

Weak-Beam Dark-Field Microscopy

CHAPTER PREVIEW

The term ‘weak-beam microscopy’ refers to the formation of a diffraction-contrast image in either BF or DF where the useful information is transferred by weakly excited beams. The DF approach has been more widely used, in part because it can be understood using quite simple physical models. It also gives stronger contrast; we see white lines on a dark gray background. This chapter will be concerned only with the DF approach. Historically, the weak-beam dark-field (WBDF often abbreviated to WB) method became important because under certain special diffraction conditions, dislocations can be imaged as narrow lines which are approximately 1.5 nm wide. Equally important is the fact that the positions of these lines are well defined with respect to the dislocation cores; they are also relatively insensitive to both the foil thickness and the position of the dislocations in the specimen. The technique is particularly useful if you are studying dissociated dislocations where pairs of partial dislocations may only be ~4 nm apart and yet this separation greatly affects the properties of the material.

We first choose a particular \mathbf{g} and bring this onto the optic axis as if intending to form a regular on-axis DF image. We then tilt the specimen to make \mathbf{s}_g large and examine the DF image using reflection \mathbf{g} . If a defect is present, the diffracting planes may be bent locally back into the Bragg-diffracting orientation to give more intensity in the DF image. The problem is that, as we increase \mathbf{s}_g , the average intensity decreases as $1/s^2$; in the DP the beam appears as a weak spot, hence the name. When \mathbf{s}_g is large, the coupling between \mathbf{g} and the direct beam becomes small and the diffracted beam is said to be ‘kinematically diffracted.’ So, this chapter is where we will discuss the ‘kinematical approximation’.

- You will sometimes see reference to the $\mathbf{g}(3\mathbf{g})$ WB condition. Beware! Sometimes you don’t need to be this weak; sometimes this is not weak enough.
- It is often not the fact that \mathbf{s} is large that is important; what is important is that ξ_{eff} is small.

This chapter is unusual in that it deals with a special imaging technique, rather than a concept or theory. Also, WBDF is only really useful when the specimen is not perfect, i.e., when you are interested in defects in the specimen or small changes in thickness. Therefore, you can skip this chapter if crystal-lattice defects are not relevant to your microscopy study. If you are interested in defects, you will find that this chapter really covers much more than WB microscopy. For example, we will use concepts developed for diffracted beams and carefully set the excitation error, \mathbf{s}_g , by referring to the Kikuchi-line pattern. In Section 27.9, we will discuss some of the ways that new developments in TEM design are changing the way we do WB microscopy and how we interpret the images.

27.1 INTENSITY IN WBDF IMAGES

We showed in Chapter 13 that, in a two-beam situation, the intensity of the diffracted beam \mathbf{g} in a perfect crystal can be written as

$$|\phi_g|^2 = \left(\frac{\pi t}{\xi_g}\right)^2 \cdot \frac{\sin^2(\pi t s_{\text{eff}})}{(\pi t s_{\text{eff}})^2} \tag{27.1}$$

Remember that when we derived this expression we assumed that only two beams, O and G, are important.

We will consider complications which arise when more beams are present in Section 27.9. The important variables in equation 27.1 are the thickness, t , and the effective excitation error, s_{eff} , which was given by equation 13.47

$$s_{\text{eff}} = \sqrt{s^2 + \frac{1}{\xi_g^2}} \quad (27.2)$$

In the WB technique we increase s to about 0.2 nm^{-1} so as to increase s_{eff} . (In most WB papers you will see this value as $2 \times 10^{-2} \text{ \AA}^{-1}$; remember 50 \AA is 5 nm .) This large value of s means that s_{eff} , and therefore the intensity, I_g , becomes independent of ξ_g except as a scaling factor for t (in the prefactor in equation 27.1). The actual value of s can be set by carefully positioning the Kikuchi lines for the systematic row of reflections, which includes \mathbf{g} . You can best appreciate this effect by calculating a range of values for s . Remember when doing this that you must specify \mathbf{g} and the kV because, as we saw in Chapter 13, ξ_g varies with both the reflection used to form the WB image and the energy of the electrons, as implied in equation 13.47

$$\xi_{\text{eff}} = \frac{\xi_g}{\sqrt{w^2 + 1}} \quad (27.3)$$

Practical Considerations: As the value of s_{eff} increases, equation 27.1 shows that the intensity of the \mathbf{G} beam decreases very rapidly. The result is that the exposure time needed to record the image on the CCD or on photographic film also rapidly increases and has, in the past, been the factor which limited the usefulness of the technique. Although manufacturers may guarantee a drift rate of less than 0.5 nm per minute for new machines, values of six times this rate are common on many older instruments. In early WB studies, this problem was partly overcome by using photographic film with a fast emulsion or by modifying photographic processing conditions. In either case, the grainy appearance of the photographic emulsion would be increased. The problem of drift can, in principle, be overcome by using a video system to record the image and capturing frames from the video. You could then, in principle, reduce the noise by frame averaging, particularly if you can take account of any drift. The causes of drift (specimen and thermal effects) and their correction or minimization are discussed in Chapter 8, but it is worth reminding you that changes in the temperature of the water in the objective lens are a major cause of drift. Although WBDF imaging may aim for 0.5 nm resolution rather than 0.2 nm in HRTEM, exposure times are often 10 times greater for WBDF than HRTEM, so drift may be even more important.

ON EQUATION 27.2

If $s \gg \xi_g^{-2}$ then $s \approx s_{\text{eff}}$ so that equation 27.2 becomes what is known as the ‘kinematical equation’; the kinematical equation cannot be applied for small s unless the thickness, t , is also very small.

27.2 SETTING s_g USING THE KIKUCHI PATTERN

Since the contrast in the WB image is so dependent on the value of s_g we need a method for determining s_g . We draw a line through the \mathbf{g} -systematic row and let the Ewald sphere cut through this line at $n\mathbf{g}$ where n is not an integer. Figure 27.1 illustrates this situation. How can we ‘see’ what the value of n is? Of course we can’t, since we are looking approximately normal to the ZOLZ. We can’t just judge n by looking at the intensities of the spots except in special circumstances.

The solution to the problem can be appreciated by looking at the Kikuchi pattern shown in Figure 27.2. When \mathbf{g} is exactly at the Bragg condition, the \mathbf{g} Kikuchi line passes through the \mathbf{g} reflection; when $3\mathbf{g}$ is exactly satisfied, the $3\mathbf{g}$ Kikuchi line passes through the $3\mathbf{g}$ reflection. We might guess that n is ~ 3.2 in Figure 27.2, but we don’t have a $3.2\mathbf{g}$ Kikuchi line; we have to deduce this value of n from the position of the $3\mathbf{g}$ Kikuchi line. Remember (from Chapter 19) that when the $3\mathbf{g}$ Kikuchi line passes through $3.5\mathbf{g}$ on the \mathbf{g} -systematic row, the $4\mathbf{g}$ Kikuchi line and the Ewald sphere pass through the $4\mathbf{g}$ reflection as shown in Figure 27.3. Therefore, when the Ewald sphere passes through $3.2\mathbf{g}$,

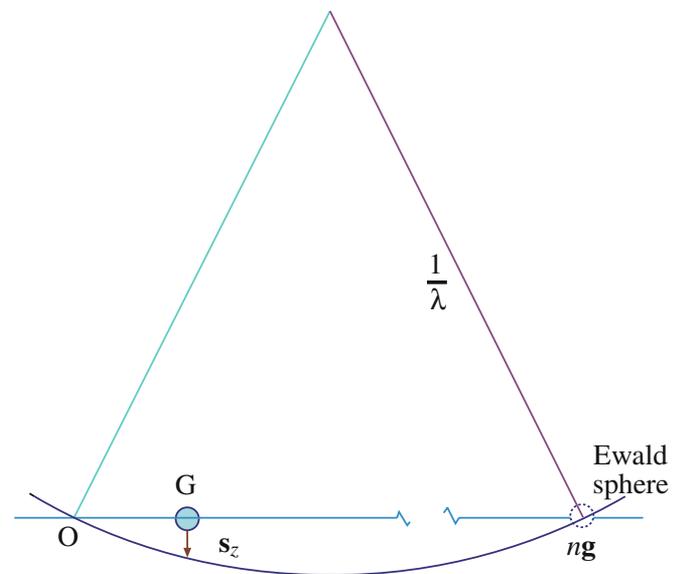


FIGURE 27.1. The Ewald-sphere construction showing the diffraction conditions used to obtain weak-beam images. The sphere cuts the row of systematic reflections at ‘ $n\mathbf{g}$ ’ where n is not necessarily an integer.

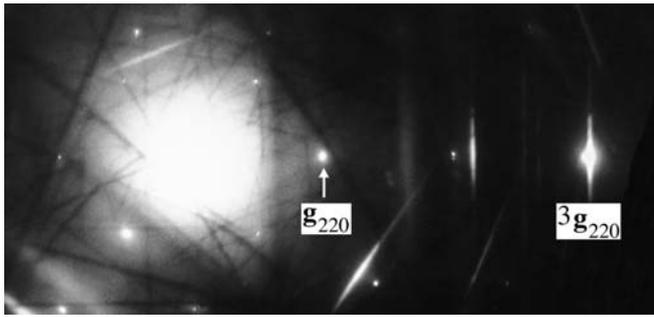


FIGURE 27.2. DP obtained when the specimen is tilted to a suitable orientation for WB microscopy. Here g is a 220 reflection and $3g$ is strong.

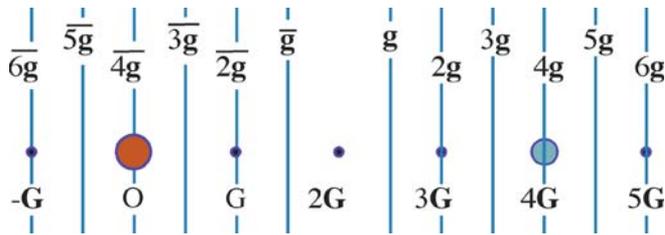


FIGURE 27.3. A schematic diagram showing the positions of the Kikuchi lines for the systematic row of reflections when $4g$ is excited.

the $3g$ Kikuchi line will pass through $3.1g$; we can express this simple geometric result as

$$n = 2m - N \quad (27.4)$$

where Ng refers to the Kikuchi line closest to ng (N is an integer) and mg is the location of the Kikuchi line as we measure it. In the example above, we can choose N to be 3 so that if m is 3.1 then n is 3.2; if, instead, we choose N to be 4, then m is 3.6 (because we measure the position of the $4g$ Kikuchi line) and n is still 3.2. Having determined n we need to estimate s . This we do using the expression

$$s = \frac{1}{2}(n - 1)|g|^2\lambda \quad (27.5)$$

which you can derive from Figure 27.4 using the intersection chord theorem ($ab = cd$) and the fact that $1/\lambda$ is much larger than s .

You can immediately appreciate some important results from this expression

- Setting $n = -1$ gives the same value of s as for $n = 3$ but the sign is reversed.
- The magnitude of s is more strongly dependent on $|g|$ than on λ but it depends on *both*.
- The specific nature of the material enters through g , the microscope affects s through λ .

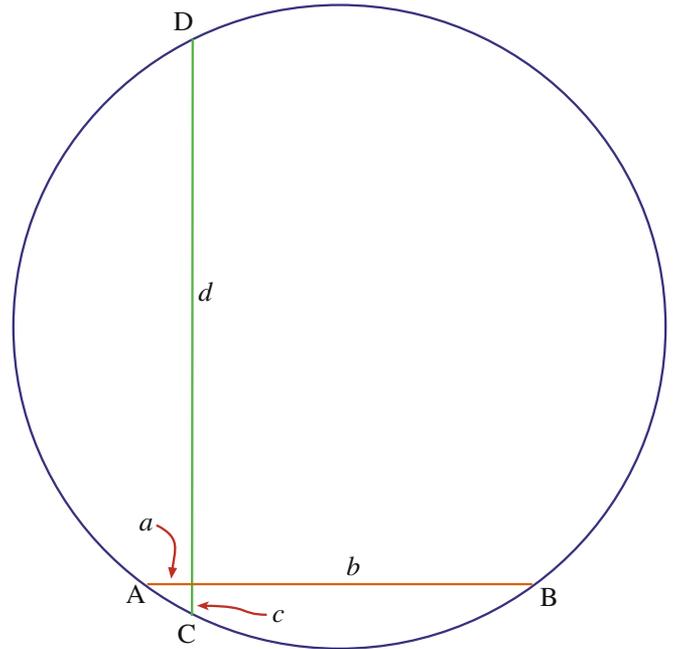


FIGURE 27.4. The intersection chord construction used to deduce the value of s_g : we approximate d to $2/\lambda$, c is s , a is $|g|$, and b is $(n-1)|g|$.

