22

Amplitude Contrast

CHAPTER PREVIEW

We've already mentioned in Chapters 2–4 that TEM image contrast arises because of the scattering of the incident beam by the specimen. The electron wave can change both its amplitude and its phase as it traverses the specimen and both types of change can give rise to image contrast. Thus a fundamental distinction we make in the TEM is between *amplitude contrast* and *phase contrast*. In most situations, both types of contrast actually contribute to an image, although we usually select conditions so that one will tend to dominate. In this chapter, we'll discuss only amplitude contrast and we'll see that there are two principal types, namely, *mass-thickness contrast* and *diffraction contrast*. This kind of contrast is observed in both TEM and STEM and in both BF and DF images. We'll discuss the important differences between the images formed in each of these two modes of operation. Then we'll go on to discuss the principles of diffraction contrast, which are sufficiently complex that it takes Chapters 24–27 to show you how this form of contrast is used to identify and distinguish different crystal defects. Diffraction-contrast imaging came into prominence in about 1956 when it was realized that the intensity in a diffracted beam depends strongly on the deviation parameter, **s**, and that crystal defects rotate the diffracting planes near the defect. Therefore, the diffraction contrast from regions close to the defect will depend on the properties (in particular, the strain field) of the defect. We'll then consider phase contrast and how it can be used to image atomic-level detail in Chapters 23 and 28. Other forms of TEM imaging and variations on these major types of contrast are gathered in the catch-all Chapter 29.

22.1 WHAT IS CONTRAST?

Before we start to describe specific types of contrast it's worth a quick reminder of what exactly we mean by the word 'contrast.' We can define contrast (C) quantitatively in terms of the *difference* in intensity (ΔI) between two adjacent areas

$$C = \frac{(I_2 - I_1)}{I_1} = \frac{\Delta I}{I_1} \tag{22.1}$$

In practice your eyes can't detect intensity changes $<5\%$ and even $<10\%$ is difficult. So unless the contrast from your specimen exceeds >5–10% you won't see anything on the screen or on the recorded image. However, if your image is digitally recorded, you can enhance low-contrast images electronically to levels at which your eyes can perceive the contrast. We'll return to image processing and contrast enhancement in Chapter 31.

So we see contrast in TEM images as different levels of green light coming from the viewing screen or the computer display. On the photograph or computer screen, contrast is seen as different gray levels and our eyes can only discern about 16 of these. If we want to quantify the contrast, we need to make direct intensity measurements, e.g., via a microdensitometer if we're using film or directly from the CCD, but usually it's only necessary to see qualitative differences in intensity. Be careful not to confuse *intensity* with *contrast* when you describe your images. We can have strong or weak contrast but not bright or dark contrast. The terms 'bright' and 'dark' refer to density (number/unit area) of electrons hitting the screen or detector, and the subsequent light emission that we see. In fact, you generally get the strongest contrast under illumination conditions that lower the overall intensity. Conversely, you can try to increase the number of electrons falling on the screen by condensing the beam onto a reduced area of the specimen, but then you'll usually lower the image contrast. These points are summarized in Figure 22.1, which defines intensity and contrast.

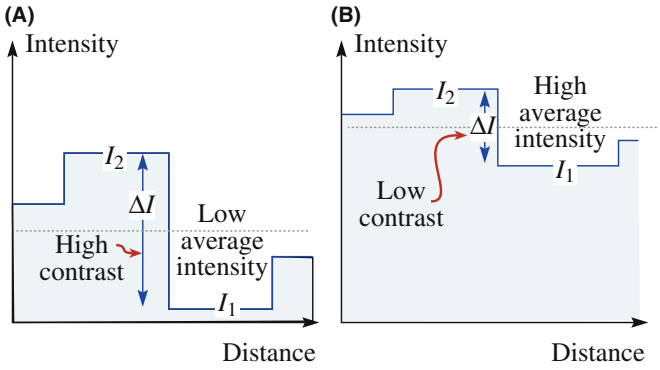


FIGURE 22.1. Schematic intensity profiles across an image showing (A) different intensity levels (I_1 and I_2) and the difference (ΔI) between them which defines the contrast. Generally, in a TEM, if the overall intensity is increased (B) the contrast decreases.

Before we discuss the two forms of amplitude contrast in detail, we need to remind you of the operational principles for creating amplitude contrast in your image. We obtain contrast in our images either by selecting specific electrons or excluding them from the imaging system. We have two choices: we can form either BF or DF images by selecting the direct or scattered electrons, respectively. So this chapter builds on what you learned about electron scattering in Chapters 2–4 and how to operate the TEM, described in Chapter 9.

22.2 AMPLITUDE CONTRAST

Amplitude contrast results from variations in mass or thickness or a combination of the two: the thickness variation can produce contrast because the electron interacts with more material (hence, more mass). Alternatively, diffraction can vary locally because the specimen is not a perfect, uniformly thin sheet.

22.2.A Images and Diffraction Patterns

If you look back at Figure 2.1, you'll see that the uniform electron intensity in the incident beam is transformed into a non-uniform intensity after scattering by the specimen. So a variable electron intensity hits the viewing screen or the electron detector, which translates into contrast on the screen. Now you also know that the DP shows you this non-uniformity because it separates the diffracted and direct beams. Therefore, a fundamental principle of imaging in the TEM is: first, *view the DP*, since this pattern tells you how your specimen is scattering. The relationship between the image and the DP is most critical for crystalline specimens showing diffraction contrast. However, you should view the DP first, no matter which contrast mechanism you want to use, or the specimen you are studying.

22.2.B Use of the Objective Aperture or the STEM Detector: BF and DF Images

In order to translate the electron scatter into interpretable amplitude contrast, we select *either* the direct beam or a diffracted beam in the SADP to form BF and DF images, respectively. (Remember that a small aperture will enhance the contrast but might decrease the resolution.) Note we are justified in using the 'beam' terminology, since the electrons have left the specimen. We've already seen in Section 9.3 that in a TEM we select the direct or a scattered electron beam with the objective aperture. Remember, if we form an image without the aperture, the contrast will be poorer (lower) because many beams then contribute to the image so we lose diffraction contrast. Furthermore, aberrations due to the off-axis electrons will make it impossible to focus your image. Your choice of the aperture size governs which electrons contribute to the image and you thus control the contrast.

Figure 22.2 shows a DP from a single-crystal Al specimen with two possible positions of an objective aperture indicated by white circles. In this figure, if the aperture is in position A it selects the direct beam only and thus a BF image will be formed in the image plane of the lens. This arrangement will produce amplitude contrast whether the specimen is crystalline (as in this case) or amorphous. If the aperture is in position B, it will select only electrons scattered in that specific direction. Thus a DF image will be formed. Traditionally (for 50 years), we have tilted the incident beam such that the scattered electrons remain on axis when forming a DF image. We thus create a centered dark-field (CDF) image, which we described back in Section 9.3. We'll discuss CDF techniques later in Section 22.5 and we'll usually assume CDF is the operational mode in DF imaging. However, you already know that it would be much better if we did not tilt the specimen but instead displaced the aperture (so the BF and DF are formed with the same diffraction conditions). We'll see later

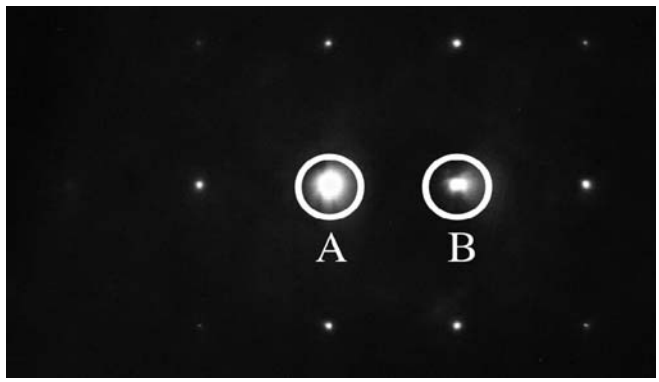


FIGURE 22.2. The relationship between the objective aperture and the diffraction pattern for forming (A) BF and (B) DF images. The circles show the location of the objective aperture.

that if you want to observe phase contrast, you have to use an objective aperture that is large enough to gather more than one beam.

BF AND DF
The two basic ways to form amplitude-contrast images.

In a STEM we select the direct or scattered beams in an equivalent way but use detectors rather than apertures. We compare the two different operational modes in Figure 22.3. Again, we saw back in Section 9.4 that we insert a BF on-axis detector, or an annular DF (ADF) detector, in a plane that's conjugate with the back focal plane. We control which electrons fall on which detector and thus contribute to the image by adjusting the post-specimen (imaging) lenses to change the camera length. Clearly, for DF imaging, the ADF detector will probably gather many more electrons than the objective aperture, which is good for imaging some specimens and bad for imaging others, as we'll see.

So, in summary, we can create BF or DF images with the direct beam or scattered beams, respectively. In order to understand and control the contrast in these images you need to know what features of a specimen cause scattering and what aspects of TEM operation affect the contrast.

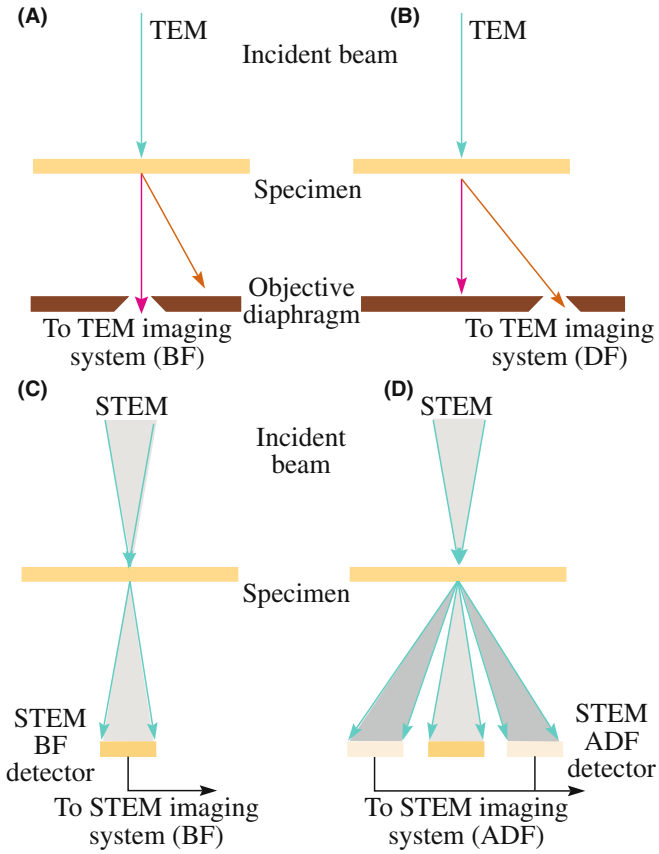


FIGURE 22.3. Comparison of the use of an objective aperture in TEM to select (A) the direct or (B) the scattered electrons forming BF and DF images, respectively. In STEM we use (C) an on-axis detector or (D) an off-axis annular detector to perform equivalent operations.

