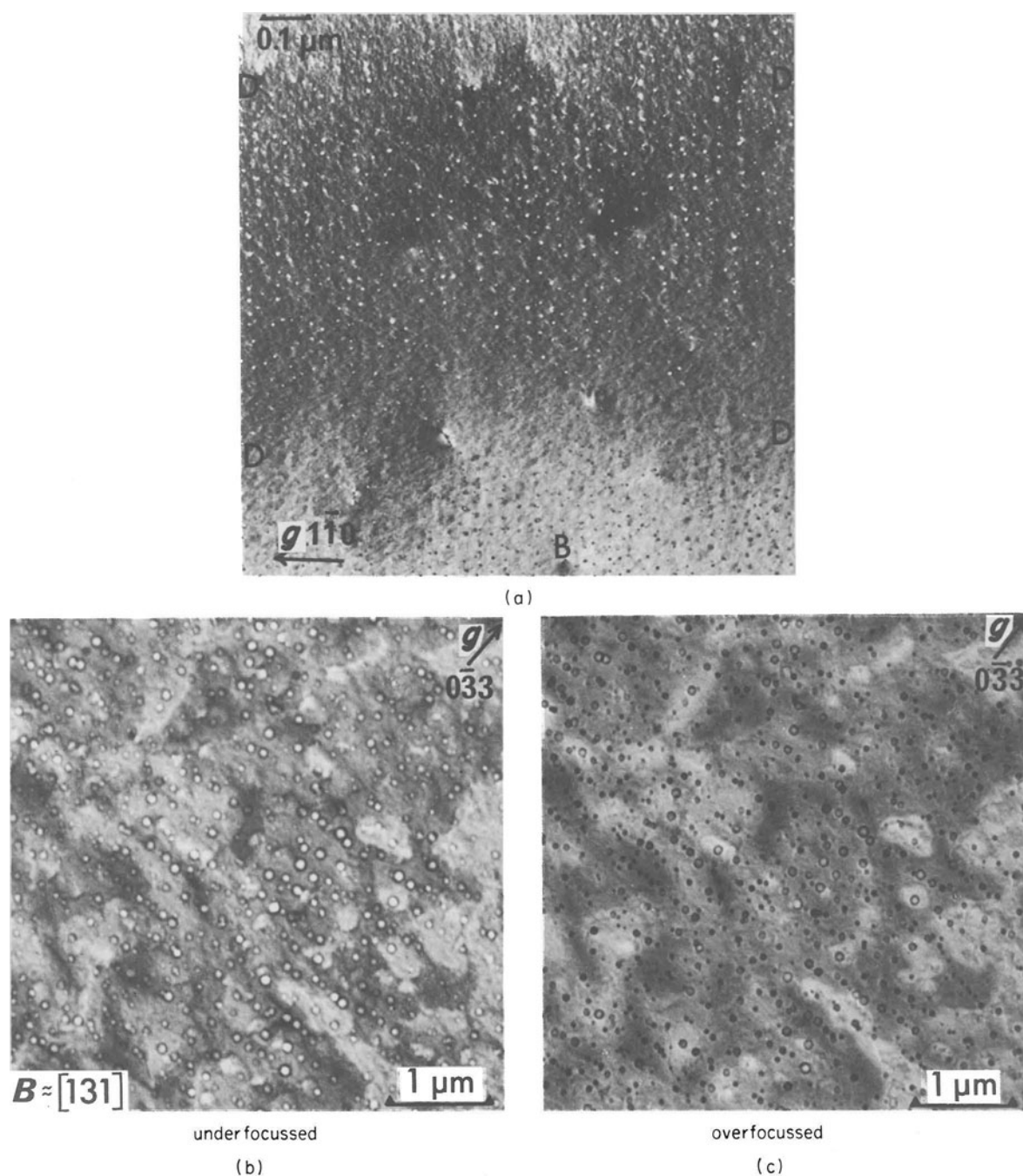


Figure 3.50 (a) An in-focus BF image of voids in neutron-irradiated molybdenum; (b), (c) underfocussed and overfocussed BF images of voids in the same material [Courtesy of B. L. Eyre]

et al., 1965; Ingram, 1969) and experimental studies have been reported by van Landuyt *et al.* (1965) and Eyre (1972).

The results which are directly relevant to experimental observations in BF are as follows.

(1) Cavities exhibit strongest contrast in thin regions of the foil ($\lesssim 3\xi_g$).

(2) At $w = 0$ contrast arises from anomalous absorption (that is they are bright on a dark background) except for very small cavities radius $r < 0.1\xi_g$ when strain contrast may make a significant contribution.

(3) Under conditions when contrast arises from anomalous absorption the image depends sensitively on foil thickness. Optimum contrast is obtained in a narrow region at the front of the first or second dark thickness fringe. Cavities are bright at the front of the dark contour D, figure 3.50(a) (thinner region) and dark at the bright (thicker region) B in figure 3.50(a).

(4) In thick foils when thickness fringes in BF are damped out (section 3.2.3.1) cavities are bright because of normal absorption, that is different effective thickness. In this case only larger cavities ($r \gtrsim 0.5\xi_g$) are visible).

(5) Small cavities are much more easily visible with the image slightly out of focus and under kinematical diffraction conditions. In an underfocus condition the cavity image is bright relative to background and the image has a dark rim. The reverse is true of an overfocus condition when the void is a dark image with a faint bright rim, see figure 3.50(b), (c). The real cavity diameter is that contained inside the rim image (underfocus).

3.12.2 Quantitative Information

Cavities may be identified using points (1)–(5) above; however, the single most useful property in this context is point (5).

The shape of large cavities may be obtained by photographing individual cavities with different simple B , and performing a crystallographic analysis of the facets. However, shape determination for small cavities $r < 0.5\xi_g$ is difficult and when $r \lesssim 0.2\xi_g$ it is almost impossible.

3.13 Gas Bubbles

Gas bubbles occur in material irradiated at high temperatures. In studies of such defects we are primarily interested in determining bubble shape, size and sense of strain field.

3.13.1 Image Characteristics

The in-focus contrast from bubbles with a surrounding strain field has been calculated for different sizes, amounts of strain, diffraction condition and foil thicknesses (Rühle, 1972). Both diffraction contrast from the cavity and strain field were included. It has been concluded that the form of the image depends on the parameters

$$P_b = gR_s^2(2\gamma - pR_s)/3\mu\xi_g \quad \text{and} \quad R_v/\xi_g$$

where R_s is the radius of the inclusion required to produce the strain field, R_v is the void radius, p is the gas pressure, γ is the surface energy and μ is the shear modulus. Figure 3.51 shows schematically the conditions under which three types of image may be formed, that is

- region A: black/white images with depth oscillations, figure 3.27
- region B: Ashby–Brown black/black or black/white with no depth oscillations, section 3.11.1
- region C: no black/white contrast mainly thickness contrast

3.13.2 Quantitative Information

For small bubbles (area A), figure 3.51, the order of magnitude of the strain may be estimated by

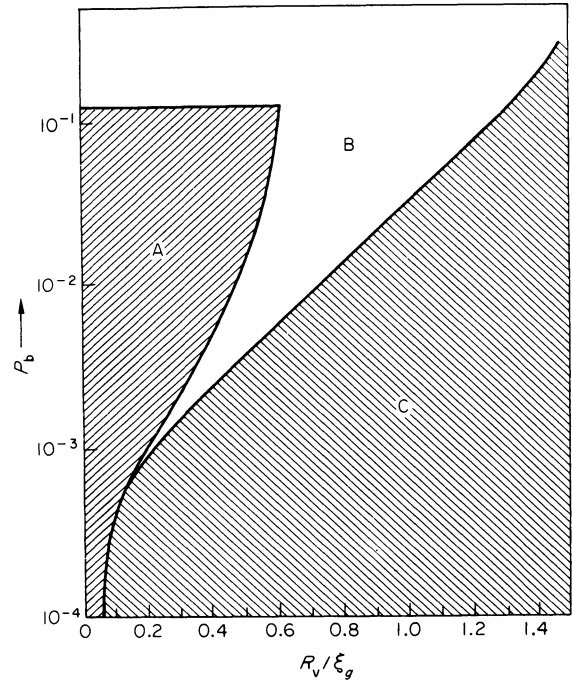


Figure 3.51 The experimental conditions under which different types of image of gas bubble may be formed. R_v is the radius of the void, ξ_g is the extinction distance and P is the normalised misfit parameter [Courtesy of M. F. Rühle]

comparing computer calculated images with experimental images. The sense of the strain field may be determined using the analysis outlined in section 3.11.2 provided the strain field is spherically symmetrical. For larger bubbles, area B, the Ashby–Brown analysis may be used to estimate the magnitude of the strain but no analysis of larger bubbles (area C) is possible because the strain field is an insignificant portion of the image.

The size and shape of bubbles should be determined using different B for kinematic BF images (see previous section, on voids) or weak beam images but there is no published work on this subject at present.

3.14 Visibility of Defects in Strain Field Diffraction Contrast Images

As pointed out in section 3.3.1 maximum transmission occurs in BF with s small and positive, see figure 3.3(b), (c), and it is under these conditions that most BF images are usually obtained. When imaging crystal defects such as dislocations, stacking faults, precipitates with strain fields, small Frank loops, etc., contrast is frequently good under these conditions, but there are two common problems.

(1) If different types of defect are present it can be difficult to make visible only the particular defect under study.

(2) If small defects are present, for example Frank loops or voids, it can be difficult to obtain

conditions for maximum resolution, that is observation of very small defects, when several types of defect occur.

The following factors are a useful guide to determine which conditions should be used to image a particular defect.

(1) The experimental conditions for maximum contrast can be different for each defect, for example dislocations with $g \cdot b = 1$ and 2 (France and Loretto, 1968), section 3.4.3.1; small Frank loops in the centre and near the surface of the foil (Wilkens, 1969), section 3.5.5; π boundaries for superlattice reflections, section 3.7.1.

(2) Defects not exhibiting strain field contrast require different imaging conditions, that is coherent precipitates and voids are readily visible at $s = 0$ or very large, whereas strain field images show best contrast in BF at s small and positive.

(3) The nature of outside fringes from stacking faults, etc., is best determined at $s = 0$ bright and dark field.

(4) Best image contrast is generally found in regions with intermediate background intensity ($w \approx 0.5$), that is at the edges of thickness fringes, see figure 3.50(a), or for thicknesses just above the maximum at which thickness fringes occur, see section 3.2.3.1 and figure 3.2(d).

(5) If complex defect arrays are present in the material, those producing detectable diffraction spots are most easily studied using CDF images.

(6) The weak-beam DF technique is a very useful method of improving resolution when a high density of closely spaced defects, for example dislocations, are present, see section 3.17.

(7) Eyre *et al.* (1971) have shown that the minimum diameter for detection of Frank loops is 14 Å and occurs for $w = 0.4$ with the loop in layer 5 at the bottom of the foil, figure 3.27. Similar results would be expected for precipitates exhibiting strain field contrast.

(8) Although voids have not been studied in detail, void images down to 10–15 Å diameter have been detected when imaged with a high order systematic reflection, for example {330} b.c.c. metals, see figure 3.50(b), (c).

If precipitates are imaged using precipitate dark field techniques sizes < 20 Å may be detected although no detailed study of the minimum observable size has been reported, errors in particle size determination arising from chromatic aberration may be significant, see section 1.2.

3.15 Differentiation Between Similar Types of Images

In two areas there are a number of types of defect that produce qualitatively similar images. Thus

large planar objects such as stacking faults, coherent or semi-coherent plate precipitates, anti-phase domain boundaries, etc., all produce fringe images. Furthermore, small defects such as dislocation loops, precipitates and gas bubbles form similar lobes of contrast. In some, although not all, cases it is possible to differentiate between different crystal defects within groups such as those above, entirely on the basis of the details of their BF and DF images or using information in diffraction patterns. The first part of section 4 summarises how this may be achieved and gives a number of examples.

3.16 Many-beam Effects

Up to this point, images have been discussed in terms of the two-beam dynamical theory. However, in practice it is often difficult to obtain good two-beam conditions in ordered materials or those with a large lattice parameter. Frequently if the (hkl) reflection is strong the systematic row of ($2h2k2l$) and ($3h3k3l$) reflections are also reasonably strongly excited. This situation is accentuated at accelerating voltages $\gtrsim 200$ kV. In practice the most important effects are on the extinction distance and the major features of the image are relatively unaffected.

3.16.1 Extinction Distance

Howie and Bazinski (1968) and Serneels and Gevers (1969) have calculated the influence of a number of operative reflections on ξ_g .

Table 3.9 shows their results for ξ_g (two beams) and $\xi_g^{(c)}$ systematic many beams for which the effective extinction distance is reduced.

Howie and Bazinski (1968) and Serneels and Gevers (1969) have also calculated ξ_g effective for multiple, non-systematic cases in copper, see

Table 3.9 Extinction distances ξ_g and $\xi_g^{(c)}$ (in ångströms) for 100 kV electrons

Reflection	111		200		220		311	
Element	ξ_g	$\xi_g^{(c)}$	ξ_g	$\xi_g^{(c)}$	ξ_g	$\xi_g^{(c)}$	ξ_g	$\xi_g^{(c)}$
Al	556	503	673	630	1058	1036	1300	1285
Ni	236	199	275	245	409	393	499	488
Cu	243	203	283	251	418	402	508	496
Ag	224	175	255	214	363	342	433	417
Pt	146	108	166	132	231	211	274	260
Au	159	116	179	141	248	227	293	277

Reflection	110		200		221		310	
Element	ξ_g	$\xi_g^{(c)}$	ξ_g	$\xi_g^{(c)}$	ξ_g	$\xi_g^{(c)}$	ξ_g	$\xi_g^{(c)}$
Fe	270	231	395	375	503	409	712	705
Nb	261	210	367	339	458	439	619	609
Mo	232	187	329	303	411	394	561	552
W	161	121	222	196	272	255	365	355

Table 3.10 Influence of non-systematic reflections in the extinction distance ξ_g for different reflections and B in Cu

Reflection	Cross grating pattern	Number of beams	Extinction distance (Å)
(111)	12 $\bar{3}$	26	188
(111)	31 $\bar{4}$	24	199
(111)	32 $\bar{5}$	20	202
(200)	023	16	249
(200)	012	24	235
(200)	013	34	192
(200)	015	22	242
(220)	1 $\bar{1}$ 3	16	385
(220)	2 $\bar{2}$ 3	16	395
(220)	3 $\bar{3}$ 2	26	332
(220)	2 $\bar{2}$ 1	16	376
(220)	3 $\bar{3}$ 1	16	396

table 3.10. The results show that the effect of non-systematic reflections is weak for the (111) reflection, more noticeable for the (200) reflection and quite important for the (220) reflection. It is probable that these changes in effective ξ_g can account for the persistence of spot patterns when a specimen is tilted from exact B (Laird *et al.*, 1966), see section 2.7.5.

3.16.2 Images

3.16.2.1 Perfect crystals

Many-beam effects introduce complications into the general contrast effects associated with bend extinction contours in perfect crystals. This effect is most important in high-voltage electron microscopy, but does occur at 100 kV in Au and some metals, and has been studied by Spring and Steeds (1970) and by Steeds (1970) using computer simulation techniques.

3.16.2.2 Dislocations

Howie and Bazinski (1968) considered the influence of four systematic reflections on dislocation images. They showed that image width was only slightly modified and that the images are qualitatively rather similar in form for two- and four-beam cases. Additional calculations have been reported by Skalicky and Papp (1972) for three non-systematic and four systematic reflections using the computer simulation technique of Head (1967). They demonstrated that good matching of computed and experimental images could be obtained but the details of the images themselves were very sensitive to the values of s for the operative reflections.

3.16.2.3 Misfitting precipitates

The Ashby–Brown calculations are only modified by $\pm 10\%$ in the determination of the misfit ϵ . The line of no contrast remains perpendicular to the row of operative reflections. Howie and

Bazinski also showed that the dark field images in weak $-g$ or $+2g$ reflections often showed higher contrast than the bright field images or the dark field image in the strong g . It is therefore possible that the visibility of defects could be improved under certain systematic many-beam conditions.

3.16.2.4 Stacking faults

Humphreys *et al.* (1967) and Metherell and Fisher (1969) showed that the main influence of systematic many-beam cases on images of stacking faults is to change the details of the fringes. In general, more fringes of a range of intensities are obtained. Although the nature of the outer fringes does not alter compared with a two-beam image, they may become difficult to detect.

It can be concluded that, for all purposes in part II of this section, two-beam theory is applicable to 100 kV accelerating potential under the following conditions.

- (1) The specimen is carefully tilted to two-beam conditions such that first-order reflections are used and s is very small, that is $w \lesssim 1.0$.
- (2) B is not too close to a simple zone axis, that is there are no non-systematic reflections visible.
- (3) The lattice parameter of the material is not too high.

3.17 Weak-beam Images

The 'weak-beam' (WB) technique is a convenient and important method of producing high-resolution diffraction contrast images of crystal defects. It was first reported by Cockayne *et al.* (1969) and has been discussed further by de Ridder and Amelinckx (1971), Ray and Cockayne (1970, 1971), Cockayne *et al.* (1971a), Stobbs and Sworn (1971a, b) and Cockayne (1972, 1973). Diffraction contrast from the core of the defect only is responsible for the image. Consequently, systematic analysis of a given crystal defect is possible at a resolution approaching that of the electron microscope in the specimen limiting case described in section 1.3. The technique seems likely to receive increasing emphasis in the future.

The major advantage of the WB approach over the two-beam dynamical technique lies in its improved resolution combined with the high contrast in the image. In comparison with direct lattice plane resolution, section 3.19, the WB technique has four advantages.

- (1) Ordinary electropolished thin foils containing a high density of defects may be used and relatively thick regions $2-4\xi_g$ may be used if maximum resolution is not required.

(2) The resolution attainable is experimentally controllable to that required for the particular problem under investigation.

(3) Since beam recombination is not required lens aberrations are not so important.

(4) The image has a very high level of contrast.

(5) The invisibility criteria $\mathbf{g} \cdot \mathbf{b} = 0$, $\mathbf{g} \cdot \mathbf{b}_e = 0$, $\mathbf{g} \cdot \mathbf{b} \wedge \mathbf{u} = 0$, see section 3.4.3.1, should be applicable at least in elastically isotropic materials (Cockayne, 1973).

The main disadvantages of the WB technique are as follows.

(1) The long exposure time required to record the image (10–30 s). Thus unusually good mechanical stability of the specimen is required for best resolution, that is almost zero specimen drift, etc.

(2) The resolution is not quite as high as the direct lattice resolution technique described in section 3.19.

Images are formed in CDF under kinematical conditions. The experimental procedure is outlined in section 1.5.3.

3.17.1 Image Characteristics

Most WB studies have involved dislocation images, although the technique is now being applied to small dislocation loops and precipitates. Based on the dislocation studies we find the following.

(1) The image lies close to the core of the strain field and accurately defines the position of the defect.

(2) The image consists of intense white regions, for example white line for dislocation, on a uniform black background, see figure 3.53(b).

(3) The image is very fine because only the high-strain region of the defect is imaged, for example dislocation images can have a half-width of $\sim 15 \text{ \AA}$ compared with $\sim \xi_g/3$ (80–200 $\text{\AA}$) for the dynamical two-beam image.

3.17.1.1 Dislocations

The essential features of the WB image of a dislocation may be understood qualitatively with reference to figure 3.52(a). Here an edge dislocation is drawn schematically such that the lattice planes are undistorted in levels I and III and all the distortion is concentrated in level II. Consider a situation in which the incident beam direction is some angle $\Delta\theta$, well away from the Bragg angle θ in layers I and III, that is s in figure 2.16 is large. It is only within level II that significant diffraction will occur and then only near the dislocation core where the rapidly varying strain field distorts the reflecting plane into the Bragg position (plane B). Clearly as s in the matrix increases the image width decreases. Furthermore, because significant

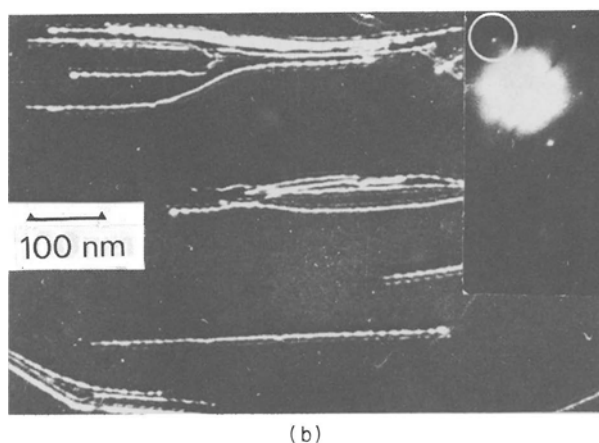
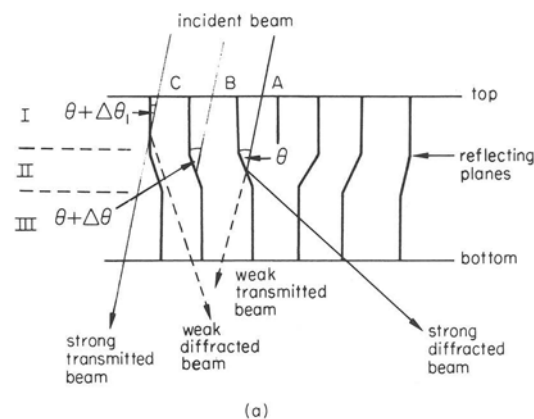


Figure 3.52 (a) A schematic diagram indicating that strong diffraction only occurs near the core of a dislocation under overall kinematical diffracting conditions. (b) A weak-beam (220) image of dislocations in silicon. Inset shows the diffracting conditions used to form the image [From Ray and Cockayne (1971b), courtesy of The Royal Society]

diffraction does not occur above or below the dislocation line in figure 3.52(a), the image width is independent of depth in the crystal and in dark field the image appears as an intense white line on a black background.

Clearly for a given crystal defect increasing the value of s in the matrix by tilting the specimen enables the experimentalist to control the image width to obtain the desired resolution. Figure 3.53(a) shows a conventional dynamical BF image of a dislocation while figure 3.53(b), (c) shows the improvement in resolution obtained with the WB image.