Here we recommend that you use a spreadsheet to calculate different values of s as you vary g or λ . A selection of these for Cu and Si is given in Table 27.1.

As an exercise, you can use equations 27.2 and 27.5 to calculate s_{eff} with $3g = 42 \text{ nm}$ for the 220 reflection in Cu at 100 kV. You can then see when s_{eff} becomes ‘independent’ of ξ_g . Next, repeat the exercise with other values of λ or for other reflections and materials.

One point you should bear in mind is that none of the above discussion requires a particular value for s and yet you will often read that s must be $\geq 0.2 \text{ nm}^{-1}$ for a WB image. This value of s is recommended when you are studying defects quantitatively, because computer calculations show that the position of the image can then be directly related to the position of the defect. You will find that smaller values of s will often give you WB images which contain the information you want and you can more easily see and record the image!

TABLE 27.1. Values of s (nm^{-1}) for Various kV					
Si	Cu	Accelerating voltage			
		100 kV	200 kV	300 kV	400 kV
n_{Si}	n_{Cu}				
4.9	2.8	0.20	0.14	0.11	0.09
6.9	3.6	0.30	0.20	0.16	0.13
8.5	4.3	0.38	0.25	0.20	0.17
9.9	4.9	0.44	0.30	0.24	0.20

For Cu, $a = 0.3607 \text{ nm}$; for Si, $a = 0.534 \text{ nm}$. The values of n are accurate to better than 0.1. In both cases the calculation is for $g = 220$.

27.3 HOW TO DO WBDF

The nature of the WB image imposes a restriction on the maximum specimen thickness which you can use because the visibility of such images decreases as the thickness increases (due to a corresponding increase in inelastic scattering). However, the orientation of the specimen is accurately set by reference to the Kikuchi lines observed in the DP (Chapter 19), and these are not visible in specimens which are too thin. Therefore, your specimen thickness must be greater than a certain minimum value. Also, if the observations you made on certain defects, in particular, dislocation ribbons and nodes, are to be interpreted as representative of the bulk and not influenced by the surface of the foil, then again the foil must not be too thin. You can generally satisfy these requirements by selecting defects for detailed study which, in the case of Cu and its alloys, lie in areas which are about 70 nm thick.

Due to the very low intensity of the WB images, exposure times required are typically on the order of 4–30 seconds using Kodak SO-163 film (CCD cameras are better!). The main factor limiting the exposure time is the inherent instability of any specimen stage. To minimize the exposure time, you can usually use a highly convergent (or divergent) beam, contrary to the assumption implicit in the simple theory. The effect of this convergence is that oscillations in image intensity and position, which result from variations in depth and thickness parameters, are diminished.

WB STEP-BY-STEP

We'll go through how to set up the $\mathbf{g}(3.1\mathbf{g})$ diffraction mode condition since this is widely used in practice. Actually, it is generally referred to as ' $\mathbf{g}(3\mathbf{g})$ with $\mathbf{s}_{3\mathbf{g}}$ positive' because we guess the value of n by estimating m . This condition ensures that the $3\mathbf{g}$ reflection is not satisfied and also that you can use the BF $\mathbf{0}(\mathbf{g})$ image, with $\mathbf{s}_{\mathbf{g}}$ slightly positive, to locate the defect and to focus your image.

The first two steps in setting up the WB conditions are illustrated in Figure 27.5, relating what happens in the Ewald-sphere model with what you see happening to the Kikuchi lines.

- Orient the specimen in BF so that \mathbf{g} is excited and $\mathbf{s}_{\mathbf{g}}$ is just greater than zero. Make sure that no other reflections are excited (see upper diagram in Figure 27.5).
- Use the DF beam-deflecting coils to bring the reflection \mathbf{g} on to the optic axis. Use the binoculars because \mathbf{g} becomes very weak; underfocus the beam before you use the high-resolution screen.

- Insert the objective aperture. In BF, check that the aperture is centered, then switch to DF and check that the spot \mathbf{G} is centered in the aperture.
- Fine-tune your conditions looking at the DP with \mathbf{G} centered.
- Go to imaging mode; you now have a WB image with the required $\mathbf{g}(3.1\mathbf{g})$ condition (see lower diagram in Figure 27.5).

Since you have inserted a small objective aperture, you should now check the objective astigmatism. We use a small objective aperture so as to remove inelastic scattering; remember that this aperture will then limit our potential resolution. If you focus the beam, you may change the position of the beam and probably the astigmatism! Remove the objective aperture and check that no other reflections are strongly excited when you are in DF. Then repeat the process starting at the third step (insert the objective aperture).

After finely focusing the image, record it together with its SADP.

If you're not sure why this 'trick' for setting the $\mathbf{g}(3.1\mathbf{g})$ condition works, go back to Chapter 19 and draw the systematic row and the corresponding Kikuchi lines. Then move the spots, while keeping the crystal, and thus the Kikuchi lines, fixed. You may see the conditions $\mathbf{g}(\bar{\mathbf{g}})$ used. This was the original condition suggested by Cockayne et al. (1969); it does give you the same values of s as the $\mathbf{g}(3.1\mathbf{g})$ condition, but the interband scattering processes are different and it is not so convenient to change from BF to WBDF. However, you may find variations on this condition useful when you need to use a $\mathbf{g}(n\mathbf{g})$ condition with a large value of n .

WB USING CDF

Note that we are using CDF imaging—we are not comparing contrast in BF and DF images.

Weak-beam microscopy becomes much easier when you are comfortable with using a CCD camera. Just being able to see whether the image is moving due to specimen drift can save boxes of photographic film. As we mentioned earlier, you could use frame averaging to reduce the noise. However, you will realize that the extra magnification from the video system tends to limit the area which can be viewed, so that film is still preferred by many WB practitioners but this is changing as higher resolution CCD cameras become affordable. You will find that $30\text{ k}\times$ magnification on the plate is a good compromise; without video, you will use $50\text{--}60\text{ k}\times$ to allow you to focus with the binoculars.

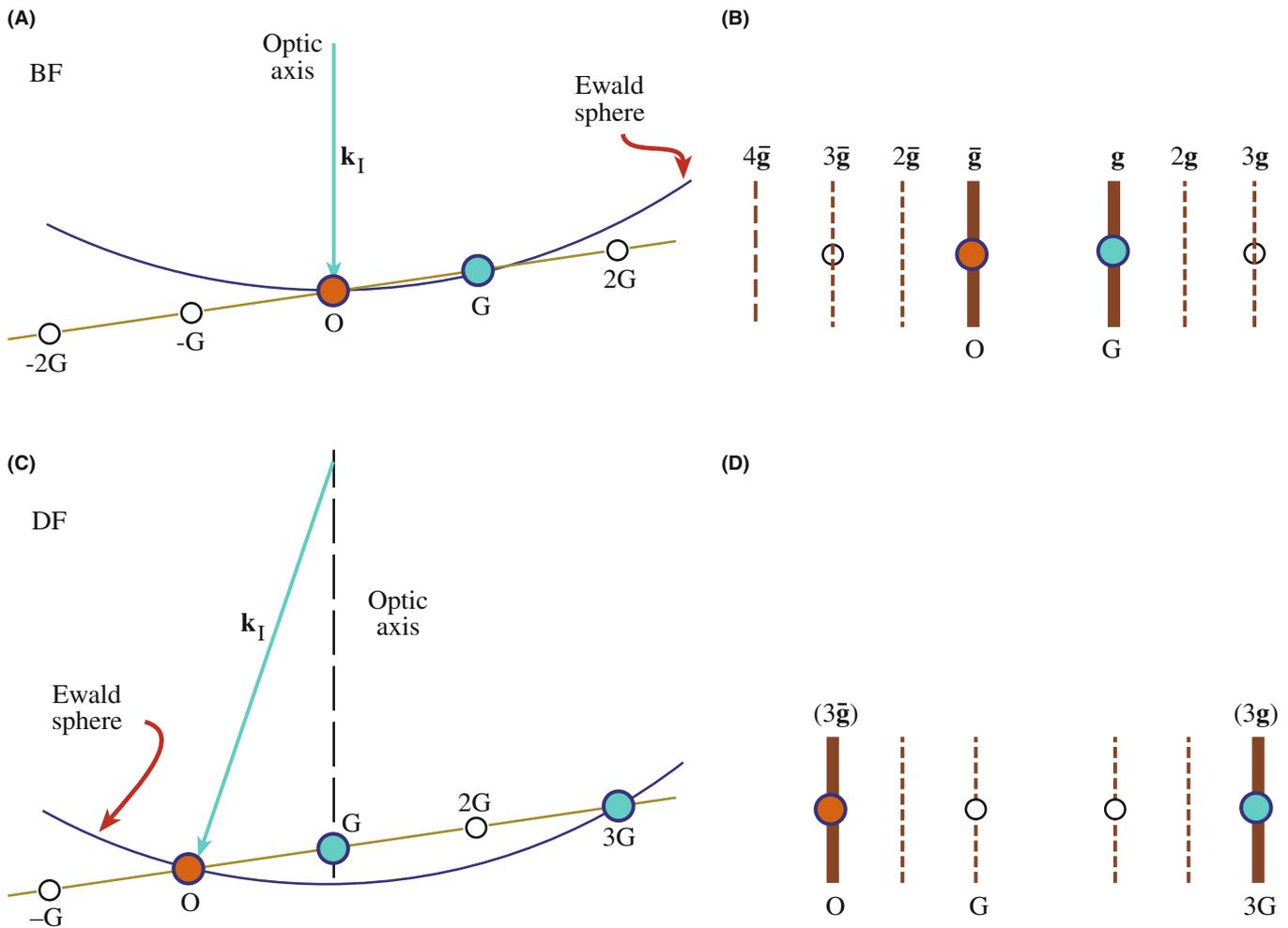


FIGURE 27.5. Relationship between the orientation of the Ewald sphere and the position of the Kikuchi lines for the $0(g)$ (A and B) and $g(3g)$ (C and D) diffraction conditions. The two pairs of diagrams are related by tilting the beam; the specimen has not tilted so the position of the Kikuchi lines is unchanged.

27.4 THICKNESS FRINGES IN WEAK-BEAM IMAGES

Thickness fringes in WB images are just like thickness fringes in strong-beam images but the effective extinction distance, ξ_{eff} , is much smaller. From equation 27.1 we can see that the intensity minimum occurs at thicknesses of $\mathcal{N}(s_{\text{eff}}^{-1})$ with maxima at $(\mathcal{N} + \frac{1}{2})\mathcal{N}(s_{\text{eff}}^{-1})$. The effective extinction distance for $s = 0.2 \text{ nm}^{-1}$ is 5 nm; this value is rather sensitive to the precise value of s so that the fringes will change if the foil bends. Using WB images we can form a rather detailed contour map of the specimen, but you must remember that both surfaces may be inclined to the beam, as shown in Figure 27.6.

The thickness effect is illustrated in Figure 27.7. These images were recorded with $s = 0.2 \text{ nm}^{-1}$. The MgO specimen has been heat-treated so that there are

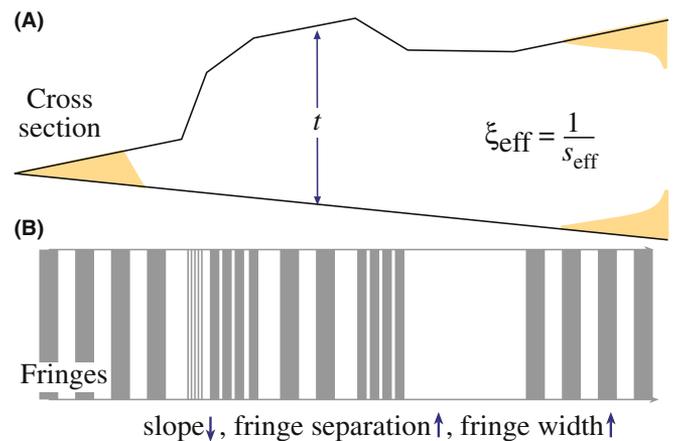


FIGURE 27.6. (A) In WB imaging, the thickness periodicity depends on the effective extinction distance, ξ_{eff} . (B) The separation of the fringes varies accordingly.

