



The Instrument

CHAPTER PREVIEW

Over the preceding four chapters we've now introduced all the essential components of the TEM and it's time to see how the guns (Chapter 5), lenses (Chapter 6), detectors/screens (Chapter 7), and specimen holders (Chapter 8) are combined to form the instrument. Just as we do for the VLM, it's convenient to divide up the TEM into three components: the illumination system, the objective lens/stage, and the imaging system. The illumination system comprises the gun and the condenser lenses and its role is to take the electrons from the source and transfer them to your specimen. You can operate the illumination system in two principal modes: parallel beam and convergent beam. The first mode is used primarily for TEM imaging and selected-area diffraction (SAD), while the second is used mainly for scanning (STEM) imaging, analysis via X-ray and electron spectrometry, and convergent-beam electron diffraction (CBED).

The objective lens and the specimen holder/stage system is the heart of the TEM. This critical region usually extends over a distance of ~ 10 mm at the center of the TEM. Here is where all of the beam-specimen interactions take place and the two fundamental TEM operations occur, namely, the creation of the various images and DPs that are subsequently magnified for viewing and recording. Clearly the objective lens is the most important lens in a TEM because its quality determines the quality of all the information about the specimen that we seek. Because of the various defects inherent in magnetic lenses, our specimens must be placed close to the center of the objective lens and this restricts our ability to manipulate the specimen and gain access to the various signals generated. However, as we've mentioned many times already, we can now overcome many of the defects that limit lens performance by inserting complex aberration-correction systems into the column, but only the most expensive TEMs are so equipped.

The imaging system uses several lenses to magnify the image or the DP produced by the objective lens and to focus these on the viewing screen or computer display via a detector, CCD, or TV camera. We'll refer to the magnifying lenses as the intermediate and diffraction lenses and the final lens as the projector lens (since it projects the final image or DP onto the viewing screen or detector). All TEM operations involve observing the electrons on a screen of some form, with or without a specimen in place. In many current TEMs you will have a button for focus, another for magnification, and another for diffraction (or a slide on the computer screen). These three components, the illumination system, stage, and imaging system, are often called the 'column', for obvious reasons. Throughout this book, the electrons will invariably be shown as traveling 'down' the column because this is the construction of most (but not all) TEMs.

The purpose of this chapter is to go through the principal functions of the many lenses in the column and give you some feel for what is happening in the microscope when you press a button, rotate a knob or trackball, or drag your mouse. The better you understand the operation of a TEM, the more you can be sure that you are getting the most out of it.

We do NOT intend to teach you how to operate a specific TEM since individual manufacturers choose different ways to manipulate the many lenses, apertures, deflectors, etc., in the TEM column to achieve the same ends. What we will do is introduce specific *operational procedures* that we hope are generic enough such that you should, in concert with the operating manual of your own TEM, be able to carry out all the basic procedures.

9.1 THE ILLUMINATION SYSTEM

The illumination system takes the electrons from the gun and transfers them to the specimen giving either a broad beam or a focused beam (often called a ‘probe’ or ‘spot’) entering the specimen. We can think of these two cases as analogous to a floodlight or a spotlight. In Chapter 5 we described how the gun produces an image of the source (called a crossover). This crossover acts as the object for the first lens in the illumination system that consists of several condenser lenses (C1, etc.). We will discuss the two different ways to use the illumination system: formation of a parallel electron beam (although it is almost never truly parallel) or a convergent beam (which may be divergent).

9.1.A TEM Operation Using a Parallel Beam

In the *traditional* TEM mode, the first two condenser lenses (C1, C2) are adjusted to illuminate the specimen with a parallel beam of electrons, typically several micrometers across at reasonable magnifications (20,000 \times –100,000 \times). As shown in Figure 9.1, the C1 lens first forms an image of the gun crossover. In the case of a thermionic source, the original crossover may be several tens of micrometers across, and this crossover is demagnified by an order of magnitude or more. In the case of a FEG, the source size may be less than the desired illumination area on the specimen so it may be necessary to magnify the crossover (so the condenser lenses don’t always condense!). The simplest way to produce a parallel beam is shown in Figure 9.1A in

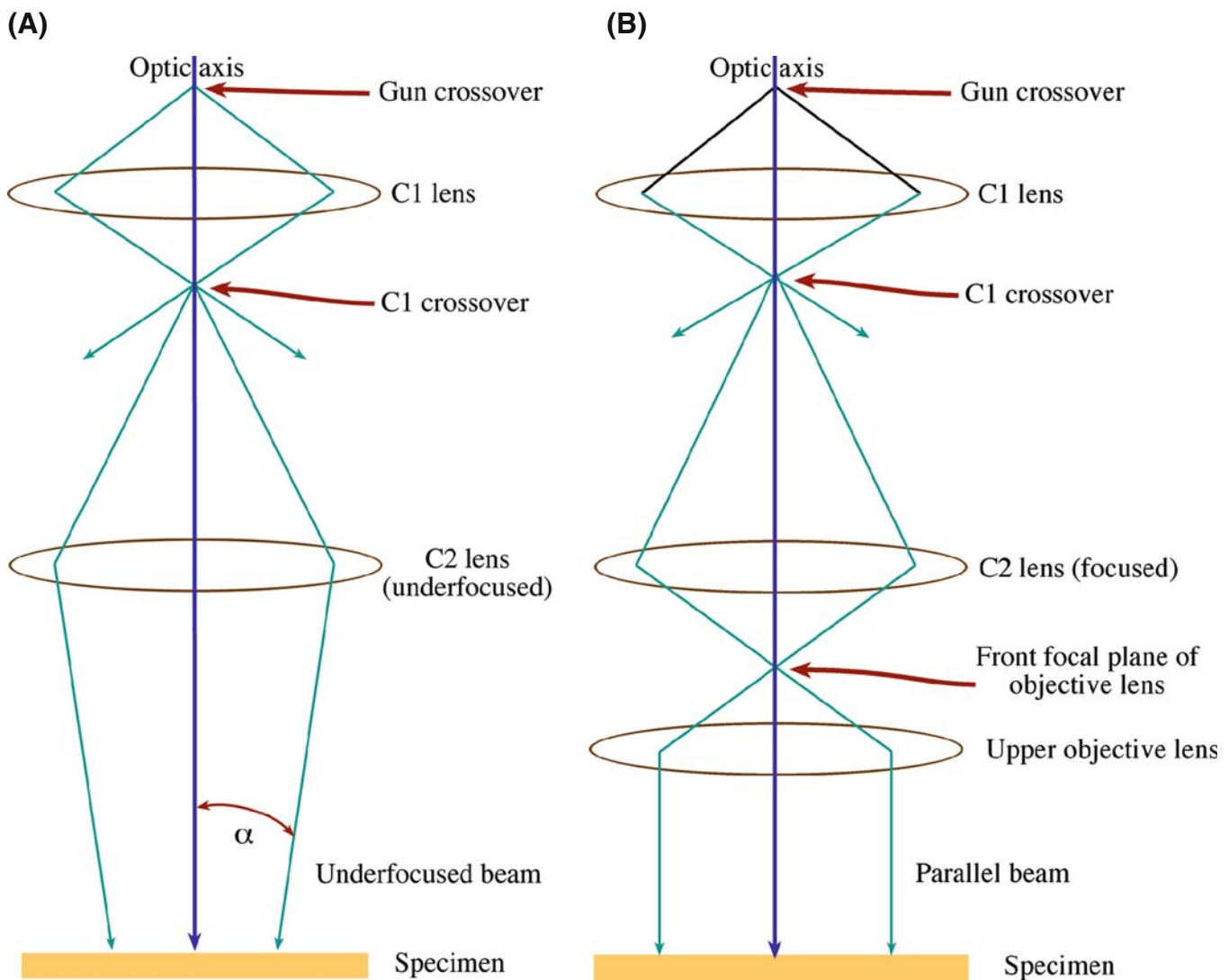


FIGURE 9.1. Parallel-beam operation in the TEM: (A) the basic principle illustrated, using just the C1 and an underfocused C2 lens. (B) The practical situation in most TEMs; using the C1 and C2 lenses to image the source at the FFP of the condenser-objective lens, thus creating a parallel beam at the specimen plane. Therefore, the upper objective is sometimes termed the C3 lens.

which the C2 lens is weakened to produce an under-focused image of the C1 crossover (although no TEM today has only two condenser lenses). Remember from Section 6.2.D and Figure 6.5 that any lens operates either in focus or out of focus and there are times when each mode is useful. In its out-of-focus condition, a lens is said to be overfocused if it is strong and the crossover occurs before the image plane and underfocused if the lens is weak and the crossover occurs after the image plane. Remember also that underfocus gives a more parallel beam at the specimen than overfocus.

CONVERGENCE ANGLES

α is small. All ray diagrams in this book are drawn with exaggerated (large) angles.

While the beam in Figure 9.1A is not exactly parallel to the optic axis, α under these conditions is $< 10^{-4}$ rads (0.0057°), which is effectively a parallel beam.

When generating the small probes we need in STEM and AEM, the upper polepiece of the objective lens acts as the C3 lens (see the next two sections) and controls the beam hitting the specimen as shown in Figure 9.1B. If the C2 lens is focused to produce an image (of the crossover) at the front-focal plane (FFP) of the upper-objective polepiece then a parallel beam of electrons is formed by the lens. The so-called condenser-objective (c/o) lens system is standard on almost all TEMs used for materials characterization but there is no c/o lens in dedicated STEMS, TEMs made before ~ 1980 and those designed primarily for imaging of biological specimens. The c/o lens system complicates the electron optics, as we shall see in Section 9.1.C below.

We'll see that parallel illumination is essential to get the sharpest selected-area DPs (SADPs) (Chapter 18) and, in principle, the best *classical* image contrast (Part 3). In fact, we usually assume that the incident beam is parallel when interpreting our *classical* images. Usually you should underfocus C2 until the illuminated area on your specimen fills the viewing screen. The higher the magnification the more you have to strengthen C2 (making the beam *less* parallel) in order to keep the illuminated area just filling the screen and thus illuminating less of the specimen.

CLASSICAL? PARALLEL?

Classical (or traditional or conventional) means... thinking in terms of waves and beams. Parallel illumination isn't really parallel, it's just not very convergent.

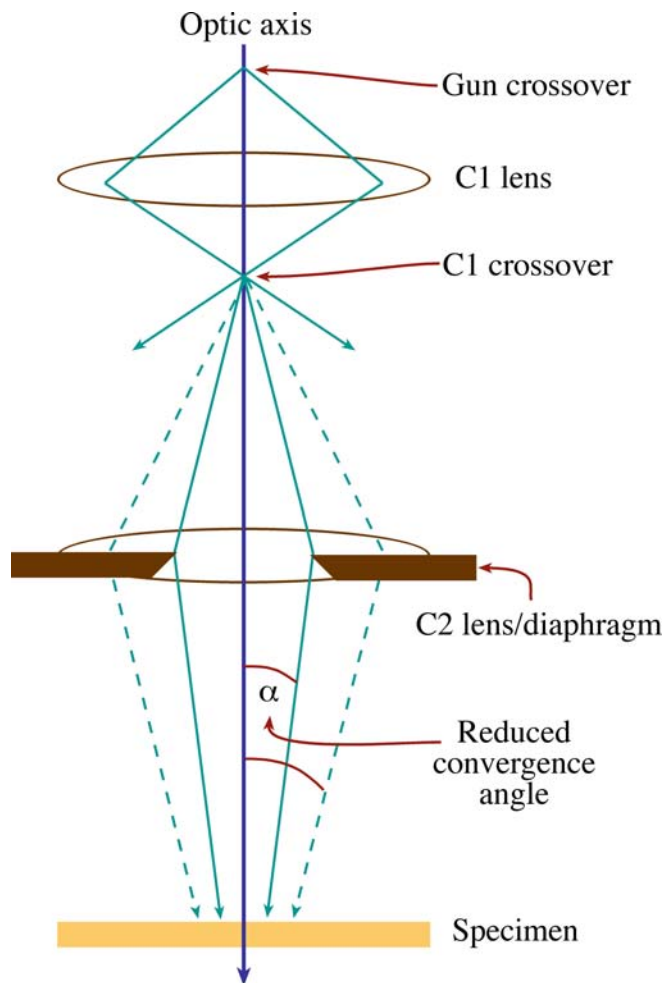


FIGURE 9.2. Effect of the C2 aperture on the parallel nature of the beam: a smaller aperture creates a more parallel beam at the expense of total number of electrons (i.e., reduced probe current) reaching the specimen.

In parallel-illumination TEM mode, such as you would use for diffraction-contrast imaging and SADP formation, there is usually no need to change C1, which is therefore kept at some intermediate setting, recommended by the manufacturer. The only other variable is the C2 aperture. A small aperture reduces the electron current reaching your specimen. However, if you use a smaller aperture, you decrease the angle of beam convergence and therefore make the beam more parallel, as is evident from Figure 9.2.

9.1.B Convergent-Beam (S)TEM Mode

Now, there are times when you may wish to focus the beam more, so that the intensity of the beam on a specific area of the specimen is increased. Let's look at various ways to do this.

If you want to *minimize* the area of the specimen that you are illuminating, you simply change the C2 lens so it is focused rather than defocused and you form an

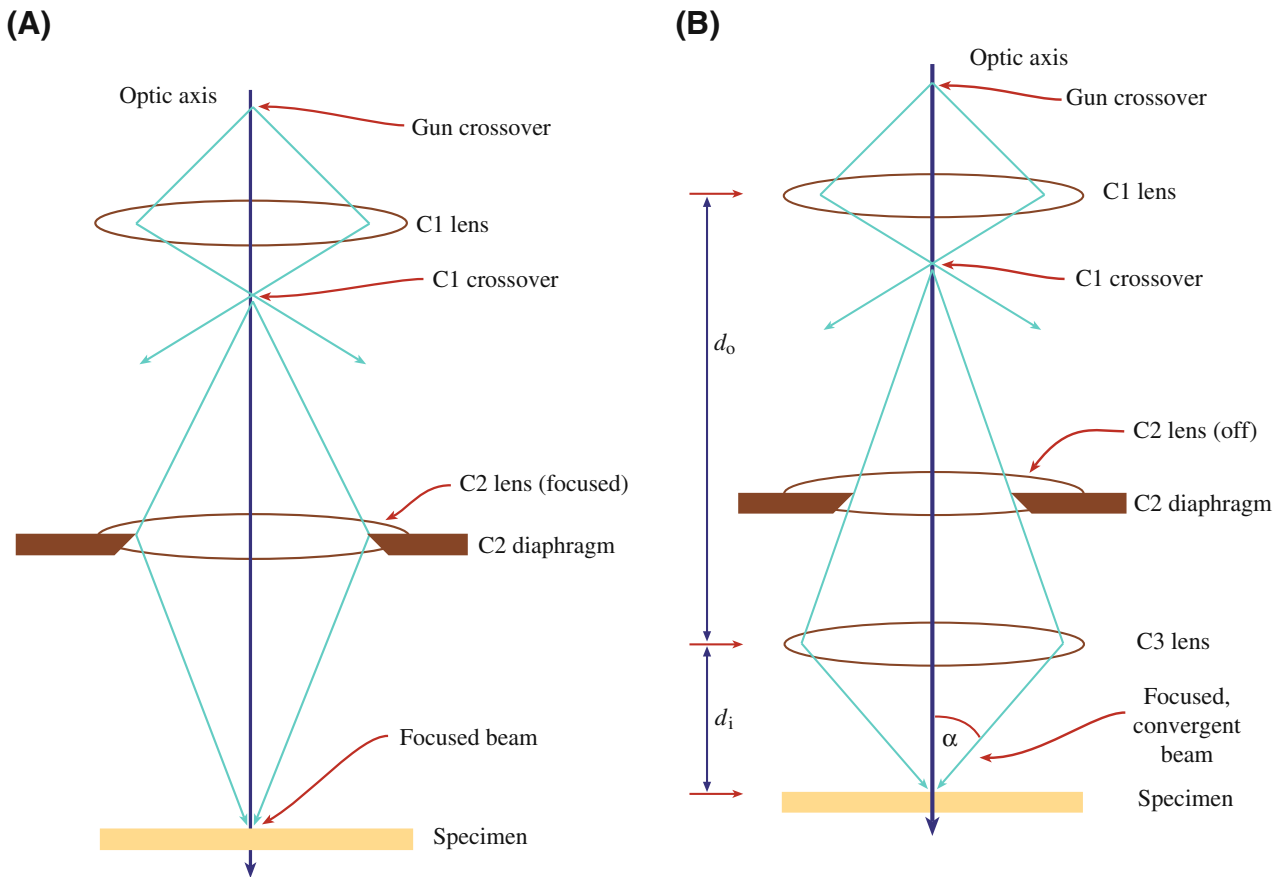


FIGURE 9.3. Convergent-beam/probe mode in the TEM. (A) The basic principle: a focused C2 lens illuminates a small area of the specimen with a non-parallel, convergent beam. (B) The practical situation in most TEMs: use of the upper-objective polepiece as the C3 lens gives the smallest possible probe and large convergence angles. Note that C2 is shown here as having no effect on the ray paths (i.e., it is effectively switched off). The large d_o/d_i ratio gives the maximum demagnification of the image of the gun crossover.

image of the C1 crossover at the specimen, as shown in Figure 9.3A. This is the condition under which you can view the source image to adjust its saturation (go back and look at Figure 5.5B and C) or to measure the dimensions of the beam (see Section 5.5.D). When C2 is focused like this, the beam is at its least parallel and most convergent. While the intensity of illumination on the viewing screen will be greatest, your image contrast will be reduced and any SADP will be distorted. Ideally, for routine TEM work, your specimen should always be thin enough so that you never have to operate with C2 focused but, in practice, you'll often find yourself focusing C2 to compensate somewhat for poor transmission through a thick portion of your specimen.