The physical model discussed above implies that the image will lie close to, but slightly displaced from, the dislocation core. Calculations by Cockayne (1972, 1973) show that, provided $|s| \gtrsim 2 \times 10^{-2} \text{ \AA}^{-1}$ and $|w| = |s\xi_g| \gtrsim 5$ and no other reflections are strongly excited, the image behaves as follows.

- (1) It is within $\sim 20 \text{ \AA}$ of the core.
- (2) It has a width at half peak height of $\sim 15 \text{ \AA}$.
- (3) It varies in position by $< \pm 5 \text{ \AA}$ with depth in the foil.

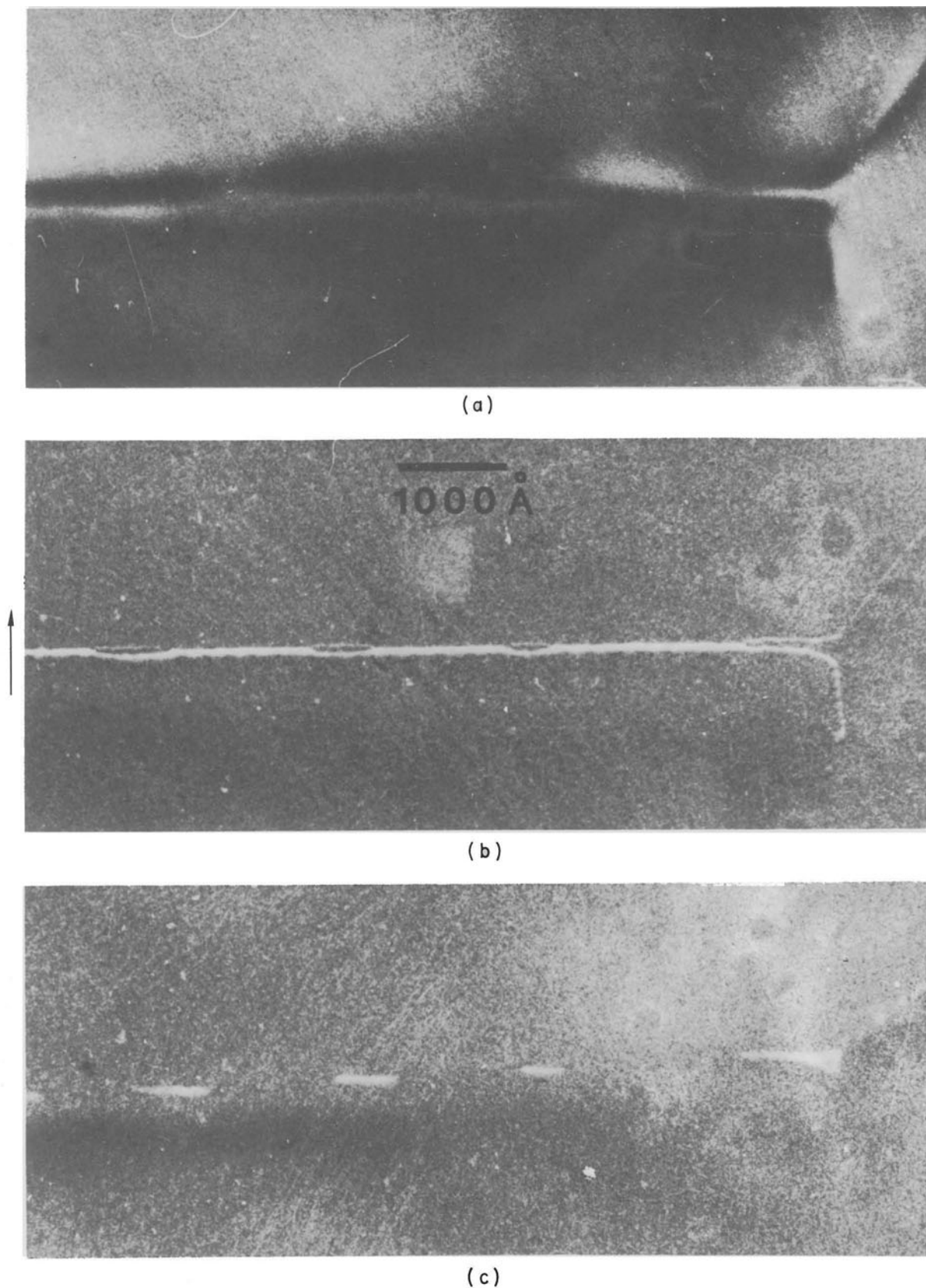
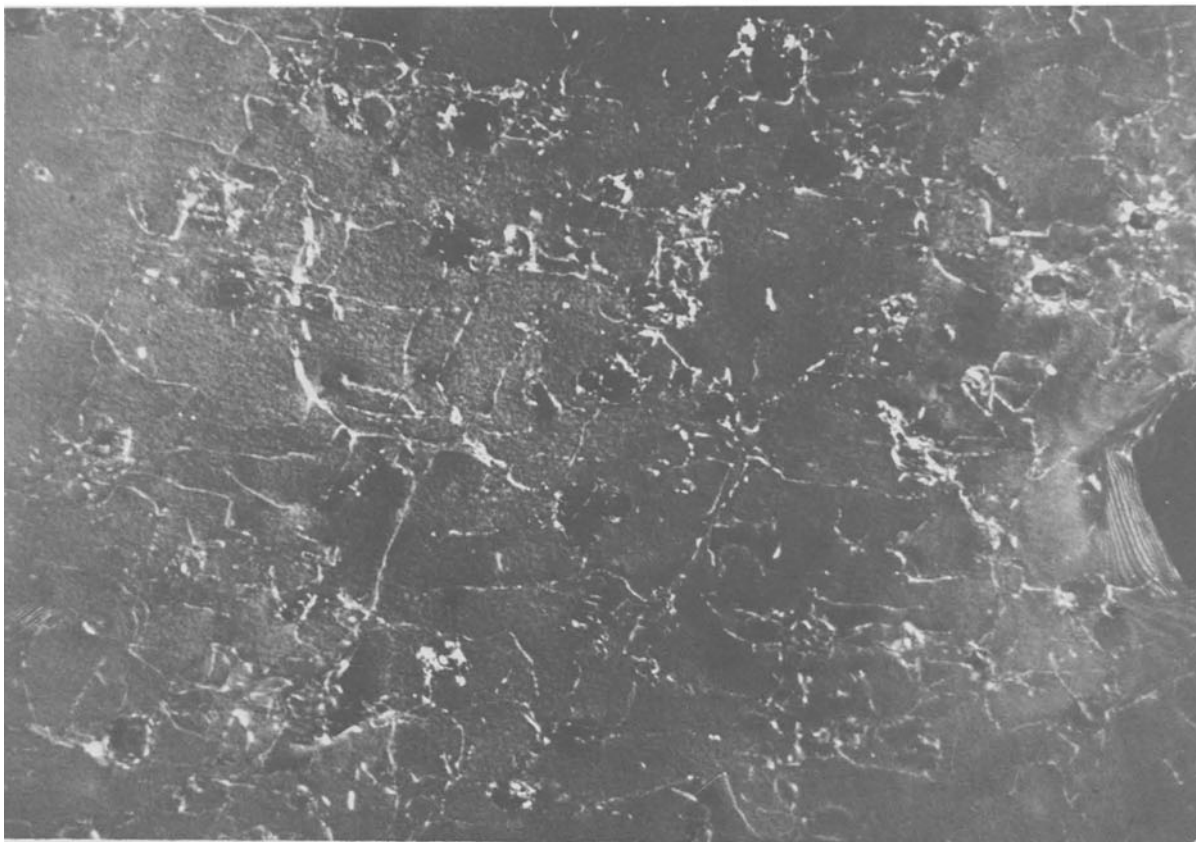


Figure 3.53 (a) BF $(2\bar{2}0)$ dynamical and (b), (c) CDF weak-beam images $g [2\bar{2}0]$ and $[11\bar{1}]$ respectively of a constricted dislocation in silicon. In (c) the stacking fault is visible whereas in (b) it is not. (d) BF and (e) CDF weak-beam images of deformed Al-4 wt % Cu containing θ' precipitates, (f) BF and (g) CDF weak-beam images of dislocation climb precipitation of TiC particles in an aged Ni-4.47 wt % Ti-1.96 wt % C alloy [(a)–(c) From Ray and Cockayne (1971b), courtesy of The Royal Society]



(d)



(e)

Figure 3.53 (continued)

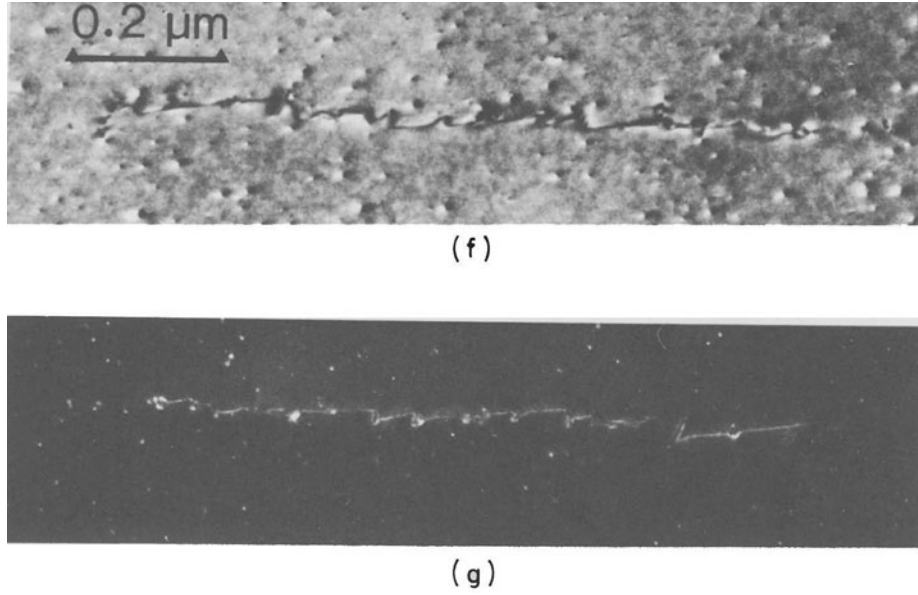
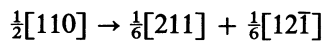


Figure 3.53 (continued)

However, the intensity of images of partial dislocations can vary markedly with depth and the image can apparently disappear, see figure 3.52(b).

From a practical standpoint, if WB images are to be used simply to increase resolution when a high density of defects is present, any suitable value of s may be chosen which gives the resolution required. However, if accurate measurements are to be made the criteria mentioned above *must* be satisfied.

Most theoretical and experimental studies have been performed on f.c.c. metals and alloys in which dislocations have dissociated into Shockley partials $\frac{1}{6}\langle 112 \rangle$ according to the reaction



The most convenient method of summarising the results is in terms of the value of $\mathbf{g} \cdot \mathbf{b}_{\text{total}}$ where $\mathbf{b}_{\text{total}}$ is the Burgers vector of the dislocation before dissociation.

(a) $\mathbf{g} \cdot \mathbf{b}_{\text{total}} = 2$. This case, where stacking fault contrast does not occur, has been of considerable interest because it is under these conditions that the most accurate value of partial dislocation separation may be obtained. Figure 3.53(b) shows a typical image for a reflection giving $\mathbf{g} \cdot \mathbf{b}_p = 1$ for both partials of a dislocation only dissociated over short distances. Provided the separation of the partials is $\geq 10 \text{ \AA}$ their individual images may be detected using microdensitometer traces across the image and it has been shown that the observed partial separation Δ_{obs} is related to actual partial separation Δ by

$$\Delta_{\text{obs}} = \left(\Delta^2 + \frac{4}{c^2} \right)^{1/2} \quad (3.13)$$

where

$$c = -s \frac{\mathbf{g} \cdot \mathbf{b}_p}{2} \left\{ 1 + \frac{1}{2(1 - \nu)} \right\} \quad (3.14)$$

assuming isotropic elasticity. Stobbs and Sworn (1971a) have concluded that, in copper, equation (3.13) is accurate to within $\pm 10\%$ and the effect of elastic anisotropy on Δ_{obs} is unimportant.

In general the quantitative measurement of partial separation is difficult below $\sim 60 \text{ \AA}$ and very difficult below $\sim 40 \text{ \AA}$. This arises from the long exposure entailed when large values of s are used to produce very fine images.

Finally, Stobbs and Sworn (1971a) have shown that, in copper, surface relaxation effects must be avoided by using only those dislocations lying at least $300 \text{ \AA}$ from the top and bottom surface. Consequently, foil thicknesses $> 600 \text{ \AA}$ must be used. Furthermore, there is an upper limit on the useful specimen thickness which is determined by exposure time and this depends upon both the material and the reflection chosen.

Situations for which $\mathbf{g} \cdot \mathbf{b}_p = 0$ also occur when $\mathbf{g} \cdot \mathbf{b}_{\text{total}} = 2$, depending on $\mathbf{b}_p$ and the operative reflection. In this case the partial dislocation should be invisible in elastically isotropic material. Only one study of the Burgers vector determination using dislocation invisibility in WB images has been reported (Ray and Cockayne, 1971b).

(b) $\mathbf{g} \cdot \mathbf{b}_{\text{total}} = 1$. Under these conditions stacking fault contrast is visible which serves as an important check that the dislocation observed under $\mathbf{g} \cdot \mathbf{b}_{\text{total}} = 2$ conditions is indeed dissociated, see figure 3.53(c). However, the fault energy may not be measured accurately under these conditions because one partial is usually in extremely weak contrast.

(c) $\mathbf{g} \cdot \mathbf{b}_{\text{total}} = 0$. Stobbs and Sworn (1971a) have reported that it was impossible to obtain good micrographs under these conditions in copper using a $\{111\}$ reflection because thin regions only could be studied because the high inelastic scattering associated with this reflection reduces penetration. Thus free surface effects complicate the image and w_{111} is low for $s_{333} \approx 0$ producing a broader image. (The normal imaging condition is with the Kikuchi line running close to $3\mathbf{g}$ and dark field on $\mathbf{g}$, see section 1.5.3.)

3.17.1.2 Dislocation loops

Preliminary results on perfect dislocation loops in molybdenum, similar to those discussed in section 3.5.5, have been reported by Perrin and Eyre (1973). It has been shown that $\mathbf{g} \cdot \mathbf{b} = 0$ images are difficult to interpret because they can be either weak or strong, sometimes being difficult to differentiate from in-contrast images with $\mathbf{g} \cdot \mathbf{b} = 1$ or 2. However, where $\mathbf{g} \cdot \mathbf{b} = 0$ images are strong the image coincides with the dislocation core and provides an accurate method of assessing loop size and shape. In cases where $\mathbf{g} \cdot \mathbf{b} = 2$ and $s \geq 2 \times 10^{-2} \text{ \AA}^{-1}$ without any strong reflections, reliable inside/outside contrast effects similar to those described in section 3.5.2 may be obtained down to loop diameters $\sim 50 \text{ \AA}$. However, it is very difficult to analyse the vacancy/interstitial nature of these loops using the approach outlined in section 3.5.2 because the loop habit plane cannot be determined accurately.

Recently Kelly and Blake (1973a, b) have reported a 'symmetric WB' imaging technique which enables the Burgers vector of dislocation loops in titanium, diameter $\gtrsim 200 \text{ \AA}$, to be determined from a single image obtained with $+\mathbf{g}$ and $-\mathbf{g}$ equally excited but with a large negative value of s . Under these conditions $|s|$ is $< 2 \times 10^{-2} \text{ \AA}^{-1}$ and medium-resolution images are obtained. WB images of prismatic loops in silicon have also enabled $\mathbf{b}$ to be obtained from a single image (Bicknell, 1973). Furthermore, inside/outside contrast experiments on large prismatic and Frank loops using the same approach outlined in section 3.5.2 have enabled their vacancy/interstitial sense to be determined (Guyot, 1973). Finally, Jenkins *et al.* (1973) have also obtained the sense of Frank loops $> 80 \text{ \AA}$ in diameter using WB images.

For loops of diameter $\lesssim 50 \text{ \AA}$ WB images suffer from similar difficulties in interpretation as conventional dynamical images discussed in section 3.5.6.

3.17.2 Applications

Because of the inherently high resolution, WB images have been used to ascertain the geometry

of small defects such as stacking fault tetrahedra (Cockayne *et al.*, 1972) and Frank loops (Jenkins *et al.*, 1973) and to resolve closely spaced dislocations (Nordlander and Thölén, 1973) and interface dislocations (Guyot, 1973). However, the major area of quantitative application has been in the study of dissociated dislocations and the measurement of stacking fault energy from the separation of partials. The materials investigated include gold (Jenkins, 1972), silver (Cockayne *et al.*, 1971a), copper (Cockayne *et al.*, 1971; Stobbs and Sworn, 1971a), copper–10 wt % aluminium alloy (Ray and Cockayne, 1971a) and silicon (Ray and Cockayne, 1971b; Cullis, 1973). In addition the APB energy of ordered alloys has been measured from the separation of dislocation pairs in iron–aluminium (Ray *et al.*, 1970; Crawford and Ray, 1970; Crawford *et al.*, 1973a, b), nickel–aluminium (Campany *et al.*, 1973) and tantalum carbide (Martin, 1973). In addition the geometry of dislocation loops in silicon has been studied (Bicknell, 1973). Finally, the Burgers vectors of dislocation loops may be obtained in irradiated zirconium (Kelly and Blake, 1973b).

In addition the technique should be useful for obtaining high-resolution images of most crystal defects with strain fields, as follows.

- (1) Improvement in resolution of complex dislocation arrays either in the matrix, see figure 3.53(d), (e), in some types of interface, figure 3.39, or in second-phase particles.
- (2) Accurate depth determinations using stereo techniques because the image is nearer the defect centre.
- (3) Determination of the size and shape of precipitates that cannot be imaged in precipitate DF, that is amorphous precipitates or those present in a low density. In this case, the image arises from the region of maximum strain, that is the interface. Thus the particle shapes could be built up using a number of different views of the precipitate with different $\mathbf{B}$ and the same or different operative reflections.
- (4) Observation of the details of the interaction of lattice dislocations with small precipitates, see figure 3.53(f), (g).
- (5) Detection of the loss of coherency of small precipitates.
- (6) Dislocation density measurements at high densities.

However, in view of the fact that, even for a dislocation, considerable care is required in performing quantitative WB experiments; quantitative interpretation of images in many of the above areas must await further image computation.

III. PHASE CONTRAST

3.18 Summary of Theory

Phase contrast effects become important when attempting to resolve image detail $\lesssim 10\text{--}15\text{ \AA}$ and their significance decreases steadily above this resolution limit. For phase contrast to be important some or all of the electrons scattered by the specimen must pass through the objective aperture and be recombined with the unscattered electrons to produce intensity differences from point to point in the image. A typical example would involve recombination of the transmitted and one diffracted beam as described in section 3.19.1 and shown in figure 3.55(b). Clearly the detail resolvable in a high-resolution phase contrast image will depend upon the phase changes introduced into the scattered electrons by the specimen. Theoretical treatments of phase shifts produced by the specimen have been given by a number of workers (Cowley, 1973a; Cowley and Moodie, 1957, 1970; Thon, 1969, 1971; Heinemann and Poppa, 1970; Howie, 1974; Hashimoto, 1974). However, in addition to phase shifts arising at the specimen, further phase shifts are introduced by the spherical aberration and defocus of the objective lens, the latter being deliberately introduced to enhance image contrast because there are no suitable phase plates for electron microscopes. These instrumental factors are discussed in section 1.5.5. In this section we are concerned with phase contrast arising at the specimen. Only the essential concepts behind the theory are discussed because of the many excellent treatments already available, see above. The aim of this section, in conjunction with section 1.5.5, is to provide the experimentalist with an understanding of the importance of those factors under his control and the limitations of image interpretation at present.

Phase contrast effects may be treated semi-qualitatively in the following way. Consider a monochromatic coherent parallel beam of electrons incident upon a very thin ($< 30\text{ \AA}$) electron microscope specimen. Most are transmitted, but some pass close to atoms and are deflected through an angle that depends upon both the atomic number and the closest distance of approach. Because of the phase differences produced by these scattering events, the electrons emerge with uniform amplitude but with phase variations over a plane surface, that is there is a corrugated surface of constant phase, as shown in figure 3.54(a). In calculating the phase shifts produced by the specimen an electron wave is considered to travel in the z direction through it, and to emerge at a point $(x\ y\ z_0)$ on the exit surface as shown in figure 3.54(b). Its accumulated

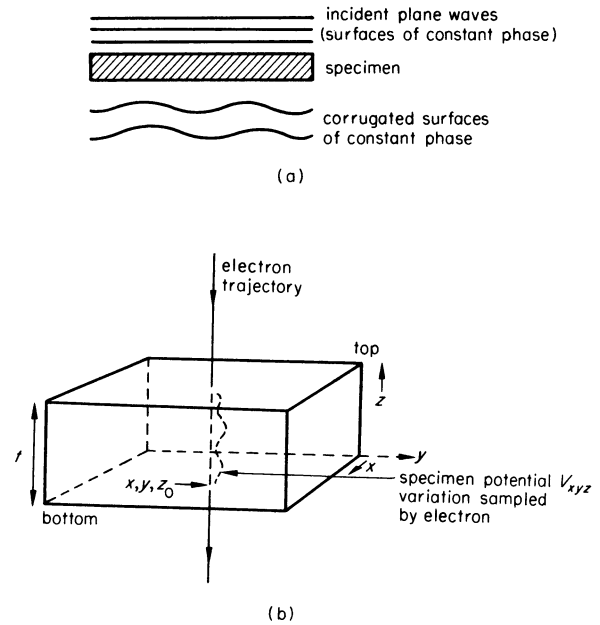


Figure 3.54 Schematic diagrams illustrating (a) the creation of phase differences by a thin transmitting specimen and (b) the influence of projected potential on the trajectory of the transmitted electron

phase shift is considered to be a result of its interaction with the projection of the specimen potential $V_{(xyz)}$ in the z direction. The wave function at the exit point is given by the weak phase grating approximation of the phase grating theory developed by Cowley and Moodie (1957) and is

$$\psi_{(x,y,z_0)} = \exp(2\pi i x z_0) \left\{ 1 + 2\pi i \frac{Me}{h^2 x} \int_0^{z_0=t} V_{(xyz)} dx \right\} \quad (3.15)$$

The following assumptions are implicit in this approach.