27.5 IMAGING STRAIN FIELDS

The principle of the technique is very simple. When the area of your specimen in which the defect of interest lies is oriented away from the Bragg position, the reflecting planes may be bent back into the reflecting position close to the defect. The region over which this occurs is very small because the strain has to be quite large to cause this bending. For the (220) planes in Cu (which fixes the plane spacing, d), the planes must rotate through an angle of $\sim 2^\circ$ to change s locally from 0.2 nm^{-1} to zero.

WHAT IS WEAK?

The intensity of the reflection that we see in the DP is still small even though a relatively intense peak may occur in the image close to the defect core because the DP averages over a large area.

When you look at dislocations in the WB image, you see bright lines on a dark background. Let's compare the WB image to a BF image of the same defect in Figure 27.8. You can see that the WBDF image is much narrower; you could make the comparison look even better if you make s very close to zero in the BF image.

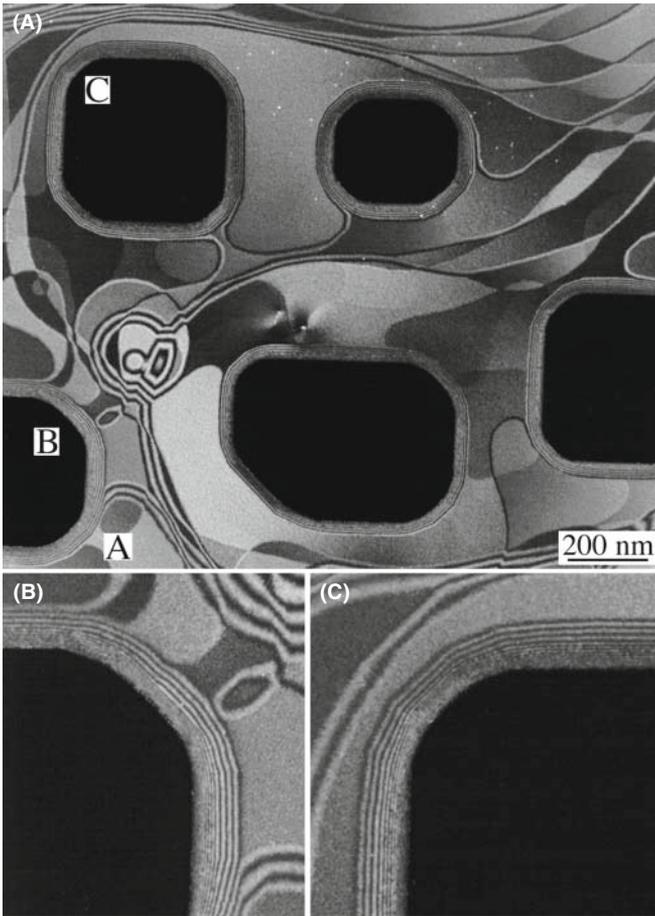


FIGURE 27.7. (A) WB thickness fringes from annealed MgO. (B, C) Higher magnifications of regions B and C. Compare to Figure 23.3.

large regions where the surface is atomically flat on both sides. Before heating, the specimen had been acid etched, which caused the holes seen in this image (they're black because we're in DF); you can see the defects causing the etch pits in some pits. The specimen shows inclined steps which curve across the surface. Where we see wide, uniform gray regions the surface is atomically flat. At A there is a large inclined step which runs into the hole B. Notice how the number of fringes around the hole B increases at A. Around the holes (Figure 27.7B and C) we see much more closely spaced fringes because the thickness changes more quickly here. Now if we look at the edge of any hole such as C, we see that the spacing of the fringes has one value away from the hole but another, smaller value close to the hole. What we find is that the inclined surface facets on different planes, with each facet becoming steeper closer to the hole. This topology is a result of the way the specimen has been prepared and would not normally be found, say, in electrochemically polished specimens, but it does illustrate the possibility of 'profiling' using thickness fringes.

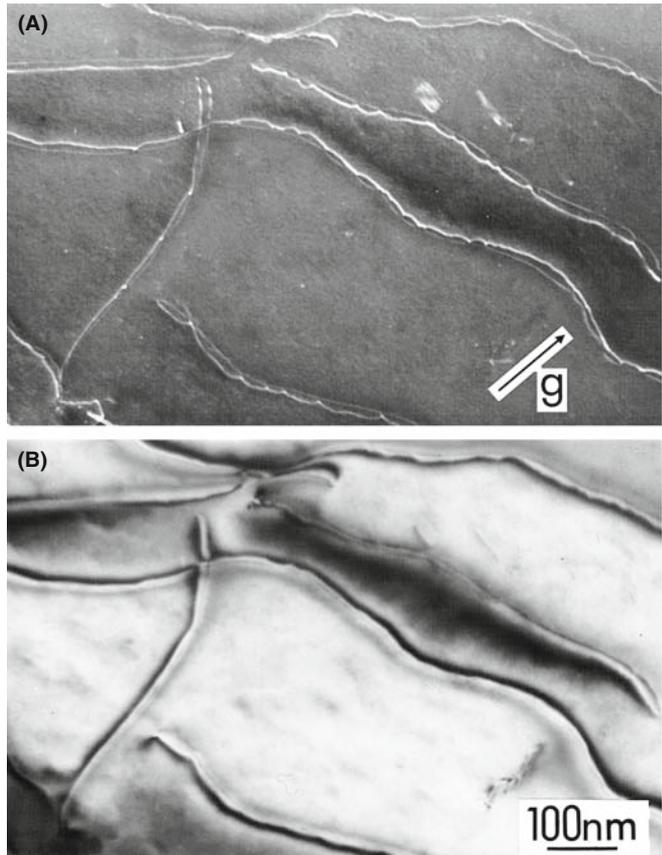


FIGURE 27.8. A comparison of dislocation images in a Cu alloy formed using (A) WB and (B) strong-beam ($s_g > 0$) conditions.

We'll keep our discussion of dislocations brief but draw your attention to a few particular points

- In the WB technique, most of the specimen is tilted so that s is large; the lattice planes in most of the specimen are then rotated away from the Bragg condition. However, as you can see in Figure 27.9, near the core of the dislocation the planes are locally bent back into the Bragg condition.
- This bending is only large close to the core of the dislocation (i.e., at the same depth from the surface).
- The peak you see in the WB image is always displaced to one side of the dislocation core. If you reverse the sign of \mathbf{g} , the peak moves to the other side of the core. If you reverse the Burgers vector, \mathbf{b} (rotate the diagram in Figure 27.9 through 180°), but keep \mathbf{g} the same, the peak again moves to the other side.
- If you increase s in the crystal, then the planes must bend more to satisfy the Bragg condition, which means the observed peak will move closer to the dislocation core.

- When we say 'position of the peak,' we are always talking about a projected position where the projection is along \mathbf{k}_D .
- There will be some situations where the local strain at the defect is not large enough to compensate for the s you have chosen. Then you will only see poor contrast in the image.

27.6 PREDICTING DISLOCATION PEAK POSITIONS

There are three ways to calculate the contrast in a WB image. Since each teaches us something new, we'll go through them in turn.

Method 1: The WB Criterion states that the largest value of $\phi_{\mathbf{g}}$ in the WB image occurs when $s_{\mathbf{R}}$, which we derived in equation 26.7, is zero. We can express this result as

$$s_{\mathbf{R}} = s_{\mathbf{g}} + \mathbf{g} \cdot \frac{d\mathbf{R}}{dz} = 0 \quad (27.6)$$

Equation 27.6 tells us that if the effective value of s (i.e., s_{eff}) is zero, even though $s_{\mathbf{g}}$ is not zero, then the direct beam and the diffracted beam, \mathbf{g} , are strongly coupled. In this situation the strain field effectively rotates the lattice planes into the Bragg-reflecting position. Therefore, the crystal can be oriented so that $\phi_{\mathbf{g}}$ is small for all columns except those near the dislocation core where it can attain a considerable magnitude due to the strong coupling with the transmitted beam as it passes through the region close to the core of the dislocation where s_{eff} is zero. This increased amplitude is then retained below the core when the coupling between the two beams is decreased again. The intensity is expected to be largest for that column where s_{eff} remains closest to zero over the longest length, and this occurs for the column where there is an inflection in the curve of \mathbf{R} versus z . Therefore, the position of the WB peak should occur when equation 27.6 is satisfied at a turning point of $\mathbf{g} \cdot d\mathbf{R}/dz$.

Method 2: The Kinematical Integral. An alternative criterion for defining the position of the WB peak was derived with the approximation that only two beams are considered and s is sufficiently large. Cockayne showed that the maximum scattering from the transmitted to the diffracted beam occurs where the kinematical integral, defined as

$$\int_{\text{column}} e^{-2\pi i(s_{\mathbf{g}}z + \mathbf{g} \cdot \mathbf{R})} dz \quad (27.7)$$

is maximized. This maximum, in general, occurs for a column which is closer to the dislocation core than predicted by *Method 1*. The reason for this difference

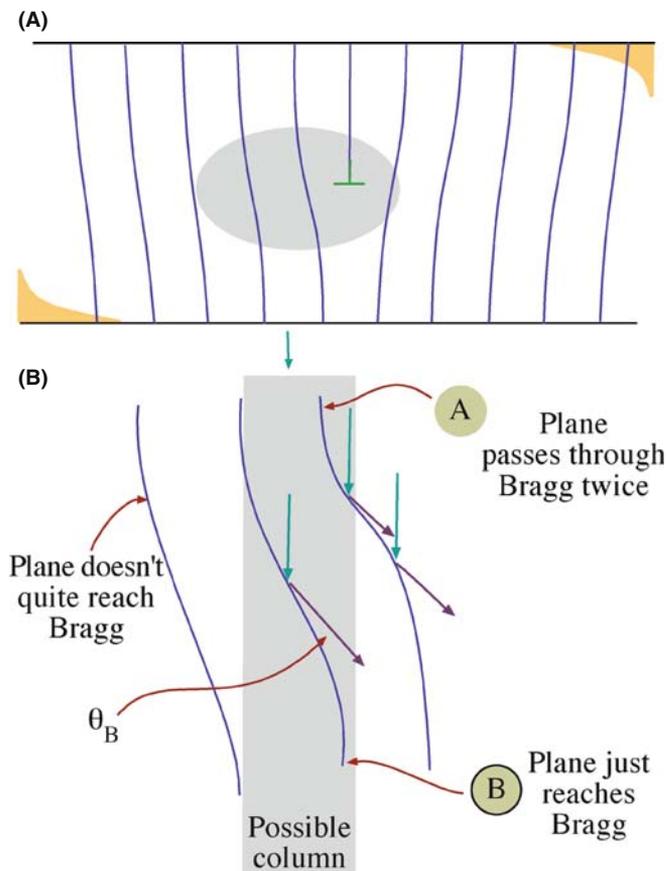


FIGURE 27.9. WB images from strained regions of the specimen show high intensity only from those regions where the diffracting planes are bent back into the Bragg condition. This illustration is for an edge dislocation. (B) shows the core region selected in (A).

is interesting: because the planes are bent, the reciprocal lattice point is, on average, nearer to the Ewald sphere. Therefore, the integral has a larger value over the length of the column.

Without doing all the math, we can illustrate how these two approaches are related. What we want to do is determine when $\phi_{\mathbf{g}}$ is large, but still kinematical (i.e., \mathbf{s} is large); we want to maximize the kinematical integral in equation 27.7. We can do this using the stationary-phase method described by Stobbs. We write the integral as

$$\int_0^t \exp\left(-2\pi i \left[\frac{z^2}{2} \cdot \frac{d^2}{dz^2}(\mathbf{g} \cdot \mathbf{R}) - \frac{z^3}{3} \cdot \frac{d^3}{dz^3}(\mathbf{g} \cdot \mathbf{R}) \right]\right) dz \quad (27.8)$$

where we have set $s + d/dz(\mathbf{g} \cdot \mathbf{R}) = 0$. If we also set $d^2/dz^2(\mathbf{g} \cdot \mathbf{R}) = 0$ (at the inflection), we go further to ensuring that the term in the square brackets is zero. This condition is what we guessed for the first method of defining the WB criterion.