However, there are times when we do need to create a focused, convergent beam at the specimen. We then use the other principal way to operate the illumination system: the convergent-beam (or probe/spot) mode. When you use this mode you won't immediately see a useful image; the convergence destroys the parallelism and reduces the image contrast. So to see an image we have to scan the beam; this mode of operation of the illumination system is standard for STEM and AEM.

PROBE

The convergent beam is a probe. We use a probe to localize the signals coming from the specimen, as in XEDS, EELS, CBED, or HAADF.

Now, if you have a FEG it's possible to use the C1 and C2 lenses to produce Å-level probes but, with a thermionic source, it isn't possible to use just those two lenses (as in Figure 9.3A) to demagnify the relatively large thermionic-gun crossover to less than several nanometers. So to get the desired probe sizes of $\ll 1$ nm for analysis, etc., the usual solution, as we just noted, is to introduce a C3 or c/o lens. We can only do this if the objective lens is split into two polepieces with separate coils and then we can make the upper polepiece of the objective lens much stronger than usual and weaken C2 or even turn it off, as shown in Figure 9.3B. In addition, C1 must be strongly excited so the image of the gun crossover is a long way from C3. Thus the C3 image distance (d_i) is much less than the object distance (d_o), which gives a large demagnification of the C1 crossover (see equation 6.2).

From Figure 9.3B you can see that, although C2 is switched off, the C2 aperture still controls the convergence angle (α) of the beam on the specimen. As was the case for parallel-beam mode, a smaller C2 aperture gives a smaller α . You'll see later in Chapter 20 that the correct choice of C2 aperture is important in CBED and also in defining the exact dimensions of the probe for XEDS or EELS analysis (see Part 4). Some TEMs such as the FEI and older JEOLs use a double-gap C1 lens (check the situation for your own TEM in the manufacturer's handbook). In these TEMs there is a single C1 lens winding but effectively four polepieces and hence two crossovers occur in the bore of the lens. In some other TEMs (e.g., the Akashi 002B, the JEOL 2010, and Hitachi 8100 and later instruments) there are actually three independent lenses. Without this additional crossover, it's not possible to get a probe $< \sim 10$ nm. An additional condenser lens will yield a probe size about $10\times$ smaller. Even though you only get to use two controls (C1 and C2) there are at least three crossovers. So, as you can see, condenser-lens configurations can get much more complicated than the basic principle shown in Figure 9.3B. The more lenses you have, the more flexible the optics and therefore the more operations that a single TEM can perform.

So, in probe-forming TEMs, the role of C1 is fundamentally different from its role in traditional, parallel-beam TEM. The C1 lens is used to control the probe size (and thus the current) at the specimen. As shown in Figure 9.4, a strong C1 gives you a small probe while a weak C1 creates a large probe. This difference occurs because increasing the strength of C1 shortens its d_i , thus lengthening d_o for the probe-forming C2 (or C3) lens and, therefore, increasing the demagnification (but remember that this is a very simplified diagram).

9.1.C The Condenser-Objective Lens

When convergent-beam TEMs were first constructed, it was not possible to design an objective lens that would give both a parallel or a convergent beam at the specimen. So you had to change polepieces in order to change operating modes, which was highly inconvenient. To solve this problem, the condenser/objective (c/o) lens was developed in the mid-1970s. In a c/o lens, the magnetic field between the polepieces of the objective lens is very strong (~ 2 Tesla). This strong field has a dual function. First, it acts as a condenser lens and effectively causes the beam to converge on the specimen. Thus, as we mentioned above, we can refer to the condenser part of the lens as the C3. Second, once the beam has penetrated the specimen, it is focused just as it would be in a conventional lens. The beam is almost never parallel in a c/o lens and so, as shown in Figure 9.5, yet another lens is introduced into the column between the C2 lens and

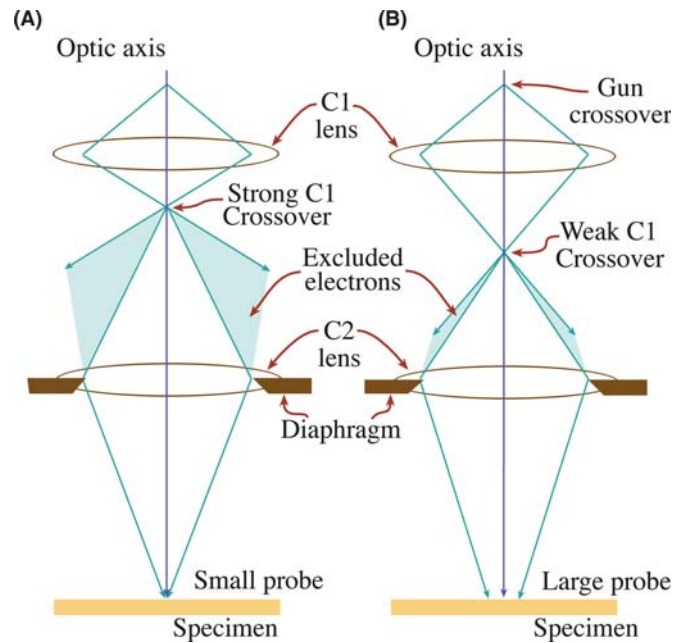


FIGURE 9.4. Effect of the C1 lens strength on probe size: a stronger C1 lens (A) results in greater demagnification by any subsequent lens (C2 and/or C3), giving a smaller electron beam at the specimen. A weaker lens (B) gives a broader probe. Note how changing C1 also changes the number of electrons that hit the C2 diaphragm and therefore, do not contribute to the current in the probe hitting the specimen.

the objective lens. This extra lens is a condenser mini-lens (called a twin-lens in Philips/FEI instruments) and it is standard on TEMs that also operate as STEMs or analytical TEMs (i.e., it's standard on all modern TEMs used in materials characterization). Without the condenser mini-lens, the action of the c/o is always similar to the objective lens in a dedicated STEM because, in such instruments, parallel-beam imaging conditions are never required. All imaging and analyses are accomplished with a convergent probe. However, in TEM where both parallel and convergent beams are needed, when the mini-lens is activated, the convergent effect of the c/o lens can be offset by forming a crossover at the FFP of the objective. Here the FFP is actually acting as the condenser part of the c/o lens. Thus, unlike in conventional objective lenses (see Figure 6.5), the parallel-beam condition is met in the *overfocus* condition in the c/o lens. If you're feeling confused, take another look at Figure 9.5.

THE c/o LENS

In older TEMs without a c/o lens, underfocus gives a more parallel beam at the plane of the specimen than overfocus. This is not the case if the objective lens is a c/o lens and the beam is more parallel at the specimen when the c/o lens is overfocused.

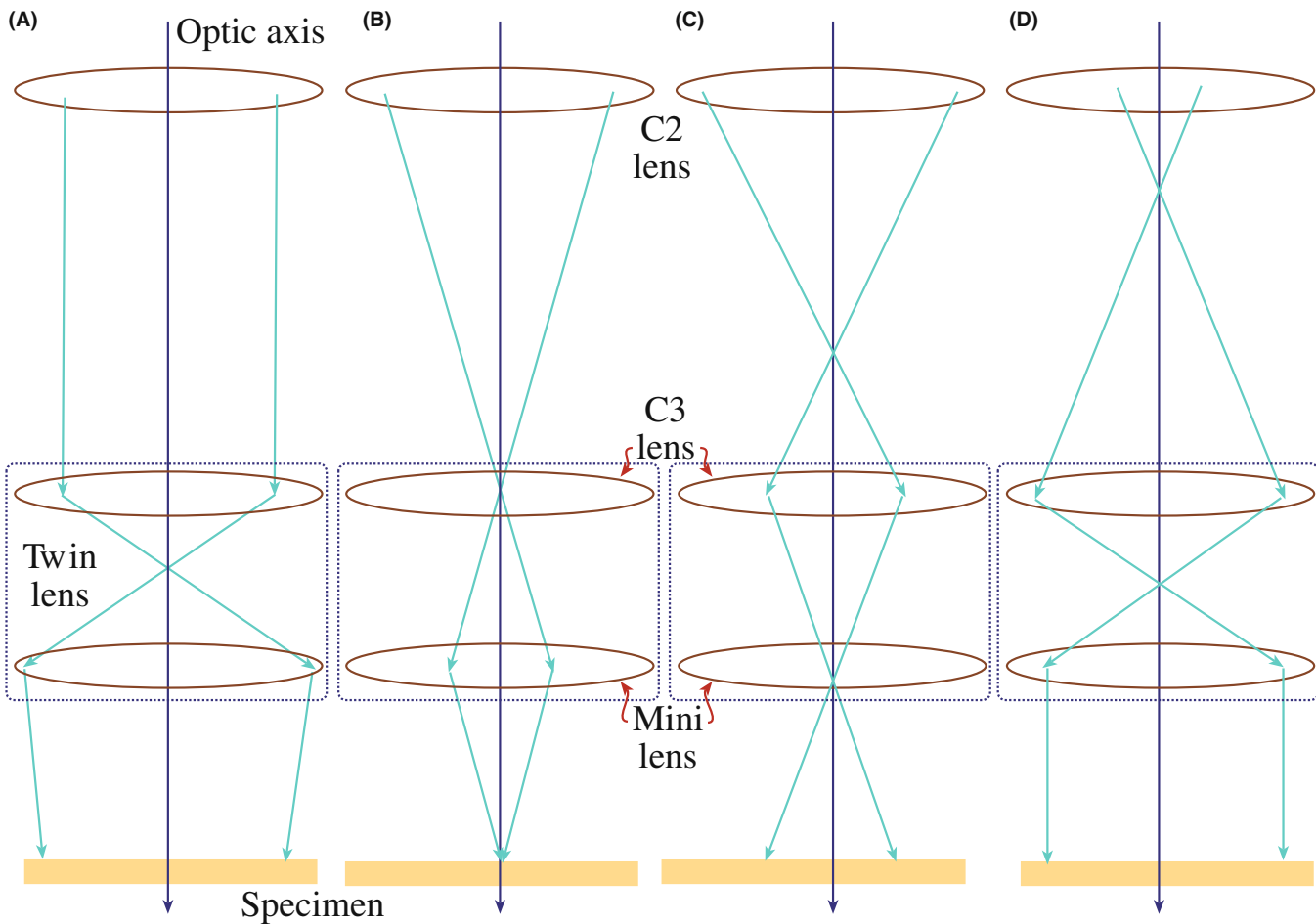


FIGURE 9.5. Condenser/objective lens operation as a function of the strength of the C2 lens. As the C2 strength is increased the beam on the specimen varies from (A) almost parallel, through (B) convergent, and (C) divergent, then (D) back to parallel. You can see that the crossover moves up the column as the C2 strength increases and the C3 lens introduces its own crossover prior to the minilens depending on the convergence of the ray paths entering it.

Figure 9.5 shows the various ray paths through a c/o lens, with a mini/twin lens, as the strength of the C2 lens is changed. As you can see, the beam at the specimen varies from parallel through convergent, divergent, and then back to parallel, as C2 is strengthened. Therefore, because of this interaction between C2 and the c/o lens, changing C2 changes the focus of the probe at the specimen and thus changes the focus of the STEM image and the CBED pattern and also changes the spatial resolution of any XEDS or EELS analyses, as we'll describe in detail in Part 4. So you have to be aware that, when you are operating in probe mode, two lenses control the focus, which can really mess things up unless you are fully aware of the situation. If you are operating in conventional parallel-beam TEM mode then you can simply use the objective lens (lower polepiece) to focus the image (see Section 9.3) and you don't need to adjust the illumination-system optics.

All recent probe-forming TEMs operate at a very specific c/o lens excitation where the STEM probe can be imaged in TEM mode on the viewing screen/

computer display. This c/o condition for simultaneous focus and image is a very limited one. (Look in the manufacturer's instructions to find out how to set this condition exactly on your own TEM.) If you don't do this, you're not at the correct objective-lens excitation and the beam can act in contrary ways; the beam convergence can decrease when you expect it to increase and the electron distribution in the probe can be spread into a broad halo rather than a fine focused point. So find out how to do this whenever you're operating in probe-forming mode.

In a c/o lens, while the convergence angle can be effectively reduced to zero, the helical path of the electrons through the strongly excited upper-objective polepiece never permits truly parallel illumination. In contrast, in the older conventional C1/C2 optics, the objective pre-field is not strong and the spiraling is greatly reduced. Therefore, image contrast and other effects arise as a result of c/o lens operation. If you read Christenson, and Eades all of this is very well explained.

9.1.D Translating and Tilting the Beam

There are certain operations where we need to translate the beam laterally on the specimen (e.g., to position a fine probe on a feature of interest for analysis). Similarly, there are times when we need to tilt the beam off the optic axis so it impinges on the specimen at a specific angle (e.g., for centered-DF imaging using a specific diffraction spot which we describe in Section 9.3.C or for hollow-cone imaging/diffraction (Section 9.3.D) and precession diffraction (Section 18.8)). Both translating and tilting are also essential for aligning the beam down the column and are accomplished by varying the current through potentiometers (which we call ‘scan coils’) to generate a local magnetic field which deflects (rather than focuses) the beam. There are several sets of scan coils in the column, some of which tilt and others which translate the beam. Ray diagrams to explain translating and tilting the beam are shown in Figure 9.6A and B.

When we create a scanning beam for STEM imaging, the beam must always move parallel to the optic axis to mimic the parallel-beam illumination conditions of standard TEM. Such scanning is accomplished by tilting the beam twice with two sets of scan coils (one above the other) to ensure that the beam crosses the optic axis at the FFP of the upper-objective-lens polepiece. Then, wherever the beam enters the upper-objective-lens field, it is tilted to follow a path parallel to the optic axis (so sometimes you have to tilt in order to translate!). You need computer control to do this rather complex adjustment (look ahead to Figure 9.17). Like

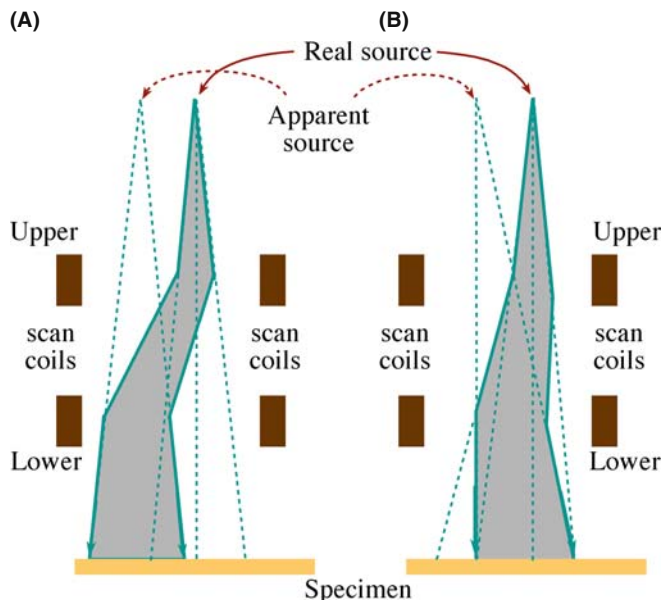


FIGURE 9.6. The use of pre-specimen scan coils for (A) traversing the beam and (B) tilting the beam. Traversing moves the beam to a different area of the specimen but it stays parallel to the optic axis. Conversely, tilting the beam illuminates the same area of the specimen, but from a different angle of incidence.

many other procedures on a modern TEM, this adjustment is made automatically when you select a particular (in this case STEM) operating mode.

9.1.E Alignment of the C2 Aperture

Instructions for alignment vary for different TEMs so we’ll simply describe the principles. Even if you won’t be doing the alignment you will want to check that the microscope is correctly aligned; you can recognize if the wheels are not aligned on your car and you know it is important to balance them for best performance of the vehicle, even if you have to ask someone else to perform the task. If you want the best out of your machine, you’ll want to be able to fine-tune this alignment.