- (1) Only a small phase change $\ll \lambda$ occurs.
- (2) The specimen is so thin ($\lesssim 30\text{ \AA}$) that scattering does not produce significant deviation of the path of the electron from the z axis in figure 3.54(b).
- (3) A single scattering event only occurs.

The concepts embodied in equation (3.15) may be used as a basis for the weak-phase grating treatment of n -beam lattice images, section 3.19.2 (Allpress *et al.*, 1972), dark field images from diffuse regions of diffraction patterns (Cowley, 1973a, b), section 3.22.

The above treatment ignores possible energy losses arising from the inelastic scattering events discussed in section 2.6.3. Because inelastic scattering is incoherent, it degrades the phase contrast

image. Thus phase contrast information from thick specimens is lost.

When equation (3.15) is generalised to the case of a perfect crystal, the description of the wave function $\psi_{(r)}$ of the electron is

$$\psi_{(r)} = \psi_{(x,y,z_0)} = \sum_g \phi_g \exp\{2\pi i(x + g + s)r\} \quad (3.16)$$

where

$$\phi_0 = 1 + \frac{\pi i t}{\xi_0} \quad (3.17)$$

$$\phi_g = \frac{\pi i t}{\xi_g} \exp(-\pi i t s) \frac{\sin(\pi t s)}{\pi s} \quad (3.18)$$

which corresponds to the Darwin–Howie–Whelan equations (3.1) re-written in terms of an electron wave function, see Hirsch *et al.* (1967). Equation (3.16) may be used as the basis for a treatment of direct resolution of lattice planes, see section 3.19, and Moiré patterns, see section 3.21.

The scattering theory summarised above is adequate for high-resolution studies of materials except for the case of highly disordered specimens, for example amorphous materials which are discussed in sections 3.22, 4.15.2, 4.16.2.

From the above discussion it is clear that the phase contrast image is sensitive to point-to-point variations in the projected specimen potential rather than the atomic displacements responsible for diffraction contrast discussed in parts I and II. Consequently, the detailed interpretation of phase contrast images in terms of the microstructure of the specimen is not simple, even for very thin specimens when overlap effects are minimised and the projected potential is relatively simply related to the structure of the specimen. Furthermore, allowance must be made for the influence of both spherical aberration and defocus of the objective lens on image detail, as pointed out in section 1.5.5. Thus theory describing the relationship between specimen microstructure and phase contrast images is not as well developed as for diffraction contrast.

IV. APPLICATIONS OF PHASE CONTRAST TO STUDIES OF CRYSTALS

Phase contrast images such as those described in the following sections cannot be simply related to the specimen structure except in a few cases. However, the weak-phase grating theory described in part III may be used to relate image detail to the structure of the specimen provided a critical value of objective lens defocus is used (see section 1.5.5) which frequently corresponds to maximum contrast. Nevertheless, detailed calculations are almost always necessary before satisfactory interpretation of images is possible.

Four areas in which phase contrast effects are important are illustrated in the following sections.

3.19 Lattice Plane Resolution (Two- or Three-beam Images)

3.19.1 The Perfect Crystal

These images of crystalline specimens are generally formed by recombining one or two diffracted beams with the transmitted beam.

A periodic fringe image is produced as a result of phase interference between these beams. Under certain well-defined conditions the spacing of the fringe image corresponds to that of the reflecting planes producing the diffracted beam.

The dynamical theory of lattice plane resolution has been discussed by Cowley and Moodie (1970), Hashimoto *et al.* (1961), Heidenreich (1964) and Amelinckx (1964) for the case of the transmitted beam lying on the optic axis. However, the dynamical case for tilted illumination (section 1.5.5) has

not been discussed, although both Komoda (1964) and Howie (1974) have given a kinematical treatment.

The main conclusions of the dynamical treatments may be illustrated with the following approximate approach. Since both the transmitted and diffracted beams contribute to the image, the wave function $\psi_{(r)}$ describing their combined effects within the column illustrated in figure 3.1 must be considered. Thus, re-writing equation (3.16) for two beams we have

$$\psi_{(r)} = \phi_0(z) \exp(2\pi i \chi \cdot r) + \phi_g(z) \exp(2\pi i \chi' \cdot r) \quad (3.19)$$

which is the wave function representation of equations (3.1) where $\chi' = \chi + g + s = \chi + g'$, see figure 2.16. If the incident wave has unit amplitude, we have

$$\psi_{(r)} = \exp(2\pi i \chi \cdot r) \{1 + \phi_g \exp(2\pi i g' \cdot r)\} \quad (3.20)$$

and putting $\phi_g = I \exp(i\delta)$, we obtain for the total intensity distribution in the direction of g' in the image

$$I = 1 + R^2 + 2R \cos(2\pi g' \cdot r + \delta) \quad (3.21)$$

It follows from equation (3.18) for ϕ_g that

$$R = \frac{\pi}{\xi_g} \frac{\sin(\pi t s)}{\pi s} \quad \text{and} \quad \delta = \frac{\pi}{2} - \pi t s \quad (3.22)$$

Since g' is nearly normal to the incident beam, a coordinate x may be chosen parallel to this

direction to give

$$I = 1 + R^2 - 2R \sin(2\pi g x - \pi s t) \quad (3.23)$$

Now for a given thickness t , and s , this expression gives a modulated sinusoidal intensity in the x direction with periodicity d the interplanar spacing, as shown in figure 3.55(a). Consequently, the lattice is 'resolved', and the fringes run perpendicular to g , see figure 3.55(a), (b).

From a practical standpoint the following factors are important.

(1) The visibility of the fringes depends upon both specimen thickness and deviation from the Bragg position. Thus if $\pi s t = n\pi$ then $R = 0$ in equations (3.22) and (3.23) and there are no fringes.

(2) The fringes do not coincide with the atomic planes. For example, if we consider lattice fringes perpendicular to and near an edge where the thickness t varies, the value of x at which the minima in the sine curve occurs will change and fringes will be curved. Furthermore, as the thickness varies black fringes will be replaced by bright ones. If the fringes are parallel to the edge, then t varies with x and the spacing of the fringes changes from d to some value depending upon the local variation of t and x .

These effects have been reported by Menter (1958) and Hashimoto and Mannami (1962).

(3) There will be a fringe shift with no change in periodicity if $s \neq 0$ because the term $\pi s t$ in equation (3.23) can be considered to be an additional phase shift.

(4) An additional image shift arises from objective lens spherical aberration and defocus, see section 1.5.5. There is no change in periodicity.

Clearly, if two-beam fringe images are formed with the transmitted beam on the optic axis, detailed interpretation of the image will be difficult, although the fringe spacing provides an estimate of instrument resolution, see section 1.12.

If axial illumination, section 1.5.5, is used, chromatic aberration effects (which destroy contrast) are minimised and the attainment of high-resolution fringe images is easier.

If three beams are allowed to contribute to the image, that is $\pm g$ and the transmitted beam lying on the optic axis, fringes corresponding to the lattice and half lattice spacing occur, although in practice the latter are too faint to be observed. This approach has the advantage that the position of the main fringes is independent of crystal thickness but the contrast depends upon defocus, being a maximum $\Delta f = 4C_s \theta^2$, where θ is the Bragg angle.

3.19.2 Crystal Imperfections

Because this technique provides high-resolution images with spacing approximately that of the reflecting planes, a number of workers have attempted to use it to study the fine details of crystal defects. Detailed studies of dislocations have been reported by Parsons and Hoelke (1969, 1970), Parsons *et al.* (1968) and Phillips and Hugo (1970). A typical image of a dislocation running normal to the foil surface and parallel to the electron beam is shown in figure 3.55(c). The distortion of the reflecting planes near the dislocation core may be clearly seen. However, detailed interpretation of such images, assuming a one-to-one correspondence between fringes and lattice planes is not in general possible (Cockayne *et al.*, 1971b). In particular terminating fringes are not to be identified with terminating lattice planes. There is only one case (Phillips, 1972) in which it is possible that correspondence of fringes and lattice planes may hold, that is when a dislocation is parallel to the foil normal and B .

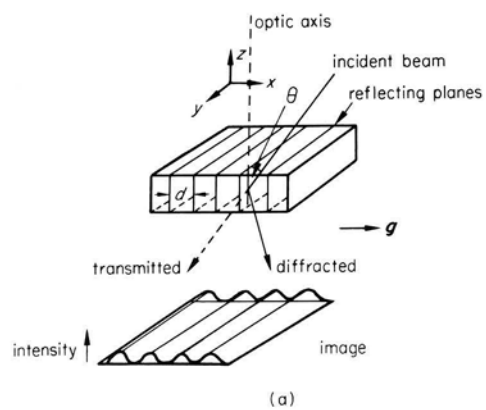
It has been reported by Cockayne *et al.* (1971b) that the Burgers vector of a dislocation may be obtained by performing a Burgers circuit around the dislocation and algebraically adding terminating fringes, giving those above and below the dislocation line positive and negative signs. Then $N = g \cdot b$, where N is the algebraic sum of the terminating fringes. However, it is not clear whether this rule applies if there is not a one-to-one correspondence of fringes and reflecting planes. However, dislocations are invisible when $g \cdot b = 0$.

This technique has also been applied to studies of the lattice distortion near GP zones in Cu-Be (Phillips and Tanner, 1973) and Al-Cu (Parsons *et al.*, 1970; Phillips, 1973). Figure 3.55(d) is a two-beam axial illumination image of the $\{002\}$ lattice planes at a GP zone in Al-3% Cu. The zone is four planes thick and 110 Å in diameter. Dashed lines show the points at which measurements of the distortion of the matrix planes were made giving rise to the reconstructed diagram in figure 3.55(e).

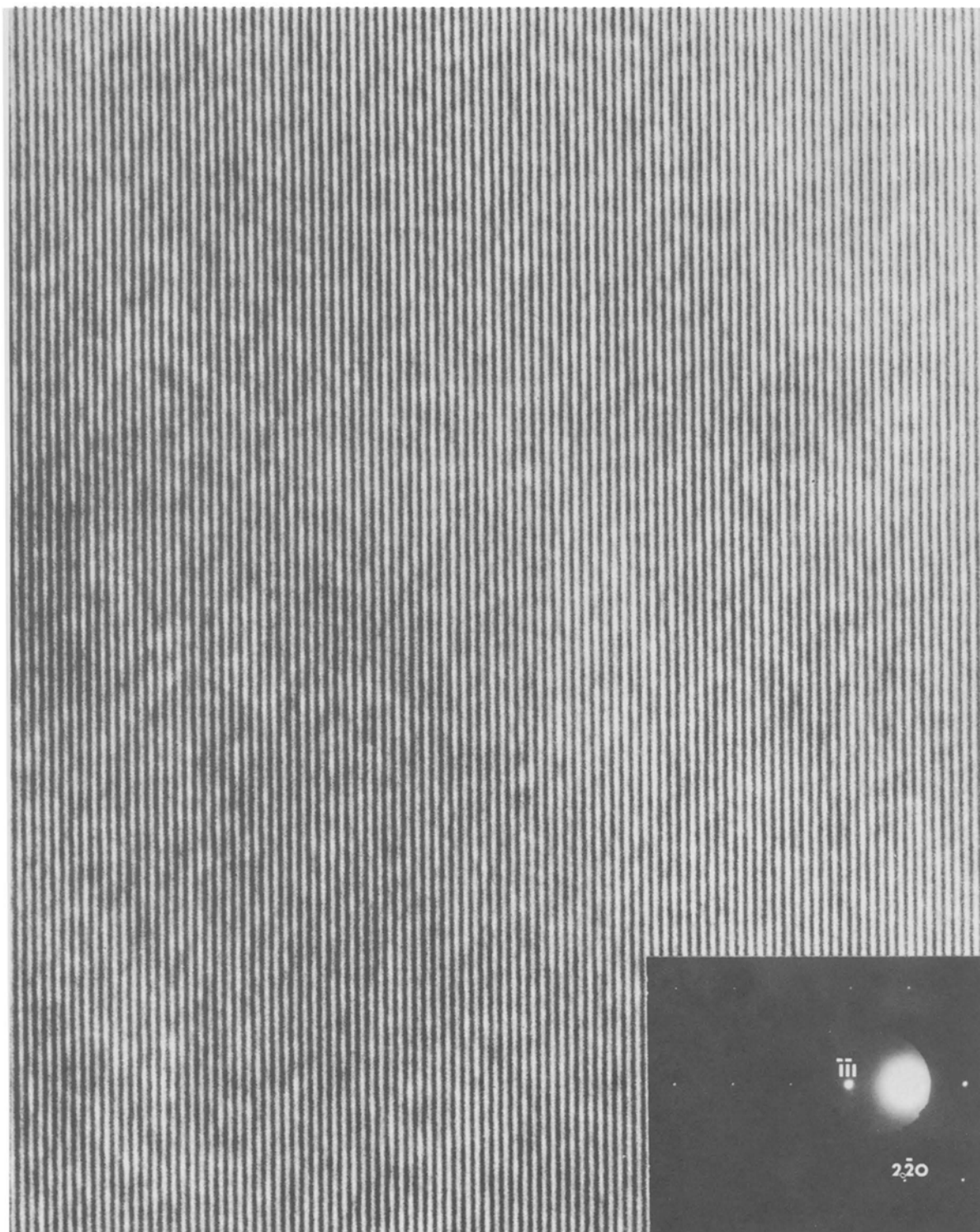
Lattice images can provide useful information in specific cases as described above. However, for many purposes where the interpretation of the details of the image is at present uncertain, such as dislocations, weak-beam images may prove to be more useful, particularly since the instrument operation conditions are not so stringent.

3.20 Multi-beam Lattice Images

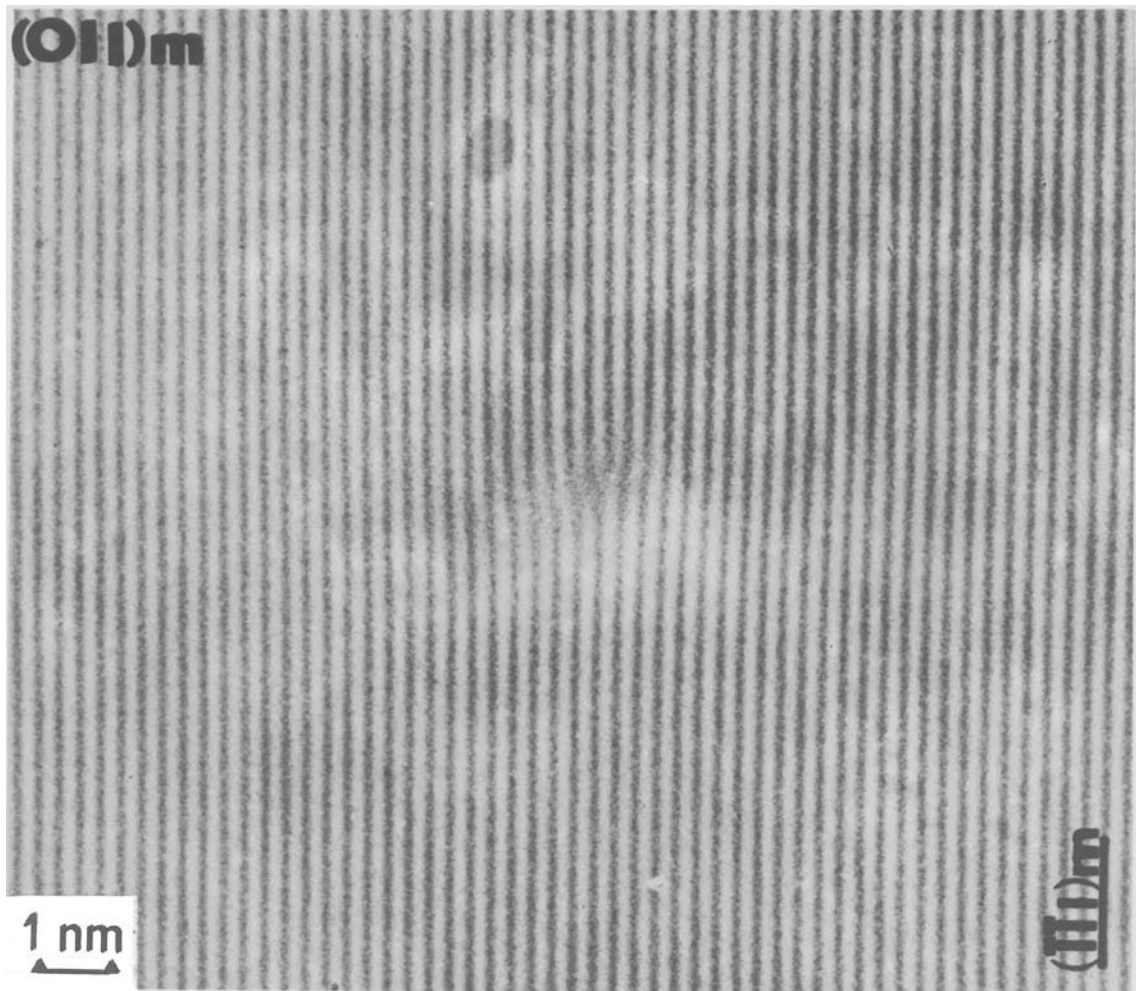
When several diffracted beams occur either a row or an array of diffracted beams may be recombined with the transmitted beam as shown in figure 3.56(a). In each case the transmitted beam lies on the optic



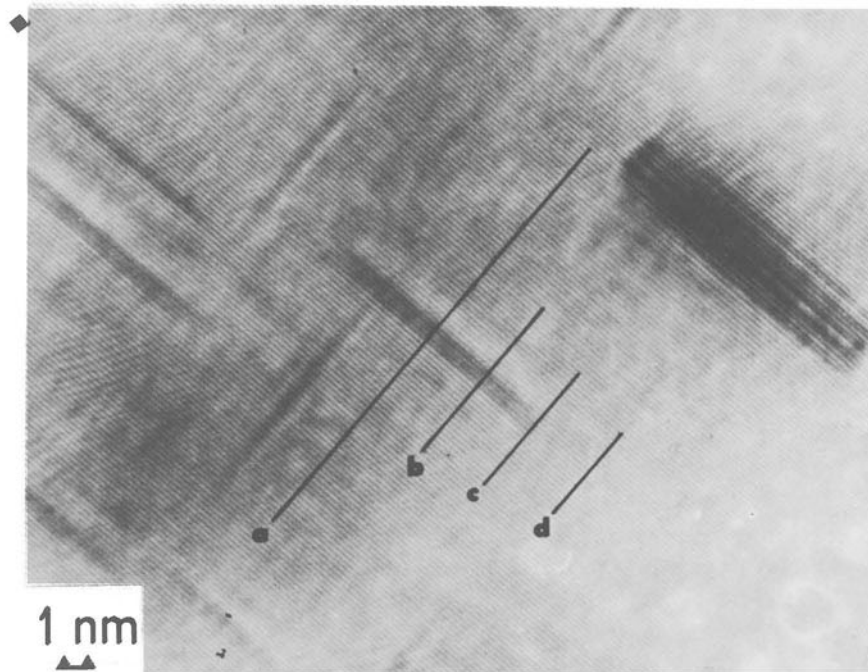
(a)



(b)



(c)



(d)

Figure 3.55 (a) A schematic diagram illustrating the image intensity parallel to the reflecting planes when the transmitted and diffracted beams are recombined to form the image. (b) A (111) lattice image in silicon, fringe spacing $3.138 \text{ \AA}$, tilted illumination. (c) A (111) lattice image in silicon, interpreted as a dislocation $b = \frac{1}{2}[10\bar{1}]$ or $\frac{1}{2}[110]$. (d) An (002) lattice image in Al-3 wt % Cu showing a four-plane thick, $110 \text{ \AA}$ diameter, GP zone. Nominal fringe spacing $2.024 \text{ \AA}$. Dashed lines show strain field measurement positions. (e) Schematic representation of fringe pattern at zone (shaded) in (d) above. The fringe displacement is magnified by 5 and the zone length contracted by a factor of 14. The zone boundary is shown by the heavy broken line. The matrix {002} plane positions are indicated by light broken lines [(b) From Phillips and Hugo (1970), (c) from Phillips (1972), (d), (e) from Phillips (1973), courtesy of *Acta Metallurgica*]

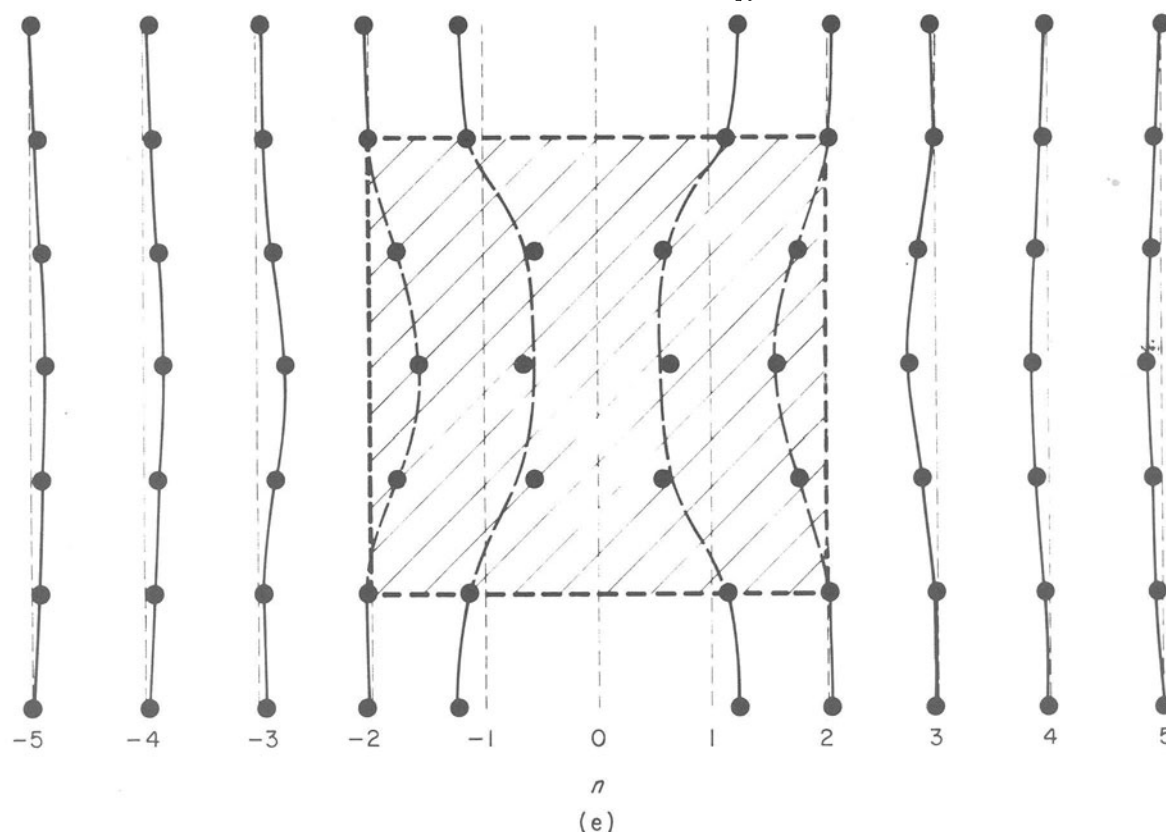


Figure 3.55 (continued)

axis and the diffracted beams included within the objective aperture (outlined) are symmetrically disposed about it. Increased detail is present in the two-dimensional images formed from an array of diffraction spots, see figure 3.56(b), and a more detailed interpretation of the structure is possible (Cowley and Iijima, 1972; Allpress and Sanders, 1972).