22.3 MASS-THICKNESS CONTRAST

Mass-thickness contrast arises from incoherent elastic scattering (Rutherford scattering) of electrons. As we saw back in Chapter 3, the cross section for Rutherford scatter is a strong function of the atomic number Z (hence the mass or the density, ρ) and the thickness, t, of the specimen. Rutherford scattering in thin specimens is strongly forward peaked. Therefore, if we form an image with electrons scattered at low angles ($<\sim 5^\circ$), mass-thickness contrast dominates (but it also competes with Bragg-diffraction contrast). However, we'll also see that at high angles ($>5^\circ$), where Bragg scattering is usually negligible, we can pick up low-intensity, scattered beams. (The former is often referred to as coherent and the latter as incoherent; we'll discuss these terms later.) The intensity of these beams depends on atomic number (Z) only. Thus we can also get so-called Z-contrast, which contains elemental information like that in BSE images in the SEM. We can obtain these images with atomic resolution, particularly in a DSTEM. It is also feasible to form BSE images in a TEM but, because the specimen is thin, the number of BSEs is so small that the images are noisy and of poor quality, so no one does it. You shouldn't waste your money buying a BSE detector (but a secondary-electron detector can be very useful).

Mass-thickness contrast is most important if you are looking at non-crystalline materials, such as polymers and it is *the* critical contrast mechanism for biological scientists. But as we'll see, any variations in mass and thickness will cause contrast. As you learned in Chapter 10, it's almost impossible to thin a bulk sample uniformly (although a FIB can get close) and so nearly all real specimens will show some mass-thickness contrast. In some cases this will be the only contrast you can see.

In this section, we'll assume that there is no contribution to the image from diffraction contrast. This is automatically so if the specimen is amorphous. If the specimen is crystalline, then remove the objective aperture or use the ADF detector to minimize any diffraction contrast. You should still use an objective aperture to enhance the mass-thickness contrast to minimize the effects of lens aberrations. You'll still create BF and DF images of amorphous materials.

22.3.A Mechanism of Mass-Thickness Contrast

The mechanism by which differences in mass and thickness cause contrast is shown in Figure 22.4 and at this

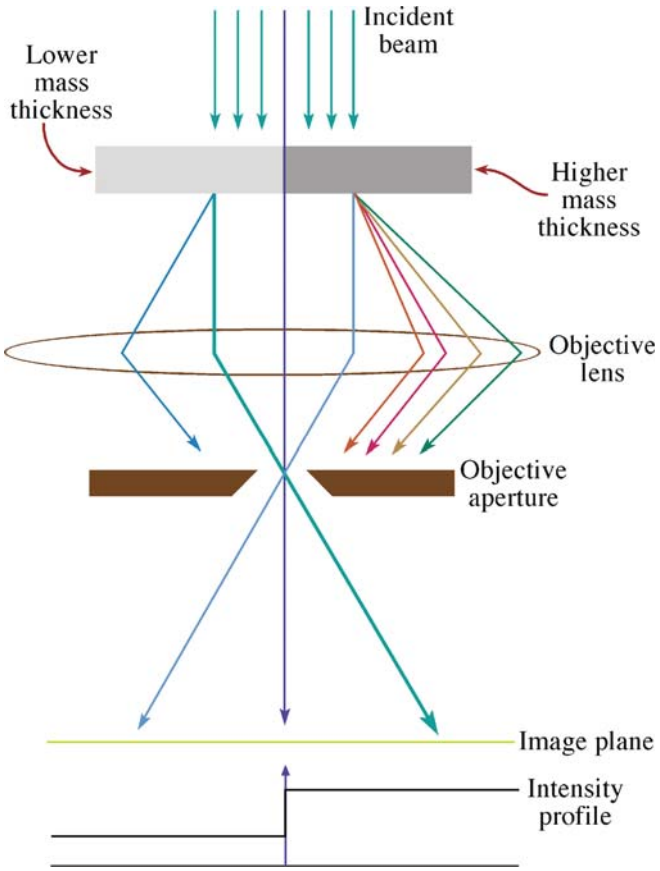


FIGURE 22.4. Mechanism of mass-thickness contrast in a BF image. Thicker or higher-Z areas of the specimen (darker) will scatter more electrons off axis than thinner, lower mass (lighter) areas. Thus fewer electrons from the darker region fall on the equivalent area of the image plane (and subsequently the screen), which therefore appears darker in BF images.

stage we'll talk about the process qualitatively. As electrons go through the specimen, they are scattered off axis by elastic nuclear interactions, i.e., Rutherford scattering. You know two factors from Chapter 3

- The cross section for elastic scattering is a function of Z.
- As the thickness of the specimen increases, there will be more elastic scattering because the mean free path remains fixed.

So, using a very simple, qualitative argument you would expect high-Z (i.e., high-mass) regions of a specimen to scatter more electrons than low-Z regions of the same thickness. Similarly, thicker regions will scatter more electrons than thinner regions of the same average Z, all other factors being constant. Usually, mass-thickness contrast images are interpreted in such a purely qualitative fashion, although we'll see a little later that it is possible to quantify the scattering intensity. So, as you can see from Figure 22.4, for the case of a BF image, thicker and/or higher-mass areas will appear darker than thinner and/or lower-mass areas. The reverse will be true for a DF image.

This is all you need to know for the simplest interpretation of mass-thickness contrast images. Sometimes mass-thickness contrast is explained in terms of different amounts of electron absorption within the specimen and so you may come across the expression 'absorption contrast.' We think that this term is misleading, because in thin foils the actual amount of electron absorption is small; scattering outside the aperture or the detector, not absorption within the specimen, causes the contrast. For much the same reason, we prefer not to use the term 'structure-factor contrast' that is sometimes used to describe this phenomenon, since this implies a Bragg contribution, which may or may not be present.

However, you should be aware that if there are small crystals of different atoms in a given foil thickness, differences in their structure factor (F) from that of the matrix will cause contrast changes, since $I \propto |F|^2$. For example, you can detect the presence of nanometer-size clusters of Ag atoms in very thin foils of Al alloys in this way. Conversely, an absence of atoms (e.g., a void) will also scatter differently, although Fresnel contrast (see Chapter 23) is a better way to detect voids and bubbles.

Let's first look at a few images showing mass-thickness contrast and see which TEM variables you can control.

22.3.B TEM Images

Figure 22.5A is a TEM BF image of some latex particles on an amorphous-carbon support film. Assuming the latex is predominantly carbon, we have a constant Z and varying t. So the latex particles are darker than the support film since they are thicker. What you are basically seeing is a shadow projection image of the latex particles. Because it is a projection image, you cannot say that the particles are spheres (which in fact they are). They could equally well be disks or cylinders. One way to reveal the shape in a single image is to shadow-coat the particles, i.e., evaporate a thin heavy metal (Au or Au-Pd) coating at an oblique angle as shown in Figure 22.5B. The shape of the shadow reveals the true shape of the particles.

Shadowing introduces some mass contrast to what was just a thickness-contrast image. If we assume the Au-Pd film is very thin compared to the carbon support film, then the contrast across the edge of the shadow is predominantly mass contrast, due to the difference in average Z of the Au-Pd and the carbon film. If the spheres are small, there may also be an intensity change across the latex spheres due to the preferential deposit of Au-Pd on the side of the sphere towards the source of the evaporated metal.

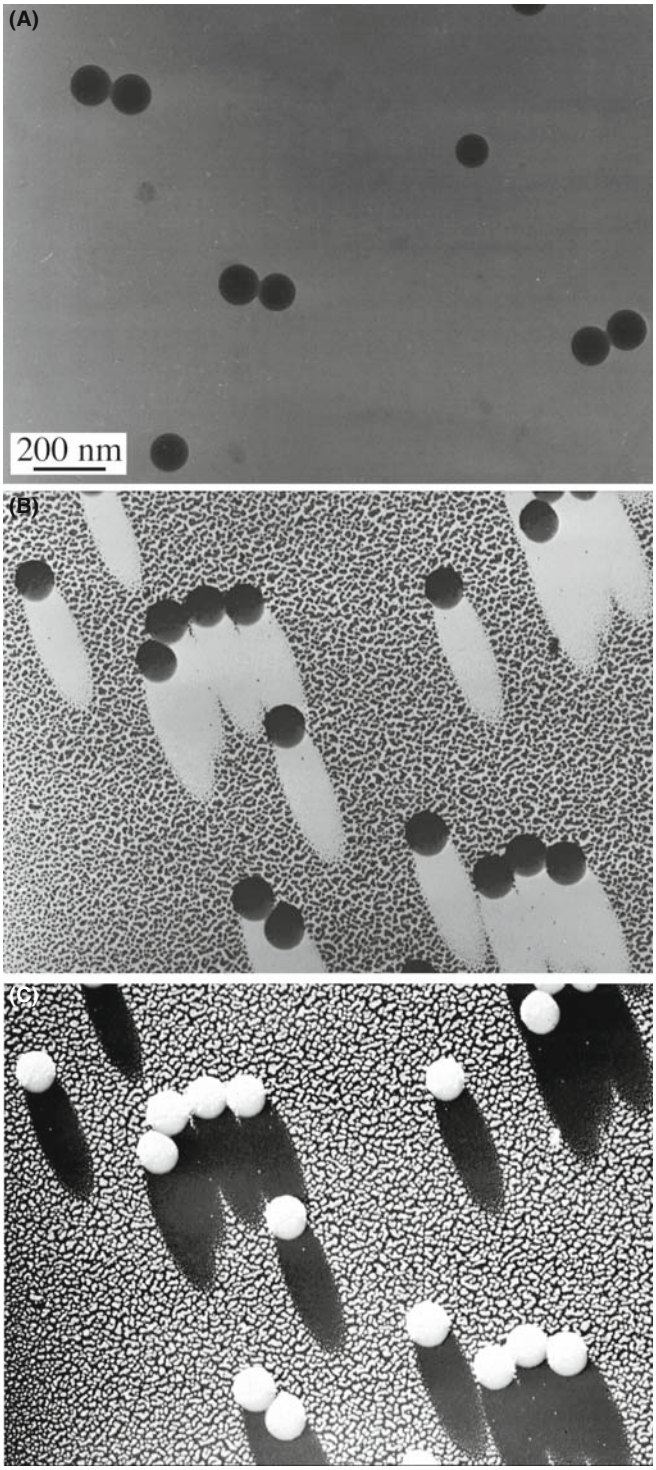


FIGURE 22.5. (A) TEM BF image of latex particles on a carbon support film showing thickness contrast only. (B) Latex particles on a carbon film shadowed to reveal the shape of the particles through the addition of selective mass contrast to the image. (C) Reverse print of (B) exhibits a 3D appearance.