If your illumination system is correctly aligned, the gun crossover is on the optic axis and the electrons can then travel down the optic axis in a straight line through the lenses and apertures until they hit the specimen. We’ll assume that your gun is aligned so that the source crossover is on the optic axis of the column, as we described back in Chapter 5. Alignment of the illumination system used to be a tedious, manual affair involving tilting and translating each of the condenser lenses and centering the apertures on axis. Now most of the lens components are machined accurately enough so that only minor electronic tilts and translations are needed and usually these are computer controlled. Under these circumstances, centering the C2 aperture remains the most critical step in obtaining the best performance out of your TEM, particularly if you intend to operate in scanning mode for STEM imaging and analysis. (Other apertures in the illumination system are usually left untouched.) The C2 alignment remains a manual operation on most TEMs, although undoubtedly this will not always be the case.

Operational Procedure #1 You must always have the C2 aperture accurately centered on the optic axis of the TEM. If the aperture is misaligned, the image of the beam on the screen moves off axis and distorts as you underfocus or overfocus C2, as shown in Figure 9.7. To adjust the diaphragm so the aperture is on axis we have to alternately focus and defocus the C2 lens (we’ll arbitrarily start by overfocusing but it actually doesn’t matter if you under- or overfocus first). It’s best to start at a low magnification so you can find the aperture image then repeat at the magnification range where you wish to operate.

- First, overfocus C2 (strengthen the lens) so the beam is spread on the screen and the image of the C2 aperture is visible (make sure any other apertures in the *imaging* system are out of the column).
- Then use the external manual drives (or computer control if you are lucky enough) to move the diaphragm so the aperture image is centered on the screen.
- Next, weaken C2 so the image of the beam is focused to its minimum diameter.

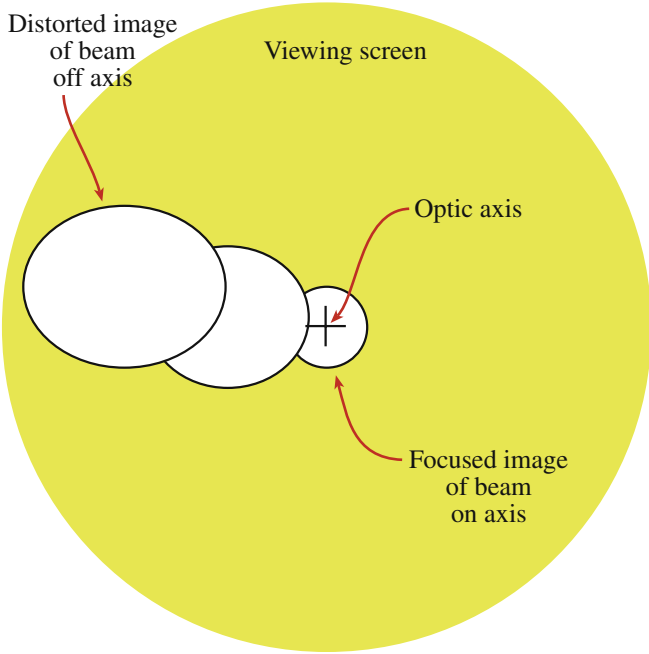


FIGURE 9.7. If the C2 aperture is misaligned, wobbling (alternately underfocusing and overfocusing the C2 lens) causes the image of the beam to sweep off axis (i.e., move across the viewing screen) and to become distorted.

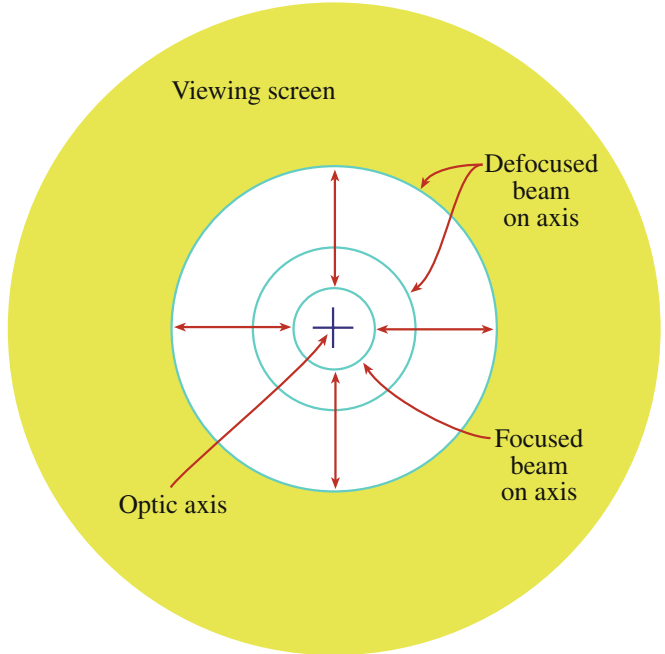


FIGURE 9.8. If the C2 aperture is aligned on axis, the image of the beam remains circular and expands or contracts about the optic axis as C2 is wobbled.

- Then, center the beam with the condenser deflector controls.
- Now underfocus the C2 lens (i.e., continue to weaken it) until you can again see the aperture image and center the image again with the external drives.
- Now strengthen the lens until the spot is focused and center it with the deflector coils.
- You have to repeat this whole operation iteratively until the image of the beam expands and contracts around the center of the screen as shown in Figure 9.8.

WOBBLING

Usually there's a control that will introduce an AC current into the lens coil, in effect 'wobbling' the lens setting either side of focus. This 'wobbler' saves you from manually underfocusing and overfocusing the lens, but think what you are doing.

In TEM mode this operation should only be necessary on the rare occasions when you change the diaphragm or the C1-lens setting but, in STEM, any change of probe size requires re-alignment of the C2 aperture.

9.1.F Condenser-Lens Defects

The illumination-system lenses suffer from all the standard lens defects we described in Chapter 6, such as

aberrations and astigmatism. These defects don't really limit the operation of the TEM in parallel-beam mode, but they are crucial if you're intent on forming the finest probe possible for STEM and analytical work. Let's look at the role of each of the major defects.

Spherical Aberration This defect plays no role in limiting parallel-beam formation. However, as we discussed in Chapter 5, in adjusting the illumination system to form the finest possible probe with the maximum available current, spherical aberration in the probe-forming lens (C3) controls the minimum possible probe size. In exactly the same manner as we control the image resolution (see Chapter 6), spherical aberration limits the probe dimensions to a minimum radius (equation 6.22) of $r_{\min} \sim 0.91(C_s\lambda^3)^{1/4}$. So the C3 probe-forming lens has a short focal length to minimize C_s . The final probe-limiting aperture in C2 needs to be carefully chosen to be the optimum value (equation 6.23) for the selected probe size $\alpha_{\text{opt}} = 0.77 \lambda^{1/4}/C_s^{1/4}$. In practice, however, there are always more C1 settings than available C2 apertures, so it is not possible to choose the optimum aperture for each probe. This can cause problems if you need a specific probe size for a certain spatial resolution, as we discuss in Chapter 36. Of course, if you are lucky enough to be using a STEM with a C_s -corrected probe then this problem is minimized.

Chromatic Aberration Remember this aberration depends on the energy spread of the electrons. Since the electrons in the beam (particularly from a FEG)

have a very small energy spread we generally regard them as monochromatic and there is no detectable degradation of the probe dimensions. However, for the very best image resolution or the very best EELS, C_c -correction via monochromation of the beam can help, as we'll describe later in Section 37.7.

Astigmatism This is the most common defect in the TEM illumination system and arises either because the final limiting aperture in C2 is misaligned or it is contaminated and charging up, thus deflecting the beam. We'll assume you've centered the C2 aperture as we just described and we'll now tell you how to detect and correct any residual astigmatism due to contaminated apertures. The condenser stigmators introduce a compensating field (like a scan coil) which corrects this distortion.

Operational Procedure #2A — Detecting Astigmatism

You can discern astigmatism in the illumination system by looking at an image of the electron source on the display/screen as follows

- Operate in image mode with no specimen inserted (or look through a hole in your specimen).
- Focus C2 so the beam is a minimum diameter (i.e., in spot mode with the size governed by C1).
- Adjust the C2 aperture and the beam traverses so the spot is in the middle of the screen and the image of the beam is circular, as you did when aligning the aperture.
- Wobble the C2 lens either side of the focal setting so the image of the beam expands and contracts about its minimum dimension.
- If there is astigmatism, the image is not circular, but distorts elliptically and rotates through 90° either side of focus, as shown in Figure 9.9.

Operational Procedure #2B — Correcting Astigmatism

- First overfocus the beam so you can see the effect of the astigmatism (i.e., the beam appears elliptical) in one direction.
- Then adjust the stigmators so the image appears circular.
- Now underfocus the beam and repeat the correction.
- Repeat the whole over/underfocus procedure iteratively until the image of the beam remains circular as you expand and contract it on the screen with the C2 lens (see Figure 9.8). Again, a wobbler can help here once you get the hang of the interplay between defocusing and the stigmators.
- If you can't make the image circular, you'll have to increase the range of strength of the stigmators. If you are on maximum strength, then there is too much contamination on the aperture and you either need to replace or flame-clean the diaphragm, as we described in Chapter 6.

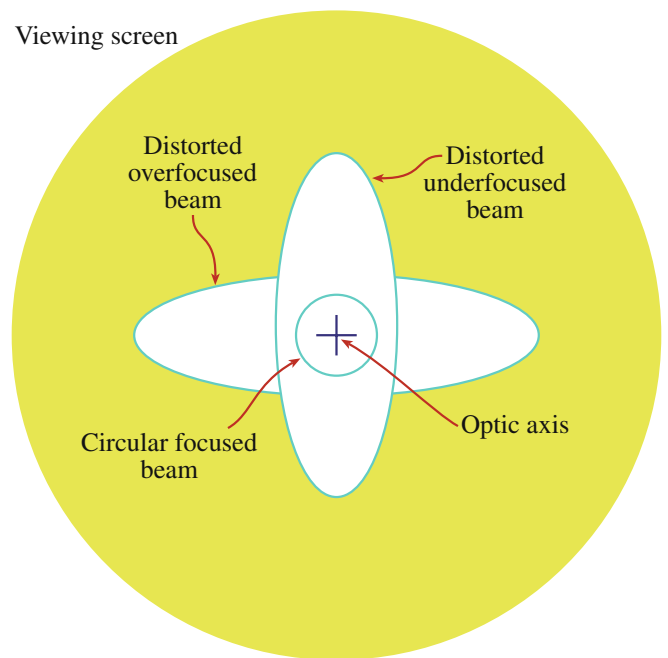


FIGURE 9.9. The effect of astigmatism in the illumination system is to distort the image of the beam elliptically as the C2 lens is wobbled. Correction of this astigmatism results in an image that remains circular as the C2 lens is defocused (as in Figure 9.8).

9.1.G Calibration

We've already described in Section 5.5 what it takes to measure the performance of the electron gun and to optimize the brightness in STEM operation so that the maximum beam current goes into the minimum probe size. We also have to calibrate the illumination system. The major variables are the probe size (and current) for various C1 settings and the convergence angle for various C2 aperture sizes.

The C1 lens strength controls the probe size at the plane of the specimen. We've described in some detail how to measure the beam dimensions at the specimen back in Section 5.5.C. in Chapter 5. Figure 9.10A shows the variation of the calculated (not measured) probe size as a function of the C1 lens setting for a typical TEM. These calculations are approximate, since they define the probe width as the FWHM and assume the C2 aperture correctly limits the Gaussian distribution. Despite these approximations you can clearly see the expected trend of decreasing probe size with increasing C1 strength. The probe current is a strong function of the probe size. If you look in the text by Goldstein et al. you'll find that the maximum probe current is proportional to (almost) the cube of the probe diameter: if you increase the probe size by a factor of 10, the current will increase by almost $1000\times$. This is an extraordinary dependence and explains why, particularly for probe-related TEM work (XEDS, EELS, STEM imaging, and CBED), we are very

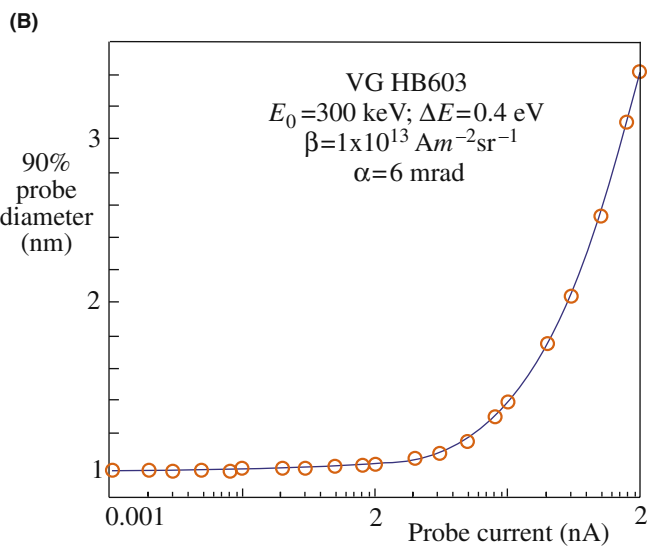
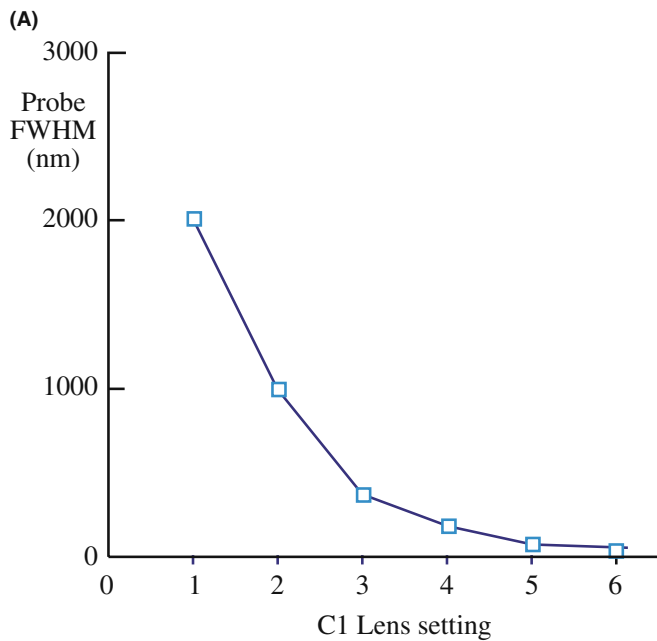


FIGURE 9.10. (A) Calibration of the illumination system requires determining the variation of the probe dimension with C1 lens strength. (B) The probe current is strongly related to the probe size. These data are from a 300-keV FEG-STEM; the dependence would be even greater in a thermionic-source TEM.

concerned about knowing and measuring the probe size and current. (There’s much more about this in Part 4.) Figure 9.10B shows the variation of probe current with probe size for a FEG AEM.

The C2 aperture size governs the convergence (semi) angle α , as we also discussed in Chapter 5 when we were determining the gun brightness.

- We measure the total convergence angle 2α from a CBED pattern (Figure 5.8).
- We increase α by increasing the C2 aperture size (Figure 9.11).

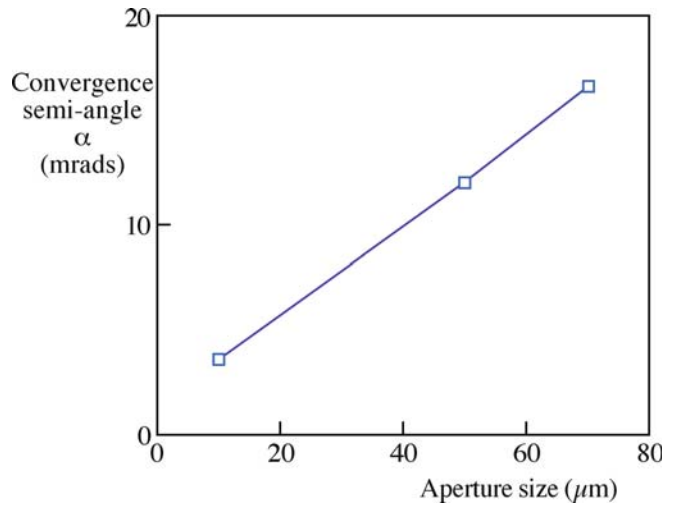


FIGURE 9.11. Increase of the beam-convergence angle, α , with increasing C2 aperture size. Increasing α will also increase the probe current.

Obviously the probe current will also be affected by the aperture size (e.g., if you double the aperture diameter you would expect four times the number of electrons to come through it). But, as you learned back in Chapter 6 (equations 6.22 and 6.23), because of the increasing lens aberrations with increasing α , there is an optimum aperture that gives the maximum probe current (and hence the best image resolution) that we just mentioned. You might well expect that, if there is a C_s corrector in the TEM illumination system, this limit can be overcome and you would be correct.