Method 3: Compute the Contrast. Now that personal computers are widely available, we can calculate the position of the WB peak and graph the results. What we then find is that the WB peak actually lies between the two values predicted by the two criteria deduced using the first two methods. We also find, using the computer, that the position and width of the image peak are affected by any strongly excited diffracted beams so these *must* be avoided. Some apparently weak beams can also affect the image. A practical point is that the computer sometimes gives a rather pessimistic view of the variability in the peak position, so we have to weight the results carefully. Remember that the important advantages of this approach are that we can include the effects of the other diffracted beams which are always present, and we can take account of other effects, such as the convergence of the beam.

In the kinematical approximation, the half-width, Δx , of the image of an undissociated screw dislocation with $|\mathbf{g} \cdot \mathbf{b}| = 2$ is given approximately by the relation derived by Hirsch et al. (1960)

$$\Delta x = \frac{1}{\pi s_{\mathbf{g}}} \cdot \frac{\xi_{\text{eff}}}{3} \quad (27.9)$$

This expression is a very useful rule of thumb. You will realize that this WB image width is special for three reasons, which arise because it doesn't depend on $\xi_{\mathbf{g}}$. So, once \mathbf{s} is fixed in a WB image, we can make several surprising statements regarding the width of the dislocation peak

- It does not depend on the material.
- It does not depend on the reflection.
- It does not depend on the kV.

Take the example of the 220 reflection in Cu at 100 kV: $\xi_{\mathbf{g}}$ is 42 nm, and the width Δx is 14 nm. So, even if equation 27.9 is slightly wrong, the image width is greatly reduced in WB. If we make $s_{\mathbf{g}} = 0.2 \text{ nm}^{-1}$, then the half-width is 1.7 nm.

Computed many-beam images confirm that dislocations in other orientations give rise to similar narrow peaks when this value of $s_{\mathbf{g}}$ is used. A series of peak profiles for different values of t is shown in Figure 27.10. Notice that although the intensity of the peak may be only about 0.1% of the incident beam, it is still much higher than the background.

THE WB 'CRITERION'

$s_{\mathbf{g}} = 0.2 \text{ nm}^{-1}$ is always a useful guide; it satisfies the requirements that the image should have a narrow width and show a high contrast between the defect and background regions.

Equation 27.9 indicates that as we increase the value of s , the half-width of the image peak decreases. However, a maximum is imposed on s by the fact that the intensity of the diffracted beam varies as s^{-2} . If we make s much larger, the contrast of the image therefore becomes too small to be of practical use.

The basic requirements governing the value of s which you must use for quantitative imaging are

- $s \geq 2 \times 10^{-2} \text{ \AA}^{-1}$ to give sufficiently narrow peaks for fine detail to be studied.
- $s \leq 3 \times 10^{-2} \text{ \AA}^{-1}$ because the intensity varies as s^{-2} in the kinematical limit.
- $s\xi_{\mathbf{g}} \geq 5$ to give sufficient contrast in the WB image.

If you use the $\mathbf{g}(3\mathbf{g})$ condition for Cu with $\mathbf{g} = 220$ and 100-keV electrons, then the value of $s_{\mathbf{g}}$ will be 0.238 nm^{-1} .

27.7 PHASOR DIAGRAMS

We sometimes find it useful to demonstrate the depth dependence of the contrast in the WB image using phasor, or amplitude-phase, diagrams which we introduced in Chapter 2. You can generally use such diagrams whenever the kinematical approximation holds; they are equivalent to a graphical integration of the two-beam equations for the case where \mathbf{s} is large. In fact, many of the early calculations of defect contrast were made using this approach before computers became widely available. We recommend that you glance at the original paper by Hirsch et al. (1960): the contrast profiles were calculated by plotting the phasor diagram.

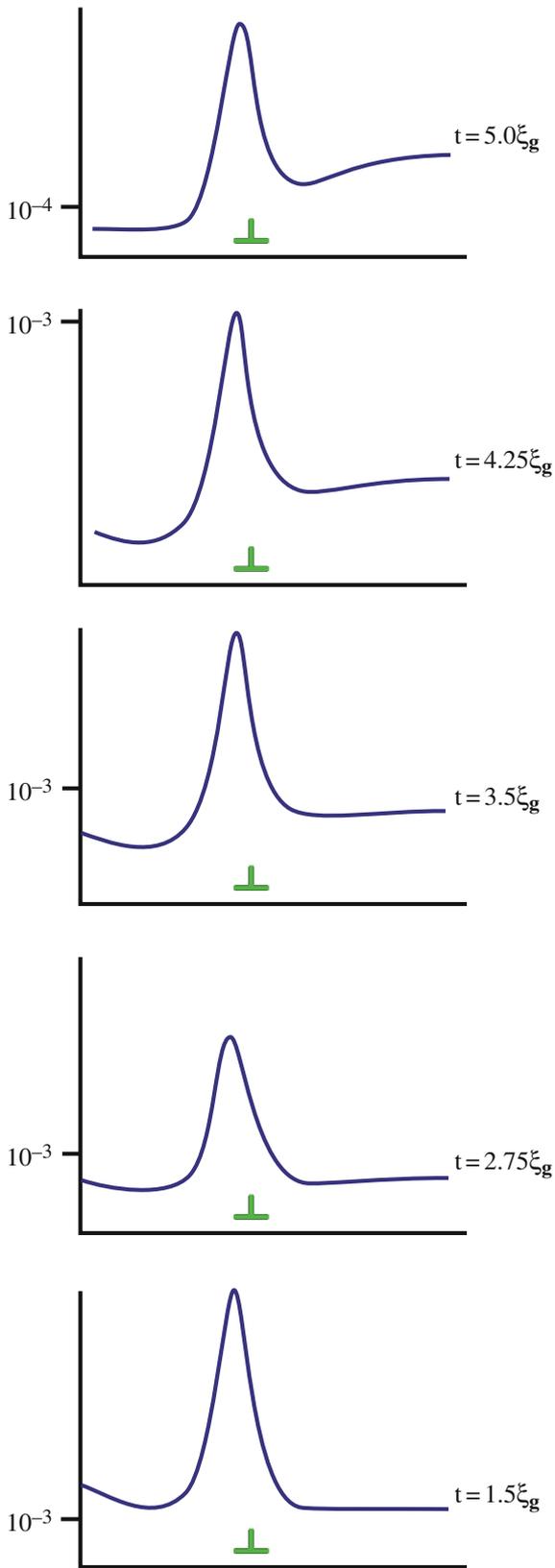


FIGURE 27.10. Examples of computer-calculated intensity peaks in WB images of an edge dislocation in Cu for different values of t . The intensity is relative to the unit incident-beam intensity. Note that the dislocation position and the peak intensity never coincide.

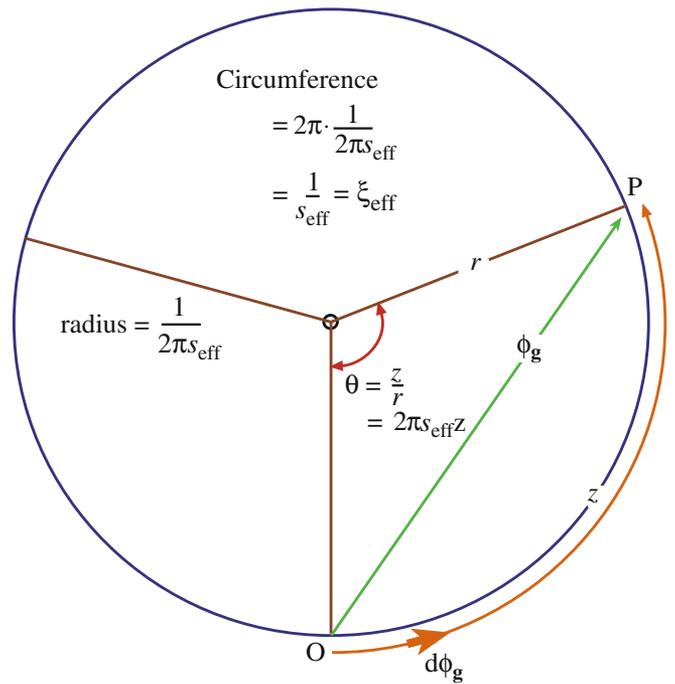


FIGURE 27.11. A phasor diagram for the WB case. The distance z is the arc OP measured around the circumference and the radius of the circle is $(2\pi s_{\text{eff}})^{-1}$. An increase in θ of 2π means the thickness increased by the ξ_{eff} . The amplitude in the WB is ϕ_g .

The basic idea is shown in Figure 27.11. We simply add all the $d\phi_g$ increments to ϕ_g . In doing so, we take account of the phase changes which occur as the beam passes through the crystal. Remember that in this approximation, no electrons leave the \mathbf{g} beam! If the crystal is perfect and our increments are sufficiently small, we will produce a smooth circle.

The circumference of this circle is ξ_{eff} as we require for the depth periodicity, and the radius is $\xi_{\text{eff}}/2\pi$ or $(2\pi s_{\text{eff}})^{-1}$. Notice that as we increase s , we decrease ξ_{eff} and the circle becomes smaller. Thus we move around the circle more quickly if s is large. In other words, our effective extinction distance is reduced, as we knew from Chapter 13.

If the diffracted beam passes through a stacking fault, it will experience an extra phase shift given by $2\pi\mathbf{g}\cdot\mathbf{R}$. Using the familiar example for an fcc crystal, we take an example where $\mathbf{R} = \frac{1}{3}[11\bar{1}]$ and $\mathbf{g} = (20\bar{2})$ which gives $\alpha = 2\pi/3 = 120^\circ$ (modulo 2π). The abrupt phase change is shown at P_3 , in Figure 27.12. Now, the value of $\phi_g(P_1P_2)$ can be much larger than in a perfect crystal. The locus of ϕ_g still travels around the first circle until it meets the planar fault at $z = n\xi_{\text{eff}} + t_1$, where z and t_1 are measured parallel to \mathbf{k}_D . It then moves onto the second circle until it reaches $z = t$. You can readily see that if we keep the depth of the fault, t_1 , fixed, we then see depth fringes which vary with periodicity ξ_{eff} as we vary the total thickness, t . (The value of α is still 120° .) The situation is a little

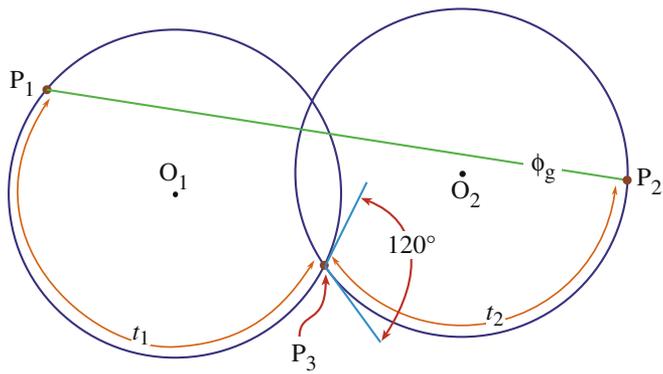


FIGURE 27.12. A phasor diagram used to explain the WB contrast from a SF at depth t_1 in a foil of thickness $t_1 + t_2$. P_1 is the top of the foil (on a circle centered on O_1); P_2 is the bottom of the foil (on a circle centered on O_2). Here, the phase change at the SF (P_3) is 120° . The total amplitude in the WB is ϕ_g .

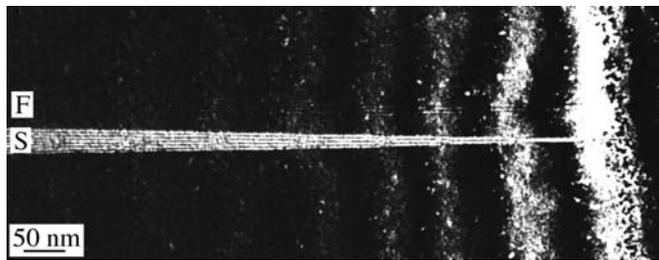


FIGURE 27.13. Illustration of thickness fringes in an experimental image of two inclined translation boundaries (S and F) in a wedge specimen of spinel. The thickness fringes from the wedge and from the inclined boundaries can be clearly seen and counted. Boundary ‘F’ shows very little contrast because it has a small R value.

more difficult to envision if we keep t fixed but vary t_1 , but the principle is the same. Images of WB fringes from inclined SFs are illustrated in Figure 27.13. The thickness fringes of the wedge specimen and those from the inclined stacking fault can both be clearly seen and counted. Notice that the number of bright fringes on the planar defect really does increase by one for every increment ξ_{eff} in the thickness of the wedge and you don’t need to know R .