It is an intriguing exercise to print Figure 22.5B in reverse (or take a DF image) as shown in Figure 22.5C. In this image, the latex spheres now appear to stand proud of the surface, even though you're still viewing a two-dimensional projected image. Because the shadows are now dark your brain interprets the picture as it would a reflected-light image and endows it with a 3D nature. While the interpretation in this case is correct, it may not always be so. Once again we stress that you must be careful when interpreting 2D images of 3D specimens.

In addition to the use of shadowing to enhance mass-thickness contrast, it is common practice to stain different areas of polymer and biological specimens with heavy metals such as Os, Pb, and U. The stain leaves the heavy metal in specific regions of the structure (e.g., at unsaturated C = C bonds in a polymer and cell walls in biological tissue) and therefore these areas appear darker in a BF image. Figure 22.6 shows a BF image of a stained two-phase polymer. Since the specimen is of constant thickness (it was ultramicrotomed) the image shows mass contrast only.

TEM VARIABLES

The TEM variables that affect the mass-thickness contrast for a given specimen are the objective aperture size and the kV.

If you select a larger aperture, you allow more scattered electrons to contribute to the BF image. So the contrast between scattering and non-scattering areas is lowered, although the overall image intensity increases. If you choose a lower kV, both the scattering angle and the cross section increase. Hence, more electrons will be scattered outside a given aperture, hitting the diaphragm, and contrast will increase at the expense of intensity. The decrease in intensity will be worse for TEMs with a thermionic source because the gun brightness decreases as the kV is lowered. Figure 22.7 shows how a smaller aperture size results in improved contrast.

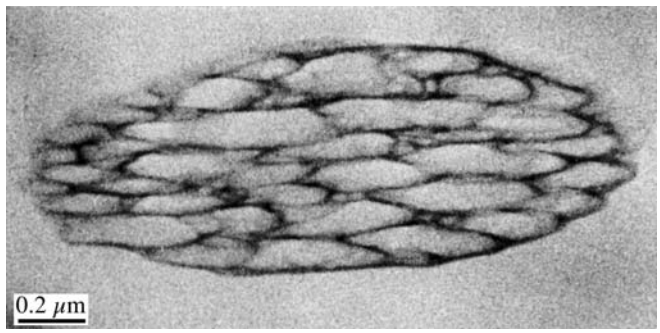


FIGURE 22.6. BF image of stained two-phase polymer exhibiting mass contrast due to the segregation of the heavy metal atoms to the unsaturated bonds in the darker phase.

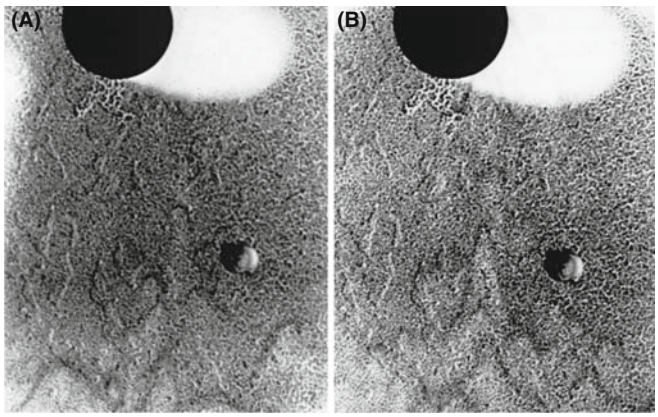


FIGURE 22.7. The effect of objective aperture size on mass-thickness contrast; the images of the shadowed latex particle were taken with an aperture size of (A) 70 μm and (B) 10 μm. A smaller aperture enhances the contrast, in a similar manner to lowering the kV.

Of course, any decrease in intensity can be offset by increased exposure times until specimen drift becomes a limiting factor.

Now for DF images, there isn't much more to be said: the images will generally show regions of contrast that are complementary to those seen in the BF (analogous to the reversed contrast in Figure 22.5B and C) but be prepared for exceptions (in Chapter 24). The overall intensity of the DF image will be much lower than the BF image (hence the relative terms 'dark' and 'bright') because the objective aperture will select only a small fraction of the scattered electrons. It's easy to remember that the BF image of a hole in your specimen will be bright and a DF image will be dark. However, remember that the corollary of low intensity is high contrast and DF images generally show better contrast.

22.3.C STEM Images

In a STEM you have more flexibility than in a TEM because by varying *L*, you change the collection angle of your detector and create, in effect, a variable objective aperture. (We'll mention the old TEMs later where you could do this and the SAD aperture was a triangle or a square.) So you have more control over which electrons contribute to the image. Even so, STEM BF images offer little more than TEM BF images. Generally, STEM images are noisier than TEM images (unless you've got an FEG STEM). Figure 22.8 shows a noisy STEM BF image of the same two-phase polymer as shown in the TEM image in Figure 22.6. The STEM images have generally shown poorer resolution because, with good thin specimens, the beam size dominates the resolution. To get reasonable intensity in a scanning image in reasonable time we have to use a large beam, as we discussed when we compared scanning and static images back in Chapter 9. Figure 22.9 shows the difference between (A) TEM and (B) STEM BF images from a low-contrast

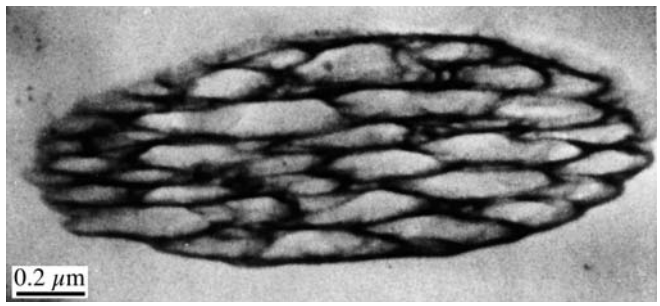


FIGURE 22.8. STEM BF image of a stained two-phase polymer. Comparison with the TEM image in Figure 22.6 shows that while the contrast is higher in STEM, the image quality is poorer.

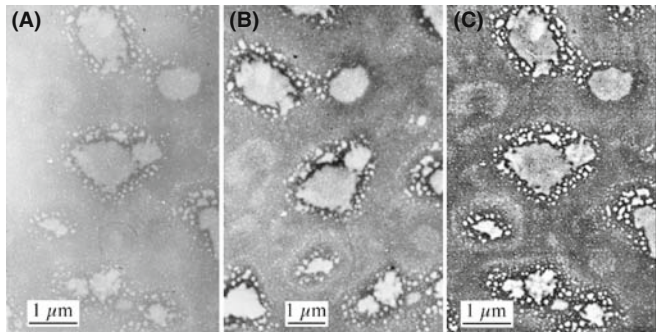


FIGURE 22.9. Comparison of TEM (A) and STEM (B) images of an amorphous SiO_2 specimen containing Cl-rich bubbles. The low mass contrast in the TEM can be enhanced in a STEM image through signal processing. (C) A similar effect can be achieved by digitizing the TEM image (A) and applying contrast-enhancement software.

specimen. The STEM image contrast has been enhanced and is considerably greater than in the TEM image, but the noise in the image is also more visible. However, if you record your TEM image using a CCD camera, or digitize the negative, you can enhance the contrast (see Chapter 31). A good way to do this is with one of the several kinds of image processing software (see Chapter 31) as you can see in Figure 22.9C.

STEM

STEM in a TEM is now a routine high-resolution technique.

In a STEM DF image, the scattered electrons fall onto the ADF detector. This gives rise to a fundamental difference between the TEM and STEM DF modes

- DF TEM images are usually formed by permitting only a fraction of the scattered electrons to enter the objective aperture.
- STEM images are formed by collecting most of the scattered electrons on the ADF detector.

CONTRAST IN STEM

Remember, you can always increase the contrast in the STEM image by adjusting the signal-processing controls, such as the detector gain and black level and the contrast and brightness controls on the computer screen; such options aren't available for analog TEM images.

Therefore, STEM ADF images are less noisy than TEM DF images, as shown in Figure 22.10. Because lenses aren't used to form the STEM image (although they are used to form the probe), the ADF images don't suffer aberrations, as would the equivalent off-axis TEM DF image.

STEM ADF image contrast is greater than TEM DF contrast: in STEM, L can be adjusted to maximize the ratio of the number of scattered electrons hitting the detector to the number of electrons going through the hole in the middle of the detector. You can thus improve the contrast quite easily, just by watching the computer screen and adjusting L.

However, as you can see from Figure 22.10, while the TEM DF image shows poorer contrast and is noisier, it still shows better resolution. Thermionic-source STEM images generally only show better resolution than TEM images when thick specimens are being imaged, because the chromatic aberration effects from thicker specimens do not affect the STEM images. If contrast is more important than resolution, then STEM is more useful. Indeed, in a STEM, you can study unstained polymer specimens which would show negligible contrast in a TEM.

DP AND STEM

The STEM must be well aligned so the DP expands and contracts on axis.