On some instruments the C2 aperture is virtual (so you have an effective aperture size), which makes it rather difficult to measure 2α . (See the Goldstein et al. text for a detailed description of this problem, which is common in SEMs.) Furthermore, if the C2 lens is excited, it can also change α and then you have to calibrate α both as a function of the aperture size and the C2 lens setting, which is an extremely tedious exercise.

9.2 THE OBJECTIVE LENS AND STAGE

This combination is the heart of the TEM. We use the stage to clamp the specimen holder in the correct position so the objective lens can form images and DPs in a reproducible manner. As we discussed in Chapter 8, there are two different types of holder, top-entry and side-entry and these determine the geometry of the polepiece and the flexibility which you have to make adjustments. We’ll only consider side-entry holders here since they are standard but top-entry holders require the same adjustment of the z -control or specimen height.

As a practical consideration, you'll find that you would like to be able to tilt the specimen without changing its height on the optic axis. Otherwise you will be continuously using the *z*-control whenever you tilt. Clearly this means that you should ensure that the region of the specimen you want to work on is located close to the tilt axis of the specimen rod.

FOCUSING AND HEIGHT

We cannot emphasize too strongly that we need to fix the height of our specimen on the optic axis so we can always work at the same objective-lens current and thus at a fixed objective-lens magnification.

This is **really** important! This is *usually* ignored!

The central requirement here is the need to define a reference plane so that our calibrations will be reproducible. The reference plane (see Chapter 6) for a side-entry holder is called the eucentric plane. This plane is normal to the optic axis and contains the axis of the specimen-holder rod; clearly there could be many such planes. What is special about the eucentric plane is that when your specimen is located at this plane and the image is in focus, the objective-lens current is a standard reference value. The position of this plane within the objective lens is known as the eucentric height. If you put your specimen in the eucentric plane, then a point on the optic axis does not move laterally when you tilt it around the holder axis, which makes many essential imaging and diffraction operations very easy. Unfortunately, if you tilt your specimen normal to the holder axis, or rotate/traverse it off axis, then the area you're examining almost invariably moves out of the eucentric plane upon tilting and the image can easily disappear from the screen. This limitation can only be overcome by complete computer control of all five axes (three translations (*x*, *y*, *z*) and two tilts (parallel and orthogonal to the axis of the holder)) and such stages are often called double-eucentric. They are becoming more commonly available and are extremely useful. Get one if you can!

Operational Procedure #3A The first thing you must always do when inserting your specimen into the TEM is to ensure that it is in the eucentric plane. To do this, first find the *z*-control, which is usually a knob on the outside of the goniometer stage near the hole where you insert the holder. Then

- Select a recognizable feature in the image and position it at the center of the screen at a reasonable magnification (20–50,000×).

- Tilt the goniometer stage a few degrees clockwise or anticlockwise until the feature moves close the limit of the viewing screen. (If the slightest tilt has this effect then go to a lower magnification until you can tilt a few degrees and still see the feature on the screen.)
- Adjust the height of the specimen holder until the feature returns to the center of the screen.
- Tilt the goniometer the other way and repeat the process.
- Continue to increase the tilt both ways and compensate with the *z*-control until the image of the specimen remains stationary when you tilt the specimen through about $\pm 30^\circ$ either side of zero tilt.
- Repeat at a higher magnification as needed.

Operational Procedure #3B: Another Way to Set the Eucentricity The eucentric position of the stage is a mechanical point located in the column. As we mentioned earlier, the optimal *c/o* lens setting in a STEM/TEM is a very well-defined and reproducible excitation of the objective lens. This lens condition also defines a mechanical point somewhere between the polepieces. We need to make the two points (eucentric position and the optimal *c/o* lens excitation position) the same. To find the eucentric point when these conditions are met, you need to

- Set the lens condition to the optimal *c/o* operating condition.
- Focus the image using the *z*-control.
- The specimen height will now be set to the eucentric position.

With practice you can set these two points to differ by $< 10 \mu\text{m}$, and $< 3 \mu\text{m}$ is not uncommon.

With computer control and auto-focusing techniques becoming common, this operation can be automated. As a result, in a completely eucentric stage, your specimen doesn't move off the optic axis and remains in focus no matter around what axis it is tilted or rotated. If you don't have such a computer-controlled stage, be cautious and develop the skill to tilt and change the *z*-control simultaneously.

The eucentric plane should also be symmetrically positioned with respect to the upper and lower objective polepiece fields. At this condition, the eucentric plane coincides with the plane at which the electron beam is imaged, in both TEM and STEM modes. If the symmetric plane and the eucentric plane are not coincident, then the images and DPs will appear at different magnifications and different focus settings in TEM and STEM. Obviously this requirement has no meaning in a DSTEM where there is no TEM mode.

Ensuring coincidence of the eucentric and symmetric planes is usually carried out by the manufacturer. You can check it by comparing the focus of a DP or an image in TEM and STEM modes. You should not have to refocus the image or DP with the objective lens when you change from one mode to the other. If you find this is a problem, talk to your technical staff.

9.3 FORMING DPs AND IMAGES: THE TEM IMAGING SYSTEM

You know already that the objective lens takes the electrons emerging from the exit surface of the specimen, disperses them to create a DP in the back-focal plane (BFP), and recombines them to form an image in the image plane (see Figure 6.3). We can use this ray diagram to introduce the basic operations for forming static-beam images and DPs in the TEM. We'll then describe how to do the same thing with a scanning beam in STEM mode.

In this discussion, we will skip many of the details and concentrate on the role of the instrument. In Chapter 11 we'll discuss the details of the diffraction process and then expand these ideas in Chapters 16 through 21. We'll then discuss the images formed in the TEM in Chapters 22 through 31.

PUSH-BUTTON TEM

There are buttons to push on the console or icons to click on the computer screen by which you choose either **image mode** or **diffraction mode**. These are the primary modes, central to TEM operation. Now TEM is so easy it has never been more difficult!

The first operation that you need to master when using the TEM is viewing the DP (diffraction mode). In all the subsequent imaging modes, we'll form our images by using the objective aperture and the DP to select electrons that have been scattered through particular angles.

- *Diffraction mode*: to see the DP you have to adjust the imaging-system lenses so that the *BFP* of the objective lens acts as the object plane for the intermediate lens. Then the DP is projected onto the viewing screen/CCD as shown in Figure 9.12A.
- *Image mode*: if you want to look at an image instead, you readjust the intermediate lens so that its object plane is the *image plane* of the objective lens. Then an image is projected onto the viewing screen/CCD, as shown in Figure 9.12B.

Let's look now at the details of these two fundamental operations from the point of view of the instrument.

In subsequent chapters, we will discuss how to understand the images/DPs and why we form them in the ways we do.

9.3.A Selected-Area Diffraction

As you can see from Figure 9.12A, the DP contains electrons from the whole area of the specimen that we illuminate with the beam. Such a pattern is not very useful because the specimen will often be buckled. Furthermore, the direct beam is often so intense that it will damage the viewing screen or saturate the CCD camera. So we perform a basic TEM operation which both selects a specific area of the specimen to contribute to the DP and reduces the intensity of the direct beam in the DP on the screen.

If you look at Figure 9.12A, there are two ways we could reduce the illuminated area of the specimen contributing to the DP.

- We could make the beam smaller
- We could insert an aperture above the specimen which would only permit electrons that pass through it to hit the specimen

The first option involves using C2 and/or C3 to converge the beam at the specimen. We use this approach to form CBED patterns, which we'll discuss in great detail in Chapters 20 and 21. Converging the beam destroys any parallelism, and spots in the pattern are not sharply defined but spread into disks. If we wish to obtain a DP with a parallel beam of electrons, the standard way is to use the selected-area aperture. Now, we can't insert an aperture at the specimen plane, because the specimen is already there! But if we insert an aperture in a plane *conjugate with* the specimen, i.e., in one of the image planes of an imaging lens, then it creates a virtual aperture at the plane of the specimen. This is exactly what we do. This operation is called selected-area diffraction or SAD, and is shown in Figure 9.12A.

The conjugate plane that we choose is the image plane of the objective lens. As shown in Figure 9.13, we create SADPs by inserting what we call the SAD aperture into the image plane of the objective lens and we center this aperture on the optic axis in the middle of the viewing screen. You can see this aperture if you want to change it or center it, by projecting the image plane onto the viewing screen/CCD, as we'll discuss in Section 9.3.B below.

Operational Procedure #4 The specific steps to form an SADP are as follows (while it looks complicated you'll soon find that it only takes a few seconds to do all these steps)

- Choose image mode so you can see an image of your thin specimen on the viewing screen.

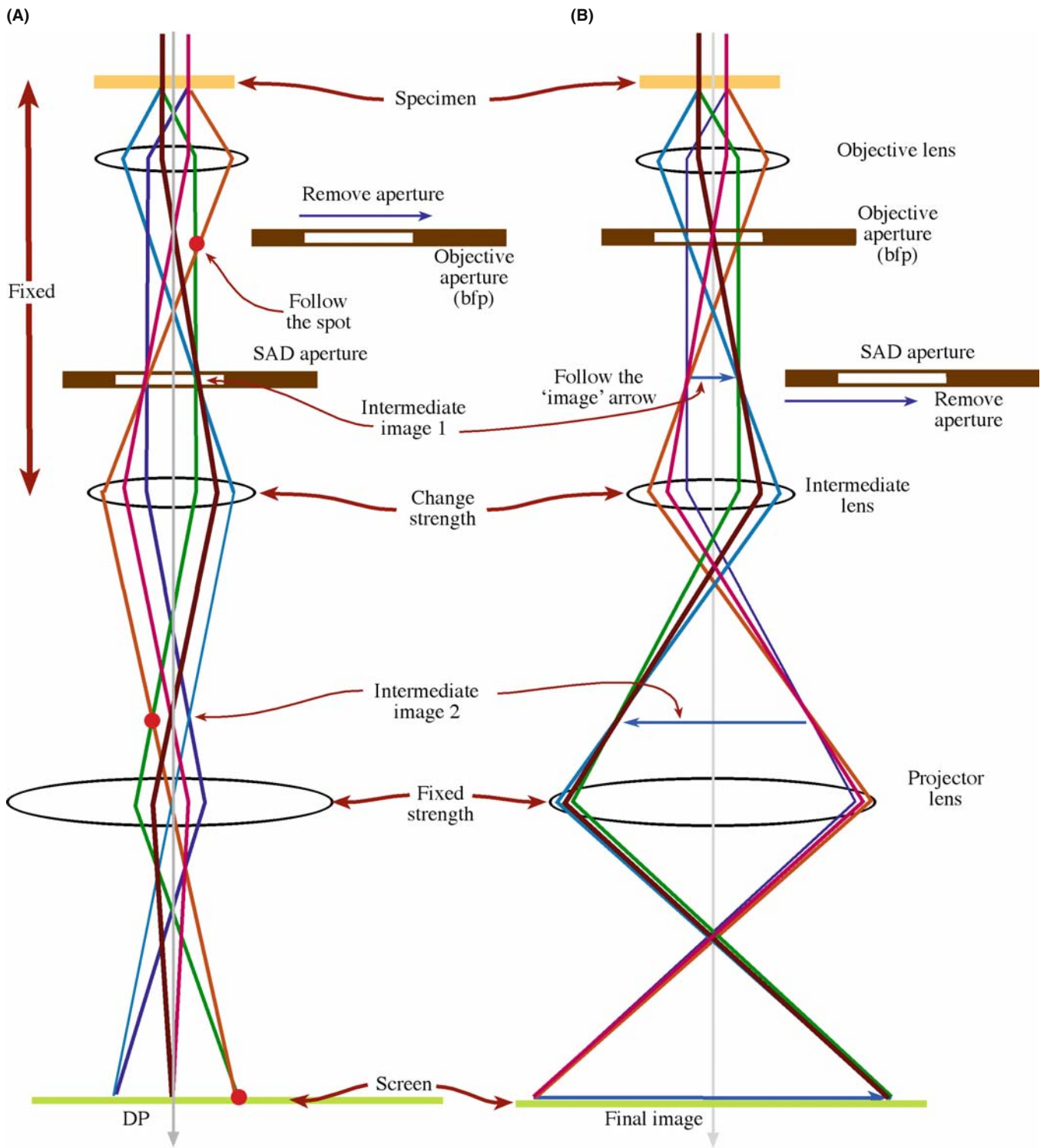


FIGURE 9.12. The two basic operations of the TEM imaging system involve (A) diffraction mode: projecting the DP onto the viewing screen and (B) image mode: projecting the image onto the screen. In each case the intermediate lens selects either the BFP (A) or the image plane (B) of the objective lens as its object. The imaging systems shown here are highly simplified. Most TEMs have many more imaging lenses, which give greater flexibility in terms of magnification and focusing range for both images and DPs. The SAD and objective diaphragms are also shown appropriately inserted or retracted. NOTE: This is a highly simplified diagram showing only three lenses. Modern TEM columns have many more lenses in their imaging systems.

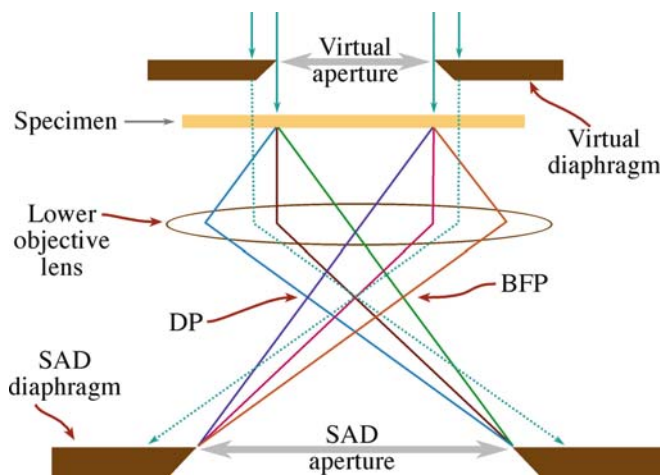


FIGURE 9.13. Ray diagram showing SADP formation: the insertion of an aperture in the image plane results in the creation of a virtual aperture in the plane of the specimen (shown here slightly above the specimen plane). Only electrons falling inside the dimensions of the virtual aperture at the entrance surface of the specimen will be allowed through into the imaging system to contribute to the SAD pattern. All other electrons (dotted lines) will hit the SAD diaphragm.

Under these conditions, with an SAD aperture inserted and the objective apertures removed, any electron that hits the specimen outside the area defined by the virtual aperture will hit the diaphragm when it travels onto the image plane. Such electrons will thus be excluded from contributing to the DP that is projected onto the viewing screen. In practice, we can't make apertures smaller than $\sim 10 \mu\text{m}$, and the demagnification back to the plane of the specimen is only $\sim 25\times$ which gives a minimum selected area of $\sim 0.4 \mu\text{m}$ —which isn't as small as we'd like, particularly in today's nano-world. We'll discuss in Chapter 11 whether or not smaller values would be useful. As always, you really need to know these values for the TEM you are using, so do the calibration.

SAD

It is a basic principle of TEM operation that when you want to look at the DP (i.e., the **BFP** of the objective lens), you put an SAD aperture into the **image plane** of the objective lens.

- Spread the beam by underfocusing C2.
- Make sure that the objective diaphragm is retracted. The image contrast will be poor and it will be difficult to focus but that's all right at this stage.
- Insert the SAD diaphragm and start by choosing the largest available aperture to make finding the aperture easy (there may be three to five different apertures depending on the specific TEM you are using).
- If the screen then goes blank and you can't see anything, the aperture you chose is not on axis and you should lower the image magnification until you can see the aperture and center it on the axis.
- If the illuminated area of the image isn't restricted when you insert the aperture then you are at too high a magnification; so lower it until you can see the image of this aperture on the viewing screen.
- Traverse the specimen if necessary to make sure the specific region you are interested in is visible through the aperture.
- You must focus the SAD aperture by adjusting the intermediate lens so it is conjugate with (i.e., exactly in the plane of) the image of the specimen that we focused with the objective lens.
- Switch to diffraction mode and an SADP will appear on the screen.
- Focus the SADP with the diffraction-focus controls so the spots are sharp. Further underfocusing C2 may also help at this stage.
- If you need a smaller aperture to select a smaller region of the specimen, simply click the diaphragm holder in or out to choose a smaller aperture and repeat the above procedure.

The SADP is often displayed on the viewing screen at a fixed magnification so that you can recognize differences in the magnitude of **g**.

By analogy with an X-ray diffractometer or pinhole camera, we define a distance called the 'camera length' (L). We think of this length as being the distance of the recording plane from the DP (look ahead to Figure 9.23); it's analogous to a real distance in a real camera. We choose the value of L such that inter-spot or ring spacings in the DP are easily discernible on the viewing screen and on the recording medium. This magnification can be changed by adjusting the intermediate lenses. We'll describe how we calibrate this magnification in Section 9.6.B.