You can imagine applying this analysis to the situation where you have several overlapping planar faults, as shown in Figure 27.14. $\phi_g (P_1P_2)$ can become very large. (Does our approximation of unit incident intensity still hold?) This situation does occur in practice, as shown in Figure 27.15. Here we have several overlapping faults. The bright ones are bright even when ϕ_g reaches its minimum, as you would expect from Figure 27.14. If you compare the WB image with its strong-beam counterpart in Figure 25.5, you will notice that there is much more detail in the WB image; as we saw in Chapter 25, overlapping faults on nearly adjacent

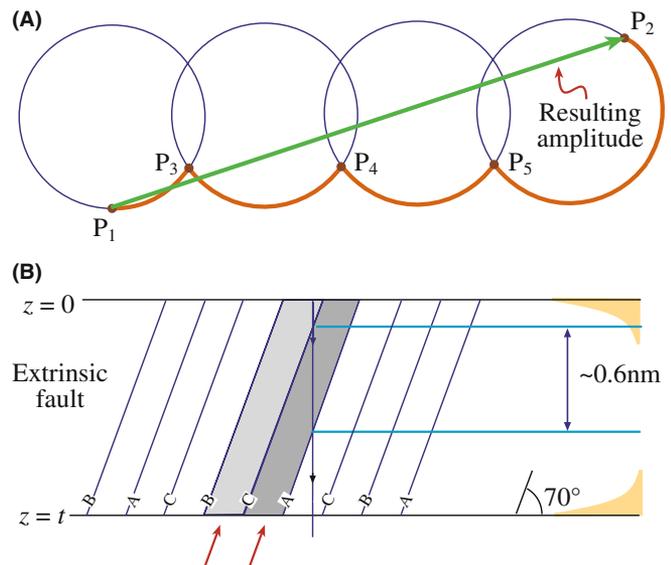


FIGURE 27.14. Phasor diagram (A) for WB encountering a series of overlapping planar defects (B) at P_3 , P_4 , and P_5 . For a $11\bar{1}$ foil normal, the inclined $11\bar{1}$ planes lie at 70° to the surface so that the spacing between adjacent planes in the direction of the beam is 0.627 nm. Compare with Figure 27.12.

planes can essentially give no contrast in the BF image. You can easily check such effects in WB by adjusting s as in Figure 27.15C–E. It’s interesting to realize that this effect can occur even when two intrinsic SFs lie on adjacent planes to give the extrinsic SF. This approach can be used to image other planar defects such as the $\{112\}$ twin boundary which can have a significant thickness. The key factor is that, under WB conditions, ξ_{eff} can become comparable to the distance that the beam travels between encountering successive planes of atoms, particularly when the interface is quite steeply inclined relative to the beam.

We can also use a phasor diagram to describe the contrast from a dislocation, but now the phase change occurs over a length of the column rather than at a particular depth in the sample. As illustrated in Figure 27.16, the phase can either add or subtract depending on the sign of $\mathbf{g} \cdot \mathbf{R}$. When the phase changes quickly with a change in t , as at the center of Figure 27.16B, it means that we are strongly coupling the incident and diffracted beams.

We’ll summarize our discussion of phasor diagrams by two points

- They should only be used when the kinematical approximation holds.
- They then give us a graphical method for understanding the variation of ϕ_g with thickness; the method is especially useful when crystal defects are present.

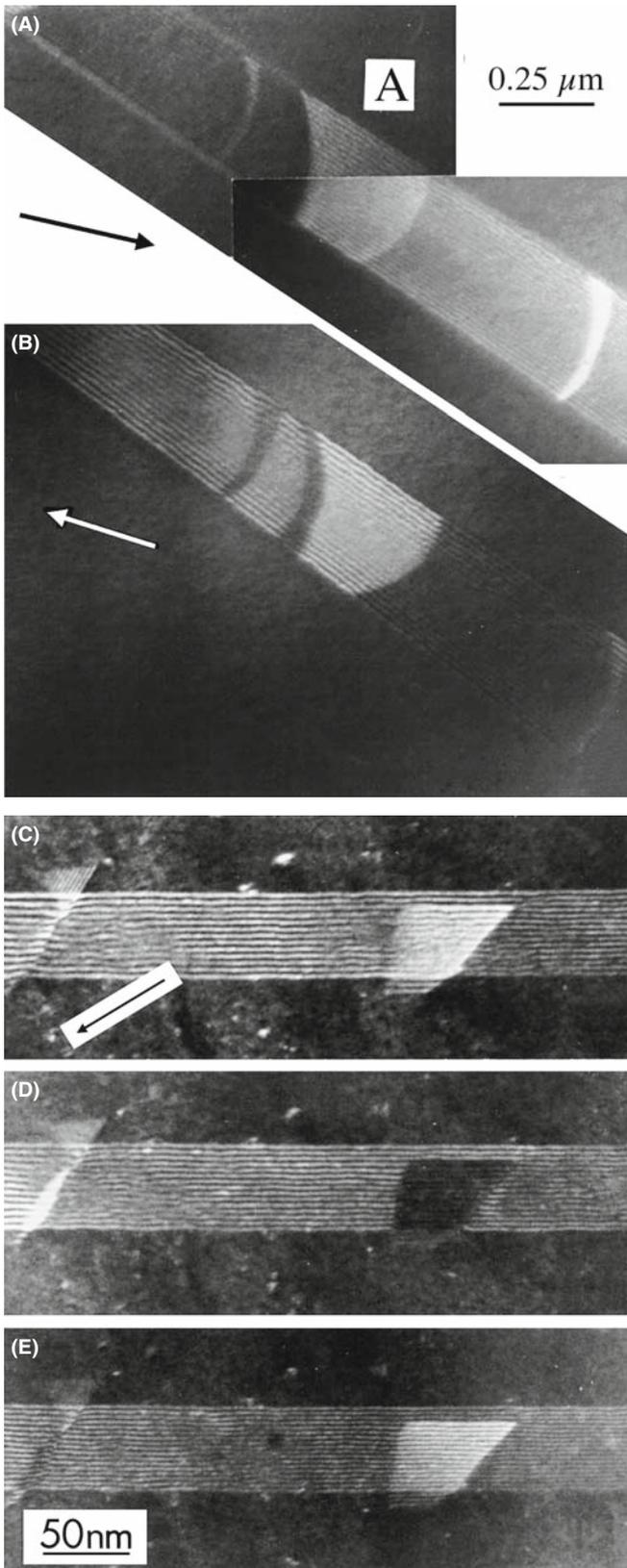


FIGURE 27.15. (A, B) Overlapping SFs imaged in WBDF using two different conditions ($\pm\mathbf{g}$). Many more fringes occur than in the corresponding BF image in Figure 24.5. Fringes are present at A where the BF image showed no contrast. (C–E) Changes in fringe spacing and intensity when one SF overlaps another; s increases from C to E. (Note the decrease in the fringe spacing.)

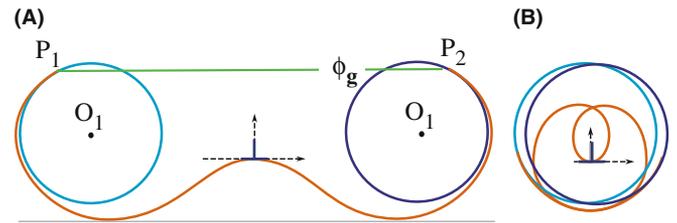


FIGURE 27.16. Phasor diagrams for a dislocation for $\pm\mathbf{g}$. The phase change is not abrupt but instead occurs over an extended distance along the column. (A) The phase change causes an increase in the amplitude scattered: $\phi_{\mathbf{g}}$ is larger than in the perfect crystal (compare to Figure 27.12). (B) When \mathbf{g} is reversed the phase change has the opposite sign and the resulting $\phi_{\mathbf{g}}$ is much smaller (so it's not shown in this confusing figure).

27.8 WEAK-BEAM IMAGES OF DISSOCIATED DISLOCATIONS

Although the study of dislocations is a very specialized topic, it beautifully illustrates the potential of the WB technique. Dissociated dislocations are common in face-centered-cubic (fcc) materials (including Si) and ordered intermetallics such as Ni_3Al . The geometry of a dissociated dislocation in Cu is summarized in Figure 27.17. We gave some general references on the theory of dislocations in Chapter 26.

Since the computed many-beam images show that the position of the dislocation image lies close to the position predicted by the WB criterion, this criterion is used in practice because it allows us to deduce an equation for the position of the image. We can thus directly relate the separation of Shockley partials, for example, to the measured separation of the two peaks observed in $|\mathbf{g} \cdot \mathbf{b}_T| = 2$ images of dissociated dislocations. Hence we can estimate the stacking-fault energy (SFE) of

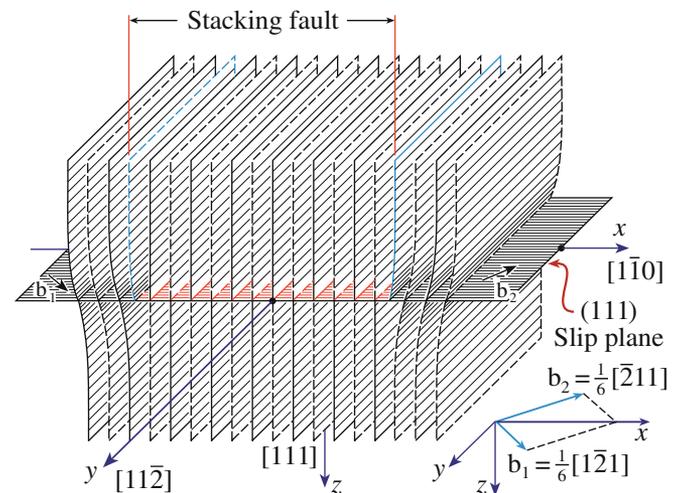


FIGURE 27.17. The geometry of a perfect dislocation in Cu. The perfect dislocation separates into two Shockley partial dislocations with Burgers vectors \mathbf{b}_1 and \mathbf{b}_2 separated by a SF on a $\{111\}$ plane. Although it might appear that partial dislocations are edge type, they are actually 60° dislocations (shown in the figure) with an edge component.

semiconductors and several fcc metals. In order to interpret WB images of extended dislocation configurations, we may need to know how the position of the image peak is related to the position of the dislocation core. This information is essential whenever we use the WB technique to collect *quantitative* data. We will now answer two questions for quantitative WB analysis

- What factors determine the position of the WB peak?
- What methods are used to determine the Burgers vectors of the dislocations?

When the WB conditions are satisfied, dissociated dislocations can be imaged in a $\{220\}$ reflection for which $|\mathbf{g} \cdot \mathbf{b}_T| = 2$, so that each partial dislocation gives rise to an intensity peak having a half-width of 1–1.5 nm. The separation of the partials can be deduced to within ± 0.7 nm providing that the peak separation is greater than ~ 2.5 nm. For many materials, we need to use anisotropic elasticity theory to relate the atomic displacements to the image, so the computer becomes essential.

Why not just use HRTEM since, as we'll see in Chapter 28, HRTEM can give detail down to below 0.2 nm, whereas WB is often limited to ~ 1 nm? If you want to interpret an HRTEM image the defect must be *absolutely straight*, parallel to the beam, and located in a very thin region of the specimen. The segment of dislocation studied by HRTEM will thus be no longer than 20 nm and less than 10 nm for the highest resolution. In a WB image the defect can be micrometers long and, in a relatively thick foil, it can even change direction. If you look back at Figure 27.8 you'll see pairs of lines which correspond to partial dislocations in the WB image. You can see other features such as constrictions. The strong-beam image may also show two or more lines for a particular dislocation but as we saw in Chapter 26, these lines are generally not related to the detailed structure of the dislocation but rather to n in the equation $\mathbf{g} \cdot \mathbf{b} = n$.

Ideally, for quantitative analysis, you should choose long, nearly straight dislocations. As shown in Figure 27.17, the two Shockley partial dislocations lie in the (111) plane of the foil. The Burgers vectors of the total and partial dislocations can be determined by imaging the dislocation in the WB mode using the $\{2\bar{2}0\}$ reflections. A sharp peak is found for a partial dislocation with $|\mathbf{g} \cdot \mathbf{b}_p| = 1$, and either no peak or a diffuse one (arising from the anisotropy of the lattice) if the partial dislocation has $|\mathbf{g} \cdot \mathbf{b}_p| = 0$. When $|\mathbf{g} \cdot \mathbf{b}_T| = 2$, the diffraction vector \mathbf{g} and the Burgers vector \mathbf{b}_T are parallel and, in the image of a dissociated dislocation, two sharp peaks are formed, one corresponding to each of the partial dislocations (both now have $|\mathbf{g} \cdot \mathbf{b}_p| = 1$).