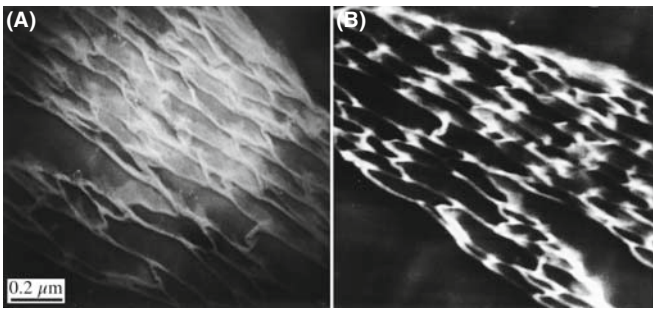


FIGURE 22.10. Comparison of (A) TEM DF and (B) STEM ADF images of the same two-phase polymer as in Figures 22.6 and 22.8. As in BF the STEM image shows higher contrast but lower resolution. Also, the ADF aperture collects more signal than the TEM objective aperture so the STEM image is less noisy.

STEM imaging is also useful if your specimen is beam sensitive, e.g., some polymers. A scanning beam lets you precisely control the irradiated region of the specimen, so it's a form of low-dose microscopy (see Section 4.6). You'll lose some image resolution unless you have access to a FEG STEM.

The comparison we've made of TEM and STEM images here is qualitative, but there have been many quantitative comparisons of STEM and TEM contrast, particularly for biological specimens. When STEMs were first introduced in the 1970s, the absence of chromatic aberration effects led to prophesies that STEM image resolution would invariably be better than TEM; there were even predictions of the end of classic TEM imaging! This hasn't happened because, as we'll see, there is more than just the chromatic aberration factor that governs the image quality, particularly for crystalline specimens. In summary, then, there are occasions when you might want to use STEM mass-thickness contrast images

- The specimen is so thick that chromatic aberration limits the TEM resolution.
- The specimen is beam-sensitive.
- The specimen has inherently low contrast in TEM and you can't digitize your TEM image or negative.
- Your specimen is ideally suited for HRTEM by Z-contrast imaging.

We'll examine the latter in the next section and in Chapter 28, and in much more detail in the companion text.

This said, be aware that STEM in the TEM has greatly improved since many of the comparison studies were carried out. Second, low-dose techniques in TEM will improve in the future. Third, you must be able to digitize your negatives now—negatives will be obsolete in the near future.

22.3.D Specimens Showing Mass-Thickness Contrast

Mass-thickness is the primary contrast source in amorphous materials, which is why we've illustrated this chapter mainly with polymer specimens. Replicas also display thickness contrast (see Figure 22.11A). Remember from Chapter 10 that replicas recreate the specimen topography, e.g., for a fracture surface. The amorphous-carbon replica can be as prepared (without shadowing) (Figure 22.11A) or shadowed (Figure 22.11B). The uneven metal shadowing increases the mass contrast and thus accentuates the topography, as for the latex particles in Figure 22.3. An extraction replica (Figure 22.11C) or a collection of particles dispersed on a support film will also show mass-thickness contrast; shadowing could be useful to reveal the shape of these particles too. If the particles are crystalline there will also be a component of diffraction contrast. Don't shadow if you want to do elemental analysis of the particles.

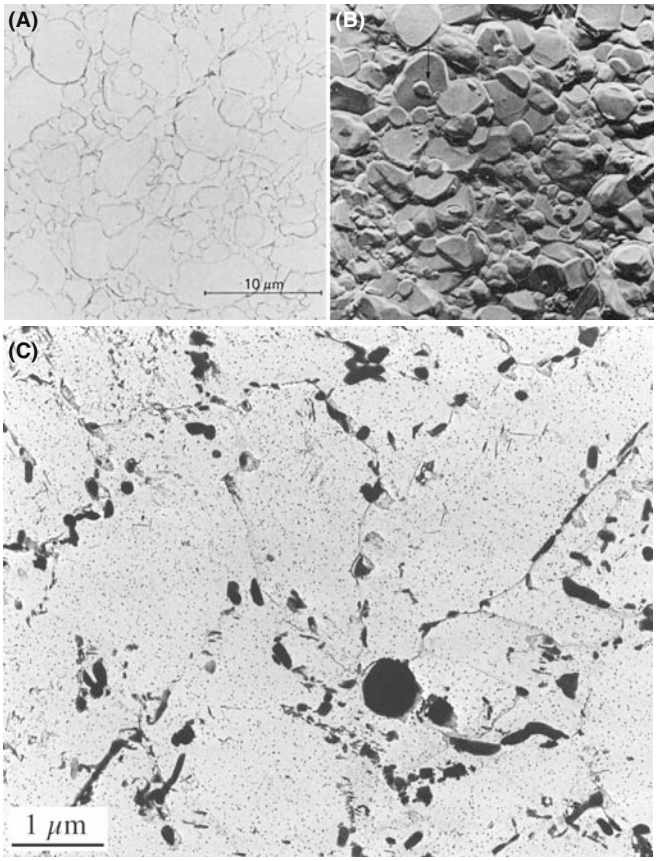


FIGURE 22.11. More examples of mass-thickness contrast: (A) a carbon replica of a fracture surface doesn't show much of either form of contrast until (B) oblique shadowing enhances the topography. (C) An extraction replica of small precipitate particles in a Cr–Mo steel weld shows both mass and thickness contrast.

22.3.E Quantitative Mass-Thickness Contrast

Because mass-thickness contrast is governed by Rutherford scattering, we can use the equations given back in Chapter 3 to predict the effect of Z and t on the scattering angle, θ, and the effect of kV on the cross section. We assume that the atoms scatter independently. (We then say that the scattering is truly incoherent.) This is actually not really the case, since even DPs from amorphous specimens show diffuse rings rather than uniform intensity (Figure 2.13A). Nevertheless, we'll still assume incoherent scattering.

As we stated at the start of this chapter, the contrast C is given by $\Delta I/I$ and it can be shown that a change in thickness, Δt, at constant atomic number Z creates contrast

$$\frac{\Delta I}{I} = 1 - e^{-Q\Delta t} \tag{22.2}$$

Q is the total elastic scattering cross section. So C becomes $Q\Delta t$ for $Q\Delta t < 1$. If 5% is the minimum contrast we can see, then the minimum Δt that we can see is

$$\Delta t \cong \frac{5}{100Q} = \frac{5A}{100\mathrm{N}_0\sigma\rho} \tag{22.3}$$

where A is the atomic weight, N_0 is Avogadro's number, σ is the single-atom scattering cross section, and ρ is the density.

A similar argument can be made if there is a ΔZ (in which case σ or ρ changes). So, if we want to calculate the contrast, we need to know σ. As we've seen in equation 3.8, for low-angle scattering, the differential Rutherford cross section is equal to $f(\theta)^2$ where $f(\theta)$ is the atomic scattering factor, given by equation 3.9

$$f(\theta) = \frac{\left(1 + \frac{E_0}{\mathrm{m}_0\mathrm{c}^2}\right)}{8\pi^2 a_0}\left(\frac{\lambda}{\sin\frac{\theta}{2}}\right)^2 (Z - f_x) \tag{22.4}$$

The Z term represents the Rutherford scattering. For unscreened Rutherford scattering (where we ignore the effects of the electron cloud), σ is therefore proportional to Z^2. This unscreened behavior is quite a good approximation for electrons scattered through angles (remember, we mean semiangles when we talk about scattering) above $\sim 5^\circ$ (e.g., for Cu) although it is dependent on E_0 and Z. At lower angles, scattering becomes increasingly screened (less dependent on Z), and more dominated by inelastic scattering and diffraction. There is no precise angle which we use to define the transition from low- to high-angle scatter but the effect of screening effectively disappears at angles $>\theta_0$, the screening parameter, defined back in equation 3.4.

We can use the atomic scattering factor (equation 22.4) to determine the probability that an electron will be scattered through greater than a given angle. To do this, we integrate $|f(\theta)|^2$ from an angle β (defined by the angle of collection of the objective aperture) to infinity. Thus

$$\sigma(\beta) = 2\pi \int_\beta^\infty |f(\theta)|^2 \theta d\theta \tag{22.5}$$

which can be evaluated to give

$$\sigma(\beta) = \frac{\left[Z\lambda\left(\frac{a_0}{Z^{0.33}}\right)\left(1 + \frac{E_0}{\mathrm{m}_0\mathrm{c}^2}\right)\right]^2}{\pi(a_0)^2\left(1 + \left(\frac{\beta}{\theta_0}\right)^2\right)} \tag{22.6}$$

where a_0 is the Bohr radius and θ_0 is the characteristic screening angle; all the other terms have their usual meaning (see Chapter 3). So in equation 22.6 you can see directly the effect of Z and kV on electron scatter and hence on contrast. As we've already described, higher-Z specimens scatter more while lowering E_0 increases scattering. The effect of thickness is deduced

from the mean free path for elastic scatter, λ (which is inversely proportional to σ). So, thicker specimens scatter more.

Let's assume that n_0 electrons are incident on the specimen and dn electrons are scattered through an angle $>\beta$. Then, from equation 22.6, ignoring any inelastic scattering (which isn't really reasonable, but we'll do it to simplify matters), the reduction in the number of electrons going through the objective aperture to form the BF image is given by

$$\frac{dn}{n} = -N\sigma(\beta)dx \tag{22.7}$$

where $N = N_0/A$ and N_0 is Avogadro's number, $\sigma(\beta)$ is given by equation 22.6, and $x = \rho t$. So this expression gives the dependence of the contrast on Z and t. If we integrate

$$\ln n = -N\sigma x + \ln n_0 \tag{22.8}$$

and if we rearrange this expression

$$n = n_0 e^{-N\sigma x} \tag{22.9}$$

which describes the exponential decrease in the number of scattered electrons (n) as the specimen mass-thickness ($x = \rho t$) increases.

As you'll have gathered, this equation is something of an approximation but it does give you a feel for the factors that control mass-thickness contrast. For a given specimen, the variables are local changes in Z and t; within the microscope, the variables are β and E_0, which you can control to change the contrast, as we saw in Figure 22.7.

In principle, you could use these equations and equation 22.1 to calculate the expected contrast arising from differences in Z or t and see if they were detectable at the 5% contrast level. In practice, however, image-contrast calculations are not carried out for simple mass-thickness contrast in materials specimens.

22.4 *Z*-CONTRAST

Z-contrast is the name given to a high-resolution (atomic), mass-thickness (Z), imaging technique. We'll talk about it here because it represents the limit of mass-thickness contrast where detectable scattering arises from single atoms or column of atoms.