At this stage, however, it's worth noting that, with a c/o lens, calibration of L can be a challenge because changing the condenser-lens focus as we just described changes the diffraction crossover and, if you re-focus the SADP with the intermediate lens, then you'll measure a different L .

Beware: in all the early TEM books, SAD is the only standard diffraction technique. As a result, some microscopists only use SAD to obtain diffraction information. However, CBED, which we discuss in Chapters 20 and 21, provides a great deal of complementary diffraction information and *must* be used by all TEM operators in the materials and nanotechnology fields. But there are still certain times when your best option is to form an SADP

- When you need to select a spot from which to form a BF or DF image (see next section)
- When diffraction spots are very close to one another and would overlap in CBED patterns (see examples in Chapters 23 and 24)

- When you are looking for fine structure in the DP such as streaks (see Chapter 17)
- When your specimen is beam sensitive

On all other occasions, when the diffraction maxima provide the most important information in the pattern, you should use CBED.

9.3.B Bright-Field and Dark-Field Imaging

When the SADP is projected onto the viewing screen/CCD, we can use the pattern to perform the two most basic imaging operations in the TEM. No matter what kind of specimen you're looking at, the SADP will contain a bright central spot that contains the direct-beam electrons and some scattered electrons (as shown in Figure 2.13A–C), the distribution of which will depend on the nature of your specimen.

IMAGING

It is another basic principle of TEM operation that if you want to view an image (i.e., the **image plane** of the objective lens), you insert an aperture called the objective aperture into the **BFP** of the objective lens.

Operational Procedure #5 When we form images in the TEM, we either form an image using the central spot or we use some or all of the scattered electrons. The way we choose which electrons form the image is

- Starting with both SAD and objective diaphragm holders retracted, select image mode to observe an image of the desired region of your specimen on the screen. It will not be a good image because there is no objective aperture inserted, but that is OK at this stage.
- Form an SADP of a selected region as we described in operational procedure #4.
- Insert the objective diaphragm so an aperture appears in the BFP of the objective lens, thus blocking out most of the DP, except that small area which is visible through the aperture. As with the SAD operation above, we use the external drives to move the aperture.
- Adjust the external diaphragm drives so the aperture selects the direct beam on the optic axis.
This centering procedure is absolutely critical to forming the best image. If you have binoculars, view the TEM screen at a much higher magnification to ensure the best possible centering; otherwise do the equivalent using the digital image.
- Return to image mode, remove the SAD aperture, and focus the image with the objective lens. We call the resultant image, formed by the direct-beam electrons, a bright-field (BF) image (Figure 9.14A).
- If instead we choose scattered electrons (e.g., a specific diffracted beam (usually) or a portion of a

diffraction ring), then we call the image a dark-field (DF) image (Figure 9.14B).

You can view the BF and DF images at any magnification simply by adjusting the intermediate lenses of the microscope and typical magnification ranges will be $\sim 25,000\times$ to $100,000\times$ for a wide field of view, but up to $10^6\times$ for high-resolution images. Usually you'll have to calibrate the actual magnification and also be able to relate directions in the image at any magnification to directions in the DP at a fixed camera length. These are the two basic calibrations required for any TEM.

BF AND DF

Select the direct beam to form a BF image.
Select only electrons that are not in the direct beam to form a DF image.

The insertion and removal of the SAD and objective apertures can be confusing to the beginner and often the wrong aperture is inserted or not removed when it should be. You have to practice obtaining SADPs and BF/DF images to get used to what aperture should be inserted and when. Both apertures are inserted below the objective lens. The objective aperture goes into the BFP, so it is closer to the lens (i.e., higher up the column) than the SAD aperture, which is in the image plane. Remember that if you're looking at a DP the (lower) SAD aperture should be inserted and the (upper) objective aperture removed. If you want to look at an image, the objective aperture should be inserted and the SAD aperture removed.

OBJECTIVE APERTURE

This is the most important aperture in the TEM. When inserted, its size controls the collection angle (β); hence, it determines the effect of the aberrations of the most important lens in the instrument and thus directly influences the resolution.

9.3.C Centered Dark-Field Operation

If you look at Figure 9.14B, the electrons that are selected by the aperture travel off the optic axis, since we displace the aperture to select the scattered electrons. The more off-axis the electrons are the greater the aberrations and astigmatism they suffer. Therefore, such a displaced-aperture DF (DADF) image is difficult to focus on an older TEM. You'll find that the image will move on the screen as you adjust the objective-lens strength. While there may be situations where you want to use such a DADF image, almost invariably you have to get any

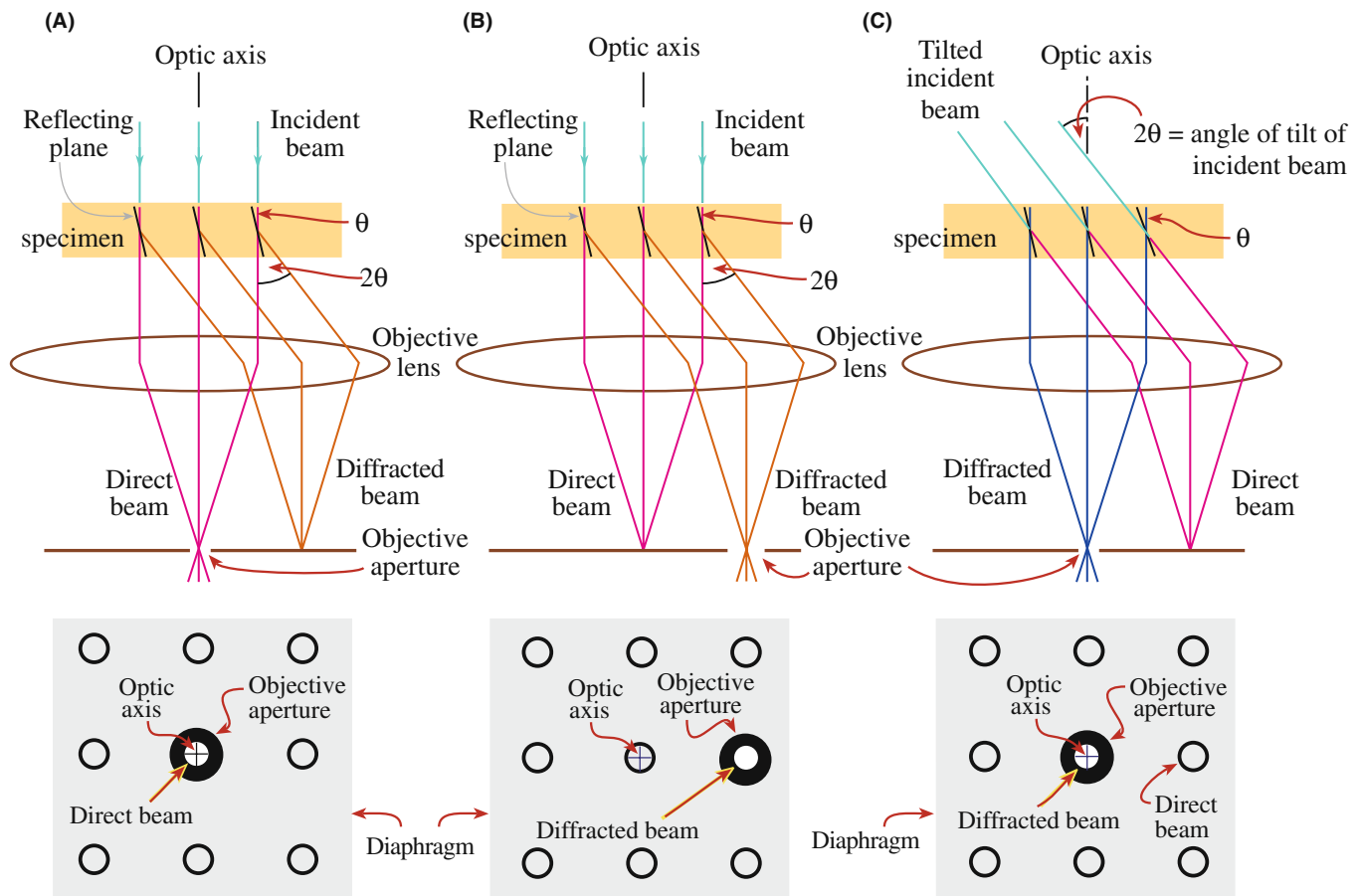


FIGURE 9.14. Ray diagrams showing how the objective lens and objective aperture are used in combination to produce (A) a BF image formed from the direct electron beam, (B) a displaced-aperture DF image formed with a specific off-axis scattered beam, and (C) a CDF image where the incident beam is tilted so that the scattered beam emerges on the optic axis. The area of the DP selected by the objective aperture, as seen on the viewing screen, is shown below each ray diagram. (Images comparing BF and DF are given in Part 3.)

scattered electrons back on the optic axis and then form a DF image under this condition. This operation is called centered dark-field (CDF) imaging. It is the conventional way to do DF imaging so we'll tell you how it's done. If you have a new TEM (with a small C_s) you should consider using only DADF for SAD because it's easier and you've eliminated the reason for forming CDF images!

Operational Procedure #6

- Start with an SADP on the screen and instead of moving the objective diaphragm so the aperture selects scattered electrons, adjust the aperture so it is on axis, as for BF imaging.
- Retract the objective diaphragm drive so you can see the SADP.
- Switch on and adjust the beam-tilt (DF) potentiometers above the objective lens so that the scattered-electron beam that you wish to use to form the CDF image moves toward the central, on-axis position.

- Looking through the binoculars, switch the potentiometers on and off to ensure that the scattered beam exactly superimposes where the direct beam was when the potentiometers are on and the direct beam is back on axis when the potentiometers are off.
- Re-introduce the objective diaphragm and check that the aperture is still centered around the on-axis scattered beam.
As with BF imaging, this aperture-centering procedure is absolutely critical to forming the best CDF image.
- Select image mode again and focus the CDF image.

What you are doing here is making the incident beam hit the specimen at an angle equal and opposite to the scattering angle. In this way the scattered electrons will now travel down the optic axis, as shown in Figure 9.14C.

We'll return to BF, CDF, DADF, and SAD operations when we discuss specific contrast mechanisms that occur in TEM images in Chapter 22.

9.3.D Hollow-Cone Diffraction and Dark-Field Imaging

One obvious limitation to either DADF or centered CDF imaging is that we are only using a small fraction of the scattered electrons. When that fraction is a specific diffraction spot from a crystalline specimen that can indeed be very useful, as we'll see in Part 3 of the book. However, if, for example, we want to see *all* the portions of the specimen or *all* the precipitate phases that are diffracting into a *set* of diffraction spots (e.g., $\{111\}$ rather than (111)) then we can either take separate CDF images from *all* the individual (111) reflections in the SADP (which is obviously a pain) or we can carry out hollow-cone (also called conical) diffraction and DF imaging. Likewise, if your specimen is micro/nanocrystalline so it generates a ring pattern, or if it is amorphous so diffuse-intensity rings are formed, then we can use the same technique to maximize the information in the images. There are two ways to do hollow-cone DF imaging: hardware and software. In essence, in either method, a specific set of diffracted beams is collected by the objective aperture when the conical-scanning beam satisfies a particular Bragg reflection condition (see Section 3.10 and Chapter 11).

- *Hardware:* Use an annular condenser aperture such that a cone of electrons illuminates the specimen at a fixed angle to the optic axis. Annular apertures fabricated by FIB techniques (see Chapter 10) are becoming more commonly available. (Think about how to make an annular aperture so that the middle doesn't just fall out of the doughnut shape leaving just a large circular hole?) So then we basically have multiple DPs formed; the direct (000) beam travels through the specimen and emerges off axis and a set of diffracted beams (depending on the cone angle) is always scattered on axis.
- *Software:* Use computer control of the scan coils to spin the incident (and thus the direct) beam around the optic axis. This method is obviously more flexible because, while an annular C2 aperture gives a fixed cone (semi) angle, the scan coils can be adjusted to give a variable cone angle. Thus, with the software approach, all the electrons scattered at a specific angle can be integrated into a single DF image which you can envisage as spinning (Figure 9.14C) around so that a set of diffraction spots always rotates through the optic axis. The principle of hollow-cone diffraction and imaging is shown in Figure 9.15.

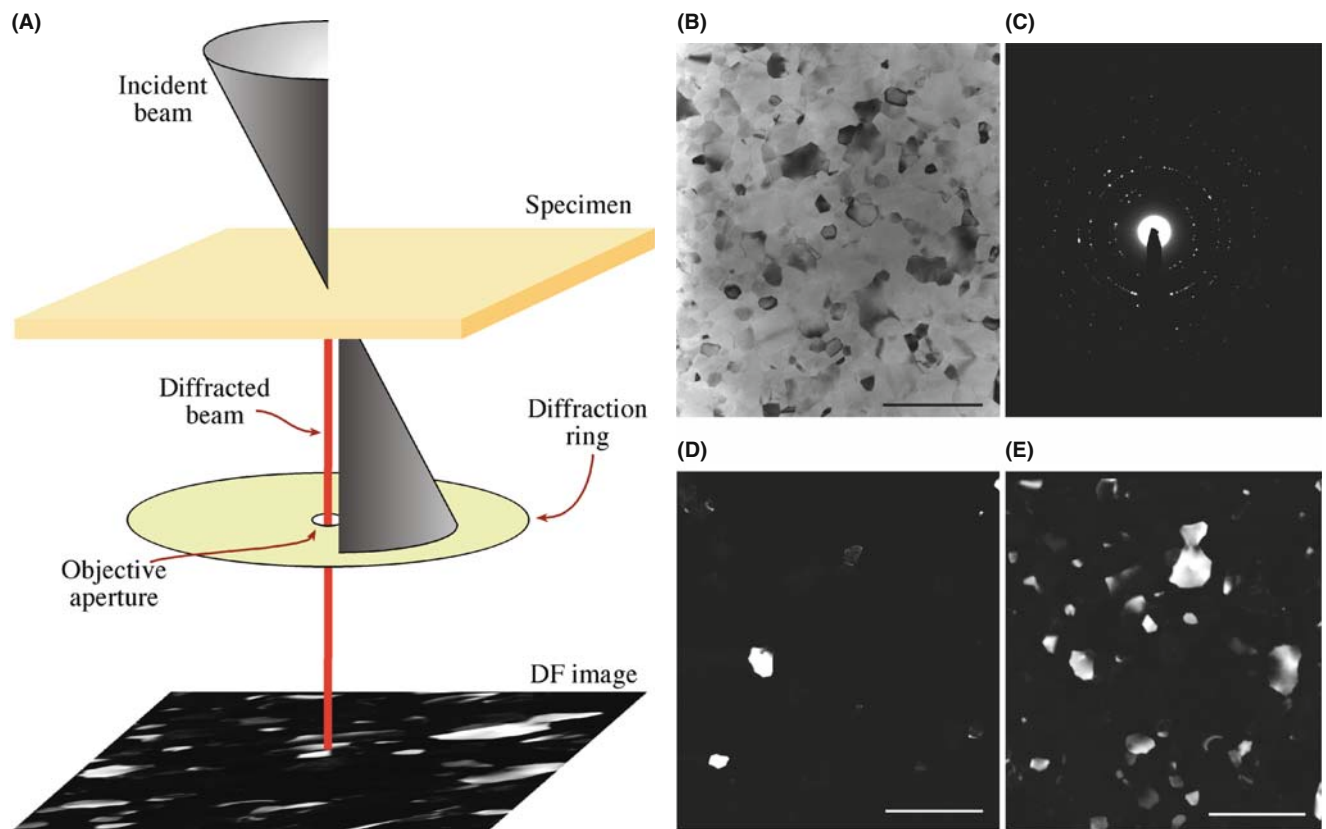


FIGURE 9.15. (A) Ray diagram showing hollow-cone illumination conditions. The direct beam is always off axis but electrons diffracted at the cone angle are always scattered on axis. (B) A BF image of a nanocrystalline Al film. (C) A hollow-cone SAD pattern from the film. Thus, while a single (220) CDF image (D) reveals only a couple of strongly diffracting crystals, a hollow-cone DF image (E) from all the $\{220\}$ reflections shows diffracted intensity from dozens of grains. The scale bar is 500 nm.

Computer control of the scan coils allows you to select the angle, i.e., the radius of the circle, thus selecting which (hkl) ring is to be on the optic axis. You can also control the speed of the rotation so you can synchronize a single rotation with sufficient exposure time to record the DF image. This process is analogous to the normal DF tilt controls that shift the DP in the x - y plane (by tilting the incident beam as in Figure 9.14C) being replaced with r - θ axis controls. If you observe the rotating DP with the objective aperture removed, the direct (000) beam describes a circle, radius r , around the optic axis. Any point in the DP that is distance r from the (000) spot will at some time pass through the optic axis during the rotation of the DP. An example of a hollow-cone DP (not spinning) is shown in Figure 9.15C. The SAD aperture was about the size of the image ($\sim 50 \mu\text{m}$ across) and the objective aperture was small enough to permit diffracted intensity from only one ring to be collected. Figure 9.15B, D and E shows, respectively, a BF image of a polycrystalline metal film from which the DP in Figure 9.15C was obtained, a conventional CDF from a few spots in the DP ring and the hollow-cone DF, revealing many more diffracting grains. You usually have to take a series of time exposures to get a good hollow-cone image. Figure 9.15E was a 20-second exposure during which time the DP rotated about 1000 times through the objective aperture!