Figure 3.56(a) is taken from $W_4Nb_{26}O_{77}$ and is interpreted in terms of the black lines corresponding to crystallographic shear planes in the material for which the spacing corresponds to a particular ordered form of the structure. Thus the regions Z and Y of figure 3.56(a) correspond to two different ordered forms of $W_4Nb_{26}O_{77}$, that is $W_3Nb_{14}O_{44}$ and $WNb_{12}O_{33}$ respectively whereas at X ordered intergrowth to form $W_4Nb_{26}O_{77}$ has occurred. Figure 3.56(b) is a specimen of $TiNb_{52}O_{132}$ after quenching from near the melting point. Most of the field contains $TiNb_{24}O_{62}$ labelled Y but there are several complex extended defects marked by arrows. Occasionally linear portions such as T are identifiable as slabs of HNb_2O_5 . Further details of interpretation may be found in the paper by Allpress and Sanders (1972).

Allpress *et al.* (1972) have discussed the influence of lens defects including spherical aberration on the image and conclude that the effects are small, provided the Bragg angles for the diffracted beams

are very small, interplanar spacing ≥ 5 Å. However, defocussing effects can destroy image detail because of beam divergence. It has been found that experimentally and theoretically the most useful lattice images for study of microdomains in oxide were obtained at small amounts of underfocus which produced maximum contrast and were close to the Scherzer optimum defocus (section 1.5.5).

In attempting to interpret the images in terms of the fine-scale defect structure of the material for the case of oxides, computer image matching techniques must be used. In this area the approach represents a powerful technique for direct observation of fine scale crystal defects on a unit cell level.

3.21 Moiré Patterns

Moiré patterns occur in electron microscope images when overlap occurs between two crystals with almost exactly equal or multiple lattice parameters. Under these circumstances Moiré patterns form if a beam diffracted by both crystals is allowed to recombine with the transmitted beam to form the final image (Dowell, 1962; Bassett *et al.*, 1958). Thus double diffraction described in section 2.16.1 is an essential prerequisite for Moiré pattern formation.

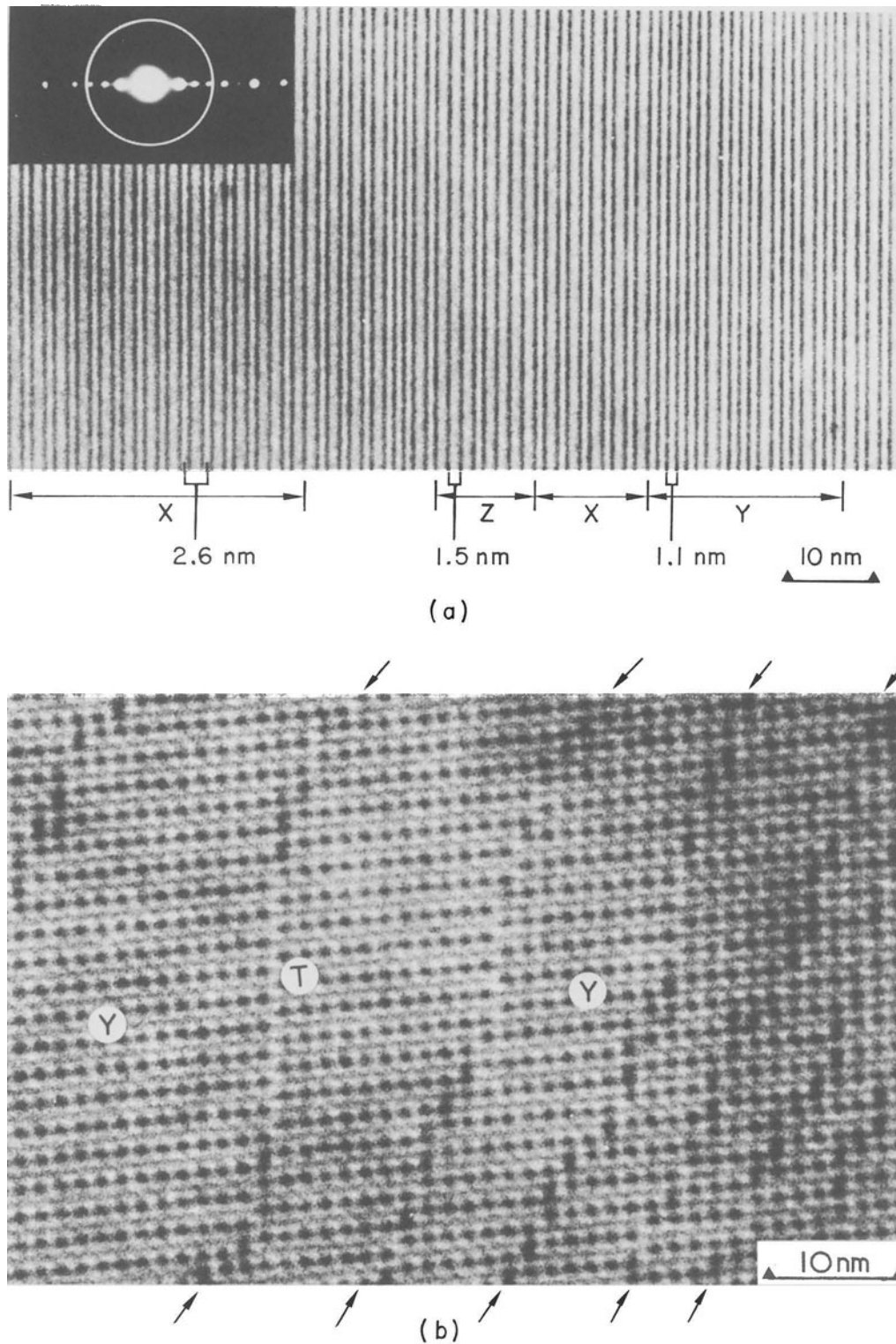


Figure 3.56 (a) A one-dimensional lattice image from a fragment of $W_4Nb_{26}O_{77}$ oriented so that the $\{00l\}$ systematic reflections appear in the diffraction pattern (inset). The image contains white bands 15 Å and 11 Å wide, corresponding to slabs of $W_3Nb_{14}O_{44}$ and $WNb_{12}O_{33}$ respectively. The regions Y and Z contain microdomains of these parent structures and at X ordered intergrowth to form $W_4Nb_{26}O_{77}$ has occurred. (b) A two-dimensional lattice image from a quenched fragment of nominal composition $TiNb_{52}O_{132}$. Most of the field contains undisturbed $TiNb_{24}O_{62}$ labelled Y, but several extended defects, marked by black arrows, wander across the fragment. Occasionally, linear portions occur (for example at T), which are identifiable as slabs of $H-Nb_2O_5$ [From Allpress and Sanders (1972), courtesy of University of California Press]

Figure 3.57(a) shows the most common experimental situation in which such effects occur. The thin foil contains a plate semi-coherent precipitate satisfying the above conditions, and the same reflection is excited in both crystals, although the reciprocal lattice vector changes by $\Delta\mathbf{g}$ on passing through the interface. If a vector $\mathbf{r}_n$ would describe a matrix atom position were the precipitate absent, a vector $\mathbf{r}_n + \mathbf{R}_n$ must be used to take account of the actual atom position in the matrix because of the strain introduced by the precipitate. Thus there will be a phase change $\alpha = 2\pi\mathbf{g} \cdot \mathbf{R}_n$, see equation (3.1), where $\mathbf{R}_n$ will depend on the displacement vector $\mathbf{r}_s$ in the interface. However, because $\mathbf{g} \cdot \mathbf{r}_n$ is an integer, we may write for small quantities

$$\alpha = 2\pi\mathbf{g} \cdot \mathbf{R}_n = 2\pi\Delta\mathbf{g} \cdot \mathbf{r}_n = 2\pi\Delta\mathbf{g} \cdot \mathbf{r}_s \quad (3.24)$$

because $\Delta\mathbf{g}$ is usually parallel to the interface. Thus α is a linear function of position in the interface and the image consists of fringes whose direction is given by $\Delta\mathbf{g} \cdot \mathbf{r}_s = \text{constant}$. This is the equation of a straight line perpendicular to $\Delta\mathbf{g}$, that is the fringes run perpendicular to $\Delta\mathbf{g}$.

In effect, in forming Moiré patterns we recombine the transmitted and double diffracted beams in the image, that is the objective aperture would admit beams T and $\mathbf{D}_3$, figure 2.57(e), for two-beam conditions in both crystals.

The dynamical theory of Moiré pattern formation has been formulated by Gevers (1962, 1963) and Hashimoto *et al.* (1961). References should also be made to papers by Menter (1958), Bassett *et al.* (1958) and Hashimoto and Uyeda (1957) for the detailed theoretical background. In practice, however, the main features of the Moiré patterns may be understood using the optical analogue of diffraction gratings. Parallel or rotational patterns may occur.

3.21.1 Parallel

Both crystals have parallel reflecting planes but different lattice parameters. Using the analogue of two line gratings a structure of larger periodicity is produced, see figure 3.57(b). Beats are formed between these two periodicities with spacing D given by

$$D = \frac{d_1 d_2}{d_1 - d_2} \quad (3.25)$$

where d_1 and d_2 are the spacings of each grating. Typically, Moiré patterns with spacings 5–500 Å can be produced in the electron microscope depending upon the combination of d spacings for the operative reflections. Also shown schematically in figure 3.57(b) are the positions of the spots in the diffraction pattern for both crystals, together

with the objective aperture. The Moiré fringes are parallel to the reflecting planes and perpendicular to $\Delta\mathbf{g}$ as pointed out above.

3.21.2 Rotational

In this case, figure 3.57(c), the two gratings have identical spacings but a slight angular rotation β . The lines in the Moiré pattern are then approximately perpendicular to the lines of the gratings (perpendicular to $\Delta\mathbf{g}$) and have a spacing given by

$$D = \frac{d}{2 \sin(\beta/2)} \approx \frac{d}{\beta} \quad (3.26)$$

3.21.3 Mixed

Overlapping gratings that are rotated relative to one another and have different spacings from mixed Moiré patterns and have a spacing

$$D \approx \frac{d_1 d_2}{\{(d_1 - d_2)^2 + d_1 d_2 \beta^2\}^{1/2}} \quad (3.27)$$

It can be shown that, if the grating with the smaller spacing is rotated, the Moiré fringe pattern rotates in the same sense but, if the larger spacing grating is rotated, the Moiré pattern rotates in the opposite sense.

3.21.4 Magnification

The magnification M of parallel Moiré patterns is $d/(d_1 - d_2)$ where $d = (d_1 + d_2)/2$. For rotational Moiré patterns $M = 1/\beta$.

3.21.5 Summary

The main features of Moiré patterns are as follows.

(1) The fringes always run perpendicular to $\Delta\mathbf{g}$, the line joining the operative $\mathbf{g}$ vectors for the two crystals, see figure 3.57(b), (c). Consequently, for a parallel Moiré the fringes run perpendicular to $\mathbf{g}$ but for a rotation Moiré the fringes are parallel to $\mathbf{g}$, see figure 3.57(b), (c). Thus parallel and rotational Moiré patterns can be clearly distinguished.

(2) Anomalous absorption does not influence fringe spacing or direction but the contrast is reduced in thick crystals (Gevers, 1962).

(3) The fringes change continuously in intensity, contrast and position when the specimen is tilted or bent.

(4) Fringe positions and directions are unaffected by surface steps if they are an integral number of ξ_g . However, in all other cases fringes may be drastically affected and may stop at surface steps.

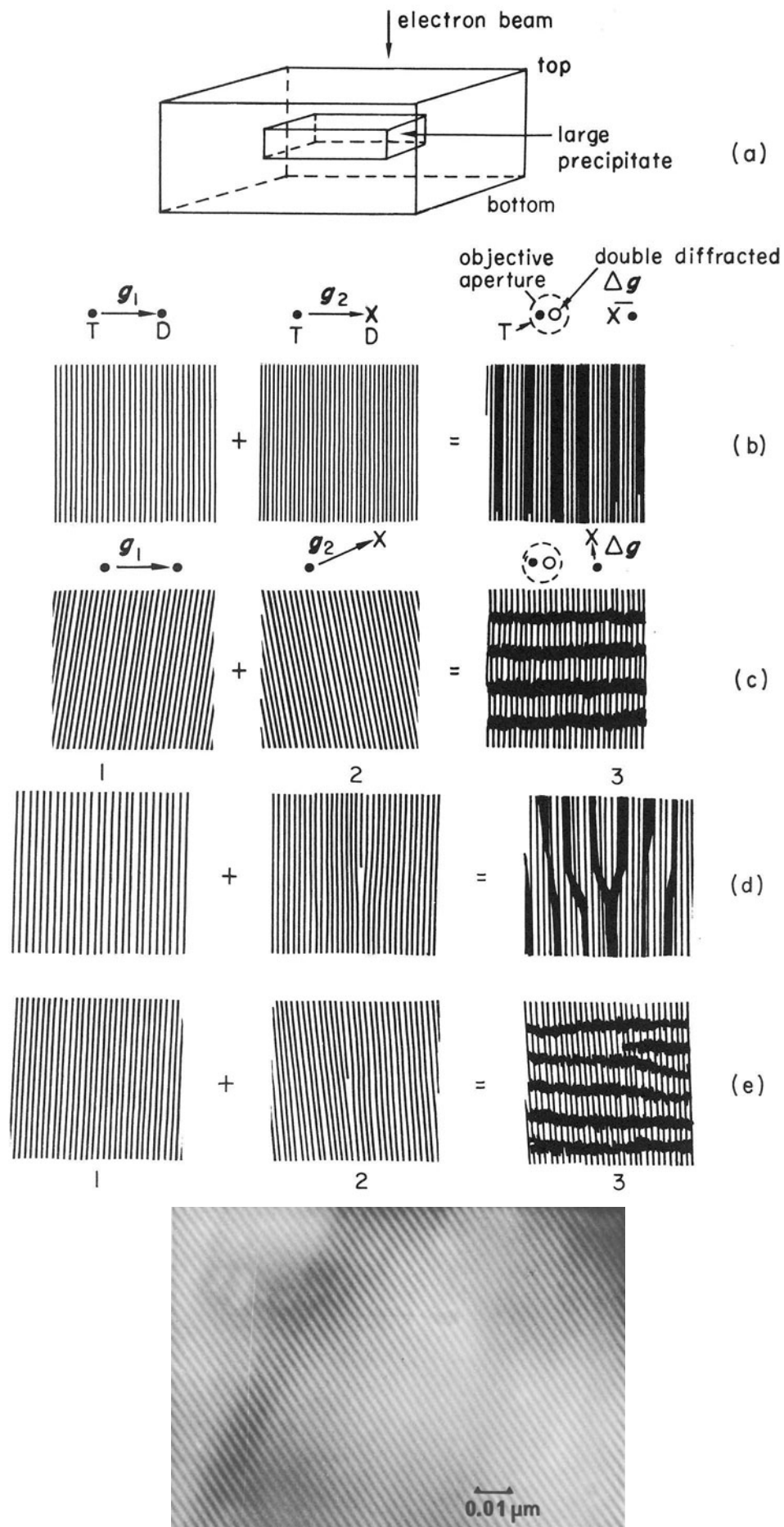


Figure 3.57 (a) A schematic diagram illustrating a large precipitate lying parallel to the foil surface. Optical analogues demonstrating the formation of (b) parallel and (c) rotational Moiré patterns by two overlapping line gratings. Diffraction patterns are also shown. Similar optical analogues showing the influence of dislocations on (d) parallel and (e) rotational Moirés. (f) A rotational Moiré pattern from overlapping graphite flakes

3.21.6 Applications

Moiré patterns have been used to make visible crystal defects such as dislocations and stacking faults because they are, in effect, a magnified image of the reflecting planes. The optical analogues in figure 3.57(d), (e) show the image to be expected from an edge dislocation for both parallel and rotational Moirés. Menter (1958) and Bassett *et al.* (1958) have reviewed the application of Moiré patterns to defect studies. However, the technique has not gained significant support in defect studies because of its limited range of application.

3.21.7 Detection

Moiré patterns formed at precipitate–matrix interfaces often confuse the interpretation of interface effects such as dislocation arrays. This may be important, for example in analysing the Burgers vector of interface dislocations when there may be a close geometrical correspondence between the Moiré pattern and the dislocation network (Pumphrey and Edington, 1974). For very fine networks, with dislocation spacing $\sim 0.3\text{--}0.5\xi_g$ (Thölén, 1970) has shown the dislocations are not resolvable using conventional two-beam techniques. A simple Moiré fringe pattern occurs independent of the presence or absence of the dislocations in the interface and of the value of g or s . Thus the form of the image cannot be used to determine the nature of the structure of the interface and alternative indirect methods must be used.

If the dislocation spacing is $>0.3\text{--}0.5\xi_g$ the network may be resolved under conventional two-beam dynamical conditions. The most useful technique available to prove it is a network is still the weak-beam approach described in section 3.17 using a series of different reflections. The methods suggested (Hirsch *et al.*, 1967) that is changing the direction of g , and obtaining images in g and $2g$ are not satisfactory.

Note that the minimum mesh-size spacing for which the weak-beam approach is applicable has not yet been defined.

3.22 Dark Field Images of Short-range Ordered (SRO) or Amorphous Materials

Material exhibiting SRO frequently gives rise to diffuse scattering effects in SADP such as those discussed in section 2.19.2, and shown in figure 2.71. When domains are small $\lesssim 10\text{--}15\text{ Å}$, CDF images formed with regions of diffuse scattering consist of white dots, but phase contrast effects are important such that the image may not be simply related to the object (Cowley, 1973a, b). A typical

image is shown in figure 3.58(d). In general, interpretation of images in the above size range may not be based on the assumption that a white dot corresponds to a single ordered microdomain, oriented such that it gives rise to a diffuse diffraction spot within the objective aperture. Such specimens should be treated as phase objects (Cowley 1973a, b) with a point-to-point variation of potential of the type shown in figure 3.58(b). This treatment is a developed form of the approach embodied in equation (3.15). There are two important complicating factors in interpreting the image.

(1) The image is determined by the form of the deviations of the potential from average *but* both positive and negative deviations give intensity maxima. Thus a vacancy or a heavy atom in a light-atom matrix would *both* produce a bright spot, and not be distinguished in the image.

(2) The image consists in practice of superimposed potentials of many ordered domains for specimens of significant thickness. Consequently, interpretation of image detail in terms of specific domains is very difficult unless the specimen is very thin.

The following example highlights some of the problems involved. Figure 3.58(a) shows an electron diffraction pattern taken from LiFeO_2 with $B\langle 100 \rangle$ and containing diffuse scattering. CDF images obtained with regions A, B, figure 3.58(a), are shown in figure 3.58(d), (e).

The SADP is consistent with SRO regions having the structure along the $\langle 100 \rangle$ axes shown in figure 3.58(b) such that the projected potential has the form shown, that is regions of positive and negative deviations from the average potential. The resulting DF intensity distribution along $\langle 100 \rangle$ is shown in figure 3.58(c); note that both positive and negative deviations from average produce white spots. Thus one expects rows of white spots spacing 4 Å , the lattice parameter. Such rows are in fact visible in figure 3.58(d) but in figure 3.58(e) the rows of spots have spacing 4 Å while within the row the spacing is $\sim 2.9\text{ Å}$ not 4 Å . There is no quantitative explanation for this latter effect at present.