Back in the 1970s, early FEG STEMs demonstrated the remarkable capability of imaging single heavy atoms (e.g., Pt and U) on low-Z substrates as shown in Figure 22.12. These images were formed by the ADF detector collecting low-angle elastically scattered electrons only. Single atoms scatter incoherently and the image intensity is the sum of the individual atomic scattering

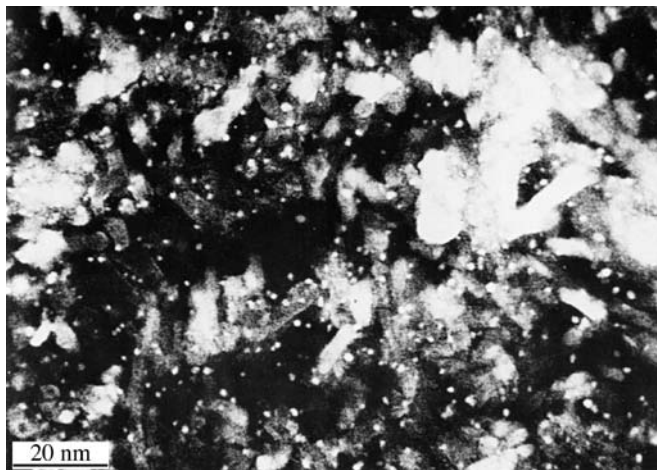


FIGURE 22.12. *Z*-contrast ADF image of individual Pt atoms or groups of atoms on a crystalline Al_2O_3 film obtained using an FEG STEM.

contributions. There was sometimes a problem with thickness changes in the substrate and contributions to the ADF signal from inelastically scattered electrons. This problem was overcome by dividing the digital ADF signal by the inelastic (energy-loss) signal from the EELS system. A drawback to this technique is that diffraction contrast (e.g., from a crystalline substrate) is preserved in the low-angle EELS signal, which can confuse the image interpretation. In Figure 22.12, the large bright regions arise from the Al_2O_3 substrate diffracting onto the ADF detector, obscuring the scatter from the Pt atoms.

HAADF

The detector is called a high-angle ADF or HAADF detector. *Z*-contrast images are also termed HAADF images. The outer diameter of the Fischione HAADF detector is 28 mm; the inner diameter is 4 mm.

Because of Bragg scattering, this early approach to *Z*-contrast was not suited to the study of crystalline specimens. Since the normal ADF detector will always collect some Bragg electrons, it was necessary to design an ADF detector with a very large central aperture. *Z*-contrast images could then be formed from thin crystals (Figure 22.13). You can decrease the camera length with the post-specimen lenses to ensure that the Bragg electrons (including any HOLZ scatter) don't hit the detector. Thus only the electrons scattered through very high angles contribute to the image.

Bragg effects are avoided if the HAADF detector only gathers electrons scattered through an angle of >50 mrad (~3°). Remember that cooling your specimen has the effect of increasing coherent HOLZ scatter, so don't cool it unless you must. Electron channeling effects

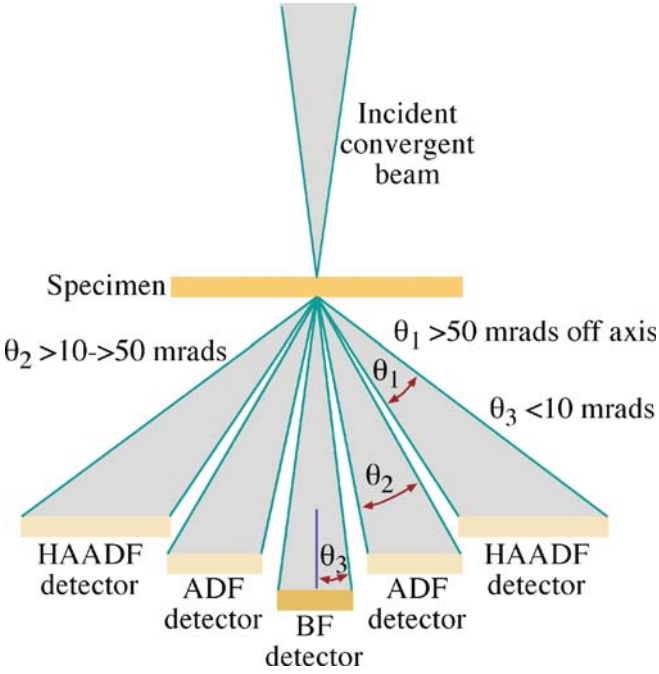


FIGURE 22.13. Schematic of the HAADF detector setup for *Z*-contrast imaging in a STEM. The conventional ADF and BF detectors are also shown along with the range of electron scattering angles gathered by each detector.

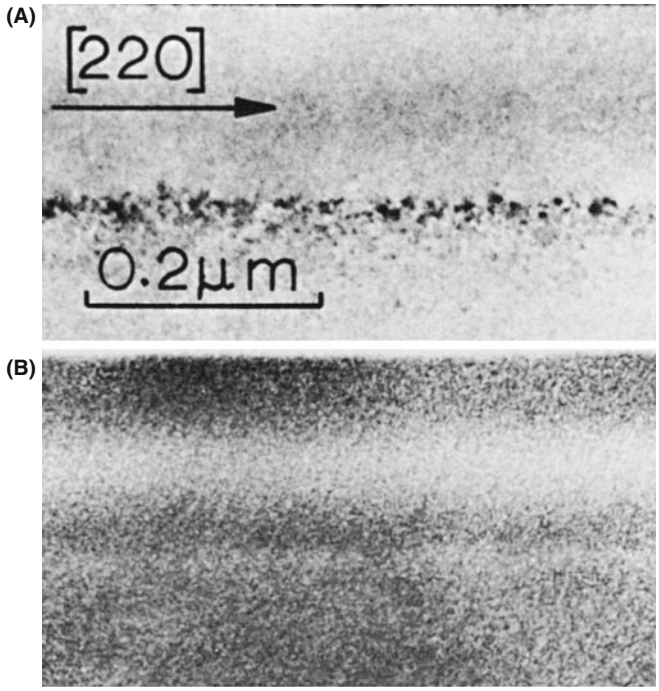


FIGURE 22.14. (A) Low-resolution TEM BF image showing a row of defects in Bi-implanted Si. In (B) obtained under *Z*-contrast conditions, the defects associated with the implant are invisible but the specimen is bright in the region implanted with Bi.

remain at high scattering angles, so imaging away from strong two-beam conditions and closer to zone-axis orientations is wise.

So, what do these *Z*-contrast images of crystals look like? Figure 22.14 shows a TEM BF image of Bi-implanted Si and below is a *Z*-contrast image. In the TEM BF image, formed from the direct beam, defects associated with the Bi implant are shown (we'll talk about such diffraction contrast from defects in Chapter 26) but otherwise there is no contrast associated with the Bi. In the *Z*-contrast image the Bi-implanted area is bright, but note that defect contrast isn't preserved in this image. You can relate the intensity differences in Figure 22.14 to an absolute measure of the Bi concentration. To do this you need to choose a suitable elastic scattering cross section. The contrast is related directly to the cross section for elastic scatter by the matrix (σ_A) and the alloying or dopant element (σ_B)

$$C = \left(\frac{\sigma_A}{\sigma_B} - F_B\right) c_B \qquad (22.10)$$

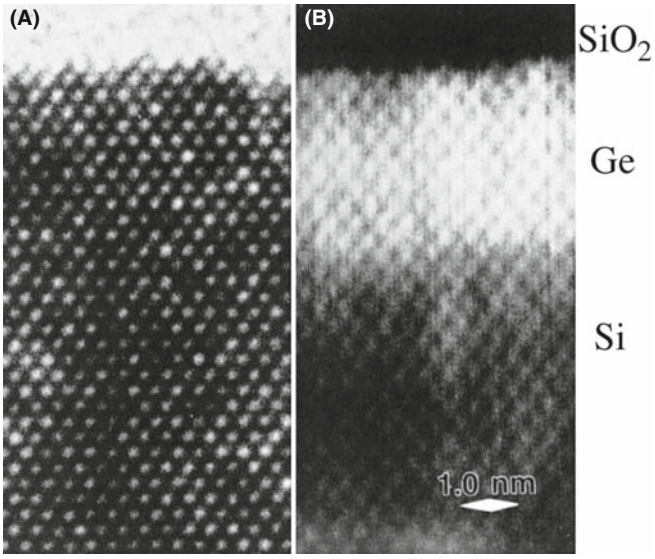
where c_B is the atomic concentration of the alloying element and F_B is the fraction of the alloying element that substitutes for matrix atoms. The intensity can be quantified to an absolute accuracy of better than $\pm 20\%$.

In an FEG with probe sizes of <0.3 nm, *Z*-contrast image resolution of close to the probe diameter is possible. Figure 22.15A shows a high-resolution phase-contrast TEM image of Ge on Si with an amorphous SiO_2 surface layer. The Si and Ge are indistinguishable by phase contrast. In Figure 22.15B, which is a STEM *Z*-contrast image of the same region, the higher-*Z* Ge crystal region is clearly visible and the lower-*Z* SiO_2 layer appears very dark. The atomic structures of the Si and Ge crystals are visible in both phase-contrast and *Z*-contrast images, although the *Z*-contrast image is noisier. Phase-contrast TEM images can show similar *Z*-contrast effects, as we'll detail in Chapter 28. Figure 22.15C shows a model of a grain boundary superimposed on a *Z*-contrast image which has been refined and processed to reduce the noise via a maximum-entropy approach. You can easily see atomic-level detail.

HAADF

HAADF has the advantage that the contrast is generally unaffected by small changes in objective lens defocus (Δf) and specimen thickness.

We'll see in Chapter 28 that interpretation of atomic-resolution phase-contrast images requires knowledge of t and Δf. Some microscopists claim that *Z* contrast will become the principal method of high-resolution imaging in the future as more FEG STEMs become available; others strongly disagree!