We can explain this difference in intensity if one peak (the weaker) arises from the region between the partial dislocations and the other from outside the dissociated dislocation. This effect cannot occur in $|\mathbf{g} \cdot \mathbf{b}_T| = 1$ images, when $|\mathbf{g} \cdot \mathbf{b}_p| = 1$ for one partial and $|\mathbf{g} \cdot \mathbf{b}_p| = 0$ for the other, and it can be used to identify the reflection for which $|\mathbf{g} \cdot \mathbf{b}| = 2$. Confirmation of the Burgers vector is always obtained using the BF mode, observing characteristic $|\mathbf{g} \cdot \mathbf{b}_T| = 2$ or $|\mathbf{g} \cdot \mathbf{b}_T| = 0$ images.

DISSOCIATED DISLOCATIONS

One of the peaks is on average more intense than the other; the order reverses when $\bar{\mathbf{g}}(3\bar{\mathbf{g}})$ is used instead of $\mathbf{g}(3\mathbf{g})$.

In a WB image with $|\mathbf{g} \cdot \mathbf{b}_T| = 2$, each of the partial dislocations will generally give rise to a single peak in the image which is close to the dislocation core. You can calculate the approximate positions of these peaks using the criterion from equation 27.6. Then, you can relate the separation of the peaks in the image to the separation Δ of the partial dislocations.

We can write the displacement, using isotropic elasticity theory, as the sum of the displacements due to the individual partial dislocations. If the Burgers vector of a straight, mixed dislocation lies in the (111) plane parallel to the surface of a foil, then at a distance x from the dislocation core

$$-s_g = \frac{|\mathbf{g}|}{2\pi} \left[\left(|\mathbf{b}_1| + \frac{|\mathbf{b}_{1e}|}{2(1-\nu)} \right) \frac{1}{x} + \left(|\mathbf{b}_2| + \frac{|\mathbf{b}_{2e}|}{2(1-\nu)} \right) \frac{1}{x-\Delta} \right] \quad (27.10)$$

Here x defines an axis perpendicular to both the dislocation line and the beam direction, and e refers to the edge component of the Burgers vectors of the partial dislocations, 1 and 2. This relation is particularly simple because, for the geometry we have chosen, the term $\mathbf{g} \cdot \mathbf{b} \times \mathbf{u}$ is zero. Using the notation

$$a = -s_g \left[\frac{|\mathbf{g}|}{2\pi} \cdot \left(|\mathbf{b}_1| + \frac{|\mathbf{b}_{1e}|}{2(1-\nu)} \right) \right]^{-1} \quad (27.11)$$

and

$$b = -s_g \left[\frac{|\mathbf{g}|}{2\pi} \cdot \left(|\mathbf{b}_2| + \frac{|\mathbf{b}_{2e}|}{2(1-\nu)} \right) \right]^{-1} \quad (27.12)$$

equation 27.10 reduces to

$$1 = \frac{1}{ax} + \frac{1}{b(x-\Delta)} \quad (27.13)$$

which has two solutions, x_+ and x_- , given by

$$x_{\pm} = \frac{ab\Delta + a + b \pm \left[(ab\Delta + a + b)^2 - 4a^2b^2\Delta \right]^{1/2}}{2ab} \quad (27.14)$$

These values of x define the positions of the peaks in the image. The separation between these peaks is then given by

$$\Delta_{\text{obs}} = \left[\Delta^2 + \frac{(a+b)^2}{a^2b^2} + \frac{2(a+b)\Delta}{ab} - \frac{4\Delta}{a} \right]^{1/2} \quad (27.15)$$

We can rearrange this equation to make it appear more symmetric in a and b . Of course, it will not be symmetric because the peak is always located on one side of the dislocation.

$$\Delta_{\text{obs}} = \left[\left(\Delta + \frac{1}{b} - \frac{1}{a} \right)^2 + \frac{4}{ab} \right]^{1/2} \quad (27.16)$$

Computed images confirm that this relation is accurate for $\Delta_{\text{obs}} > 2.5$ nm, to within ± 0.7 nm. This uncertainty is due to the variation of the peak position with the depth of the dislocation in the foil and the foil thickness. A small uncertainty arises when you have not determined the actual direction of \mathbf{b}_T , i.e., whether it is in the direction of \mathbf{g} or $\bar{\mathbf{g}}$. Stobbs and Sworn found that the relation (equation 27.16) subject to the ± 0.7 nm uncertainty is still a good approximation when anisotropic elasticity theory is used.

As a simple exercise, consider the WB images of a dissociated screw dislocation and a dissociated edge dislocation. You should pay particular attention to ‘ a ’ and ‘ b ’ because in one case, \mathbf{b}_{1e} and \mathbf{b}_{2e} have the same sign, while in the other the sign is opposite. Does the image always have the same width when you reverse \mathbf{g} ?

Example 1. Even if you never want to calculate the actual separation of two dislocations from observations of two peaks, you can learn new ideas about dislocations from such images. Figure 27.18 is a famous set of images showing a dislocation in Si which is constricted along part of its length and dissociated along the rest. Even if you don’t know the precise details of the dislocation structure, you know that it can adopt two variants; the rest of the task is modeling the defect.

Example 2. The WB image of the node pair in Figure 27.19A, tells you very quickly that the two nodes are different; if we form images using other \mathbf{g} vectors (Figure 27.19B–D), we find that one of the partial dislocations is out of contrast in the image. The extended node contains the same type of intrinsic stacking fault that is present in the dissociated dislocation; $\mathbf{g} \cdot \mathbf{R}$ is zero in this image for the stacking fault. The other node is constricted,

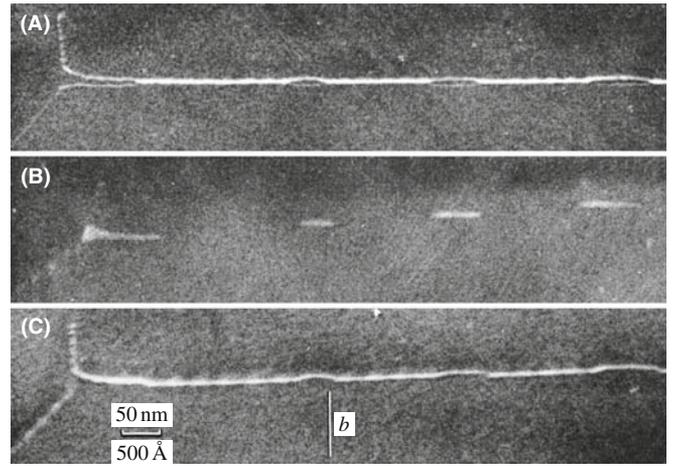


FIGURE 27.18. WB image of a dislocation in Si which has both dissociated and constricted segments: (A) $\mathbf{g} \cdot \mathbf{b} = 2$; both partial dislocations are visible. (B) $\mathbf{g} \cdot \mathbf{b}_T = 0$ showing SF contrast. (Note: no fringes.) (C) $\mathbf{g} \cdot \mathbf{b} = 1$; only one partial dislocation is visible.

within the detection limit of the WB technique. Comparison with the BF images in Figure 26.8 is instructive. Note that in Figure 27.19A the SF in the node does not show fringes because the SF plane is parallel to the surface of the specimen; we just tilted the specimen.

Example 3. We mentioned that the peak moves to the other side of the dislocation if we reverse \mathbf{b} . This is exactly what happens for a dislocation

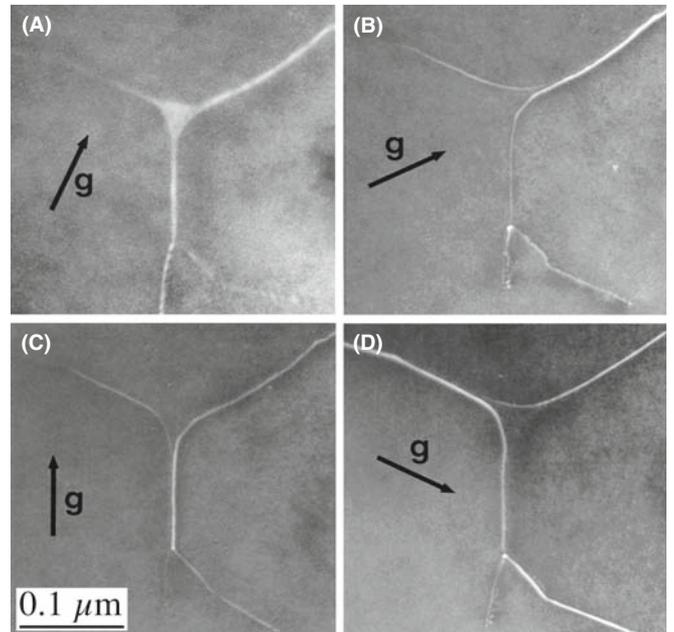


FIGURE 27.19. WB images of a pair of dislocation nodes formed by the dissociation of interacting dislocations lying on a {111} plane in a Cu alloy. The SF is imaged in (A), and imaging with different reflections (B–D) the partial dislocations are out of contrast when $\mathbf{g} \cdot \mathbf{b} = 0$. Compare with the BF image in Figure 26.8 and note the difference in magnification.

dipole as you can see in Figure 27.20. This is a complicated figure except that you can use it not only to see the inside-outside contrast in WB

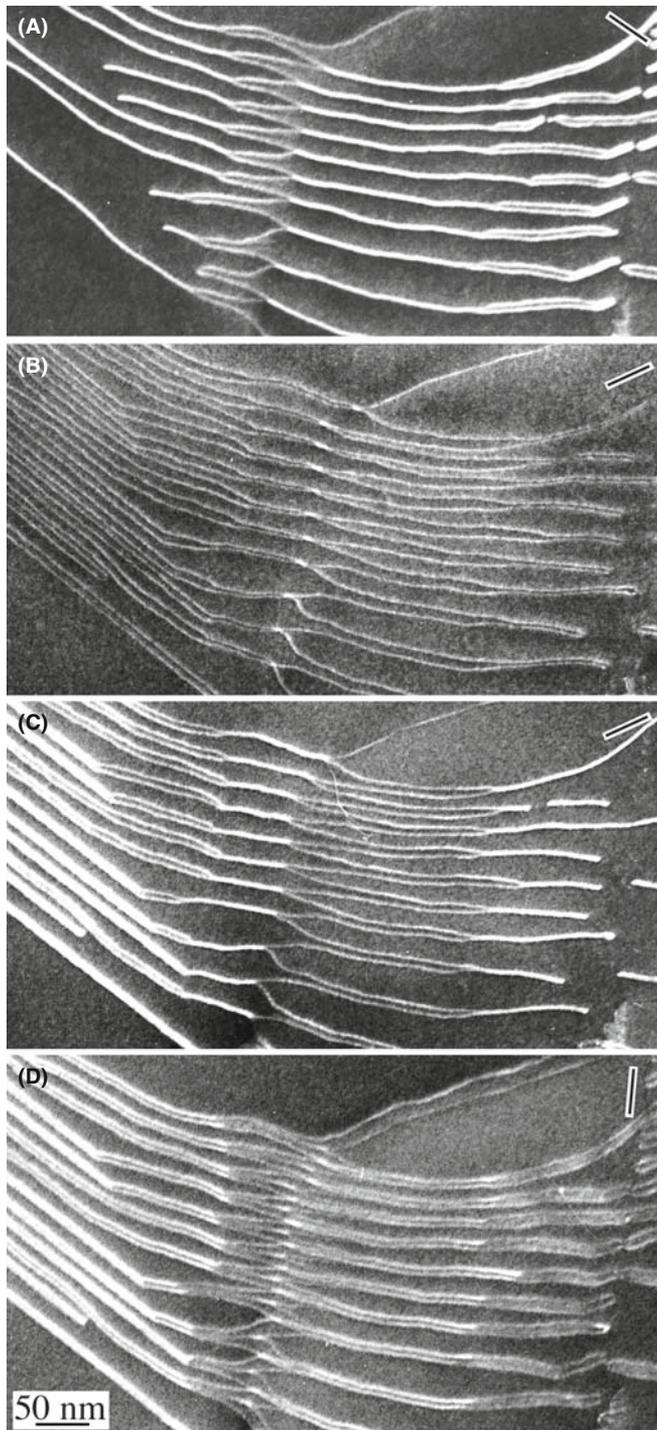


FIGURE 27.20. Four WB images showing an array of dislocation dipoles in a Cu alloy having a low stacking-fault energy. The reflections are all 220-type and the dislocations all lie on (111) planes which are nearly parallel to the surface of the specimen. All the dislocations are dissociated. All the Shockley partial dislocations are in contrast in (D) while half are out of contrast in (A–C). Notice that the narrower images are brighter than the wider ones; the strain is large in between the dislocations but decreases rapidly outside the dipole since the total Burgers vector of a dipole is zero.

images of dislocation dipoles but also as an exercise in $\mathbf{g} \cdot \mathbf{b}$ analysis. A dislocation dipole is a pair of dislocations identical in every way, apart from the sign of the Burgers vector. If we now reverse \mathbf{g} , then both peaks move to the other side of their respective dislocations. This change in contrast is referred to as inside-outside contrast and is commonly seen on dislocation loops, which are themselves closely related to these dipoles. Loops are just more ‘equiaxed’ than dipoles and always consist of one dislocation (which may be dissociated). The images shown in Figure 27.20B and C illustrate the dramatic change in contrast which you can see on reversing \mathbf{g} . Some of the dipoles completely disappear in Figure 27.20A–C because they are a special form of defect known as a faulted dipole. Such dipoles usually give very low contrast in strong-beam BF images because the dislocations are always very close together so that their strain fields overlap and cancel, and the lattice is thus only distorted over very small distances. All the dislocations are in contrast in Figure 27.20B: it’s the $\mathbf{g} \cdot \mathbf{b} = 2$ image. When we use the WB techniques, we are probing the structure on these very small dimensions and the contrast can be high. Again compare with the BF image in Figure 26.13.