3.23 Magnetic Domain Boundaries (Lorentz Microscopy)

Observation of magnetic domain boundaries involves phase contrast electron microscopy, although high resolution is not in general required. Magnetic domain boundaries separate regions (domains) of a ferromagnetic material that are magnetised in different directions. A ferromagnetic specimen that has been completely demagnetised in the absence of a magnetic field consists of

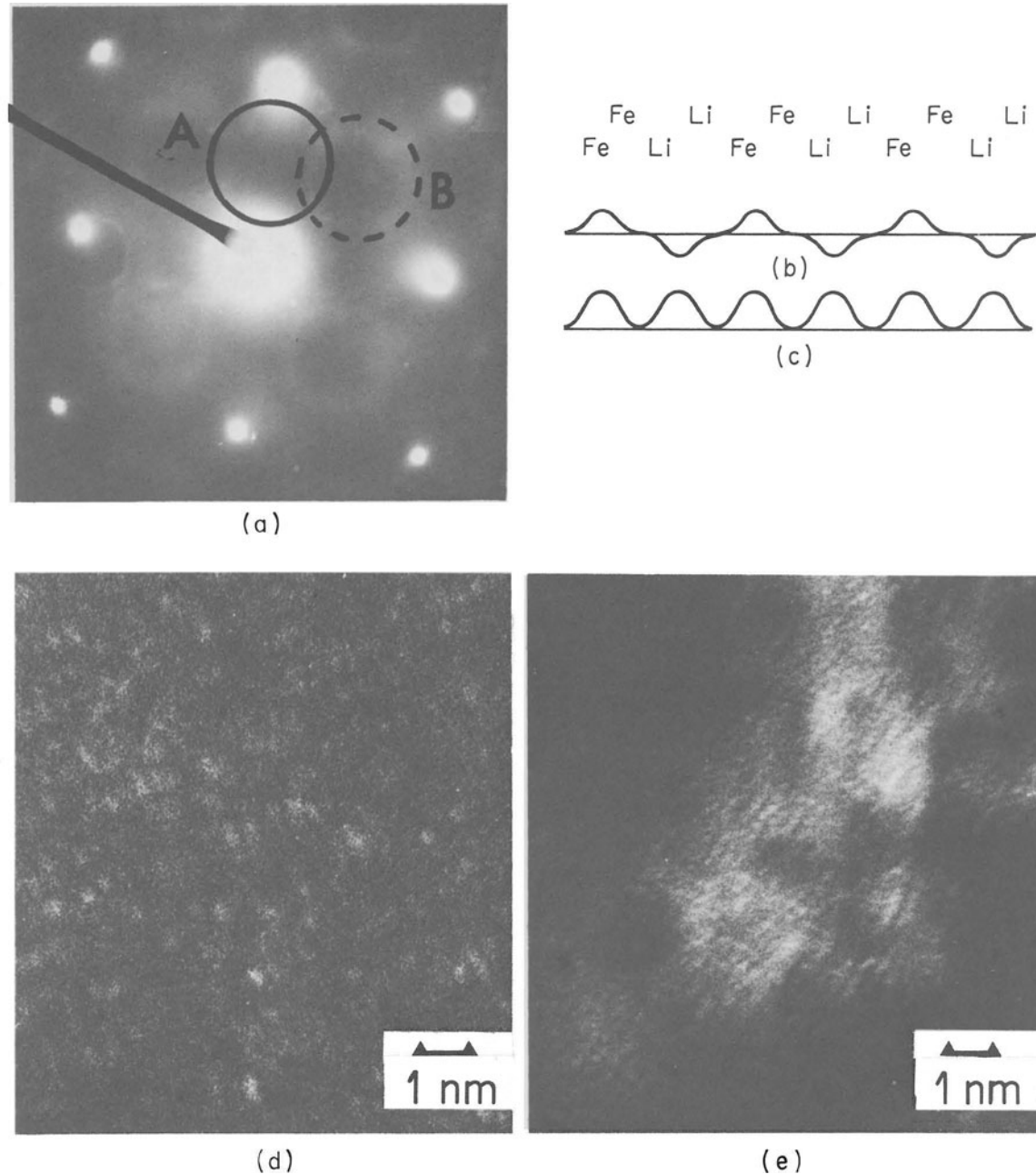


Figure 3.58 SADP of a LiFeO_2 specimen $B = \langle 100 \rangle$ showing diffuse scattering: (b) the possible configuration of Li and Fe ions giving rise to the potential distribution shown and (c) the corresponding CDF intensity distribution, (d) CDF image obtained from the diffuse scattering, that is A in (a), (e) CDF image from B in (a) [From Cowley (1973a, b), courtesy of *Acta Crystallographica*]

saturated magnetic domains with different directions of magnetisation such that the total magnetic moment of the specimen is very small. In the presence of an external applied field magnetisation proceeds initially by the preferential growth of domains whose magnetisation vector is oriented near to the field direction. As a result a specimen situated in its normal position in the bore of the objective lens is strongly magnetised and may have only a few magnetic domains. However, if it is situated above the high-field region, the magnetisation is less and the magnetic domain boundary density is increased. Thus

magnetic domains may be observed directly in the electron microscope. The technique is known as Lorentz microscopy and experimental procedures for producing images of domain boundaries have been summarised in section 1.7.2. The subject has been reviewed by a number of workers, for example Grundy and Tebble (1968) and Wade (1971).

3.23.1 Types of Domain Boundaries

On a scale large with respect to atomic dimensions the magnetisation distribution in a ferromagnetic

material can be regarded as a vector field. The transition between two neighbouring domains magnetised in different directions is effected by a rotation of the local magnetisation vector across a finite width boundary region.

Essentially, three types of domain boundary may occur in thin foils.

(1) *Bloch walls*. Figure 3.59(a) in which two 180° domains are separated by a wall requiring rotation about an axis B perpendicular to the domain wall and in the plane of the thin film. Domain walls have a finite thickness of the order of several hundred ångströms. These walls occur at specimen thicknesses ≥ 700 Å (Brown and LaBonte, 1965).

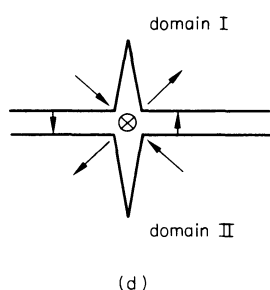
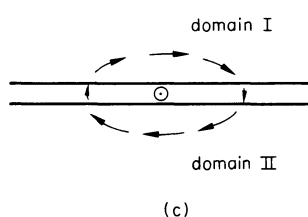
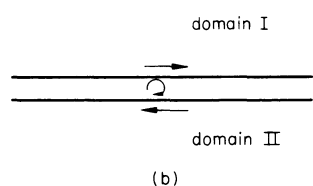
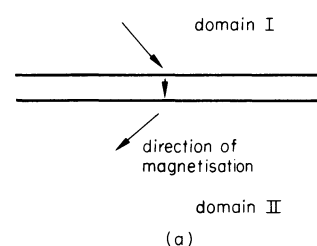


Figure 3.59 Schematic diagrams illustrating (a) Néel domain wall, (b) a Bloch domain wall, (c) an intermediate Bloch line, (d) a cross-tie Bloch–Néel wall

(2) *Néel walls*. Figure 3.59(b) in which two 180° domains are separated by a wall involving rotation

about an axis N perpendicular to the plane of the thin film and in the domain wall. The Néel wall usually occurs in very thin sections 200–500 Å thick (Néel, 1955; Middelhoek, 1963).

(3) *Cross-tie walls*. Figure 3.59(d) which are alternating Néel segments separated by segments termed Bloch lines, figure 3.59(c). Discontinuities of this type allow a domain wall to be subdivided into segments of alternate magnetic moment and occur at intermediate thicknesses, that is transition between Bloch and Néel walls.

In addition complex three-dimensional wall structures can occur (Hubert, 1969, 1970; LaBonte, 1969). All types of wall have been extensively studied in the electron microscope; for a review see Grundy and Tebble (1968). The influence of magnetic domain walls on diffraction patterns will now be discussed, together with the techniques that may be used to image them.

3.23.2 Diffraction Pattern Effects

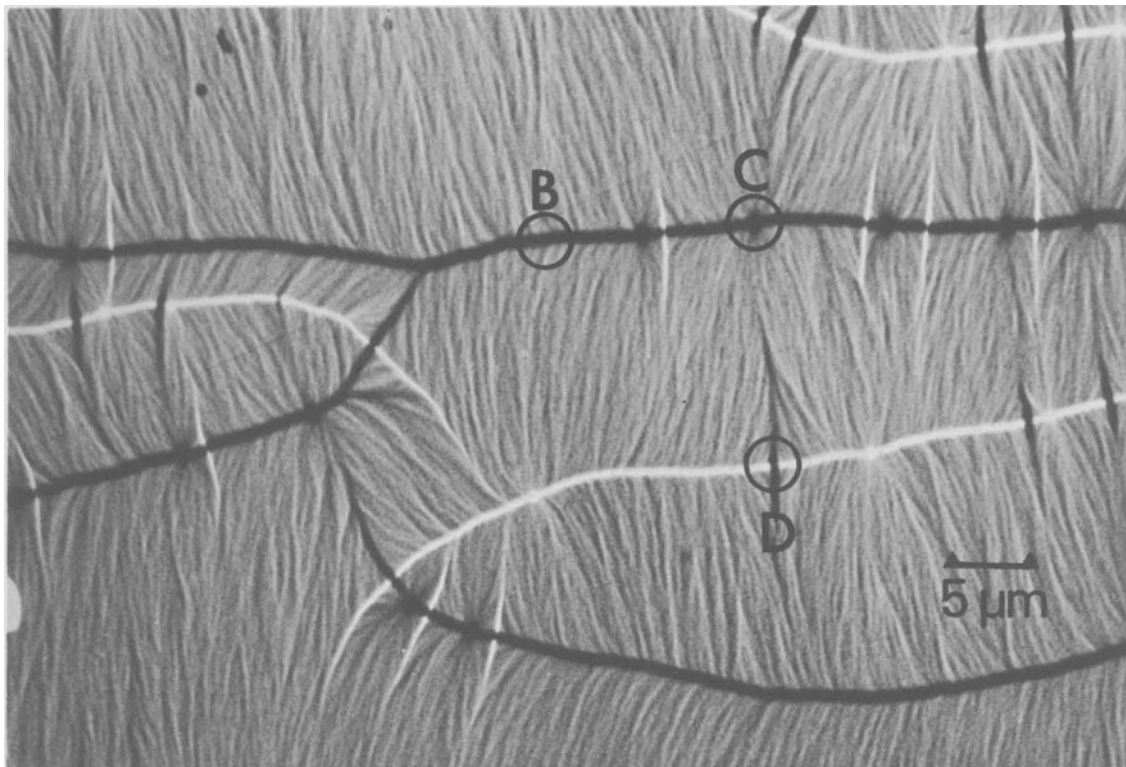
As a result of the deviations of the electron beam by the differently oriented magnetic domains in the specimen, the spots in the diffraction pattern are split in a manner dependent on the type of boundary, see figure 3.60(b)–(d). The split diffraction spots are separated by a streak due to demagnetisation field associated with the wall. In the case of the intermediate Bloch line the arcs correspond to the magnetisation distribution in the domain on either side of the wall. The cross-tie wall produces four spots corresponding to the magnetisation distribution near the wall. When crossing the wall the magnetisation vector rotates through 90° .

3.23.3 Image Characteristics

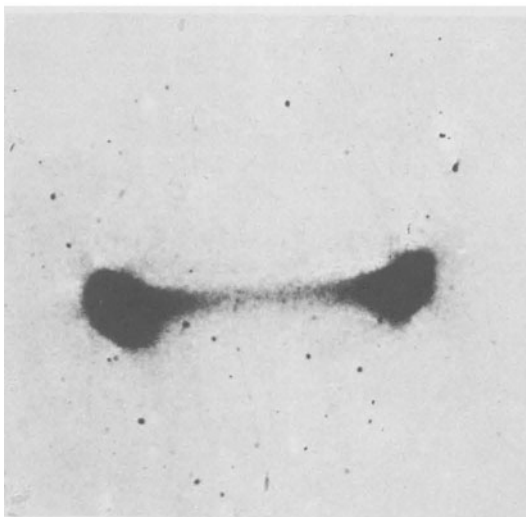
Two different methods may be used as described below. However, in both cases it is important to use an incident beam with as small an angular aperture as possible to obtain sharp images. Consequently, condenser 1 is strongly excited and condenser 2 is either switched off or strongly excited.

3.23.3.1 Fresnel images (out of focus)

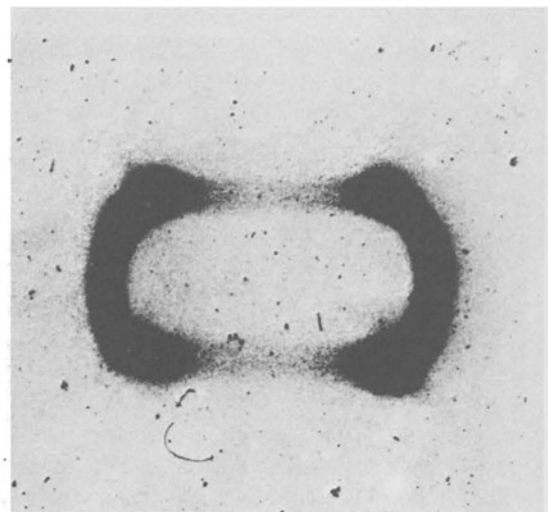
The simplest case is that of the boundary between 180° domains. The path of the electron beam is illustrated schematically in figure 3.61. Ignoring the domain wall structure and specifying the direction of magnetisation of the domain to be magnetised parallel to the plane of the section and



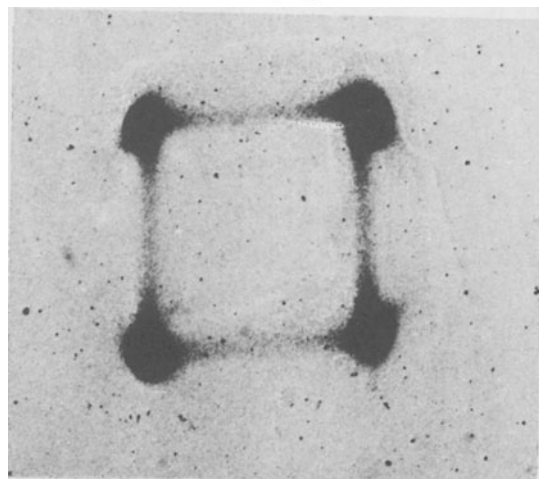
(a)



(b)



(c)



(d)

Figure 3.60 (a) A defocused image of a cross-tie wall in an evaporated Ni-20 wt % Fe film. The diffraction spots (b), (c), (d) are taken respectively from B, C, D, figure 3.60(a), that is a Néel wall, an intermediate Bloch line and a cross-tie wall. The single streak arises from the magnetic field associated with the Néel wall and the wall magnetisation distribution. The double arc streaks correspond to the magnetisation distribution in the intermediate Bloch line on either side of it. The square array of streaks correspond to the magnetisation distribution near the cross-wall where crossing a wall the magnetisation vector rotates through 90° [Courtesy of R. H. Wade]

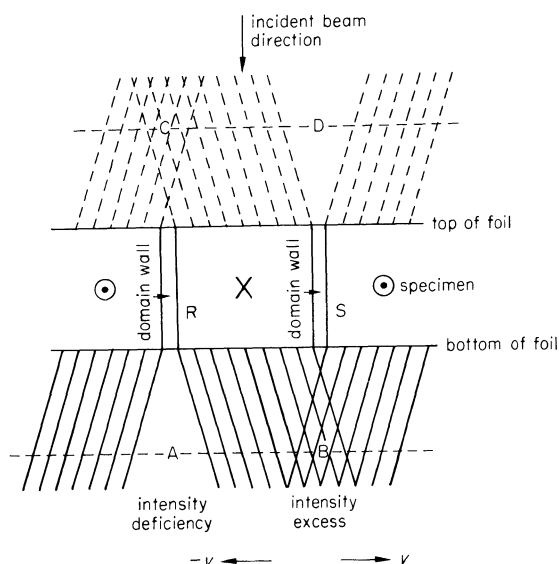


Figure 3.61 Observation of magnetic domains by the defocus method. Electrons are deviated in opposite directions in adjacent 180° domains leading to a deficiency of electrons at A and an excess at B. The contrast of virtual images C and D is reversed

in the $\pm y$ directions the electrons follow the paths shown as a result of their interaction with the magnetic field of the domains themselves. The force F on the electron is $F = 4\pi e V I_y / c$, where V is the velocity, I_y is the intensity of magnetisation of the material. With I_x and $I_z = 0$, the angular deflection ψ is $4\pi e I_y t / mc V$, where t is specimen thickness. Typical values for ψ are 10^{-4} radians (see table 3.11). As a result, for normal bright field images, no variation of intensity is observed because all magnetically deflected electrons pass through the axially centred objective aperture. However, if the objective lens is focussed on a plane above or below the specimen the domain boundary may be imaged because of the local increase and decrease of intensity produced by the mean deflection shown in figure 3.61. As a result, on level AB, figure 3.61, boundary S shows up as a bright line and boundary R as a dark line, see figure 3.61. Above the specimen the reverse is true (that is underfocussed objective lens). Figures 3.62(a), (b) show this reversal of contrast.

It is the beam deflections produced in the domains on either side of a wall which are primarily responsible for the defocussed image contrast.

Table 3.11 Deflection due to saturation magnetisation in foils 2000 Å thick for 100 kV electrons

Metal	Deflection ($\times 10^{-4}$ rad)
Fe	3.86
Co	3.20
Ni	1.09

Consequently, both Néel and Bloch walls image as alternate black and white lines and in cross-tie walls the main wall segments are in opposite contrast to the cross segments, see figure 3.60(a). Here the background striation is 'ripple'. This occurs in fine grain deposits and is caused by small aperiodic variations in the direction of magnetisation associated with small local variations in the magnetic anisotropy. The effect on the image is to produce striations lying at right angles to the main direction of magnetisation in the domain, see figure 3.60(a).

The best conditions for observation depend on the specimen size, thickness, domain magnetisation, the consequent deflection and the divergence of the illumination. As a result it is necessary to vary the mode of operation of the microscope, that is the various lens currents, to obtain the best results from a given material. Typical defocussing distances are $1.0\text{--}10^{-2}$ mm and boundary image widths are $1.0\text{--}0.01$ μm .

Note that the finite width of these boundary images is a result of the imaging method used not the width of the domain boundary and depends on the amount of defocussing.

3.23.3.2 Foucault images (in focus)

All spots in a diffraction pattern, including the transmitted spot, will be split into two if the diffraction pattern is taken from an area including magnetic domains oriented in two different directions, like figure 3.60(b). By displacing the aperture in BF one of the spots may be cut off, that is those parts of the electron beam arising from one domain will be eliminated from the image. As a result one domain will appear bright and the other dark, see figure 3.62(c), which shows a series of 180° domains in cobalt visible as alternate light and dark bands. Contrast reversal occurs when the aperture displaced in the opposite sense, figure 3.62(d). The main advantage of this method is that the image is in focus so that the defect structure of the specimen may be observed at the same time.

3.23.3.3 Diffraction contrast images

In single-crystal or large-grained polycrystalline foil the magnetic deflection causes local changes in the diffracting conditions, for example bend extinction contours are displaced in the region of domain boundaries. In-focus normal bright field images may therefore be used with this technique.

3.23.4 Applications

A major advantage of using transmission electron microscopy to observe magnetic domain walls is

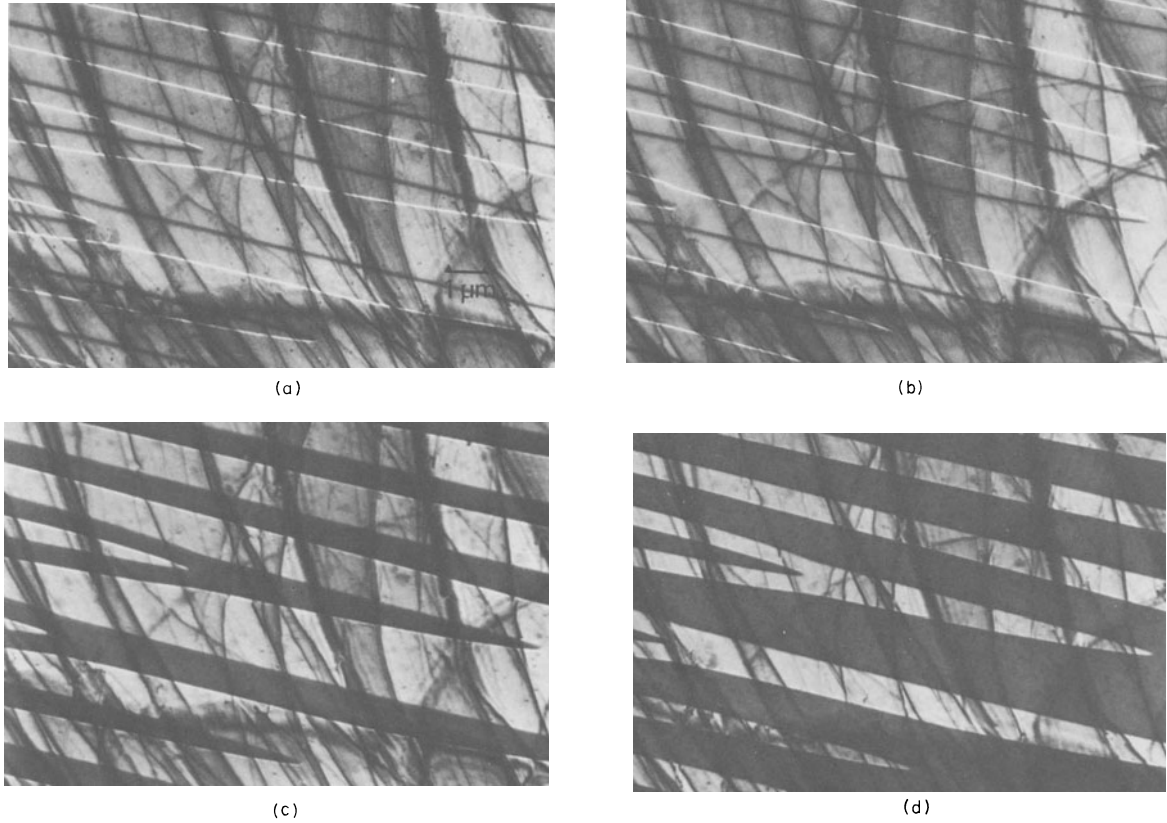


Figure 3.62 Bloch walls in a cobalt film (a) underfocussed and (b) overfocussed images. Note the reversal of contrast at the domain walls. (c, d) Foucault images from opposite ends of the streaks in the DP like figure 3.60(b). Note the reversal of contrast [Courtesy of R. H. Wade]

the high resolution obtainable compared with light optical techniques. As a result, it is possible to observe the fine-scale interaction of domain walls with crystal defects, for example dislocations (Silcox, 1963a, b; Rodgers, 1965), particles (Keh and Johnson, 1963; Wade, 1962), martensite plates (Grundy and Tebble, 1968) and small holes (Rodgers, 1965). Furthermore, the effect of temperature on domain structure may also be studied (Grundy and Tebble, 1964, 1968) as well as domain wall movement under applied magnetic fields (Wade, 1964; Fuchs and Pfisterer, 1962).

3.23.4.1 Determination of magnetisation direction

The relative orientation of the in-plane components of magnetisation may be determined from the splitting of the spots in the diffraction pattern, shown in figure 3.60. The direction of splitting is normal to the magnetisation.

3.23.4.2 Measurement of domain wall thickness

The boundary thickness has been measured in different ways by a number of workers (Fuller and Hale, 1960a, b; Warrington, 1964; Wade, 1962, 1964) using the out-of-focus Fresnel image. The in-focus Foucault method has been used by Grundy (1965), Jakubovics (1966b) and Rodgers (1965).