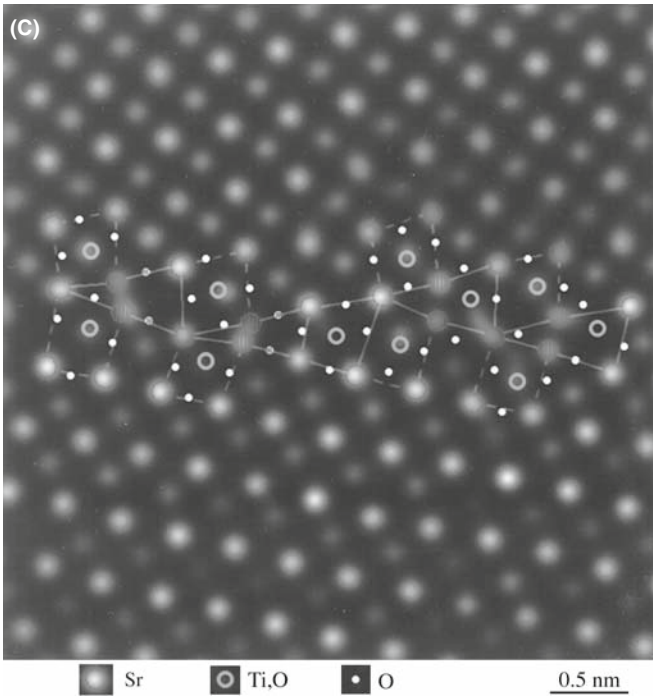


FIGURE 22.15. (A) High-resolution phase-contrast image of epitaxial Ge on Si with an amorphous SiO_2 surface. The bright array of dots common to the crystalline region represents atomic rows and the Ge and Si regions are indistinguishable. (B) The high-resolution *Z*-contrast STEM image shows the atom rows but with strong contrast at the Si–Ge interface and low intensity in the low-*Z* oxide. (C) Model structure of a boundary in $SrTiO_3$ superimposed on a processed *Z*-contrast image.

We can think of the image in Figure 22.15B as a direct map of the $f(\theta)$ variation in the specimen. In that respect, it is similar to an X-ray map showing the distribution of a certain element.

RESOLUTION
The $f(\theta)$ map can have atomic-level resolution, which XEDS imaging can't provide (yet!).

So why do we need a STEM for *Z*-contrast imaging? We are constrained in TEM if we use an analog screen rather than a digital detector to form the image. Nevertheless, we can do *Z*-contrast imaging in a TEM but we have to create electron-optical conditions which are equivalent to those used in STEM. So the beam-convergence angle in TEM must equal the collection angle of the HAADF detector. This is an example of the so-called 'principle of reciprocity' which we'll discuss in more detail in the next section. To converge the TEM beam to the required angular range, we use so-called 'hollow-cone' illumination, which requires an annular C2 aperture. However, the highest incidence angles possible in hollow-cone illumination are typically a few mrad rather than the 50–150 mrad (up to ~9°) collected by the STEM HAADF detector. So TEM *Z*-contrast images are not equivalent to STEM and will always contain some diffraction contrast from crystalline specimens. This leads us into the topic of diffraction contrast, which is the other form of amplitude contrast we see in TEM images.

The future (see Section 39.10): *Z*-contrast images can be collected at the same time and from the same location as the EELS data. There is no other combination of techniques to match this, if it gives the information you need.

22.5 TEM DIFFRACTION CONTRAST

Bragg diffraction, as we discussed in Part 2, is controlled by the crystal structure and orientation of the specimen. We can use this diffraction to create contrast in TEM images. Diffraction contrast is simply a special form of amplitude contrast where the scattering occurs at special (Bragg) angles. We've just seen how incoherent elastic scattering causes mass-thickness contrast. Now we'll see how coherent elastic scattering produces diffraction contrast. As you know, crystalline specimens usually give a single-crystal DP, such as in Figure 22.2. So, as for mass-thickness contrast, we can form BF images by placing the objective aperture around the direct beam (Figure 22.2A) and DF images by selecting any of the diffracted beams (Figure 22.2B). Remember that the incident electrons must be parallel in order to give sharp diffraction spots and strong diffraction contrast. So, if you can, underfocus C2 to spread the beam.

22.5.A Two-Beam Conditions

There is one major difference between forming images to show mass-thickness contrast or diffraction contrast. We can use *any* scattered electrons to form a DF image showing mass-thickness contrast. However, to get good strong diffraction contrast in both BF and DF images

we tilt the specimen to *two-beam conditions*, in which only one diffracted beam is strong. Of course, the direct beam is the other strong spot in the pattern.

Remember: the electrons in the strongly excited *hkl* beam have been diffracted by a *specific* set of *hkl* planes and so the area that appears bright in the DF image is the area where the *hkl* planes are at the Bragg condition. Hence the DF image contains *specific* orientation information, not just general scattering information, as is the case for mass-thickness contrast.

We can tilt the specimen to set up several different two-beam conditions. Figure 22.16A includes a zone-axis DP from a single-crystal specimen in which the beam direction is [011]. The surrounding patterns are a series of two-beam conditions in which the specimen has been tilted slightly so that different *hkl* spots are strongly excited in each pattern. We can form DF images from each strongly diffracted beam after tilting the specimen, and each will give a different image.

TWO-BEAM CONDITIONS

If you're working with crystalline materials, you'll spend a lot of time tilting the specimen to set up different two-beam conditions.

As you can see in Figure 22.16B and C, the BF and DF images show complementary contrast under two-beam conditions. We'll explain the image contrast in detail in Chapter 23. Obviously, to set up a series of two-beam conditions, we need precise tilt control, which explains why a double-tilt eucentric holder is the holder of choice for viewing crystalline specimens.

We'll see in the following chapters that two-beam conditions are not only necessary for good contrast but they also greatly simplify interpretation of the images. This is why we emphasized two-beam theory in our discussion of diffraction in Part 2.

22.5.B Setting the Deviation Parameter, s

Setting up two-beam conditions is very simple. While looking at the DP, tilt around until only one diffracted beam is strong, as in Figure 22.16. As you can see, the other diffracted beams don't disappear because of the relaxation of the Bragg conditions, but they can be made relatively faint. Now if you just do as we've described, the contrast might still not be the best. For reasons we'll discuss in detail in the next chapter, to get the best contrast from defects, your specimen shouldn't be *exactly* at the Bragg condition ($\mathbf{s} = 0$) as in Figure 22.17A. Tilt your specimen close to the Bragg condition but make **s** small and positive. (The excess *hkl* Kikuchi line, just outside the *hkl* spot; go back and check the text for Figures 19.10 and 19.11.) This will give you the best possible strong-beam image contrast as in Figure 22.17B. If you tilt the specimen slightly, so **s** increases further as shown in Figure 22.17C, the defect images become narrower but the contrast is reduced.

USE $\mathbf{s}>0$

Never form strong-beam images with **s** negative; it's easier to see the defects when $\mathbf{s}>0$.

22.5.C Setting Up a Two-Beam CDF Image

We described the basic mechanism of forming BF and DF images back in Chapter 9 (Figure 9.14A). To produce the best BF diffraction contrast, tilt to the desired two-beam condition as in Figure 22.18A, and insert the objective aperture on axis as in Figure 22.2A. A two-beam CDF image is not quite as simple. You might think it involves just tilting the incident beam so the strong *hkl* reflection moves onto the optic axis. If you do that, you'll find that the *hkl* reflection becomes weaker as you move it onto the axis and the $3h3k3l$ reflection becomes strong, as shown in Figure 22.18B. What you've just done is in fact set up a *weak-beam* image condition, which we'll discuss in Chapter 27. To set up a *strong-beam* CDF image, tilt in the $\bar{h}\bar{k}\bar{l}$ reflection which was initially weak, and it becomes strong as it moves on axis, as shown in Figure 22.18C. The CDF technique is absolutely crucial for obtaining and interpreting diffraction-contrast images, so we will take you through it in detail

- Look at the SADP and tilt the specimen until the desired *hkl* reflection is strong. Make sure the incident beam is well underfocused.
- Now tilt the specimen until the $\bar{h}\bar{k}\bar{l}$ reflection is strong: *hkl* will now be weak.
- Use the DF tilt controls to move the 000 reflection towards the strong $\bar{h}\bar{k}\bar{l}$ reflection. The weak *hkl* reflection will move towards the optic axis and become strong.
- When *hkl* is close to the axis, switch off the DF deflectors, insert and carefully center the objective aperture around 000.
- Switch the DF tilt coils on and off while looking through the binoculars. Check that the *hkl* and 000 reflections appear in the same position. Make fine adjustments to the DF coils until you can see no shift between 000 and *hkl* when the deflectors are off and on, respectively.
- Switch to image mode. If necessary, condense the beam slightly with C2 until you can see the CDF image. If you can't see an image, either the *hkl* reflection is too weak (unlikely) or your tilt coils are misaligned (common). In the latter case, realign the coils (see the manufacturer's handbook).