Hollow-cone DPs are also discussed in Section 18.6. A closely related technique, precession diffraction, is covered in Section 18.8. The STEM equivalent of hollow-cone DF imaging is annular-DF imaging, which we'll talk about in the next section.

9.4 FORMING DPs AND IMAGES: THE STEM IMAGING SYSTEM

If you want to use a fine probe to form STEM images then the objective-lens optics are a little more complex than in TEM. The key feature to remember is that the scanning beam must not change direction as the beam is scanned (unlike in an SEM where the scanning beam simply pivots about a point above the specimen). If the incident direction varies then the electron scattering (particularly the diffraction) processes would change as the beam intercepts the specimen at different angles. So interpreting the image contrast would be rather difficult to say the least.

STEM

The beam must scan parallel to the optic axis at all times so that it mimics the parallel beam in a TEM even though it's scanning.

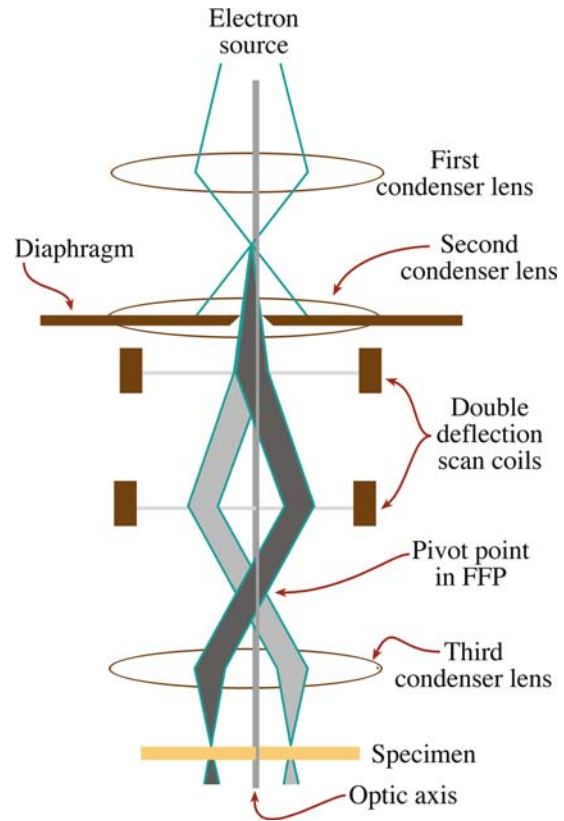


FIGURE 9.16. Scanning the convergent probe for STEM image formation using two pairs of scan coils between the C2 lens (usually switched off) and the upper-objective polepiece. The double-deflection process ensures that the probe remains parallel to the optic axis as it scans across the specimen surface.

As we show in Figure 9.16, the way we achieve parallel incidence is to use two pairs of scan coils to pivot the beam about the FFP of the upper objective (C3) polepiece. The C3 lens then ensures that all electrons emerging from the pivot point are brought parallel to the optic axis and an image of the C1 lens crossover is formed in the specimen plane. Now, if the objective lens is symmetrical, and the lower objective polepiece is similarly strong, then a stationary DP is formed in the BFP (this pattern does not move, even though the beam is scanning, since it is conjugate with the FFP, as shown in Figure 9.17). If we stop the beam from scanning, then we have a CBED pattern in the BFP and we can project that onto the TEM computer screen if we wish. So let's first discuss how to form STEM images.

THE STEM IMAGING LENS?

The STEM image quality depends on the probe. The probe has aberrations because we use a lens to form it. So the STEM image quality does depend on a lens (just not on an imaging lens).

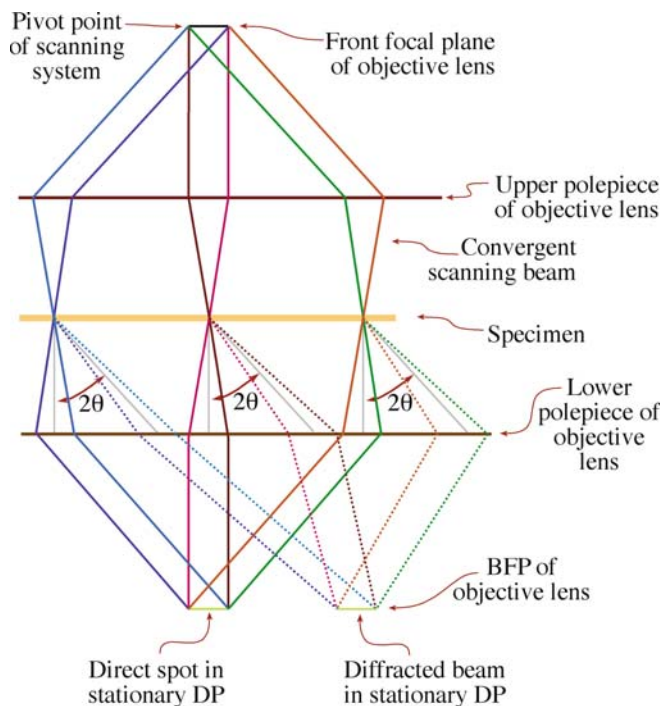


FIGURE 9.17. The creation of a stationary (convergent-beam) DP in the BFP of the objective lens is a necessary prerequisite for STEM imaging. Note that electrons scattered through the same angle (2θ) at different points in the specimen are focused at the same point in the BFP.

One potentially very big advantage for STEM is that, just like in an SEM, we don't use lenses to form the image. So defects in the *imaging lenses* do not affect our image resolution, which is limited by the beam dimensions. Hence chromatic aberration which (as we saw back in Section 6.5.B) can seriously limit TEM image resolution, is absent in STEM images. This is a great advantage if you're dealing with a thick specimen. However, there are drawbacks also, as we'll discuss below and in Part 3, and STEM images aren't widely used, particularly for crystalline specimens.

9.4.A Bright-Field STEM Images

Image formation in the scanning mode is fundamentally different from static-beam TEM image formation. As you've just seen in the TEM, we select a portion of the electrons emerging from an area of the specimen and project that distribution onto a screen. The principle of scanning-image formation is shown in Figure 9.18. Simply stated, we scan the beam on the specimen by adjusting the scan coils; these same coils are used to scan the computer display synchronously. The electron detector acts as the interface between the electrons coming from the specimen and the image viewed on the display screen. Since it takes up to 2048 scan lines to build up an image on the recording screen, the whole process of creating a STEM image is much slower than TEM imaging: it's serial recording instead of parallel recording.

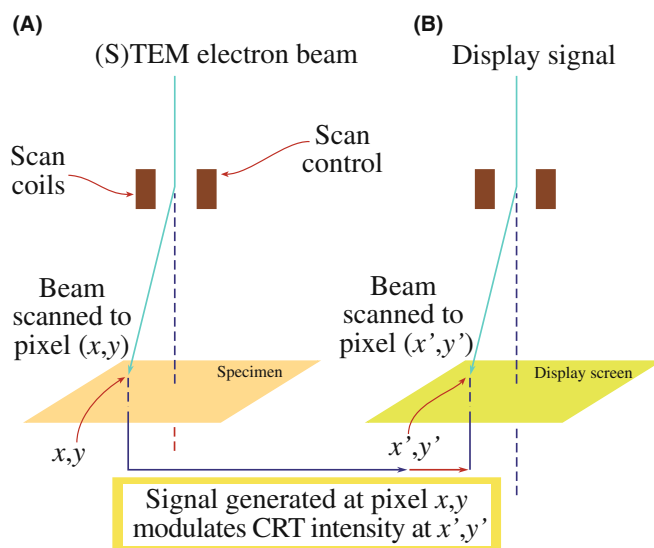


FIGURE 9.18. The principle of forming a scanning image, showing how the same scan coils in the microscope control (A) the beam-scan on the specimen and (B) the beam-scan on the computer display screen of the STEM. Thus no lenses are required to form the image.

THE STEM SIGNAL

The signal is generated at a point on the specimen, detected, amplified, and a corresponding signal displayed at an equivalent point on the computer display. The image builds up over several seconds or even minutes.

This process is exactly the same principle as used in any scanning-beam instrument such as an SEM or an STM (scanning-tunneling microscope). Remember that to form a TEM-BF image, we inserted an aperture into the plane of the TEM DP and only allowed the direct electrons through it into the imaging system. In STEM mode we use an electron detector, in exactly the same way as we use the aperture: we only allow the electrons that we want to contribute to the image to hit the detector. So we insert a BF (either a semiconductor or scintillator-PM) detector onto the axis of the microscope and it intercepts the direct-beam electrons no matter where the beam is scanning on the specimen, as shown in Figure 9.19A. So a variable, direct-beam signal travels from the detector via an amplification system to modulate the signal on the computer display, thus building up a BF image as also shown in Figure 9.19D.

BF DETECTOR

The BF detector picks up the direct beam which varies in intensity depending on the specific point on the specimen illuminated by the probe at that specific time.

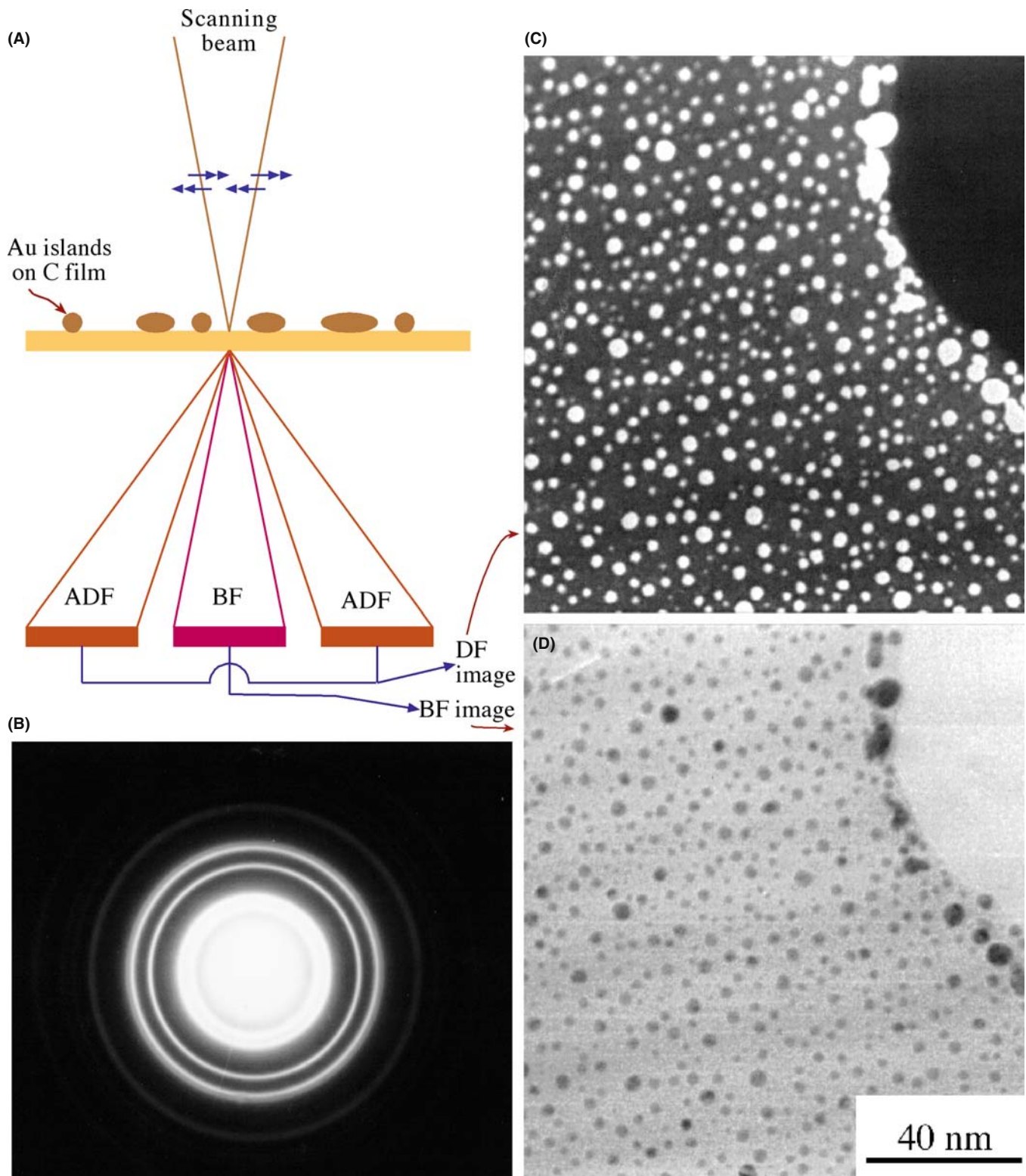


FIGURE 9.19. STEM image formation: A BF detector is placed in a conjugate plane to the BFP to intercept the direct beam (A) and a concentric annular DF detector intercepts the diffracted electrons whose distribution is shown in the SAD pattern in (B). The signals from either detector are amplified and modulate the STEM computer display. The specimen (Au islands on a C film) gives complementary ADF (C) and BF (D) images.

Now in a TEM we can't physically put the detector in the BFP of the objective lens to form a STEM image, because it would interfere with the objective aperture. Therefore, we usually insert the detector into a conjugate plane to the stationary DP (Figure 9.19B). So when you form a STEM image in a TEM, you operate the TEM in diffraction mode and insert a detector into the viewing chamber of the TEM, either above or below (in which case you raise) the screen. The stationary DP falls on the detector and the signal goes to the display. In a DSTEM, there may not be any imaging-system (or post-specimen) lenses, in which case the detector is indeed positioned immediately after the objective lens. Much of what we've just said is automatically done when you 'hit the STEM button'. The message is the same: understand what is happening in your microscope and why.

9.4.B Dark-Field STEM Images

The approach is analogous to that of TEM. We form a DF image by selecting any or all of the scattered electrons, rather than the direct-beam electrons. Remember, in a TEM we tilt the incident beam so the scattered electrons we want to form the image travel down the optic axis and are selected by the objective aperture. In a STEM, we do things rather differently.

DF STEM

If we want a specific beam of scattered electrons to fall on the BF detector, we can simply shift the stationary DP so that the scattered beam is on the optic axis and hits the BF detector.

It's simple to do this with the DP centering controls or you could also displace the C2 aperture. The former is to be preferred since doing the latter misaligns the illumination system.

9.4.C Annular Dark-Field Images

Rather than using the BF detector for DF imaging, we usually use an annular detector, which surrounds the BF detector, and then all the scattered electrons fall onto that detector. We call this process annular dark-field (ADF) imaging and it has certain advantages, depending on the contrast mechanism operating in the specimen, as we'll see in Chapter 22. As we show in Figure 9.19A, the ADF detector is centered on the optic axis and has a hole in the middle, within which the BF detector sits. The resultant ADF image in this simple example (Figure 9.19C) is complementary to the BF image (Figure 9.19D). As you'll see in Chapter 22, we can also use another annular detector that sits around

the ADF and picks up the electrons scattered out to even higher angles forming so-called high-angle (HA) ADF (or Z-contrast) images in which Rutherford-scattering effects are maximized and diffraction-contrast effects are smoothed out.

We can take this idea further and make detectors of any size or shape we wish. For example, we could design a detector in which the annulus is split into two halves or four quadrants and electrically isolate each part of the detector. Then we can form different images from electrons that fall on different parts of the detector. It's impossible to do this in a TEM, because the objective aperture that does the selecting is a hole and can't be cut up like a semiconductor detector. We'll talk more about these kinds of detectors when we discuss specific contrast mechanisms in TEM and STEM images in Chapter 22.

9.4.D Magnification in STEM

All the STEM images that we have just described appear on the computer screen at a magnification that is controlled by the scan dimensions on the specimen, *not* the lenses of the TEM. This is a fundamental difference between scanning and static image formation.

STEM IMAGES

STEM images are *NOT* magnified by lenses.

Because scanning images are not magnified by lenses, they are not affected by aberrations in the imaging lenses. They are, however, affected by aberrations of the probe itself and, therefore, can be improved by (illumination-system) aberration correctors.

If the scanned area on the specimen is 10 mm × 10 mm, and the resultant image is displayed on a computer screen with an area 100 mm by 100 mm, then the image magnification is 10×. If the scan dimension is reduced to 1 mm, the magnification on that same screen is 100×, and so on, up to magnifications in excess of 10⁷×, which are common in dedicated STEMs. As with the TEM, we have to calibrate the STEM magnification and the camera length of the DP we use to create the images.