However, in all of this work the errors are significant, $\pm 30\%$ being common. Probably, the best method of obtaining domain wall thickness is that due to Boersch *et al.* (1962). The special case of the interference fringes produced at a convergent domain wall with nearly parallel illumination was treated in detail. The general form of the image is shown in figure 3.63(a). The fringes arise from the bending of the beam obtained near the domain wall with both condenser lenses strongly excited or with condenser 1 strongly excited and condenser 2 switched off. Effectively there are two virtual sources S_1 and S_2 , figure 3.63(b), and interference produces a fringe pattern at the domain wall. This physical picture is not strictly valid but it is satisfactory.

On the analysis of Boersch *et al.* the intensity $|A|^2$ of the interference pattern in the plane z is given by the Kirchhoff–Fresnel diffraction integral which in the form used by Boersch *et al.* is

$$A = C \int_{-\infty}^{+\infty} dx \exp \left(i \left[k \left\{ \left(\frac{1}{l} + \frac{1}{z} \right) \frac{x}{2} - \frac{xu}{z} \right\} + \frac{te}{hc} \int_0^x I_y(x) dx \right] \right) \quad (3.28)$$

where c is a constant. The terms in the exponential represent the phase difference between the axial

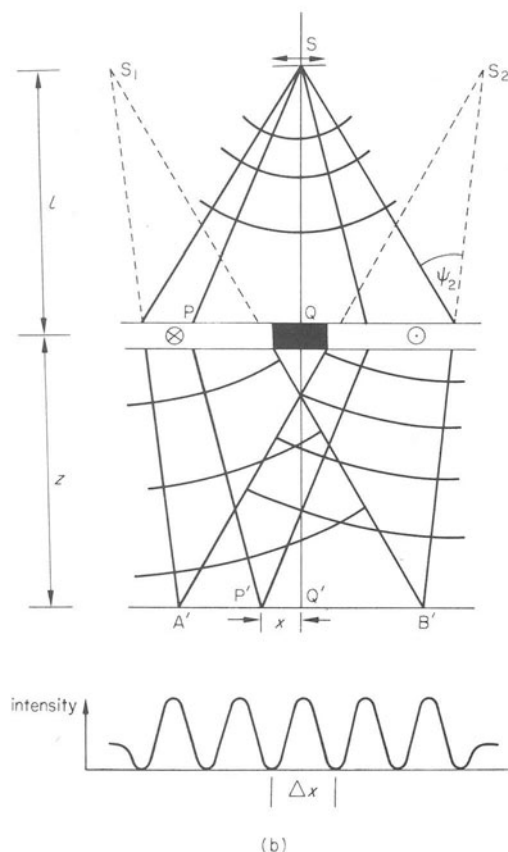
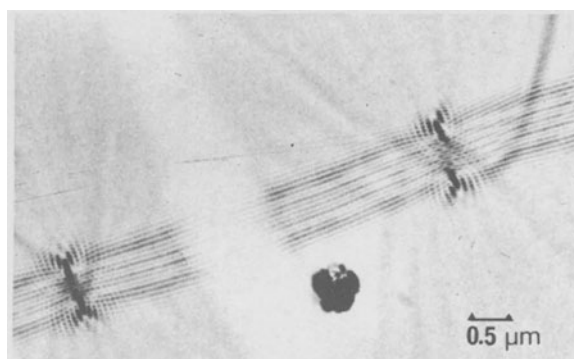


Figure 3.63 (a) A typical interference image defocused by $\sim 0.8 \mu\text{m}$ of part of a cross-tie domain wall in a Ni-20 wt % Fe film. (b) The formation of the above image in a converging domain wall image. The waves deflected by passing through two adjacent oppositely magnetized domains appear to come from vertical sources S_1 , S_2 and interfere to form fringes in the region of overlap A'B' [(a) Courtesy of R. H. Wade]

and deflected rays. The last term depending on $I_y(x)$ is the phase shift caused by the ferromagnetic film with respect to the centre of the boundary $x = 0$. The method of deriving the domain wall structure depends on obtaining a fit between the measured intensity distribution in the fringes and the intensity given by the above equation assuming different I_y functions. However, for full details the student is referred to the original papers by Boersch *et al.* (1962) and Hothersall (1969, 1971).

Cohen (1967) and Wohllenben (1967) have investigated theoretically the interpretation of Lorentz microscopy images and have emphasised the inherent errors in measurement of domain boundary widths. The defocus mode is superior to the in-focus mode for quantitative investigation because of the difficulty in accurately placing the objective aperture, the long exposure times and the difficulty in quantitative image interpretation of the Foucault in-focus method (Wade, 1971). However, there is a fundamental difficulty in obtaining precise domain wall measurements associated with the weak-phase object nature of the domain walls present in thin films.

3.23.4.3 Determination of magnetisation

Where the specimen thickness is known the in-plane component of magnetisation of the film may be determined from the equation

$$\psi_x = \frac{4\pi e I_y t}{mc V} \quad (3.29)$$

because ψ_x may be obtained from diffraction spot splitting $\delta' = 2\psi L$, where L is the camera length. This technique has been used to determine the direction of magnetisation in foils of cobalt of various orientations (Silcox, 1963b).

3.23.4.4 Determination of Curie temperature

The Curie temperature may be estimated by measuring the spot splitting as a function of temperature and extrapolating to zero splitting to obtain the Curie temperature. Reasonable accuracy is obtainable $\pm 5 \text{ degC}$.

3.23.4.5 Determination of anisotropy constant K

Evaporated thin films may be magnetically anisotropic and the parameter may be measured using Lorentz microscopy. The anisotropy may be induced by (a) deposition and annealing in a magnetic field, (b) the nature of the substrate and (c) the vacancy concentration in the film. The anisotropy constant is $K = H_K I_s / 2$, where H_K is the anisotropy field and I_s is the intensity of magnetisation. The determination of K requires the following steps.

(1) Determination of the easy magnetisation axis of the film. The easy axis is perpendicular to the hard axis which corresponds to the maximum density of domain walls at remanence when a field has been applied in the hard direction. Thus the hard axis (and therefore the easy axis) may be determined using a specimen holder with facility

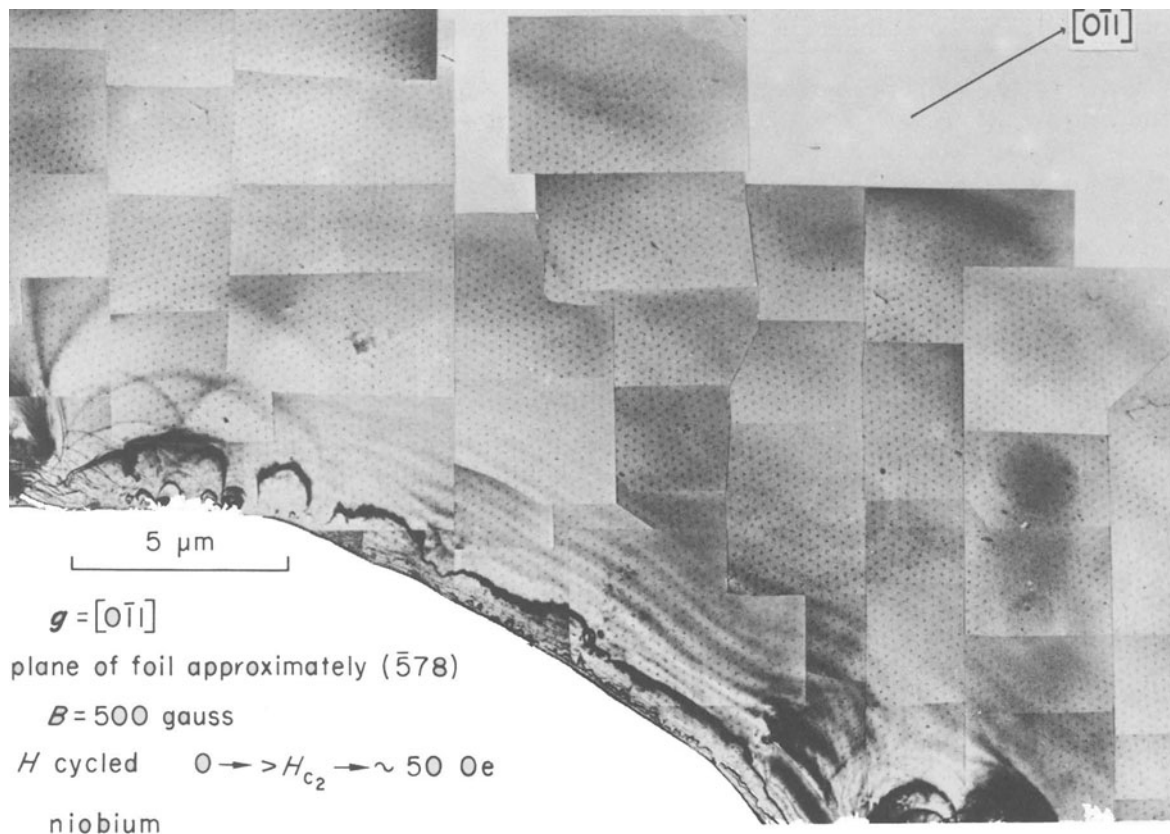


Figure 3.64 The positions of flux lines in niobium decorated by iron powder [Courtesy of C. Herring]

for applying magnetic fields in known directions to the specimen.

(2) The anisotropy constant K may now be determined by measuring the rate of rotation of the magnetisation away from the easy axis (after saturation) resulting from the application of a field H in the hard direction. The relation $H/\sin \beta = H_K$ is used to obtain H_K and K from the above equation, where β is the angle between the actual and easy directions of magnetisation. Experimentally the first procedure is to saturate the film by applying and removing a field in the easy direction. This produces only one domain. There is a range of field applied in the hard direction and the resulting domain structure is photographed so that I_s may be determined from the direction of the magnetic vector which lies at right angles to the ripple. H_K is then derived from a plot of $\sin \beta$ against H . A review of measurements of this type is given by Cohen (1965a, b).

3.23.5 Observations of Magnetic Flux Lines

Magnetic flux lines positions can be decorated in superconducting materials using fine ($\sim 25 \mu\text{m}$ diameter) iron or cobalt particles. Herring (1974) has shown that iron or cobalt may be evaporated above the surface of a niobium thin film held at liquid He temperature. The evaporated materials collide to form particles above the surface of the specimen which strike the specimen surface where

the magnetic flux lines emerge. Thus the flux line positions are decorated and are visible as islands, see figure 3.64.

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APPENDIX 3. ELASTIC CONSTANTS

For the convenience of those wishing to pursue the computer image matching techniques, described in sections 5.4.3, 5.4.7 and 5.5.5, values of the anisotropic elastic constants of a number of materials are listed in tables A3.1–A3.4.

For cubic crystal structures the anisotropy factor is given by

$$A = \frac{2C_{44}}{C_{11} - C_{12}}$$

whereas for hexagonal crystal structures

$$A_{\text{shear}} = \frac{2C_{44}}{C_{11} - C_{12}}$$

$$A_{\text{compression}} = \frac{C_{11} + C_{12} - 2C_{13}}{C_{33} - C_{13}}$$

where C_{11} , C_{12} , C_{13} , C_{33} and C_{44} are elastic stiffness constants.

The data in the tables are taken from the compilation of Chung and Buessem (1968).

The term A^* is the % anisotropy.

Table A3.1 Elastic stiffness constants and anisotropy factors for cubic crystal structures

Material and reference temperature ^a (°K)	Elastic stiffness constants			Anisotropy factor (A)	Percent elastic anisotropy (%A*)	References
	c_{11} ($\times 10^{-11}$ dyn cm ⁻²)	c_{12}	c_{44}			
Al	10.70	6.08	2.83	1.23	0.49	Schmunk and Smith (1959), Kamm and Alers (1964), Vallin <i>et al.</i> (1964), Hearmon (1966)
Cu	16.84	12.14	7.55	3.21	15.46	Schmunk and Smith (1960), Waldorf (1960), Hearmon (1966)
Ag	12.40	9.37	4.61	3.04	14.13	Neighbours and Alers (1958), Schmunk and Smith (1960), Hearmon (1966)
Au	19.23	16.31	4.20	2.87	12.78	Daniels and Smith (1958), Schmunk and Smith (1960), Hearmon (1966)
Ni	24.36	14.94	11.96	2.54	10.12	Alers <i>et al.</i> (1960), Slagle (1965), Hearmon (1966)
Pb	4.95	4.23	1.49	4.12	22.09	Waldorf and Alers (1962), Simmons (1965), Hearmon (1966)
Th	7.53	4.89	4.78	3.54	18.58	Armstrong <i>et al.</i> (1959), Simmons (1965), Hearmon (1966)
Li(298)	1.35	1.14	0.88	8.38	43.82	Trivissono and Smith (1961), Hearmon (1966)
Li(195)	1.34	1.12	0.96	8.85	45.51	Nash and Smith (1961), Hearmon (1966)
Na(300)	0.74	0.62	0.42	7.16	38.88	Daniels (1960), Hearmon (1966)
Na(195)	0.76	0.63	0.50	7.87	41.87	Diederich and Trivissono (1966)
Na(78)	0.82	0.68	0.58	8.38	43.81	
K(295)	0.37	0.31	0.19	6.71	36.85	Marquardt and Trivissono (1965), Smith and Smith (1965)
K(195)	0.39	0.33	0.22	6.89	37.66	
K(78)	0.41	0.34	0.27	7.68	41.09	Marquardt and Trivissono (1965)
K(4.2)	0.42	0.34	0.29	7.63	40.86	
Rb(293)	0.241	0.21	0.095	6.13	34.00	Robert and Meister (1966)
Rb(80)	0.296	0.244	0.16	6.15	34.12	
V	22.80	11.90	4.26	0.78	0.73	Alers (1960), Hearmon (1966)
Nb	24.65	13.45	2.87	0.51	5.26	Bolef (1961), Simmons (1965), Hearmon (1966)
Pd	22.71	17.60	7.17	2.81	12.29	Rayne (1960), Hearmon (1966)
Ta	26.09	15.74	8.18	1.58	2.50	Bolef (1961), Hearmon (1966)
Cr	35.00	6.78	10.08	0.71	1.35	Bolef and de Klerk (1963), Hearmon (1966)
Mo	44.08	17.24	12.17	0.91	0.12	Bernstein (1963), Hearmon (1966), Chung and Buessem (1967)
W	53.33	20.45	16.07	0.98	0.006	Bolef and de Klerk (1962), Hearmon (1966), Chung and Buessem (1967)
α -Fe	23.14	13.46	11.64	2.41	8.97	McSkimin and Bond (1957), Rayne and Chandrasekhar (1961), Rotter and Smith (1966)
Si	16.60	6.40	7.96	1.56	2.36	McSkimin and Andreatch (1964), Hearmon (1966)
Ge	13.00	4.90	6.70	1.65	3.01	Koppelman and Landwehr (1959), McSkimin and Andreatch (1963), Hearmon (1966)
C(diamond)	102.00	25.00	49.20	1.28	0.72	Krishnan <i>et al.</i> (1958), Hearmon (1966)
C(diamond)	107.60	12.50	57.58	1.21	0.44	McSkimin and Bond (1957)
alloys						
NiAl				3.28		Wasilewski (1969)
Ni ₃ Al				3.30		Ono and Stern (1969)

Table A3.1 (continued)

Material and reference temperature ^a (°K)	Elastic stiffness constants			Anisotropy factor (A)	Percent elastic anisotropy (%A*)	References
	c_{11} ($\times 10^{-11}$ dyn cm ⁻²)	c_{12}	c_{44}			
Ag-Mg(3.07%a)	11.98	8.98	4.60	3.07	14.32	Simmons (1965), Hearmon (1966)
Ag-Mg(7.33%a)	11.59	8.66	4.52	3.09	14.47	
Ag-Zn(2.40%a)	12.30	9.33	4.61	3.10	14.62	
Ag-Zn(3.53%a)	12.09	9.16	4.58	3.13	14.79	
Ag-In(8.36%a)	11.66	8.90	4.50	3.26	15.83	
Ag-Cd(1.34%a)	12.28	9.25	4.61	3.04	14.13	
Ag-Cd(1.92%a)	12.15	9.14	4.59	3.05	14.19	
Ag-Sn(3.17%a)	12.10	9.22	4.58	3.18	15.21	
Ag-Pd(6.22%a)	12.77	9.58	4.81	3.02	13.92	
Ag-Au(25%a)	16.6	13.2	4.86	2.86	12.67	
Ag-Au(50%a)	14.8	11.3	5.08	2.90	13.02	Zirinsky (1956), Simmons (1965), Hearmon (1966)
Ag-Au(75%a)	13.8	10.4	4.87	2.87	12.71	
Au-Cd(47.5%a)	10.2	9.60	4.23	14.10	59.36	
Au-Cd(50%a)	9.52	8.72	4.37	10.93	51.97	Schwarz and Muldower (1958), Hearmon (1966)
β -AuZn	5.3	3.5	3.0	3.33	16.39	
Cu ₃ Au	19.1	13.8	6.63	2.50	9.76	Simmons (1965), Hearmon (1966)
Cu ₃ Au(disordered)	18.49	13.53	6.87	2.77	11.95	Flinn <i>et al.</i> (1960), Simmons (1965), Hearmon (1966)
Cu ₃ Au(ordered)	18.71	13.49	6.78	2.60	10.55	Simmons (1965), Hearmon (1966)
Cu-Ga(1.58%a)	16.50	11.92	7.43	3.25	15.71	
Cu-Ga(4.15%a)	16.52	12.10	7.41	3.35	16.54	
Cu-Si(4.17%a)	16.78	12.42	7.48	3.43	17.13	
Cu-Si(5.16%a)	16.08	11.88	7.49	3.57	18.14	
Cu-Si(7.69%a)	16.58	12.64	7.41	3.76	19.57	
Cu-Ge(1.03%a)	16.66	12.10	7.50	3.29	16.05	
Cu-Ge(1.71%a)	16.31	11.82	7.50	3.34	16.45	
Li-Mg(1.09%a)	1.374	1.166	0.891	8.57	44.51	
Li-Mg(2.26%a)	1.394	1.184	0.903	8.60	44.63	
Li-Mg(3.01%a)	1.396	1.185	0.912	8.65	44.79	Alers and Karbon (1966)
Li-Mg(4.28%a)	1.429	1.217	0.924	8.72	45.05	
Po-Tl(17.4%w) ^b	4.314	3.440	1.804	4.13	22.15	Waldorf (1960), Hearmon (1966)
Cu-Mn(1.25%a)	16.70	12.06	7.52	3.24	15.68	
Cu-Mn(2.6%a)	16.53	11.95	7.47	3.26	15.84	
Cu-Mn(3.5%a)	16.27	11.75	7.46	3.30	16.14	
Cu-Mn(5.0%a)	16.04	11.60	7.43	3.35	16.49	
Cu-Mn(5.8%a)	15.95	11.53	7.43	3.36	16.61	Schmunk and Smith (1960), Hearmon (1966)
Cu-Ni(2.3%a)	16.93	12.18	7.63	3.21	15.46	
Cu-Ni(3.02%a)	16.94	12.18	7.67	3.22	15.54	
Cu-Ni(4.49%a)	17.01	12.19	7.73	3.21	15.42	Schmunk and Smith (1960), Hearmon (1966)
Cu-Ni(6.04%a)	17.11	12.24	7.81	3.21	15.42	
Cu-Ni(9.73%a)	17.23	12.26	7.91	3.18	15.23	Rayne (1958), Hearmon (1966)
α -Cu-Zn(4.1%a)	16.33	11.77	7.44	3.26	15.85	
α -Cu-Zn(9.1%a)	15.71	11.37	7.23	3.33	16.38	
α -Cu-Zn(17.1%a)	14.99	10.98	7.15	3.57	18.14	Lazarus (1949), Hearmon (1966)
α -Cu-Zn(22.7%a)	14.47	10.71	7.13	3.79	19.79	
β -Cu-Zn(48.2%a)	12.91	10.97	8.24	8.50	44.24	McManus (1963), Hearmon (1966)
β -Cu-Zn(45%a)	12.58	10.93	7.97	9.66	48.23	
β -Cu-Zn(46%a)	12.36	10.73	7.94	9.86	48.87	
β -Cu-Zn(47%a)	12.66	11.05	7.97	9.90	48.99	
β -Cu-Zn(48%a)	12.79	10.89	7.86	8.27	43.42	
β -Cu-Zn(50%a)	13.36	10.38	7.34	4.93	27.30	
Tl-In(28.13%a)	4.007	3.949	0.833	28.72	76.25	Ni-Fe(27.2%a)
Tl-In(30.15%a)	4.073	3.998	0.851	22.69	71.33	
Tl-In(35.15%a)	4.071	3.952	0.869	14.61	60.33	
Tl-In(39.06%a)	4.076	3.924	0.869	11.43	53.33	
Ni-Fe(27.2%a)	23.0	14.4	11.9	2.77	11.93	Hearmon (1966)
Halides						
AgBr	5.63	3.30	0.720	0.62	2.76	
AgCl	6.01	3.60	0.625	0.52	5.09	
CsBr	3.07	0.84	0.75	0.67	1.88	
CsCl	3.64	0.92	0.80	0.59	3.34	
CsI	2.45	0.67	0.63	0.71	1.43	
KBr	3.46	0.56	0.515	0.36	12.32	
KCl	4.05	0.66	0.629	0.37	11.34	
KF	6.56	1.46	1.25	0.49	5.98	
KI	2.75	0.45	0.369	0.32	14.71	
LiBr	3.94	1.87	1.93	1.87	4.59	
LiCl	4.94	2.28	2.46	1.85	4.47	
LiF	11.20	4.50	6.32	1.89	4.76	
NaBr	3.97	1.06	0.99	0.68	1.77	
NaCl	4.85	1.25	1.27	0.71	1.45	
NaF	9.70	2.44	2.81	0.77	0.79	
NaI	3.03	0.89	0.734	0.69	1.70	
RbBr	3.14	0.48	0.383	0.29	17.44	

Table A3.1 (continued)