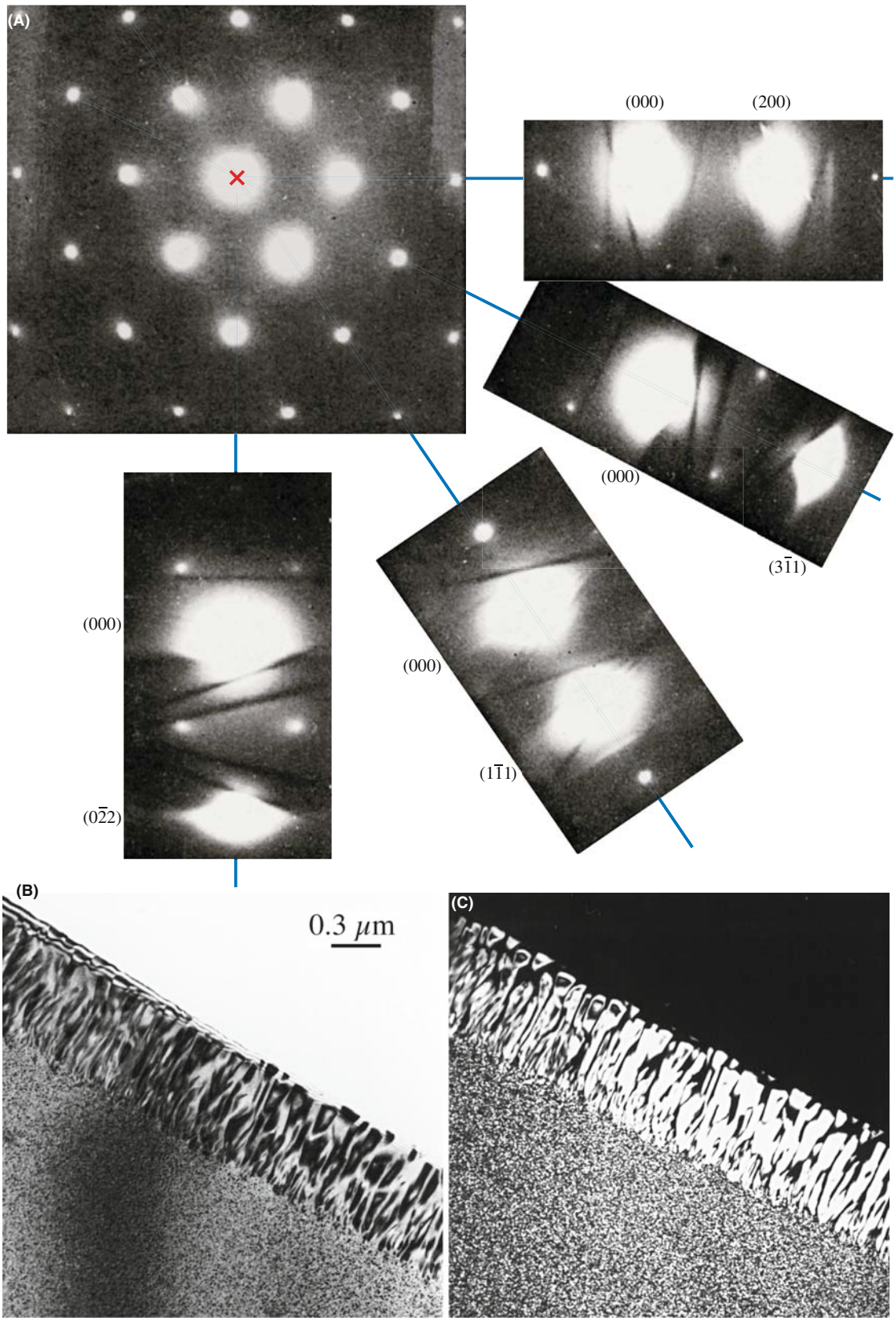


FIGURE 22.16. (A) The [011] zone-axis diffraction pattern has many planes diffracting with equal strength. In the smaller patterns, the specimen is tilted so there are only two strong beams, the direct 000 on-axis beam and a different one of the *hkl* off-axis diffracted beams. Complementary (B) BF and (C) DF images of Al-3 wt% Li taken under two-beam conditions are shown also. In (B) the Al_3Li precipitate phase (present as tiny spheres in the grain and coarse lamellae at the boundary) is diffracting strongly and appears dark. In (C), imaged with a precipitate spot, only the diffracting precipitates appear bright.

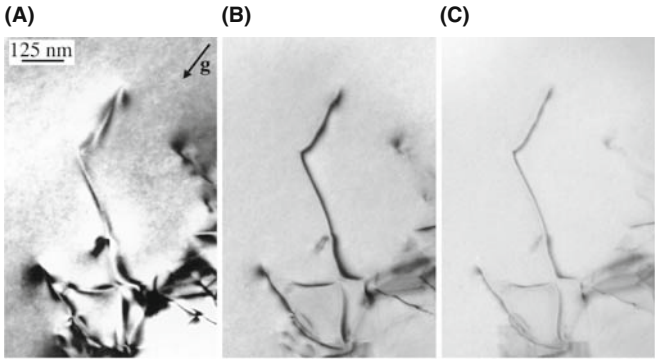


FIGURE 22.17. Variation in the diffraction contrast when **s** is varied from (A) zero to (B) small and positive and (C) larger and positive.

Now go back and study Figure 9.14C carefully. You'll see that the beam was tilted through an angle $2\theta_B$ to bring the weak beam in Figure 9.14B onto the optic axis.

Notice that $\bar{\mathbf{g}}$ is excited in the DF condition; **g** was excited in the BF condition. If you want to form a CDF image with **g** excited, you must tilt the beam *and* the specimen. Alternatively, just displace the aperture but be cautious.

22.5.D Relationship Between the Image and the Diffraction Pattern

From what we've just described, there is clearly an important relationship between the DP and a diffraction-contrast image. If we change the DP in any way, the contrast in the image will change. So it is critical to relate the DP to the image. We need to indicate the direction of the **g** vector in the image. To relate the two, remember that you may have to calibrate the rotation between the image and the DP if, whenever you change magnification, your image rotates but your DP does not. We described this calibration in Section 9.6.C. You should usually show the **g** vector in any BF or DF two-beam, diffraction-contrast image after correcting for any rotation between the image and the DP. Remember: you should always check the calibration of any microscope, especially a TEM, and be careful with possible 180° rotations.

ROTATION CALIBRATION
Do it even if only to convince yourself that you don't need to!

We will expand on diffraction contrast in far more detail in the subsequent chapters, making use of the fundamental operational principles we have just described.

22.6 STEM DIFFRACTION CONTRAST

The principle of forming BF and DF images in STEM is just the same as for mass-thickness contrast; i.e., use the BF detector to pick up the direct beam and the ADF

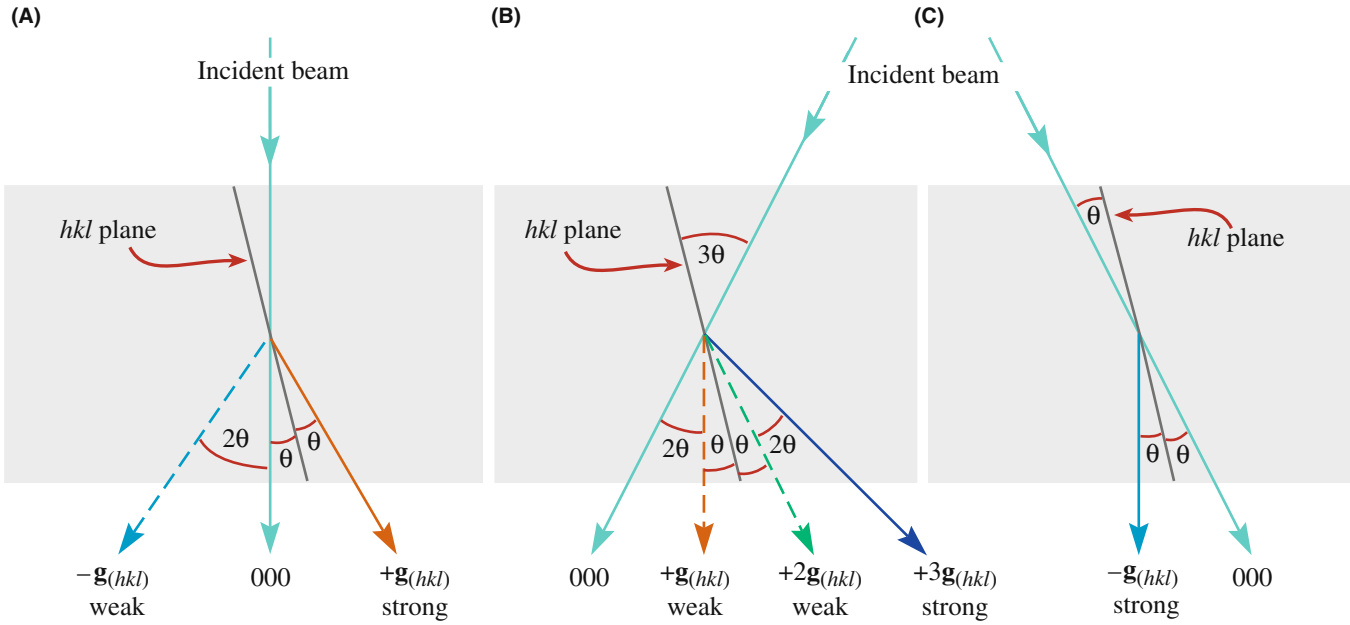


FIGURE 22.18. (A) Standard two-beam conditions involve the 000 spot and the *hkl* spot bright because one set of *hkl* planes are exactly at the Bragg condition. (B) When the incident beam is tilted through 2θ so that the excited $\mathbf{g}_{hkl}$ spot moves onto the optic axis, the $\mathbf{g}_{hkl}$ intensity decreases because the $\mathbf{g}_{3h3k3l}$ spot becomes strongly excited. (C) To get a strong *hkl* spot on axis for a CDF image, it is necessary to set up a strong $-\mathbf{g}_{hkl}$ condition first of all, then tilt the initially weak $\mathbf{g}_{hkl}$ maximum onto the axis.

detector to pick up the diffracted beams. To preserve two-beam conditions, the ADF detector must only pick up one strong diffracted beam and this can be ensured by inserting the objective aperture and selecting only one diffracted beam. Alternatively, the DP could be displaced so the chosen *hkl* reflection falls on the BF detector. Either way the computer screen will display a DF image.

However, the diffraction contrast observed in the STEM image will generally be much poorer than TEM contrast; the normal STEM operating conditions are not equivalent to the TEM conditions that ensure strong diffraction contrast. To understand the contrast in STEM images you need to know the beam convergence and detector collection angles. It's rare in fact that you'll need to do this, but we showed you how to determine the beam convergence angle back in Section 5.5. To calculate the collection angle, you need to carry out a similar exercise as we use to determine the EELS spectrometer collection angle in Section 37.4.

Remember, there are three conditions that must be fulfilled for strong contrast in your image

- The incident beam must be coherent, i.e., the convergence angle must be very small.
- The specimen must be tilted to a two-beam condition.
- Only the direct beam or the one strong diffracted beam must be collected by the objective aperture.

This condition is shown schematically in Figure 22.19A. We define the TEM convergence angle as α_T and the objective aperture collection angle as β_T. In a STEM, the equivalent angles are the beam convergence angle α_S and the STEM detector collection angle β_S as shown in Figure 22.19B. Therefore, we have identical operating conditions if

$$\alpha_T = \alpha_S \tag{22.11a}$$

$$\beta_T = \beta_S \tag{22.11b}$$

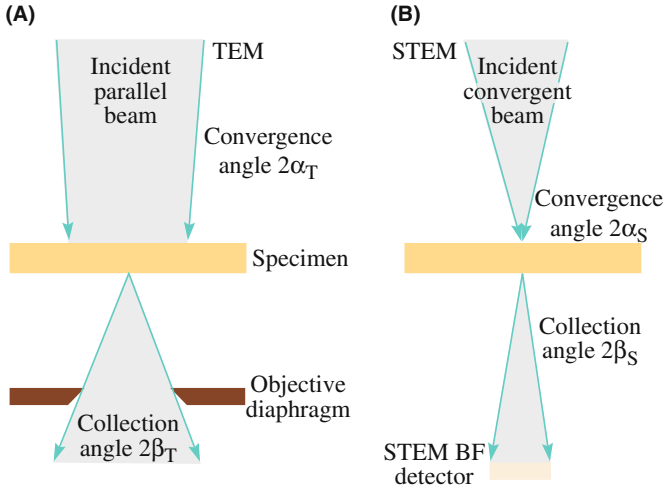


FIGURE 22.19. Comparison of the important beam-convergence and divergence angles (A) in TEM and (B) in STEM. (Note that we show the full convergence and divergence angles, not the semiangles that are usually described in the text.)