9.5 ALIGNMENT AND STIGMATION

9.5.A Lens Rotation Centers

You only need to perform two alignments to ensure that the imaging system is operating correctly. By far the most important is the alignment of the objective lens center of rotation and the second is the alignment of the DP on the optic axis. To get the best out of your TEM, you *must* master these two fundamental alignments.

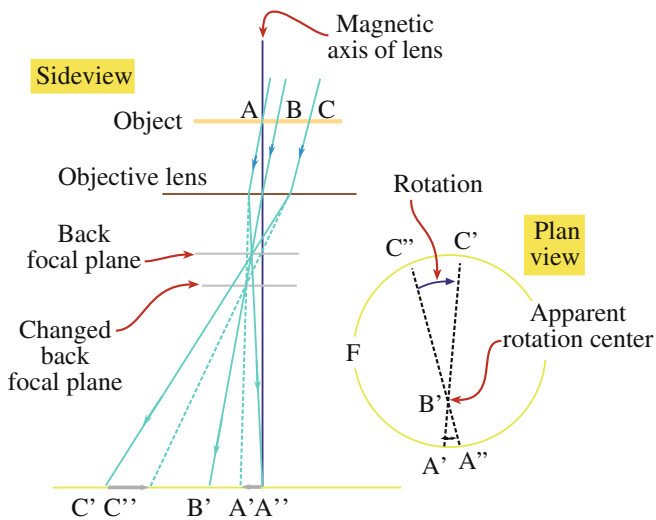


FIGURE 9.20. When the objective-lens center of rotation is misaligned, the image appears to rotate about a point away from the center of the viewing screen when the lens is wobbled about focus. When the rotation center is correctly aligned, the image will rotate about the center of the screen.

Basically, the idea of the objective-lens rotation alignment is to ensure that the objective lens field is centered around the optic axis, so that the direct-beam electrons emerging from the specimen see a symmetric field as they pass through the lens. If the field is off-center, then the electrons will move off axis, suffer more aberrations, and your image will rotate about a position off-axis as you change the objective lens (focus), as shown schematically in Figure 9.20.

Operational Procedure #7

- To center the objective rotation, start at a relatively low magnification (say 10,000 \times), select an obvious reference point in the image and move it to the middle of the screen, and observe the way the point rotates as you wobble the objective lens from overfocus to underfocus and back again. If the point rotates without moving off center, the lens is aligned, but check the accuracy of the alignment by going to much higher magnifications ($>10^5\times$).
- If the point moves off center at 10,000 \times then use the beam tilts to move the point in the image that is the center of rotation to the middle of the screen while continuously wobbling the objective. Repeat the process at higher magnifications.
- Above $\sim 10^5\times$ the wobbler may introduce too large a rotation, so you may have to defocus the objective lens manually. The actual steps to do this are instrument-dependent, so consult the manufacturer's handbook. This process is also called 'current centering.'

- When the image wobbles (rotates) about the center of the screen at magnifications $>10^5\times$, the objective lens rotation center is well aligned. The higher the magnification at which you can achieve this, the better the alignment and the better quality all your pictures will be.

This operation may be computer controlled in modern TEMs. In some instruments you can also perform 'voltage centering' in which a varying voltage is applied to the gun and the objective lens is aligned to ensure that the electrons remain on axis through the lens as their energy varies. Not all instruments are capable of this alignment.

If you change the DP magnification (i.e., the camera length, L) the whole pattern will move off axis if the diffraction center is misaligned. To align the center you have to adjust the projector lens until the central spot in the DP is on axis and it rotates around the axis as L is changed. Check the manufacturer's handbook on exactly how to do this alignment.

Centering the DP is useful for STEM-image formation, since you have to center the DP such that the direct beam hits the BF detector and the scattered beams hit the ADF detector. Apart from this simple operation, the STEM imaging system needs no lens alignment.

9.5.B Correction of Astigmatism in the Imaging Lenses

After you've centered the image and DP, the main cause of problems in the imaging system is astigmatism in the objective and intermediate lenses.

ASTIGMATISM

Objective-lens astigmatism occurs if the objective aperture is misaligned, so you must always carefully center the aperture on the optic axis, symmetrically around the electron beam used to form the BF or DF image.

Despite careful centering of the objective aperture, residual contamination may also cause astigmatism and then you have to use the objective stigmators to introduce a compensatory field. You'll find that the effects of objective astigmatism are harder to see than condenser astigmatism, which is easily visible on the screen as we just described. Correct objective astigmatism as follows:

Operational Procedure #8 Either look for a small hole in your specimen or look at a rough corner where the specimen edge curves through $> 90^\circ$. Ideally you might use a holey carbon film to correct residual astigmatism, *before* you insert your specimen, especially while learning this procedure, which we illustrate in Figure 9.21.

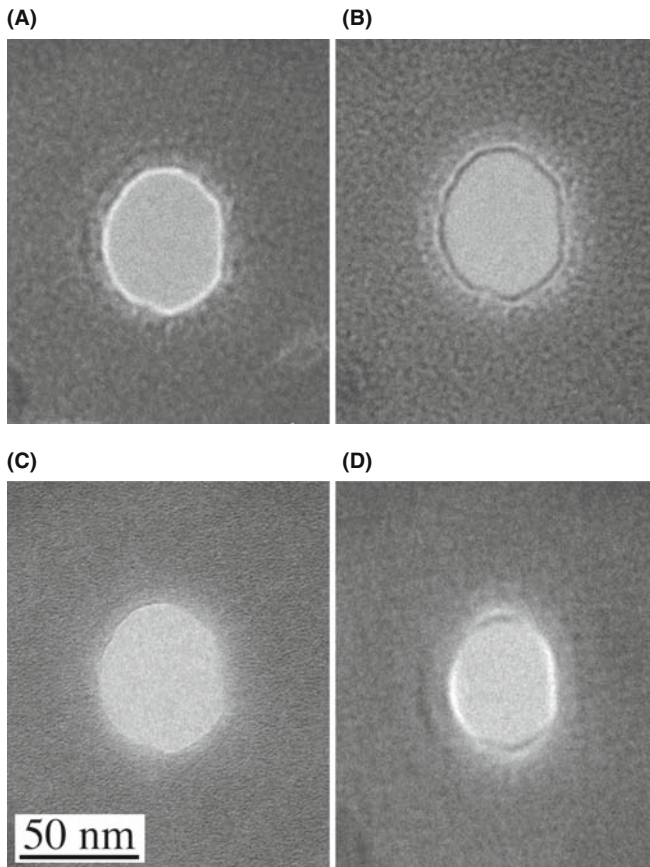


FIGURE 9.21. The image of a hole in an amorphous carbon film illuminated with a parallel beam showing that (A) with the beam underfocused, a bright Fresnel fringe is visible; (B) with the beam overfocused, a dark fringe is visible; (C) at exact focus there is no fringe; and (D) residual astigmatism distorts the fringe. Correcting the astigmatism means changing any image similar to (D) to one similar to (A) or (B).

- Often you can only see objective astigmatism at the highest magnifications so form a BF image of the hole or the specimen edge at high magnification ($>10^5\times$). First, adjust the illumination system to ensure a reasonably parallel beam. (Remember: how to do this depends on whether you have a *c/o* lens or not.)
- Now defocus the objective lens (either over- or underfocus; it doesn't matter). A Fresnel fringe (which is a phase-contrast effect, as we'll see in Chapter 27) should be visible at the thin edge of your specimen.
- Alternately underfocus and overfocus the objective lens (i.e., use the wobbler again).
- If there is astigmatism, you should see streaking in the image and this streaking effect will rotate through 90° as you wobble either side of exact focus. The streaking is most easily seen by watching the Fresnel fringe at the edge, as we describe below for Figure 9.21.
- Adjust the objective stigmators to compensate for the streaking at overfocus and then again at

underfocus (until you're very skilled, you'll probably have to switch off the wobbler to do this and manually defocus the objective).

- Repeat these steps until there is no obvious image streaking as you defocus and the image merely blurs when going out of focus.
- Go to a higher magnification and repeat the procedure. As with the rotation center, the higher the magnification at which you can correct the astigmatism, the better and $>250,000\times$ should be possible. But remember that at higher magnifications the image intensity will be reduced. Don't try and compensate for this reduced intensity by condensing the beam because doing so will destroy the parallelism, thus reducing the Fresnel-fringe (phase) contrast.

As shown in Figure 9.21A, when you underfocus the objective lens, there is a bright Fresnel fringe round the edge of the hole. If this fringe is uniform around the hole, then there is no astigmatism. If the fringe varies in intensity, as in Figure 9.21D, then the focus of the lens is changing around the hole because of astigmatism. Then you have to adjust the objective stigmators to make the fringe uniform. The same operation must be repeated at overfocus, when there is a dark fringe around the edge of the hole (Figure 9.21B). At exact focus, you should see no fringe and the image contrast is minimized (Figure 9.21C).

FRESNEL FRINGES

Underfocus = bright fringe
Overfocus = dark fringe

This method of correcting the astigmatism is reasonable at magnifications up to several hundred thousand times.

In practice, if you're working at such high magnifications, you'll probably have to check the astigmatism throughout your TEM session, so you should get used to looking at the Fresnel fringes on a thin, curved edge of your specimen rather than the ideal holey carbon film.

For high-resolution imaging at magnifications of $>300,000\times$, we have to use the streaking in the image to correct for astigmatism. We'll talk about this when we discuss HRTEM in Chapter 28.

Intermediate-lens astigmatism is of secondary importance and only affects the DP. Because the DP is at zero magnification in the objective lens, the intermediate lenses are responsible for magnifying it. So if there is residual astigmatism in these lenses, then the DP will show orthogonal distortions as you take it through focus. This effect is small and can only be seen in the binoculars as you focus the DP with the diffraction

focus (intermediate lens) control. Make sure that the incident beam is strongly underfocused to give the sharpest spots. As with objective astigmatism in the image, simply adjust the intermediate stigmators to compensate for any spot distortion at underfocus, and over-focus until the spots expand and contract uniformly in all directions through focus. You should be aware that not all instruments have the requisite intermediate stigmators to carry out this correction.

9.6 CALIBRATING THE IMAGING SYSTEM

Your TEM should be calibrated when it is first installed and then periodically throughout its life, especially if you wish to carry out accurate measurements from images or DPs. If the instrument is modified substantially (e.g., a burned-out lens coil is replaced), then it must be recalibrated. In all cases you must specify a set of standard conditions under which the calibrations are carried out (e.g., objective lens current and other lens settings, eucentric height, etc.). You can find a full description of all the detailed concerns with all the TEM calibrations in Edington (1976). Remember, if you're serious about TEM don't rely on someone else's calibrations.

THE LAST USER

Since you usually will not be the first user, you should take the time to check the existing calibration. Don't assume it is correct.

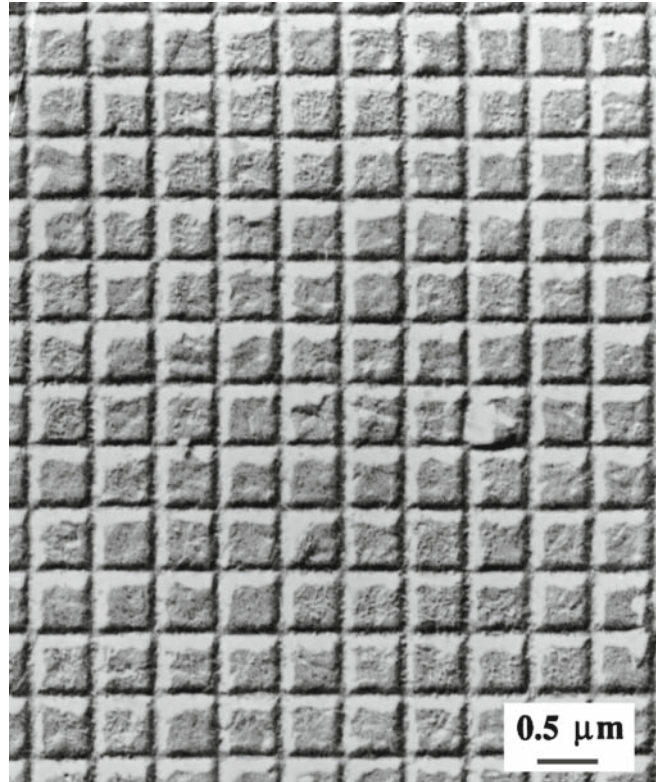
9.6.A Magnification Calibration

We use standard specimens to calibrate the magnification. The most common specimen we use is a thin carbon-film replica of an optical-diffraction grating of known spacing, as shown in Figure 9.22A. The typical linear density of lines in the replica is 2160/mm (giving a line spacing of $0.463\ \mu\text{m}$ with an error that will depend on how many grating spacings you can measure). Grating replicas enable calibration up to magnifications of $\sim 200,000\times$. Above this magnification, individual grating spacings are wider than the recording film. So we can then use small latex spheres (50–100 nm diameter) although they are susceptible to beam damage and shrinkage under electron bombardment. At the highest magnifications, phase-contrast images of known crystal spacings, such as the 0002 spacing in the graphite structure (0.344 nm) can be used or the 111 fringes of Si. What we are doing is just using a known periodicity in the crystal; careful consideration of the objective-lens defocus and specimen thickness is required before the phase-contrast lattice image can be directly interpreted, as we discuss in detail in Chapters 27 and 28.

MAGNIFICATION

Magnification calibration is so sensitive to so many variables that some users deposit a standard material on the material they are studying so that the calibration will be done under exactly the same conditions and will appear in the same image as your area of interest.

(A)



(B)

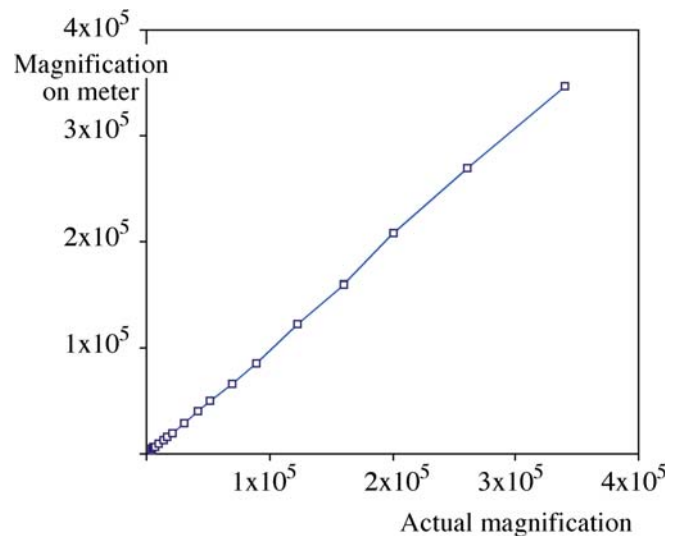


FIGURE 9.22. (A) An image of a diffraction-grating replica for which the actual spacing of the grating is known. (B) The TEM magnification can thus be calibrated, permitting specific magnification settings (usually with approximate/calculated magnifications) to be assigned more accurate, experimental magnifications. If the plot for your TEM is not linear then there are serious problems either with your measurements or with the microscope.

Operational Procedure #9

- Insert the calibration specimen and ensure the holder is at the eucentric height.
- Ensure the illumination is parallel and the objective aperture is well centered on the 000 spot in the SADP.
- Switch back to image mode and then focus the BF image (see below for the best way to be consistent in focusing).
- Record images of the diffraction grating at all magnification settings, taking care to re-check the focus after each change of magnification.
- Calculate the magnifications experimentally from the images knowing the true spacing.

Figure 9.22B shows the classical magnification calibration for a Philips CM30 TEM. Today, you'll use a crystal lattice at $\sim 300 \times$, relate it to the same area at say $50 \times$, repeat at $10 \times$, and then at all magnifications in between.

HYSTERESIS

Electromagnetic lenses have hysteresis. Everything you do must be reproducible if you want your results to be.

You have to calibrate the magnification because the TEM imaging system does not give stable and reproducible lens strengths. The lens strengths will change with ambient temperature (e.g., how many people are in the room), with the efficiency of the cooling system of the lenses and with lens hysteresis. Therefore, if you want to make accurate measurements from your TEM images, you must carry out the magnification calibration *at the time you make the measurements*. In particular, you have to minimize the lens hysteresis by always approaching image focus consistently from overfocus or underfocus and/or reversing the lens polarity several times before finally coming to focus. Also, you must remember that there may be barrel or pincushion distortions in the image particularly at very low magnification ($< 5000 \times$) where the lens is not designed to work well. (As students you ought to be able to work out what the first distortion does to your image, but you might not be familiar with the latter distortion at this early stage of your life!)

THE MAGNIFICATION

The image size will change during recording, printing and publishing. *Never* state an image magnification in the caption. Indicate a distance on the recorded image that corresponds to a distance on the actual specimen at the time the image was recorded.