Material and reference temperature ^a (°K)	Elastic stiffness constants			Anisotropy factor (A)	Percent elastic anisotropy (%A*)	References
	c_{11} ($\times 10^{-11}$ dyn cm ⁻²)	c_{12}	c_{44}			
RbCl	3.63	0.62	0.465	0.31	15.65	Haussühl (1960), Hearmon (1966) Hearmon (1966)
RbF	5.52	1.40	0.925	0.45	7.50	
RbI	2.56	0.36	0.280	0.26	20.76	
Alloys of halides						
KCl-KBr(16.8 %m) ^b	3.925	0.688	0.607	0.38	11.01	Slagle (1965), Slagle and McKinstry (1967) Slagle (1965)
KCl-KBr(17.1 %m)	3.922	0.691	0.608	0.38	11.03	
KCl-KBr(38.2 %m)	3.764	0.658	0.581	0.37	11.16	
KCl-KBr(38.7 %m)	3.762	0.660	0.580	0.37	11.17	
KCl-KBr(57.8 %m)	3.655	0.632	0.557	0.37	11.49	
KCl-KBr(59.8 %m)	3.630	0.630	0.552	0.37	11.52	
KCl-KBr(79.5 %m)	3.544	0.615	0.530	0.36	11.90	
KCl-KBr(80.0 %m)	3.545	0.605	0.531	0.36	11.94	
Antimonides, arsenides, sulphides, selenides, tellurides						
AlSb	8.939	4.427	4.155	1.84	4.41	Bulef (1960)
InSb(293)	8.329	4.526	3.959	2.08	6.32	Gerlich (1963)
GaSb	8.85	4.04	4.32	1.80	4.06	Huntington (1958), Einspruch and Manning (1963)
GaAs	11.920	5.986	5.938	2.00	5.67	Huntington (1958), Bateman <i>et al.</i> (1959), Corll (1962)
PoS	12.70	2.98	2.48	0.51	5.34	Huntington (1958), Hearmon (1966)
β -ZnS(302)	9.76	5.90	4.51	2.34	8.41	Berlincourt (1963), Einspruch and Manning (1963), Gerlich (1964)
PbSe(302)	10.70	0.77	1.30	0.26	20.04	Einspruch and Manning (1963)
ZnSe	8.10	4.88	4.41	2.74	11.70	Gerlich (1964)
CdTe	5.351	3.681	1.994	2.39	8.83	Zarembovitch (1963), Gerlich (1964)
ZnTe	7.13	4.07	3.12	2.04	5.98	Gerlich (1964)
Fluorides						
CaF ₂	16.40	4.70	3.39	0.58	3.53	McSkimin and Thomas (1962), Haussühl (1963), Slagle (1965), Hearmon (1966)
SrF ₂	12.35	4.30	3.13	0.78	0.76	Huffman and Norwood (1960)
BaF ₂	9.03	4.09	2.547	1.03	0.01	Bergmann (1957), McSkimin and Thomas (1962), Gerlich (1964), Chung (1967)
PbF ₂	8.88	4.72	2.454	1.18	0.33	Wasilik and Wheat (1965), Hearmon (1966)
Oxides						
ThO ₂	36.7	10.6	7.97	0.61	2.89	Macedo <i>et al.</i> (1964), Hearmon (1966)
UO ₂	39.5	12.1	6.41	0.47	6.77	
HgO(297)	29.60	9.51	15.557	1.55	2.28	Slagle (1965), Hearmon (1966), Chung and Buessem (1967)
SrTiO ₃	31.56	10.27	12.36	1.16	0.27	Bolef and de Klerk (1963), Macedo <i>et al.</i> (1964)
BaTiO ₃ (423)	17.28	8.2	10.82	2.38	8.79	Krishnan <i>et al.</i> (1958), Wachtman <i>et al.</i> (1963)
MgAl ₂ O ₄	30.0	15.2	15.9	2.15	6.86	Poindexter and Giardini (1958), Slagle (1965), Hearmon (1966)
Garnets						
YAl garnet	33.32	11.07	11.50	1.03	0.01	Verma (1960)
YGe garnet	29.03	11.73	9.55	1.10	0.12	
YFe garnet-I	26.80	11.06	7.66	0.97	0.01	Summer and Smith (1963)
EuFe garnet	25.10	10.70	7.62	1.06	0.04	
Carbides						
β -SiC	35.23	14.04	23.29	2.20	7.27	Tolpygo (1961), Balerman (1966)
TiC-I	50.0	11.3	17.5	0.90	0.12	Kucher (1963), Hearmon (1966)
TiC-II	38.91	4.33	20.32	1.18	0.31	Gilman and Roberts (1961)
TiC-III	52.45	9.80	18.09	0.85	0.32	de Klerk (1965)
TiC _{0.91} -IV	51.45	10.60	17.88	0.88	0.21	Williams and Lye (1963)
UC	32.0	8.4	6.47	0.55	4.27	Hearmon (1966), Chang and Graham (1966)
ZrC-I	42.80	4.08	14.60	0.75	0.95	de Klerk (1965)
ZrC _{0.94} -II	47.20	9.87	15.93	0.85	0.30	Chung (1967)

^a All data refer to 20 °C unless specified.^b %w, wt %; %m, mole %.

Table A3.2 Elastic stiffness constants and anisotropy factors for hexagonal crystal structures

Material and reference temperature ^a (°K)	Elastic stiffness constants						Elastic anisotropy				References
	c_{11}	c_{12}	c_{13} ($\times 10^{-11}$ dyn cm ⁻²)	c_{33}	c_{44}	c_{66}	A_{comp}	A_{comp}^* (%)	A_{shear}	A_{shear}^* (%)	
Be	29.23	2.67	1.40	33.64	16.25	13.28	0.90	0.10	1.22	0.46	Smith and Arbogast (1960), Simmons (1965) Garland and Silverman (1960), Corll (1962), Chang and Himmel (1966)
Cd	11.45	3.95	3.99	5.085	1.985	3.75	6.78	8.73	0.53	8.87	
Co	30.70	16.50	10.30	35.80	7.55	7.10	1.04	0.009	1.06	2.63	
Hf	18.10	7.70	6.60	19.70	5.57	5.20	0.96	0.007	1.07	0.32	
Mg	5.92	2.57	2.14	6.14	1.64	1.68	1.05	0.01	0.98	0.34	
											Long and Smith (1957), Slatsky and Garland (1957), Eros and Smith (1961), Hearmon (1966)
Re	61.26	27.00	20.60	68.27	16.25	17.13	0.99	0.001	0.95	1.02	Hirth and Lothe (1968) Ferris <i>et al.</i> (1963), Simmons (1965), Hearmon (1966)
Tl	4.08	3.54	2.90	5.28	0.726	0.27	0.77	0.15	2.69	15.56	
Ti	16.20	9.20	6.90	18.10	4.67	3.50	1.04	0.005	1.33	1.74	Fisher and Renken (1964), Hearmon (1966) Smith and Gjerre (1960), Spencer <i>et al.</i> (1965) Hearmon (1966)
Y	7.79	2.92	2.00	7.69	2.431	2.44	1.18	0.16	0.998	0.39	
Zn	16.50	3.10	5.00	6.20	3.96	6.70	8.00	9.88	0.59	13.73	Alers and Neighbours (1958), Simmons (1965), Hearmon (1966) Fisher and Renken (1964), Hearmon (1966)
α -Zr	14.40	7.28	6.53	16.50	3.21	3.56	0.87	0.07	0.90	1.08	
apatite	16.70	1.31	6.60	14.00	6.63	7.70	0.65	0.45	0.86	5.26	Simmons (1965), Hearmon (1966)
beryl	28.20	9.94	6.95	24.80	6.86	9.13	1.36	0.53	0.75	1.33	
biotite	18.60	3.24	1.16	5.40	0.58	7.68	4.60	18.37	0.08	54.61	Spencer <i>et al.</i> (1963), Simmons (1965), Hearmon (1966) Summer and Smith (1962)
CaMg ₂	5.625	1.59	1.50	6.163	1.805	2.02	0.90	0.06	0.90	0.36	
ice(257)	1.35	0.65	0.52	1.45	0.317	0.35	1.03	0.004	0.91	1.05	Green and MacKinnon (1956), Bass <i>et al.</i> (1957), Hearmon (1966) Proctor (1966)
ice(4.2)	1.710	0.85	0.71	1.82	0.362	0.43	1.03	0.003	0.84	1.28	
miscovite	17.80	4.24	1.45	5.49	1.220	6.78	4.74	17.83	0.18	29.79	Alexandrov and Ryzhova (1961), Simmons (1965), Hearmon (1966)
phlogopite-I	17.90	3.24	2.58	5.17	0.56	7.33	6.17	17.19	0.08	53.26	
phlogopite-II	17.80	3.02	1.52	5.10	0.65	7.39	4.97	18.07	0.09	49.88	Bentle (1966) Simmons (1965)
BeO	47.0	16.8	11.9	49.4	15.3	15.1	1.07	0.02	1.01	0.44	
β -quartz(873)	11.66	1.67	3.28	11.04	3.606	4.995	0.87	0.09	0.72	1.22	Simmons (1965)
ZnO	20.97	12.11	10.51	21.09	4.247	4.429	1.14	0.05	0.96	0.50	
BaTiO ₃ (293)	16.80	7.82	7.10	18.90	5.46	4.49	0.88	0.06	1.22	0.54	Huntington (1958)
(Ca, Ba)TiO ₃ (293)	17.41	7.93	8.00	16.88	4.74	4.74	1.05	0.008	1.00	0.02	
CdS	8.16	4.95	4.79	8.08	1.43	1.61	1.07	0.01	0.89	0.23	Gutsche (1958), Simmons (1965) Kucher (1963), Hearmon (1966)
TiB ₂	69.0	41.0	32.0	44.0	25.0	14.0	3.83	4.40	1.79	6.32	
α -SiC[6H]	50.4	9.8	5.6	56.6	17.0	20.3	0.96	0.01	0.84	1.05	Arlt and Schodder (1965)
Mg-Ag(0.26%a) ^b	5.968	2.615	2.172	6.168	1.649	1.677	1.06	0.02	0.98	0.34	
Mg-Ag(0.37%a)	5.975	2.620	2.179	6.201	1.652	1.678	1.05	0.01	0.99	0.35	Eros and Smith (1961), Hearmon (1966)
Mg-In(1.02%a)	5.941	2.620	2.234	6.276	1.620	1.661	1.01	0.001	0.98	0.37	
Mg-In(1.35%a)	5.955	2.626	2.201	6.143	1.625	1.665	1.06	0.01	0.98	0.33	
Mg-In(1.96%a)	5.944	2.634	2.225	6.190	1.611	1.665	1.04	0.007	0.97	0.35	
Mg-Sn(0.21%a)	5.945	2.611	2.174	6.154	1.634	1.667	1.06	0.01	0.98	0.35	
Mg-Sn(0.46%a)	5.932	2.613	2.180	6.148	1.624	1.660	1.06	0.01	0.98	0.35	
Mg-Sn(0.67%a)	5.964	2.629	2.189	6.138	1.631	1.668	1.07	0.02	0.98	0.34	
Mg-Sn(0.94%a)	5.951	2.642	2.211	6.177	1.615	1.655	1.05	0.01	0.98	0.36	

^a All data refer to 20 °C unless specified.^b at. %.

Table A3.3 Elastic stiffness constants and anisotropy factors for trigonal crystal structures

Material and reference temperature ^a (°K)	Elastic stiffness constants						Elastic anisotropy				References
	c_{11}	c_{12}	c_{13} ($\times 10^{-11}$ dyn cm ⁻²)	c_{14}	c_{33}	c_{44}	A_{comp}	A_{comp}^* (%)	A_{shear}	A_{shear}^* (%)	
Bi	6.35	2.47	2.45	0.72	3.81	1.13	2.88	3.26	0.58	14.64	Eckstein <i>et al.</i> (1960, 1961), Simmons (1965), Hearmon (1966)
CaCO ₃	14.45	5.71	5.34	-2.05	8.31	3.265	3.19	4.09	0.75	16.10	
α -Al ₂ O ₃	49.68	16.84	11.09	-2.35	49.81	14.74	1.15	0.11	0.90	1.67	Wachtman <i>et al.</i> (1960), Bernstein (1963), Simmons (1965), Hearmon (1966)
α -Fe ₂ O ₃	24.20	5.49	1.57	-1.27	2.28	8.53	1.25	0.47	0.91	1.53	
α -quartz	8.680	0.704	1.191	-1.804	10.575	5.820	0.75	0.65	1.46	7.65	McSkimin and Andreatch (1964) Huntington (1958), Simmons (1965), Hearmon (1966)
Hg(83)	3.60	2.89	3.03	0.47	5.05	1.29	0.21	1.91	3.63	42.28	
NaNO ₃	8.67	1.63	1.60	0.82	3.74	2.13	3.32	7.57	0.61	9.00	Malgrange (1963, 1964), Simmons (1965)
Sb	7.92	2.48	2.61	1.06	4.27	2.85	3.12	4.28	1.05	10.33	
Te	3.28	0.86	2.50	1.23	7.22	3.14	-0.18	16.59	2.60	31.92	Huntington (1958), Simmons (1965)
tourmaline	27.07	6.93	0.87	-0.77	16.07	6.68	2.12	5.60	0.66	3.45	

^a All data refer to 20 °C unless specified.

Table A3.4 Elastic stiffness constants and anisotropy factors for tetragonal crystal structures

Material and reference temperature ^a (°K)	Elastic stiffness constants						Elastic anisotropy				References
	c_{11}	c_{12}	c_{13} ($\times 10^{-11}$ dyn cm ⁻²)	c_{33}	c_{44}	c_{66}	A_{comp}	A_{comp}^* (%)	A_{shear}	A_{shear}^* (%)	
BaTiO ₃	28.261	18.645	14.159	17.807	8.065	11.312	5.10	5.40	0.71	8.00	Huntington (1958), Simmons (1965)
TiO ₂ (rutile)-a	27.3	17.6	14.9	48.4	12.5	19.4	0.45	2.13	0.64	10.86	Poindexter and Giardini (1958), Birch (1960), Simmons (1965), Hearmon (1966)
TiO ₂ (rutile)-b	26.6	17.3	13.6	47.0	12.4	18.9	0.50	1.79	0.66	11.32	Waldorf and Alers (1962), Hearmon (1966)
Sn-a	7.53	6.16	4.41	9.55	2.19	2.34	0.95	0.01	0.94	11.88	House and Vernon (1960), Simmons (1965), Hearmon (1966)
Sn-b	7.23	5.94	3.58	8.84	2.20	2.40	1.14	0.07	0.92	13.86	Rayne and Chandrasekhar (1960), Simmons (1965), Hearmon (1966)
ZrSiO ₄	7.35	0.90	-0.54	4.60	1.38	1.60	1.82	1.96	0.86	8.33	Simmons (1965), Hearmon (1966)

^a All data refer to 20 °C unless specified.**Appendix 3: References**

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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

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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	1.5							

APPENDIX 10. EXTINCTION DISTANCES IN SOME COMMON MATERIALS

These values have been calculated using the relation (3.2) and the values of f_0 in appendix 8.

Note that, in the case of ordered materials, the extinction distances in the following tables are unlikely to apply in practice because it is often impossible to obtain two-beam conditions. Frequently a line of systematic reflections is excited and this has the effect of reducing the extinction distances described above.

Appendix 10: References

Oblak, J. M., and Kear, B. H. (1972). *Electron Microscopy and Structure of Materials* (eds. G. Thomas, R. M. Fulrath and R. M. Fisher), University of California Press, California, p. 566.

Table A10.1 Pure metals with the f.c.c. crystal structure

Reflection	Al	Cu	Ni	Ag	Pt	Au	Pb
111	556	242	236	224	147	159	240
200	673	281	275	255	166	179	266
220	1057	416	409	363	232	248	359
311	1300	505	499	433	274	292	418
222	1377	535	529	455	288	307	436
400	1672	654	652	544	343	363	505
331	1877	745	745	611	385	406	555
420	1943	776	776	634	398	420	572
422	2190	897	896	724	453	477	638
511	2363	985	983	792	494	519	688
333	2363	985	983	792	494	519	688
440	2637	1126	1120	901	558	587	772
531	2798	1206	1196	964	594	626	822
600	2851	1232	1221	984	606	638	838
442	2851	1232	1221	984	606	638	838

Table A10.2 Pure metals with the b.c.c. crystal structure

Reflection	Fe	Nb
110	270	261
200	395	367
211	503	457
220	606	539
310	712	619
222	820	699
321	927	781
400	1032	863
411	1134	944
420	1231	1024
332	1324	1102
422	1414	1178
510	1500	1251
431	1500	1251
521	1663	1390

Table A10.3 Pure metals with the c.p.h. crystal structure

Reflection	Mg	Co	Zn	Zr	Cd
1100	1509	467	553	594	519
1120	1405	429	497	493	438
2200	3348	1027	1180	1151	1023
1100	1001	306	351	379	324
2201	2018	620	704	691	608
0002	811	248	260	317	244
1102	2310	702	762	837	683
1122	1710	524	578	590	501
2202	3917	1215	1339	1333	1140

Table A10.4 Non-metals with cubic crystal structures

Reflection	Diamond	Si	Ge	LiF	MgO
111	476	602	430	1717	2726
200				645	461
220	665	757	452	942	662
311	1245	1349	757	2199	11797
222				1210	852
400	1215	1268	659	1463	1033
331	1972	2046	1028	3352	10756
420				1710	1201
511	2613	2645	1273		
333	2613	2645	1273		
440	2151	2093	1008		

Table A10.5 Alloys with the f.c.c. crystal structure (from Oblak and Kear, 1972)

Reflection	Ni	Matrix IN 100	Matrix Udimet 700	Matrix Mar-M-200
111	331	334	335	322
200	377	383	383	368
220	547	565	567	539
311	669	696	698	661
331	991	1045	1051	988
420	1033	1092	1097	1031
422	1209	1285	1292	1210
511	1350	1441	1448	1354

Table A10.6 Ordered compounds with L1₂ crystal structure

Reflection	Type of reflection ^a	Ni ₃ Al	γ'IN 100	γ'Udimet 700	γ'Mar-M-200	Cu ₃ Au	Ni ₃ Mn
100	S	3362	9073	9408	15 473	820	10 300
110	S	3205	6406	6780	306 144	930	10 600
111	F	369	351	352	335	209	250
200	F	425	404	404	385	238	293
210	S	3501	5910	6270	17 762	1290	14 900
211	S	3680	6067	6422	16 716		
220	F	632	600	599	568		
300	S	4346	8865	7234	17 481		
311	F	777	739	738	697		
331	F	1160	1120	1107	1044		
420	F	1211	1161	1156	1089		
422	F	1422	1368	1360	1279		
511	F	1592	1535	1524	1430		

^a S, superlattice reflection; F, fundamental reflection.

Table A10.7 Ordered compounds with the B2 crystal structure

Reflection	Type of reflection	CuZn	Fe ₃ Al
100	S	22 700	2470
110	F	256	302
111	S	30 200	3020
200	F	376	450
210	S	36 300	3860
211	F	481	585

Table A10.9 Ordered compounds with the DO₃ and L2₁ crystal structure

<i>hkl</i>	Type of reflection	Cu ₂ MnAl	Fe ₃ Al
111	S	2700	2200
200	S	1680	2470
220	F	308	302
311	S	3380	2960
222	S	2170	3020
400	F	459	450

Table A10.8 Ordered compounds with the DO₁₉ crystal structure

<i>hkil</i>	Type of reflection	Mg ₃ Cd	Ti ₃ Sn	Tl ₃ Al
10 $\bar{1}$ 0	S	2700	2903	13 351
10 $\bar{1}$ 1	S	1613	1917	5809
11 $\bar{2}$ 0	S	1461	1810	4689
20 $\bar{2}$ 0	F	991	633	772
0002	F	531	336	415
20 $\bar{2}$ 1	F	646	408	506
10 $\bar{1}$ 2	S	3477	4512	9518
12 $\bar{3}$ 0	S	3791	4938	10 067
11 $\bar{2}$ 2	S	1969	2561	5185
12 $\bar{3}$ 1	S	2342	3045	6134
20 $\bar{2}$ 2	F	1457	916	1165
10 $\bar{1}$ 3	S	2750	3537	7132
22 $\bar{4}$ 0	F	872	548	705
20 $\bar{2}$ 3	F	1118	702	911
40 $\bar{4}$ 0	F	2644	1293	1234
22 $\bar{4}$ 2	F	1050	661	861

Table A10.10 θ' and S precipitates in aluminium alloys

Reflection	θ'	S
110	3836	6900
101	676	
111		2308
200		569
020		913
021		2327
112	488	976
121	1193	
013	1178	
031	1626	
310	38 380	
130		2015
131		846
113		950
022		2550
202		2386
004		989
114	39 192	
141	2390	
023		1507
041		991
221		4592
321	2011	
132	958	1211
123	1614	
223		2499

Table A10.11 Multi-beam values of extinction distance for two superlattice reflections in γ' phases L1₂ crystal structure in Udimet 700 and Mar-M-200 alloys

<i>hkl</i>	Two-beam			Twelve-beam		
	Ni ₃ Al	Udimet 700	Mar-M-200	Ni ₃ Al	Udimet 700	Mar-M-200
100	3362	9408	15 473	1650	2440	7000
300	4346	7234	17 481	1930	43 200	58 800

APPENDIX 11. VALUES OF $g \cdot b$ FOR PERFECT DISLOCATIONS IN f.c.c. AND c.p.h. CRYSTAL STRUCTURES

The term g is defined in section 2.2.3: $g \cdot b = |g| \wedge |b| \cos \phi$, where ϕ is the angle between g and b and $|g|$ and $|b|$ are their magnitudes. Thus $|g| = 1/d$ which may be determined from the relevant formulae in table A1.2. The magnitude of b may be determined from the crystal structure. Thus $\frac{1}{2}\langle 110 \rangle = 1/(2)^{1/2}$, $\frac{1}{2}\langle 111 \rangle = (3)^{1/2}/2$, etc. The angle between g and b may be obtained from the formulae in table A1.1. In this context, the angle between two directions is the same as that between the planes normal to them. Consequently, since the actual indices of the direction $[uvw]$ normal to the plane (hkl) may be non-rational in non-cubic crystal structures, care must be taken to define g and b in the same way, that is either as actual directions or as planes, before using the formulae in table A1.1.