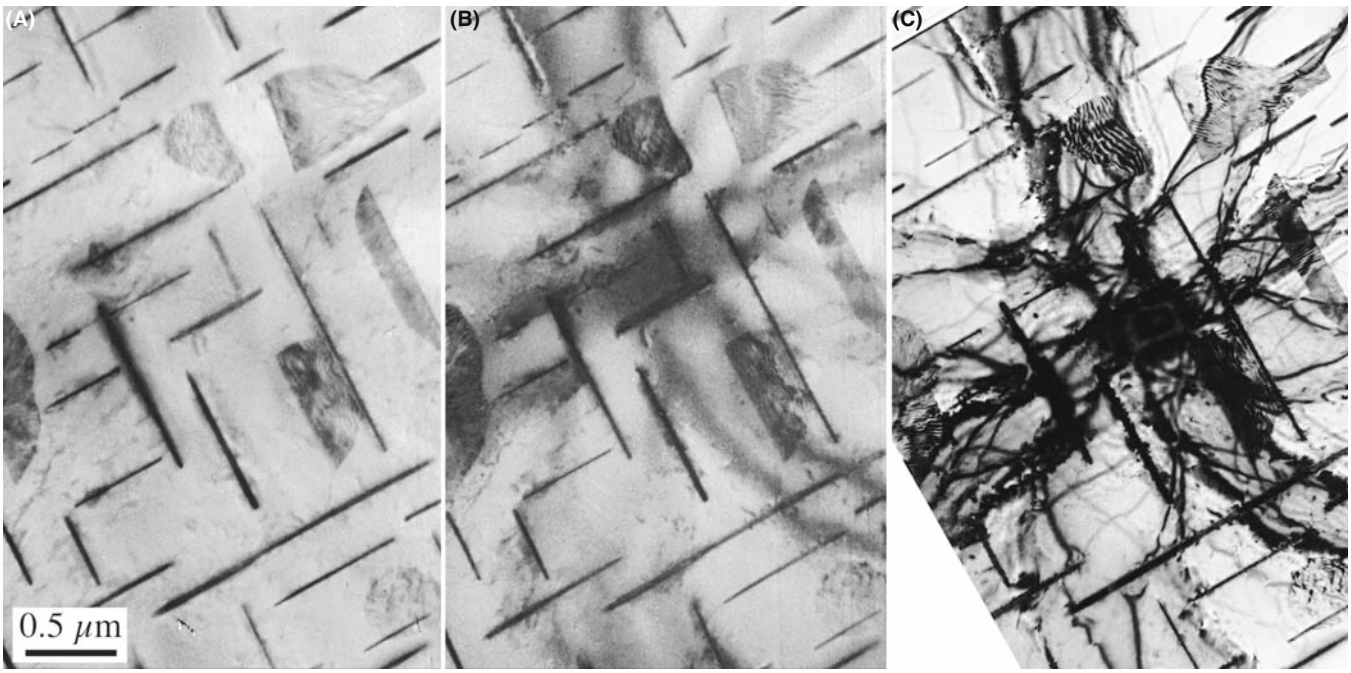


FIGURE 22.20. (A) A BF STEM image of an Al-4 wt% Cu specimen showing weak diffraction contrast in the form of bend contours. As the STEM detector collection angle is lowered (B), the diffraction contrast increases slightly at the expense of increased noise in the image. Even at the smaller collection angle, comparison with the contrast in the TEM image (C) is unfavorable. Note that the Cu-rich θ′ precipitates maintain strong mass contrast in all the images.

Now it should be immediately clear that we can't get such equivalence in a STEM because the convergence angle of the beam is very much greater than in a TEM (since in STEM we deliberately create a convergent rather than a parallel beam). However, there is a way around this dilemma and it depends on a theorem that is often used in electron optics called the *principle of reciprocity*. In essence this principle says that so long as the electron ray paths contain equivalent angles (of convergence and collection) at some point in the electron optical system, the image contrast will be identical.

In other words, while the conditions in equations 22.11 can't be fulfilled, we can create conditions such that

$$\alpha_S = \beta_T \tag{22.12a}$$

$$\alpha_T = \beta_S \tag{22.12b}$$

Under these circumstances the electrons in TEM and STEM do see equivalent angular constraints, although not at the equivalent points of convergence and collection.

- Since the objective-aperture collection angle in TEM is about equal to the convergence angle in STEM, the first of this pair of equations is easily satisfied.
- To satisfy the second pair, we have to make a very small STEM collection angle β_S.

We can't simply increase α_T, because we must keep a parallel beam to get good TEM diffraction contrast and making the beam non-parallel (large α_T) destroys the contrast.

There is an obvious drawback to making β_S small. The signal falling on the STEM detector becomes very small and the STEM image becomes noisy. So STEM diffraction-contrast images become noisier as we attempt to increase the amount of diffraction contrast, as in Figure 22.20. (See Chapter 24 for an explanation of the contrast (bend contours) in this figure.) Having an FEG helps to offset this increase in noise, but in general, STEM diffraction-contrast images (in both BF and DF) compare so unfavorably with TEM images (see Figure 22.20C) that, while they may be useful if you're performing analysis, they are rarely used to show diffraction-contrast images of crystal defects. This is solely the domain of TEM, as we'll discuss in detail in the next few chapters.

CHAPTER SUMMARY

Mass-thickness contrast and diffraction contrast are two forms of amplitude contrast. Both arise because the specimen scatters electrons. The operational procedures to produce BF and DF images are identical. Interpretation of mass-thickness contrast is generally simpler than interpretation of diffraction contrast. In fact, the interpretation of diffraction contrast is sufficiently complex that we need to devote several subsequent chapters to the various forms arising in perfect and imperfect crystals.

We can summarize the characteristics of mass-thickness contrast

- Areas of greater Z and/or t scatter electrons more strongly (in total), and therefore appear darker in BF images and brighter in DF images. The contrast can be quantified if necessary.
- TEM mass-thickness contrast images are better quality (lower noise and higher resolution) than STEM images, but digital STEM images can be processed to show higher contrast than analog TEM images.
- STEM mass-thickness contrast images are most useful for thick and/or beam-sensitive specimens.
- Z-contrast (HAADF) images can show atomic-level resolution.

We can summarize the characteristics of diffraction contrast

- Diffraction contrast arises when the electrons are Bragg scattered.
- To form a diffraction-contrast image in TEM, the objective aperture selects one Bragg-scattered beam. Often, the STEM detectors gather several Bragg beams which reduce diffraction contrast.
- Diffraction-contrast images in TEM always show better contrast than in STEM images, which are always noisier and almost never used.

Some of the statements made in the First Edition are not now valid! FEGs are now common. The original DSTEM was discontinued in ~1985; but DSTEMs are now made by Hitachi, JEOL, and Nion. C_s correctors are becoming more available. Modern TEMs all use digital recording. Z-contrast imaging has greatly improved on commercial machines and rivals CTEM in many applications.

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Pennycook, SJ 1992 *Z-Contrast Transmission Electron Microscopy: Direct Atomic Imaging of Materials* Annu. Rev. Mat. Sci. **22** 171–195.

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THE COMPANION TEXT

The chapter on simulating images is particularly relevant but you must understand what you are simulating first. The companion text also includes a full chapter on HAADF.

SELF-ASSESSMENT QUESTIONS

Q22.1 What do we mean by the term minimum contrast and can we quantify it?
Q22.2 When considering contrast (either qualitatively or quantitatively), why do we immediately consider the DP?
Q22.3 Will a TEM be able to resolve two atoms next to each other in the periodic table?
Q22.4 You are shown a polymer sphere in a TEM image. Why would you be cautious?
Q22.5 How would you prepare an unstained polymer sample for TEM imaging?
Q22.6 Why is the resolution in STEM not as good as in TEM on most TEMs?
Q22.7 Why is resolution on some STEMs as good as, or better than, many TEMs?
Q22.8 How are diffraction-contrast images different from mass-thickness contrast images?
Q22.9 No matter how hard a particular student tries to obtain a two-beam condition, he or she still sees the direct beam, a strongly diffracted beam, and several weak spots in the DP. Could the student be at the two-beam condition?
Q22.10 What is the difference between a phase-contrast image and an amplitude-contrast image?
Q22.11 What causes mass-thickness contrast?
Q22.12 What influences the intensity of mass-thickness contrast?
Q22.13 For what materials is mass-thickness contrast most useful?
Q22.14 What microscope controls can affect the mass-thickness contrast?
Q22.15 What features of a STEM give more flexibility in mass-thickness contrast imaging?
Q22.16 What is diffraction contrast?

Q22.17 How can you obtain good diffraction contrast in BF and DF images?
Q22.18 How can you obtain the best strong-beam contrast from defects?
Q22.19 How are *Z*-contrast imaging and HAADF imaging related?
Q22.20 Does a BF STEM image show strong contrast from bend contours? Explain after you've read Chapter 24.

TEXT-SPECIFIC QUESTIONS

T22.1 Where is the Ewald sphere in Figure 22.2? Give a full explanation.
T22.2 How was the specimen prepared in Figure 22.12 and why is this information important?
T22.3 Redraw Figure 22.13 showing all the angles and distances to scale and with typical values in mrad and degrees. Are the values of θ correct/reasonable/sensible? Explain your reasoning carefully.
T22.4 What do you guess is the geometry of the specimen shown in Figure 22.14?
T22.5 Draw an atomistic diagram to explain the contrast seen in Figure 22.15B.
T22.6 Draw and label direction vectors on Figure 22.15C for both grains.
T22.7 Index all the spots in the diffraction pattern in Figure 22.16.
T22.8 Explain why the bend contours in STEM images are much less pronounced than in TEM diffraction-contrast images.
T22.9 In Figure 22.17, why are some of the defects in C almost out of contrast?
T22.10 Suggest how the contrast in Figure 22.16C is influenced by the fact that the Al_3Li precipitate on the grain boundary is a lamella.