Examples 4 and 5. The WB technique allows us to see features which would be hidden if we used strong-beam imaging. Figure 27.21 gives a simple example where an inclined SF is cutting through several dissociated dislocations. The interaction of the two defects would be masked by the SF fringes in the strong-beam image but is clearly visible in the WB image. Seeing small particles close to dislocations is difficult in strong-beam imaging. Although not easy in WB, Figure 27.22 does illustrate that it can be done. This image shows, for example, that the behavior of the dissociated dislocation is different on each side of the particle.

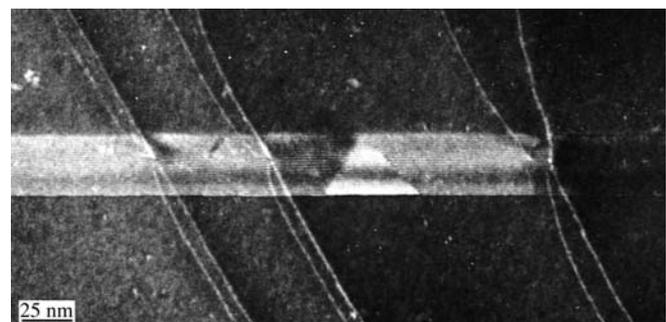


FIGURE 27.21. WB image of an inclined SF cutting through a series of dissociated dislocations lying parallel to the surface of the Cu-alloy specimen.

27.9 OTHER THOUGHTS

27.9.A Thinking of Weak-Beam Diffraction as a Coupled Pendulum

We can illustrate the principle which underlies the increase in intensity in the WB image close to a dislocation using the mechanical analog of a coupled pendulum. A diagram is shown in Figure 27.24. The two pendula are connected (coupled) by a third string. If we start the left pendulum swinging but hold the connecting string, the right-hand pendulum remains stationary. Now release the connecting string. You will see that the right-hand pendulum now begins to swing. If we let the process continue, eventually the right-hand pendulum is swinging as much as the original one did, but the original one is stationary: this is the strong-beam analog! All the kinetic energy has been transferred from one pendulum to the other. Given more time, the pendulum will return to the original condition. Now repeat the exercise but hold the connecting string again after the right-hand pendulum has begun to swing; you will notice that both pendula continue to swing, each with a constant amplitude. The role of the connecting string is to couple the two pendula (beams) so that we transfer energy from one beam to the other. In WB TEM, the defect acts as the connecting string. The two beams are only coupled over a short length as they travel past the defect. We can plot this amplitude (or intensity); try this as an exercise.

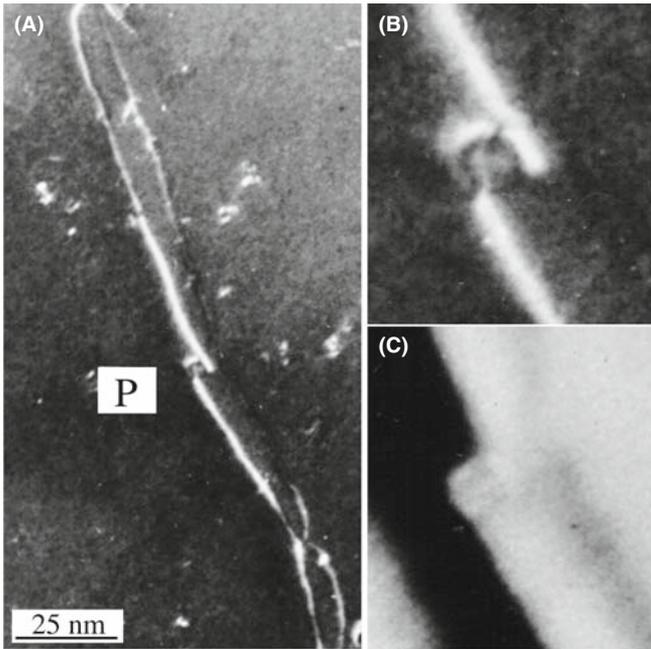


FIGURE 27.22. (A) WB image of a dissociated dislocation interacting with a particle (P) in a Cu alloy. (B) and (C) are enlargements of the WB and corresponding BF images, respectively. Note the difference in detail.



FIGURE 27.23. WB image showing the dissociation of a group of dislocations which are inclined to the foil surface and give wedge-shaped SFs. The shape of the SFs is caused by surface stresses.

Figure 27.22B and C compares magnified WB and BF images from the region close to the particle, and again stresses the advantage of WB over BF imaging.

Example 6. We noted in Section 26.8 that the surface of the specimen can affect the geometry of the defects we are examining. In general, the specimen needs to be thinner for WB imaging than for strong-beam imaging. Therefore, the surface can affect not only the appearance of the image but also the actual geometry of the defect. Figure 27.23 shows an example where this effect is particularly clear. Dislocations that were uniformly separated in the bulk material now appear wedge-shaped: the effects of the two surfaces are different in this case.

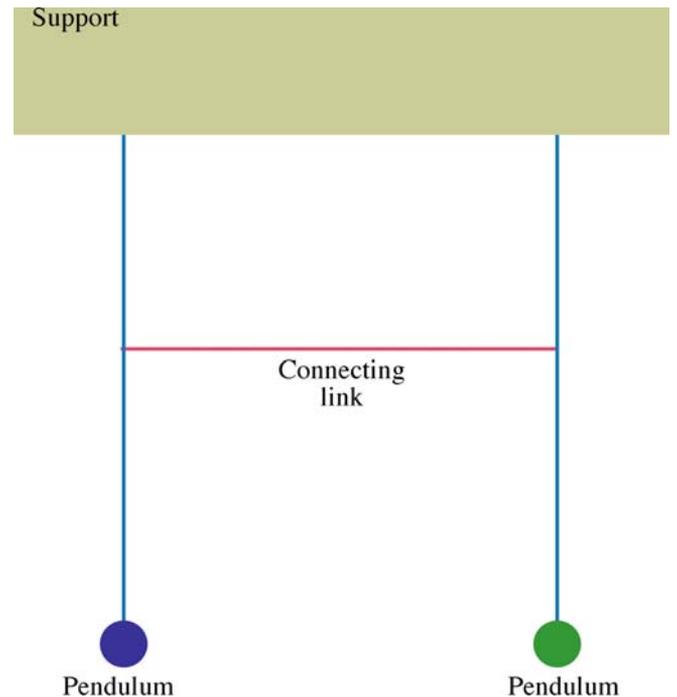


FIGURE 27.24. The coupled pendulum: a thought experiment.

27.9.B Bloch Waves

We discussed Bloch waves in Chapter 14. The difficulty in applying Bloch-wave analysis to the WB situation is that we are now usually interested in defects while Bloch waves are properties of the perfect crystal. However, we can offer some basic comments. For the reflection \mathbf{g} to give a WB image, $|\phi_{\mathbf{g}}|$ must be much smaller than unity in the regions of perfect crystal but, in strained regions, a change $\Delta\psi^{(j)}$ in the amplitude of the Bloch wave j can give rise to a change $\Delta\phi_{\mathbf{g}}$. Cockayne has shown that the appreciable contrast which can then be present in the WB image is due, in the two-beam approximation, to the interband scattering from Bloch wave 1 to Bloch wave 2. In the general case, the scattering is from the branch of the dispersion surface with the largest $\psi^{(j)}$ to those branches with the largest $C_{\mathbf{g}}^{(j)}$, i.e., from the Bloch wave with the largest amplitude to the one which is most strongly excited. The dispersion surface for the $\mathbf{g}(3\mathbf{g})$ diffraction geometry is shown in Figure 27.25. It's an instructive exercise to reread this paragraph thinking how each statement relates to this figure and to consider other diffraction geometries, e.g., $\mathbf{0}(2\mathbf{g})$.

The Bloch-wave analysis of the problem leads to two further points which simplify the interpretation of WB images

- The diffraction conditions should be such that only one interband scattering process is important.
- In the two-beam approximation, in order for the image peaks to show sufficient contrast, it is generally found that $w (= s\xi_{\mathbf{g}})$ is greater than ~ 5 .

You can satisfy the first requirement by ensuring that no reflections are strongly excited. The second condition is usually already satisfied because of the more stringent requirement that s should be greater than 0.2 nm^{-1} . For example, for a $\{2\bar{2}0\}$ reflection in copper with 100-keV electrons, w is automatically greater than 8 since $\xi_{\mathbf{g}}$ is 42 nm.

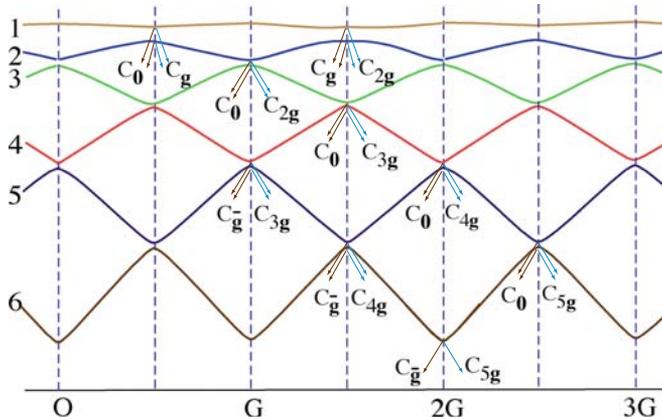


FIGURE 27.25. Dispersion-surface construction which is used to discuss the $\mathbf{g}(3\mathbf{g})$ geometry. The BZB is then at $1.5\mathbf{G}$ and tells you which reflections are strongly coupled; see Figure 15.9.

The $\mathbf{g}(3.1\mathbf{g})$ condition may also be preferable to $\mathbf{g}(-1.1\mathbf{g})$ on theoretical grounds if images are to be compared with computed profiles made using the column approximation; i.e., it simplifies your interpretation. The basis for this suggestion is that the region of the dispersion surface from which the scattering occurs is flatter for the $\mathbf{g}(3.1\mathbf{g})$ diffraction geometry than for the $\mathbf{g}(\bar{\mathbf{g}})$ case.

27.9.C If Other Reflections are Present

Several times in the previous discussion, we have said that no reflections should be strongly excited. Much of our thinking has been based on the two-beam approximation we introduced in Chapter 13. When you are using WB conditions, you must be even more careful. Consider the $\mathbf{g}, 3\mathbf{g}$ geometry shown in Figure 27.5. We form the WB image using reflection \mathbf{g} so electrons are weakly scattered from the O beam into the G beam. However, once in the G beam, they can be strongly scattered into the $2\mathbf{G}$ beam. We can picture this process by drawing the new Ewald sphere for the 'new' incident beam, G; this sphere passes through $2\mathbf{G}$!