Table A11.1 Values of $g \cdot b$ for the first seven reflections and the $a/2\langle 110 \rangle$ Burgers vectors in f.c.c. metals

Reflec- tion	Possible Burgers vector (times $a/2$)					
	$\pm[110]$	$\pm[1\bar{1}0]$	$\pm[101]$	$\pm[10\bar{1}]$	$\pm[011]$	$\pm[01\bar{1}]$
111	± 1	0	± 1	0	± 1	0
$\bar{1}\bar{1}\bar{1}$	∓ 1	0	∓ 1	0	∓ 1	0
$\bar{1}11$	0	∓ 1	0	∓ 1	± 1	0
$1\bar{1}\bar{1}$	0	± 1	0	± 1	∓ 1	0
$1\bar{1}1$	0	± 1	± 1	0	0	∓ 1
$\bar{1}1\bar{1}$	0	∓ 1	∓ 1	0	0	± 1
$11\bar{1}$	± 1	0	0	± 1	0	± 1
$\bar{1}\bar{1}1$	∓ 1	0	0	∓ 1	0	∓ 1
200	± 1	± 1	± 1	± 1	0	0
$\bar{2}00$	∓ 1	∓ 1	∓ 1	∓ 1	0	0
020	± 1	∓ 1	0	0	± 1	± 1
$0\bar{2}0$	∓ 1	± 1	0	0	∓ 1	∓ 1
002	0	0	± 1	∓ 1	± 1	∓ 1
$00\bar{2}$	0	0	∓ 1	± 1	∓ 1	± 1
220	± 2	0	± 1	± 1	± 1	± 1
$\bar{2}\bar{2}0$	∓ 2	0	∓ 1	∓ 1	∓ 1	∓ 1
$\bar{2}20$	0	± 2	± 1	± 1	∓ 1	∓ 1
$2\bar{2}0$	0	∓ 2	∓ 1	∓ 1	± 1	± 1
202	± 1	± 1	± 2	0	± 1	∓ 1
$\bar{2}0\bar{2}$	∓ 1	∓ 1	∓ 2	0	∓ 1	∓ 1
$20\bar{2}$	± 1	± 1	0	± 2	∓ 1	± 1
$\bar{2}02$	∓ 1	∓ 1	0	∓ 2	± 1	∓ 1
022	± 1	∓ 1	± 1	∓ 1	± 2	0
$0\bar{2}\bar{2}$	∓ 1	± 1	∓ 1	± 1	∓ 2	0
$02\bar{2}$	± 1	∓ 1	∓ 1	± 1	0	± 2
$\bar{0}22$	∓ 1	± 1	± 1	∓ 1	0	∓ 2
311	± 2	± 1	± 2	± 1	± 1	0
$\bar{3}\bar{1}\bar{1}$	∓ 2	∓ 1	∓ 2	∓ 1	∓ 1	0
$\bar{3}11$	∓ 1	∓ 2	∓ 1	∓ 2	± 1	0
$3\bar{1}\bar{1}$	± 1	± 2	± 1	± 2	∓ 1	0
$\bar{3}\bar{1}1$	± 1	± 2	± 2	± 1	0	∓ 1
$31\bar{1}$	∓ 1	∓ 2	∓ 2	∓ 1	0	± 1

Table A11.1 (continued)

Reflec- tion	Possible Burgers vector (times $a/2$)					
	$\pm[110]$	$\pm[1\bar{1}0]$	$\pm[101]$	$\pm[10\bar{1}]$	$\pm[011]$	$\pm[01\bar{1}]$
$31\bar{1}$	± 2	± 1	± 1	± 2	0	± 1
$\bar{3}\bar{1}1$	∓ 2	∓ 1	∓ 1	∓ 2	0	∓ 1
131	± 2	∓ 1	± 1	0	± 2	± 1
$\bar{1}\bar{3}\bar{1}$	∓ 2	± 1	∓ 1	0	∓ 2	∓ 1
$\bar{1}31$	± 1	∓ 2	0	∓ 1	± 2	± 1
$1\bar{3}\bar{1}$	∓ 1	± 2	0	± 1	∓ 2	∓ 1
$\bar{1}3\bar{1}$	∓ 1	± 2	± 1	0	∓ 1	∓ 2
$\bar{1}\bar{3}1$	± 1	∓ 2	∓ 1	0	± 1	± 2
$13\bar{1}$	± 2	∓ 1	0	± 1	± 1	± 2
$\bar{1}31$	∓ 2	± 1	0	∓ 1	∓ 1	∓ 2
113	± 1	0	± 2	∓ 1	± 2	∓ 1
$\bar{1}\bar{1}\bar{3}$	∓ 1	0	∓ 2	± 1	∓ 2	± 1
$\bar{1}13$	0	∓ 1	± 1	∓ 2	± 2	∓ 1
$1\bar{1}\bar{3}$	0	± 1	∓ 1	± 2	∓ 2	± 1
$\bar{1}\bar{1}3$	0	± 1	± 2	∓ 1	± 1	∓ 2
$\bar{1}1\bar{3}$	0	∓ 1	∓ 2	± 1	∓ 1	± 2
113	± 1	0	∓ 1	± 2	∓ 1	± 2
$\bar{1}\bar{1}\bar{3}$	∓ 1	0	± 1	∓ 2	± 1	∓ 2
222	± 2	0	± 2	0	± 2	0
$\bar{2}\bar{2}\bar{2}$	∓ 2	0	∓ 2	0	∓ 2	0
$\bar{2}22$	0	∓ 2	0	∓ 2	± 2	0
$2\bar{2}\bar{2}$	0	± 2	0	± 2	∓ 2	0
$\bar{2}2\bar{2}$	0	± 2	± 2	0	0	∓ 2
$2\bar{2}2$	0	∓ 2	∓ 2	0	0	± 2
$\bar{2}22$	± 2	0	0	± 2	0	± 2
$\bar{2}\bar{2}2$	∓ 2	0	0	∓ 2	0	∓ 2
400	± 2	± 2	± 2	± 2	0	0
$\bar{4}00$	∓ 2	∓ 2	∓ 2	∓ 2	0	0
040	± 2	∓ 2	0	0	± 2	± 2
$\bar{0}40$	∓ 2	± 2	0	0	∓ 2	∓ 2
004	0	0	± 2	∓ 2	± 2	∓ 2
$\bar{0}0\bar{4}$	0	0	∓ 2	± 2	∓ 2	± 2
313	± 2	± 1	± 3	0	± 2	∓ 1
$\bar{3}\bar{1}\bar{3}$	∓ 2	∓ 1	∓ 3	0	∓ 2	∓ 1
$\bar{3}13$	∓ 1	∓ 2	0	∓ 3	± 2	∓ 1
$3\bar{1}\bar{3}$	± 1	± 2	0	± 3	∓ 2	± 1
$\bar{3}\bar{1}3$	∓ 1	∓ 2	∓ 3	0	∓ 1	∓ 2
$31\bar{3}$	± 2	± 1	0	± 3	∓ 1	± 2
$\bar{3}\bar{1}3$	∓ 2	∓ 1	0	∓ 3	± 1	∓ 2
331	± 3	0	± 2	± 1	± 2	± 1
$\bar{3}\bar{3}\bar{1}$	∓ 3	0	∓ 2	∓ 1	∓ 2	∓ 1
$\bar{3}31$	0	∓ 3	∓ 1	∓ 2	± 2	± 1
$3\bar{3}\bar{1}$	0	± 3	± 1	± 2	∓ 2	∓ 1
$\bar{3}\bar{3}1$	0	∓ 3	∓ 2	∓ 1	± 1	± 2
$33\bar{1}$	± 3	0	± 1	± 2	± 1	± 2
$\bar{3}31$	∓ 3	0	∓ 1	∓ 2	∓ 1	∓ 2
113	± 1	0	± 2	∓ 1	± 2	∓ 1
$\bar{1}\bar{1}\bar{3}$	∓ 1	0	∓ 2	± 1	∓ 2	± 1
$\bar{1}13$	0	∓ 1	± 1	∓ 2	± 2	∓ 1
$1\bar{1}\bar{3}$	0	± 1	∓ 1	± 2	∓ 2	± 1
$\bar{1}\bar{1}3$	0	± 1	± 2	∓ 1	± 1	∓ 2
$\bar{1}1\bar{3}$	0	∓ 1	∓ 2	± 1	∓ 1	± 2
113	± 1	0	∓ 1	± 2	∓ 1	± 2
$\bar{1}\bar{1}\bar{3}$	∓ 1	0	± 1	∓ 2	± 1	∓ 2

Reflection		Burgers vectors of perfect dislocations ($\times \frac{1}{2}$)									
		$\pm[11\bar{2}0]$	$\pm[\bar{1}2\bar{1}0]$	$\pm[\bar{2}110]$	$\pm[11\bar{2}3]$	$\pm[\bar{1}2\bar{1}3]$	$\pm[\bar{2}113]$	$\pm[11\bar{2}3]$	$\pm[\bar{1}2\bar{1}3]$	$\pm[\bar{2}113]$	$\pm[0003]$
1	10 $\bar{1}0$	± 1	0	∓ 1	± 1	0	∓ 1	± 1	0	∓ 1	0
	01 $\bar{1}0$	± 1	± 1	0	± 1	± 1	0	± 1	± 1	0	0
	$\bar{1}100$	0	± 1	± 1	0	± 1	± 1	0	± 1	± 1	0
2	0002	0	0	0	± 2	± 2	± 2	∓ 2	∓ 2	∓ 2	± 2
3	10 $\bar{1}1$	± 1	0	∓ 1	± 2	± 1	0	0	∓ 1	∓ 2	± 1
	10 $\bar{1}\bar{1}$	± 1	0	∓ 1	0	∓ 1	∓ 2	± 2	± 1	0	∓ 1
	01 $\bar{1}1$	± 1	± 1	0	± 2	± 2	± 1	0	0	∓ 1	± 1
	01 $\bar{1}\bar{1}$	± 1	± 1	0	0	0	∓ 1	± 2	± 2	± 1	∓ 1
	$\bar{1}101$	0	± 1	± 1	± 1	± 2	± 2	∓ 1	0	0	± 1
	$\bar{1}\bar{1}0\bar{1}$	0	± 1	± 1	∓ 1	0	0	± 1	± 2	± 2	∓ 1
4	10 $\bar{1}2$	± 1	0	± 1	± 3	± 2	± 1	∓ 1	∓ 2	∓ 3	± 2
	10 $\bar{1}\bar{2}$	± 1	0	± 1	∓ 1	∓ 2	∓ 3	± 3	± 2	± 1	∓ 2
	01 $\bar{1}2$	± 1	± 1	0	± 3	± 3	± 2	∓ 1	∓ 1	∓ 2	± 2
	01 $\bar{1}\bar{2}$	± 1	± 1	0	∓ 1	∓ 1	∓ 2	± 3	± 3	± 2	∓ 2
	$\bar{1}102$	0	± 1	± 1	± 2	± 3	± 3	∓ 2	∓ 1	∓ 1	± 2
	$\bar{1}\bar{1}0\bar{2}$	0	± 1	± 1	∓ 2	∓ 1	∓ 1	± 2	± 3	± 3	∓ 2
5	11 $\bar{2}0$	± 2	± 1	∓ 1	± 2	± 1	∓ 1	± 2	± 1	∓ 1	0
	$\bar{1}2\bar{1}0$	± 1	± 2	± 1	± 1	± 2	± 1	± 1	± 2	± 1	0
	$\bar{2}110$	∓ 1	± 1	± 2	∓ 1	± 1	± 2	∓ 1	± 1	± 2	0
6	10 $\bar{1}3$	± 1	0	∓ 1	± 4	± 3	± 2	∓ 2	∓ 3	∓ 4	± 3
	10 $\bar{1}\bar{3}$	± 1	0	∓ 1	∓ 2	∓ 3	∓ 4	± 4	∓ 3	± 2	∓ 3
	01 $\bar{1}3$	± 1	± 1	0	± 4	± 4	± 3	∓ 2	± 4	∓ 3	± 3
	01 $\bar{1}\bar{3}$	± 1	± 1	0	∓ 2	∓ 2	∓ 3	± 4	∓ 2	± 3	∓ 3
	$\bar{1}103$	0	± 1	± 1	± 3	± 4	± 4	∓ 3	± 4	∓ 2	± 3
	$\bar{1}\bar{1}0\bar{3}$	0	± 1	± 1	∓ 3	∓ 2	∓ 2	± 3	∓ 2	± 4	∓ 3
7	11 $\bar{2}2$	± 2	± 1	∓ 1	± 4	± 3	± 1	0	∓ 1	∓ 3	± 2
	11 $\bar{2}\bar{2}$	$\pm $									

APPENDIX 12.

PREPARATION OF HIGH-RESOLUTION TEST SPECIMENS

Conventionally, three types of specimen are used as high-resolution test specimens.

- (1) Single-crystal gold films with (001) foil plane.
- (2) Partially graphitised carbon black.
- (3) Shadowed replicas for point-to-point resolution.

The preparation techniques are described below.

A12.1 Single-crystal (100) Gold Films

Single-crystal gold films have been shown to produce crossed fringe images (Komoda, 1966) with spacing 2.039, and 1.442 Å, figure 1.21a. In general these test specimens must be very thin 30–40 Å with a low defect density. To achieve such a thin film it is essential to vacuum evaporate the material. The sequence of events involves evaporation of an intermediate layer of silver, ~1000 Å thick, onto a freshly cleaved {100} rocksalt substrate held at 450 °C (Bruck, 1936; Shirai, 1943; Basset, 1965). This is followed by evaporation of a thin layer 30–40 Å of gold onto the silver at the same temperature. The combined silver–gold film may be floated off in distilled water, transferred to 50 % nitric acid to dissolve the silver, washed in distilled water and mounted on a 200 mesh grid. These films are not in general completely continuous and a few polycrystalline regions may occur.

Direct evaporation of gold onto rocksalt at 450 °C is not recommended because the films tend to be much thicker to maintain continuity.

A12.2 Partially Graphitised Carbon Black

This test specimen was first suggested by Heidenreich *et al.* (1968) who also describe its preparation. A picture is shown in figure A12.1. Standard carbon black is used (U.S. designation I.S.A.F., ASTM-N-220) which consists of small particles ~250 Å diameter primarily used to impart high abrasion resistance to tyre treads. This powder may be partially graphitised by heating at 2600–3000 °C for 30 min under argon in a graphite crucible.

The carbon black may then be dusted directly onto a holey carbon support film. Alternatively it may be dispersed ultrasonically in chloroform and then transferred to the support film by dipping.

A12.3 Replicas for Point-to-point Resolution Tests

These test specimens consist of a thin carbon film shadowed with platinum. The preparation procedure described here is that due to Nauta and Van Wijngaarden (1964). The form of the specimen on its support grid is shown in figure A12.2. Several methods have been suggested to produce the support film, and the most successful is as follows.

Prepare 6 ml of 0.25 % Formvar in chloroform and 0.05 ml of 25 % Teepol in distilled water. Mix to form a milky stock solution. Add 0.35 ml of this suspension to 10 ml of the 0.25 % Formvar in chloroform then dip a clean glass slide into the solution. When the film is dry it is floated off on distilled water and mounted on grids. For this stage lay a number of support grids on a coarse mesh gauze and use the gauze to scoop up the Formvar film. Dry on a filter paper and pick off the grids with tweezers.

Holes in the Formvar film may now be produced by placing the support grids in a dish filled with 20–25 % amyl acetate in benzene. After 15–30 min the grids are removed and dried on a filter paper. As with other films, the speed with which the glass is withdrawn from the solution influences the nature of the films. Fast withdrawal results in a thick film, slow withdrawal in a thin one. With a thick film, larger holes result and such a film is subsequently suitable as a basis for supporting a thinner film or delicate specimens (the so-called microgrid). In a thin film, smaller holes result and this is suitable for astigmatism correction.

The size of the holes is, however, also influenced by the time of immersion in the amyl acetate/benzene bath, the longer times producing, in general, larger holes.

The holey film is then covered with evaporated carbon and then gold, to build up the support grid for the final high-resolution film. The thin

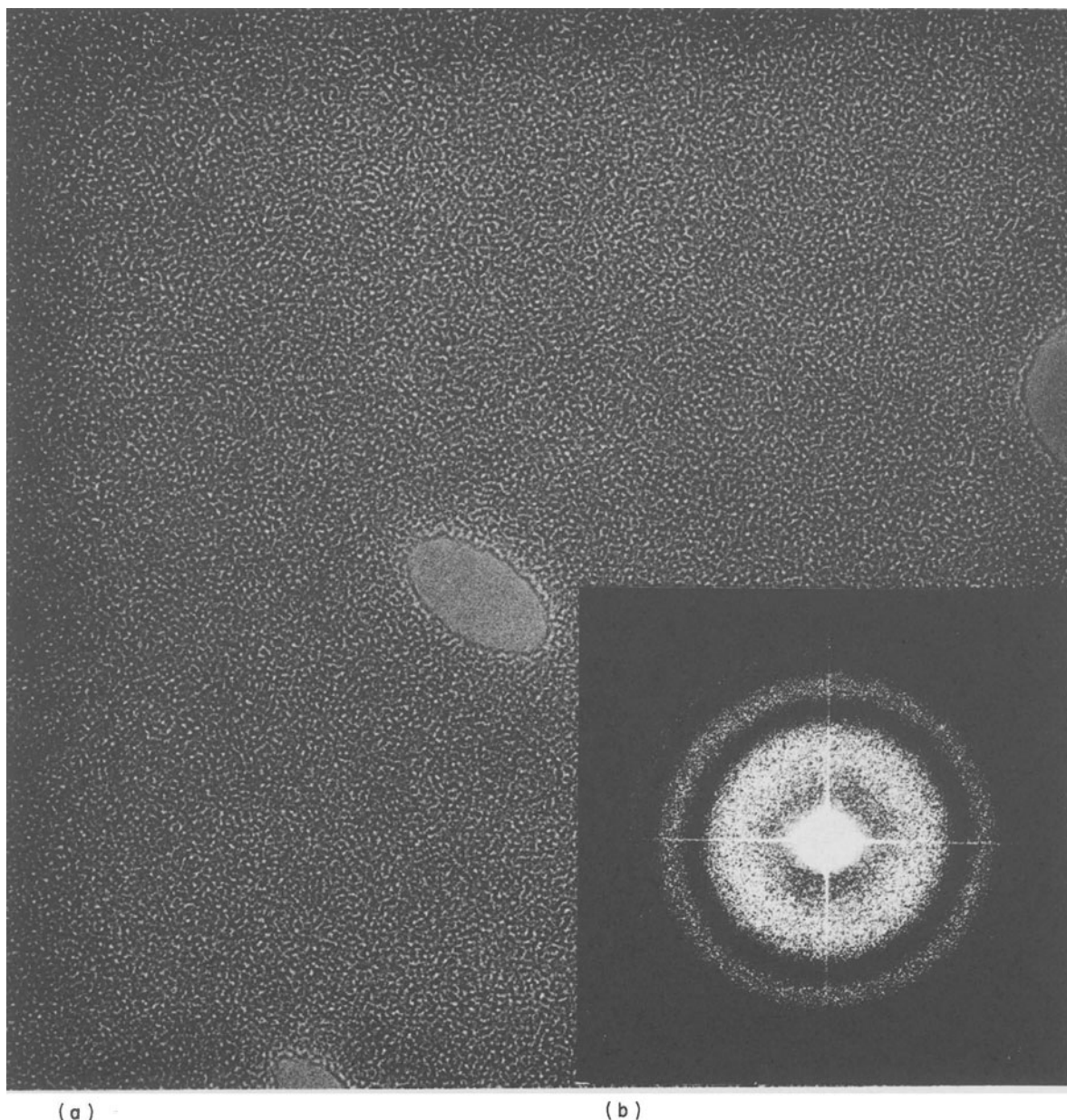


Figure A12.1 (a) A typical image of a high-resolution carbon replica showing minimum point-to-point resolution of 5 Å. (b) The corresponding light optical diffraction pattern. The inner ring is the 5 Å spacing [Courtesy of P. N. T. Unwin]

carbon film is evaporated onto a piece of freshly cleaved mica, the thinner the film the better, then stripped onto water and collected on the prepared grids as for the holey Formvar film above. Allow to dry.

The final step is to evaporate a little platinum or

other heavy metal onto the film. The platinum islands themselves may be used to check resolution above 10 Å but the structure of the support film itself is more suitable below this value.

Figure A12.1(a) shows a typical example of an image from a high-resolution replica. The resolution

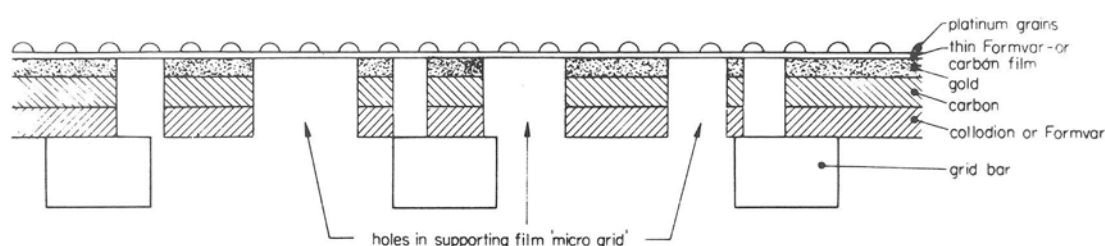


Figure A12.2 A schematic diagram showing the layer structure of a high-resolution carbon replica for point-to-point resolution tests [Courtesy of R. Nauta and J. D. van Wijngaarden]

is best checked by optical diffractometry (Thon, 1971), see figure A12.1(b).

Appendix 12: References

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APPENDIX 13.

HOLEY CARBON REPLICAS

These replicas are commonly used for astigmatism correction by observation of the Fresnel fringes at a small hole. Several preparation techniques have been suggested (Harris, 1962; Fukami and Adachi, 1965; Johnston and Reid, 1971). The technique due to Harris (1962) is easy to use and is described below. An emulsion of glycerol in 0.25 % Formvar solution is used. A cold (0 °C) glass microscope slide is dipped in the solution, allowed to dry and the Formvar replica floated off onto distilled water. This film contains holes arising from the droplets of glycerol. It is scooped up on a coarse grid with several support grids (200 mesh) on

it. After drying an ~ 100 Å thick carbon film is evaporated onto the film *in vacuo*. The Formvar is dissolved away in chloroform and the support grids then contain a holey carbon film suitable for observation in the microscope.

Appendix 13: References

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