For the mathematically inclined, you can go back to the many-beam equations, which we introduced briefly in Chapter 14. The coupling of beams \mathbf{g} and \mathbf{h} is determined by $(s_{\mathbf{g}} - s_{\mathbf{h}})$ and has an extinction distance given by $(\xi_{\mathbf{g}-\mathbf{h}})$. If $s_{\mathbf{g}}$ and $s_{\mathbf{h}}$ are equal, then the coupling between these beams will be strong. Furthermore, the characteristic length for the coupling in this example will be $\xi_{2\mathbf{g}-\mathbf{g}}$ or $\xi_{\mathbf{g}}$ which is what you would have guessed by thinking about Figure 27.5A and C.

27.9.D The Future Is Now

Several new developments are changing how we practice the WB technique. The main point here is that you should remember the principles because they will not change.

- Slow-scan CCD cameras give a very linear response and therefore make quantitative analysis of WB images possible. For this to happen, computer modeling of the defect and simulation of the image will be needed. We will return to this topic in Chapter 30 but mainly for HRTEM.
- An FEG and energy filtering allow us either to minimize the effect of variations in the energy (decrease diffuse scattering) or to form WB images using particular sections of the energy-loss spectrum. To make use of this extra information, we need to extend the theory.
- Image processing and frame averaging should allow us to reduce the noise, aid quantification, and use WB microscopy for in-situ studies.

Image simulation, as we described in Section 26.11, will allow us to be more quantitative in our interpretation of WB images when we actually have quantitative recording.

CHAPTER SUMMARY

The basic idea of the WB technique is very simple: using a large value of s gives a small ξ_{eff} and hence a narrow image of most defects, since the width of a dislocation is related to $\xi_{\text{eff}}/3$. What you should remember is that the value of s for a particular diffraction condition $\mathbf{g}(n\mathbf{g})$ depends not only on n and \mathbf{g} , but also on the lattice parameter of the crystal and the wavelength of the electrons. You will see the ‘magic number’ $s = 0.2 \text{ nm}^{-1}$ quoted often.

- Remember that this number gives a rule of thumb if you want to do quantitative analysis. It does not usually correspond to $\mathbf{g}(3\mathbf{g})$.
- Don’t use the $\mathbf{g}(3\mathbf{g})$ condition without calculating the value of $\mathbf{s}_{\mathbf{g}}$.
- The term $\mathbf{s}\cdot\mathbf{R}$ has been neglected in this analysis; we usually assume that the deformable model from Section 25.13 is valid.

You can often get all the information you need with less effort using a somewhat smaller value of s . As always, the longer you take to perfect the image, the more likely you are to alter your specimen, especially the defect structure.

Finally, remember that the diffracted beam travels parallel to \mathbf{k}_D . Therefore, the image of any defect is also projected in this direction. Even though the Bragg angle is small, this means, for example, that the apparent separation of defects in the image may not be equal to their horizontal separation relative to their glide planes if the defects are located at different heights in the specimen. This projection error can vary depending on the \mathbf{g} and s used to form the image and the orientation of the specimen.

THE TECHNIQUE

Cockayne, DJH 1972 *A Theoretical Analysis of the Weak-Beam Method of Electron Microscopy [Defectoscopy]* *Z. Naturf.* **27a**, 452–460. Derives equation 27.2.

Cockayne, DJH 1981 *Weak-Beam Electron Microscopy* *Ann. Rev. Mater. Sci.* **11**, 75–95. A review of WB.

Cockayne, DJH, Ray, ILF and Whelan, MJ 1969 *Investigations of Dislocation Strain Fields Using Weak Beams* *Phil. Mag.* **20**, 1265–1270. The original paper; derives equations 27.10 and 27.15.

Hirsch, PB, Howie, A and Whelan, MJ 1960 *A Kinematical Theory of Diffraction Contrast of the Electron Transmission Microscope Images of Dislocations and Other Defects* *Proc. Roy. Soc. London A* **252**, 499–529. Always worth another look.

Stobbs, WM 1975 *The Weak Beam Technique in Electron Microscopy in Materials Science*, vol. **II** (Eds. U Valdrè and E Ruedl), p. 591–646, CEC Brussels. A masterful review which also compared WB and a Stradivarius.

Stobbs, WM and Sworn, C 1971 *The Weak Beam Technique as Applied to the Determination of the Stacking-Fault Energy of Copper* *Phil. Mag.* **24**, 1365–1381. WB calculations using anisotropic elasticity.

SOME DEFECTS

Carter, CB 1984 *What’s New in Dislocation Dissociation?* in *Dislocations –1984* (Eds. P Veysière, L Kubin, and J Castaing), p. 227, Editions du CNRS Paris. A 50th birthday review of dissociated dislocations.

Carter, CB and Holmes, SM, 1975 *The Study of Faulted Dipoles in Copper Using Weak-Beam Electron Microscopy* *Phil. Mag.* **32** (3), 599–614.

Carter, CB, Mills, MJ, Medlin, DL and Angelo, JE 1995 *The 112 Lateral Twin Boundaries in FCC Metals in 7th International Conference Intergranular and Interphase Boundaries in Materials*, Lisbon, Portugal. The ‘thickness’ of a plane is important in WB! (Section 27.7.)

Föll, H, Carter, CB and Wilkens, M 1980 *Weak-Beam Contrast of Stacking Faults in Transmission Electron Microscopy* *Phys. stat. sol. (A)* **58**, 393–407. Discusses the anomalous WB contrast from SFs.

Gerthsen, D and Carter, CB 1993 *Stacking-Fault Energies of GaAs* *Phys. stat. sol. (A)* **136**, 29–43. An experimentalist’s comparison of WB and HRTEM.

Hazzledine, PM, Karnthaler, HP and Wintner, E 1975 *Non-parallel Dissociation of Dislocations in Thin Foils* *Phil. Mag.* **32**, 81–97. Used WB to show the unambiguous effect of surfaces on dislocation core splitting: a paper with broad implications for the microscopists.

Wilson, AR and Cockayne, DJH 1985 *Calculated Asymmetry for Weak Beam Intrinsic Stacking Fault Images* *Phil. Mag.* **A51**, 341–354. More on the WB contrast from SFs.

SELF-ASSESSMENT QUESTIONS

- Q27.1 Write down the equation relating s_{eff} to s .
- Q27.2 Write down the equation relating ξ_{eff} to $s_{\mathbf{g}}$.
- Q27.3 Write down the equation relating s to n , \mathbf{g} , and λ .
- Q27.4 If the $3\mathbf{g}$ reflection is excited, where are the \mathbf{g} and $3\bar{\mathbf{g}}$ Kikuchi lines?
- Q27.5 What is the optimum value of s for WB and why is this a compromise value?
- Q27.6 If an Si specimen is set to $\mathbf{g}, 3\mathbf{g}$ conditions at 100 kV, how wide will the image of a dislocation be?
- Q27.7 How will the image differ if we use 300 kV rather than 100 kV for $\mathbf{g}, 3\mathbf{g}$ conditions?
- Q27.8 Name the principal methods used for calculating WB images.
- Q27.9 What is the radius of the phasor diagram of a perfect crystal in the WB condition?
- Q27.10 Why is the separation of the two lines in a $\mathbf{g}, \mathbf{b}=2$ WB image not equal to the separation of the two partial dislocations which cause these lines?
- Q27.11 A WB image can show two lines when $\mathbf{g}, \mathbf{b}=2$ for a non-dissociated dislocation. How can this happen?
- Q27.12 Why is the circumference of the circle in a phasor diagram of a perfect crystal imaged under WB conditions interesting/instructive?
- Q27.13 Why might the contrast from an extrinsic stacking fault differ from that from an intrinsic SF under WB conditions?
- Q27.14 Sketch the phasor diagrams which show the difference behavior of a dislocation image in $\pm\mathbf{g}$.
- Q27.15 What extinction distance do you expect to have for the coupling of \mathbf{g} and $2\mathbf{g}$ when $3\mathbf{g}$ is excited?
- Q27.16 In WB images, we usually set $w \geq 5$. Why?
- Q27.17 Why do we avoid exciting other reflections when using the WB technique? Is it more important than when forming BF images?
- Q27.18 What is the classic value for $s_{\mathbf{g}}$ in WB images and why was this value originally chosen?
- Q27.19 Why is the condition with s positive preferred over that with s negative even though both conditions may have the same value of s ?
- Q27.20 Why is WB microscopy more difficult as we go to higher kV or to lower kV? (Higher and lower indicate relative to 100 kV.)

TEXT-SPECIFIC QUESTIONS

- T27.1 Starting from Figures 27.1 and 27.4, deduce equation 27.5.
- T27.2 Check Table 27.1 (any errors?) and add a column for 120-keV electrons. (Why?)
- T27.3 Consider the lowest part of Figure 27.7C (a line at and parallel to the bottom of the figure). Draw an accurate sketch of what the cross section of the sample might be and justify your answer.
- T27.4 If the sample in Figure 27.8 is close to the (111) plane, deduce the character of the dislocations in this image which lie in the (111) plane. Explain your answer very carefully.
- T27.5 If we have $\mathbf{g}(3\mathbf{g})$ conditions, through what angle must the planes near the dislocation bend in order to be in the Bragg condition in Figure 27.9?
- T27.6 Using equation 27.9, deduce the width of the dislocation image for $\mathbf{g}(3\mathbf{g})$ and $\mathbf{g}(4\mathbf{g})$ conditions if \mathbf{g} is $2\bar{2}0$ and the sample is parallel to the (111) plane.
- T27.7 Consider Figure 27.15. Deduce how many $\{111\}$ planes apart the continuous and dark (in D) SFs are. Explain all the assumptions and steps in your answers.
- T27.8 Consider Figure 27.19. Deduce the character of all of the perfect and partial dislocations in this image assuming that they all lie in the (111) plane.
- T27.9 Consider Figure 27.23. If the sample is (111) and the $\mathbf{g}(3\mathbf{g})$ conditions were used to form the image, deduce the thickness of the sample. Explain all your assumptions and arguments.
- T27.10 Consider Figure 27.21. What and where (indices and direction) is the most likely \mathbf{g} used to form this image? Explain your reasoning.
- T27.11 (An extra challenge question) Consider Figure 27.16. When will you see the left phasor and when will you see the one on the right, assuming you are looking at the same dislocation.
- T27.12 Determine the value of s for 220 taking 100 kV and Cu (Figure 27.2).
- T27.13 Figure 27.8. Estimate the value for the SFE. Assume a Cu fcc alloy, $\mathbf{g}, 3\mathbf{g}$ conditions.
- T27.14 Estimate the maximum thickness of the sample in Figure 27.13. Spinel with 220, $\mathbf{g}, 3\mathbf{g}$, 100 kV.
- T27.15 Figure 27.14. Phasor diagram-100 kV. For Cu, $\mathbf{g}=220$ and $\mathbf{g}, 3\mathbf{g}$ conditions. How thick is the sample?
- T27.16 Challenging. Derive equation 27.8 explaining all your assumptions.
- T27.17 Challenging: Figure 27.9 greatly exaggerates the curvature of the dislocations. Estimate the length of the column that contributes to \mathbf{g} for $\mathbf{g}, 3\mathbf{g}$ and 100-kV electrons.
- T27.18 What value of n would you use to produce WB images of dislocation for these reflections assuming 100-, 200-, and 300-kV electrons? (a) $(11\bar{2}0)$ in Al_2O_3 (alumina); (b) (220) in MgAl_2O_4 (spinel); (c) (111) in MgAl_2O_3 ; (d) (222) in YAG; (e) (220) in Si; (f) (111) in Si; (g) (110) in W; (h) (200) in Au.

- T27.19 If the specimen is oriented for $g,3g$ (Cu, 100 kV) throughout, what angle must you tilt to set up $g,4g$ conditions? (Answer in degrees and radians.)
- T27.20 Imagine four overlapping SFs in a crystal. What is the maximum intensity you could produce in the image? Give your answer as a multiple of the intensity possible for a perfect crystal. Use Cu 100 kV and $g,5g$ for $g = 111$.
- T27.21 Discuss how you might use weak-beam imaging to profile the topography of a thin sample even though the thickness can be changed by undulations on either surface. Hint: this question is related to electron tomography.
- T27.22 Discuss how weak-beam images will be improved using a new energy-filtered TEM equipped with a drift-compensated stage. Will you still use the same value of s ?
- T27.23 Professor J wants to use weak-beam imaging to obtain information on the core of dislocations. What factors will ultimately limit the information she can obtain? How will these limitations depend on the nature of the sample?