Remember that the area of the specimen you are working on must be at exactly the right 'height' in the column. Because of the magnification error, the TEM is not the best instrument for absolute measurement of particle sizes, etc. However, relative measurement is easily done with reasonable accuracy ($\pm 5\%$), so long as you note the precautions we've just described. Without a calibration, the digital readout is probably no better than $\pm 10\%$ accurate, and so it is unwise to state magnifications to better than $\pm 10\%$. You should be suspicious of any micrographs that you see in the literature with a magnification that is more precise than three (or even two) significant figures such as $52,550 \times$. It may indicate that the microscopist does not understand the limitations of their instrument and the work should be interpreted with due caution.

We used to call these lines 'micron bars' for obvious reasons but TEM magnifications have become larger and 'nano bars' would often be more appropriate (even though neither one is related to barrel distortion). All modern TEMs automatically put such a marker on the negative. However, its accuracy is only as good as your operational skills.

You can use an identical procedure to calibrate the STEM-display magnification despite the fact that the digital STEM-image magnification is, in principle, easily calculated from the scan-coil strengths. The image magnification differs from the digital readout because of variations in the objective lens. Table 9.1 shows the difference between a typical digital STEM magnification and the experimentally determined magnification using a diffraction-grating replica.

9.6.B Camera-Length Calibration

As mentioned already, we describe the magnification of the DP by the camera length (L), a term that arises from X-ray projection-diffraction cameras which operate without lenses (because focusing X-rays is very difficult). In such cameras, magnification is increased by moving the recording film further away from the specimen, in exactly the same way that you magnify your computer-slide presentations onto a screen by moving the projector further away. (You can always tell non-microscopists who try to *focus* their slides in the same way, or those

TABLE 9.1. Magnification Calibration for a Philips EM400T in STEM Mode at 120 kV

Digital readout	Calculated magnification
3,200	3,420
6,400	6,850
12,500	12,960
25,000	27,000
50,000	54,000
100,000	108,000

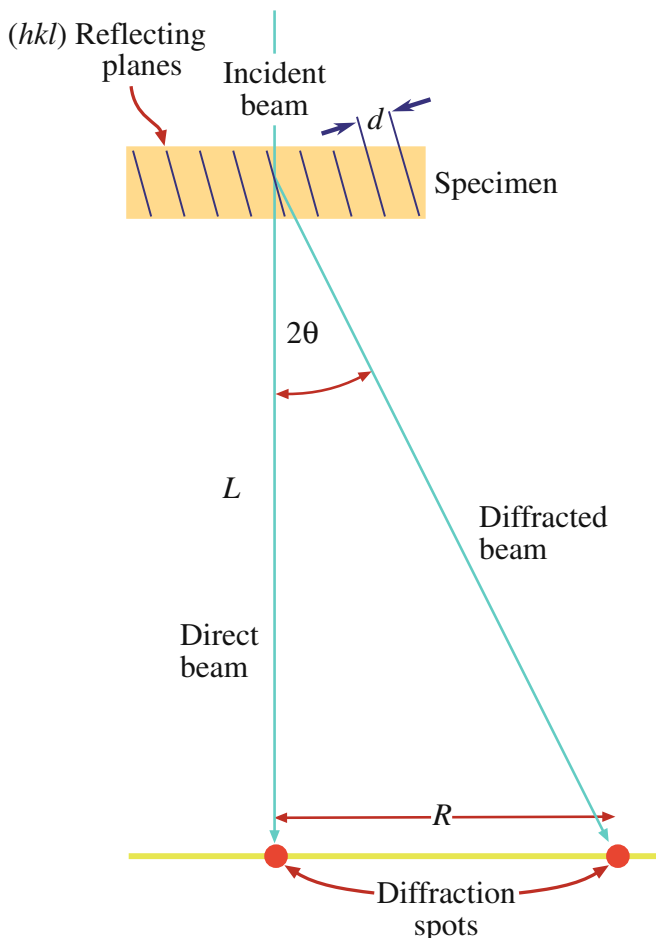


FIGURE 9.23. The spacing R between the direct beam and a scattering maximum (such as a diffracted beam or the radius of a ring of diffracted intensity) is related to the camera length, L . Increased magnification corresponds to effectively increasing L , although in practice, this is accomplished with lenses.

who try to change the magnification by adjusting the projector lens focus!)

The camera-length concept can be applied in the TEM as shown in Figure 9.23. This figure represents the imaging system, but without the lenses drawn in.

CAMERA LENGTH

If we increase the magnification of the lenses between the specimen and the viewing screen, we increase the effective distance L between the specimen and the screen.

The camera length is a calculated value rather than a physical distance. If electrons are scattered through an angle 2θ at the specimen (as in a typical diffraction event described in Chapters 2 and 11), then the distance between the direct and diffracted beams as measured on the screen (R) is determined by L , since

$$\frac{R}{L} = \tan 2\theta \sim \theta \quad (9.1)$$

From the Bragg equation (equation 3.21) we know that $\lambda/d = 2 \sin\theta \sim 2\theta$ and so we can write

$$Rd = \lambda L \quad (9.2)$$

Operational Procedure #10 To calibrate the magnification of the DP we need to record DPs from a specimen with a known crystal spacing (d) such as a thin film of polycrystalline Au or Al which gives a ring pattern (see Figure 2.11).

- Make sure your specimen is at the eucentric height and the BF image is in focus.
- Insert the SAD aperture and switch to diffraction mode.
- Ensure the beam is parallel so that the DP spots or rings can be focused to the smallest size. If the pattern is not focused then focus with the first intermediate lens (diffraction focus) control. Be careful if you have a c/o lens because changing the illumination conditions (e.g., trying to make the beam more parallel) will change the DP focus. If you then sharpen the DP by changing the diffraction focus, the camera length will change. If in doubt, consult the manufacturer's handbook.
- Since we know the lattice parameter of the metal, we can measure the ring radius R on the photographic film or computer display for any plane that is diffracting (see Chapter 18 to find out exactly how we do this).
- Since we know λ we can easily determine L from equation 9.2.

A typical TEM camera-length calibration is shown in Table 9.2. The STEM camera-length calibration may be different to the TEM if the objective lens setting is not exactly the same in TEM and STEM modes, and this depends on the vintage and make of your instrument. So you should check with the manufacturer before taking the time to perform the calibration.

TABLE 9.2. Comparison of Experimentally Measured Camera Length (and Camera Constant) with the Digital Readout for a Philips EM400T Operating at 120 kV ($\lambda = 0.0335 \text{ \AA}$)

Camera length setting	Digital readout (mm)	Measured camera length, L (mm)	Camera constant λL (mm \AA)
1	150	270	9.04
2	210	283	9.47
3	290	365	12.22
4	400	482	16.14
5	575	546	18.28
6	800	779	26.08
7	1150	1084	36.29
8	1600	1530	51.22
9	2300	2180	72.99
10	3200	3411	114.20

It's worth explaining in more detail the effect of a c/o lens on the SAD calibration. In a c/o lens, as shown back in Figure 9.5, the beam can go from divergent to convergent conditions at the specimen plane. However, unless the beam is parallel, your DP focus will change with illumination angle. In this case, the diffraction-lens setting at the point of focus of your DP will differ for different illumination conditions. So L will change (by as much as 15%) depending on the angle of convergence/divergence. In order to ensure the same L , you must pick one condition and use it for every calibrated DP. There are several ways to do this.

- Set the diffraction lens to the same excitation for each pattern and focus the DP with the C2 lens.
- Focus the pattern at the objective aperture (especially if the BFP of the objective and the objective aperture share the same position).
- Go to the maximum value of the C2 lens, focus the DP, and use the C1 lens to adjust the brightness of the DP.

There are other possibilities which you'll be able to work out as you get more skilled but, above all, be consistent and always *use the conditions you chose when you did the calibration*.

9.6.C Rotation of the Image Relative to the DP

Anyone studying crystalline materials must determine the angle between directions in the image and directions in the DP. At a fixed camera length, the DP always appears on the screen in a fixed orientation. But if you record images at different magnifications, the images will rotate by an angle ϕ with respect to the fixed DP. (In some TEMs this rotation has been removed by the addition of a compensating projector lens and, in this case, there is always a fixed rotation (ideally 0°) between directions common to both image and DP.)

THE GOLDEN RULE

Always do the calibrations yourself. Do not rely on factory calibrations. The conditions you use in your laboratory may differ from those chosen by the manufacturer.

Operational Procedure #11 To determine this rotation, we often use a specimen of α -MoO₃, because it forms thin, asymmetric crystals with a long edge known to be parallel to the 001 direction in the crystal.

- Take care to ensure that, as usual, the image is focused with the specimen at the eucentric plane.
- Insert the SAD aperture and ensure that it is focused (using the intermediate lenses) to coincide with the image plane.

- Switch to diffraction mode with the beam parallel and adjust the diffraction focus to give sharp diffraction maxima.
- Take a double exposure of the DP and the image as shown in Figure 9.24A.
- Repeat the whole exercise for different magnifications and plot out the variation of the angle ϕ as shown in Figure 9.24B.
- You can do the same, if necessary, for different values of L , which introduce a systematic change in ϕ . For this reason, we recommend that you carry out all your SAD work at a standard value of L ; 500–1000 mm is usually optimum.

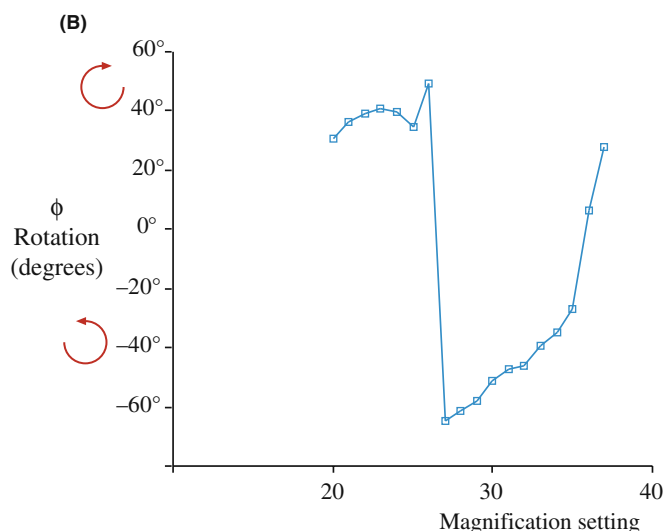
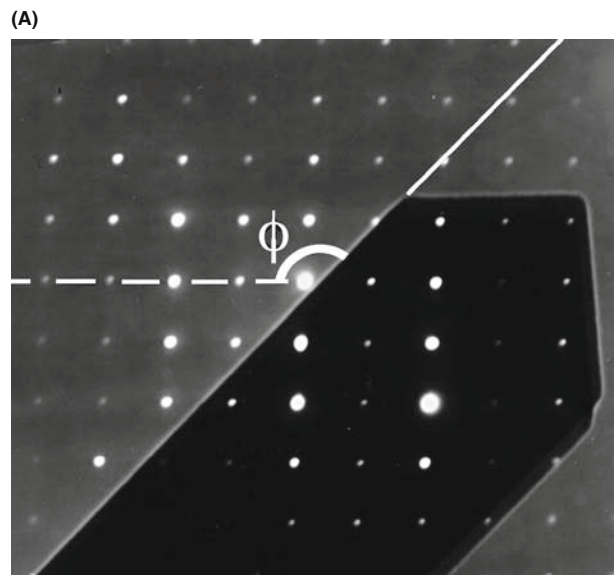


FIGURE 9.24. (A) A double exposure showing the superposition of an image of an α -MoO₃ crystal on a DP from the same crystal, defining the rotation angle ϕ . (B) The rotation calibration gives the angle ϕ between equivalent directions in the image and the DP as the magnification is varied. The calibration assumes a constant camera length. The rotation angle will change significantly if the imaging system switches lenses on or off as the magnification is changed (e.g., between settings 26 and 27).

A further complicating factor is that, as the image magnification is increased, the TEM lens control logic may switch off, or switch on, one of the imaging-system lenses. When this happens, a 180° inversion is introduced into the image. You can see this happen if you watch the image carefully as you change the magnification. This inversion has to be included in the rotation calibration, otherwise a 180° error will be made in the assignment of directions in the image. One way to see if the image has a 180° inversion is to look at the DP and defocus it slightly so the BF image in the direct beam can be seen directly at very low magnification. The 180° inversion is immediately obvious, as shown in Figure 9.25. As we already noted, some manufacturers adjust the imaging-system lenses to compensate for the rotation, thus keeping a constant image-DP rotation angle at all magnifications. Likewise, in STEM mode, since the imaging lenses are not used for magnification, there is a fixed angle between the images and DPs.

McCaffrey and Baribeau used molecular-beam epitaxy to create a cross section TEM specimen made from a single-crystal Si/Ge wafer specimen that can perform these three major calibrations (magnification, camera-length, and image/DP rotation). Similar specimens are

available from suppliers of TEM consumables (see URLs 1–3, for example). These suppliers also offer a range of standard specimens for other TEM needs (e.g., XEDS and EELS analysis). If you are serious about your microscopy, buy your own standards, keep them safe and clean, and use them often.

9.6.D Spatial Relationship Between Images and DPs

If you don't use a double exposure when comparing images and DPs (or indeed when comparing directions in any two films) you need a fixed reference line. This line must be independent of slight variations that may arise depending on the film size, how you loaded it, etc. The best reference line is the edge of the plate-numbering system that is superimposed on each film.

Yesterday's advice:

Whenever you're comparing images and DPs, it is essential to compare the photographic negatives with the *emulsion side up*. This is contrary to usual photographic practice, but it's necessary to preserve the relationship between manipulations of your specimen and what you see happening on the screen. *If you don't do this*, it is easy to introduce a 180° error into the relationships between images and DPs.

Today's advice:

Don't use film. If you use a CCD, you'll never think of inverting the image!

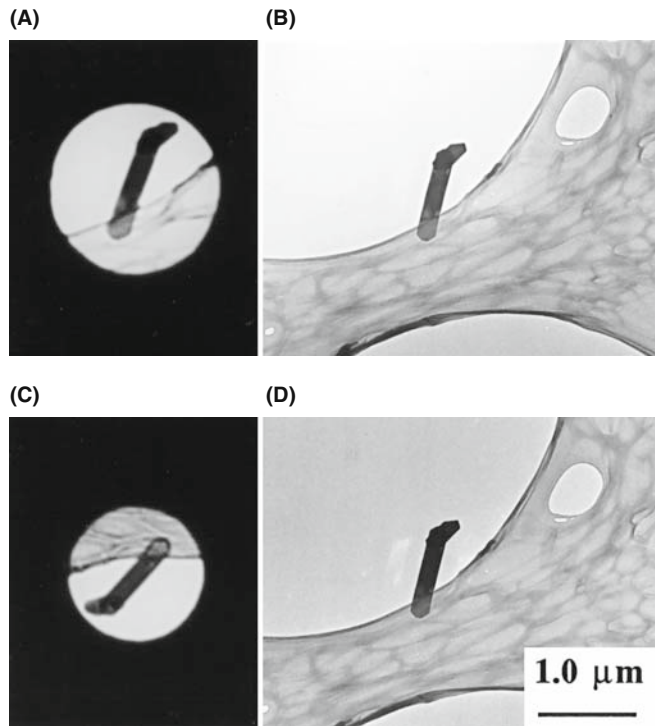


FIGURE 9.25. Defocused direct beam in a DP from α -MoO₃ compared with a BF image, showing how to determine if a 180° inversion exists or not. If the image of the specimen inside the expanded image of the beam is rotated with respect to the image on the screen, as in (C) and (D), then a 180° inversion is required to determine the correct angle between directions in the DP and directions in the image. In (A) and (B), no rotation occurs between the DP and BF image.

9.7 OTHER CALIBRATIONS

The accelerating voltage: The selected voltage may differ from the absolute voltage by detectable amounts. There are several ways to determine the actual voltage: First, you can measure the electron wavelength, λ , by measuring the angle, ϕ , between two Kikuchi line pairs (see Chapter 19) that intersect a distance R from the direct beam

$$\tan \phi = \frac{R}{L} = \frac{\lambda}{d} \quad (9.3)$$

Alternatively, you can match simulations of HOLZ lines to experimental lines in CBED patterns (see Chapter 21) and determine which λ gives the best match. Finally, in principle, you could get your XEDS computer system to display the X-ray spectrum (Chapter 34) out to E_0 , the beam energy, and the bremsstrahlung intensity vanishes to zero at the exact beam energy (this is called the Duane-Hunt limit). However, XEDS computer displays rarely show energies > 30–40 keV.

The specimen tilt axis and the sense of tilt: In a side-entry stage, the principal tilt axis is parallel to the specimen-holder rod. Since the image is usually rotated on the screen relative to the specimen, how can you locate this tilt-axis direction? Well it's easy if you can move the specimen in a known direction. From this movement, you can determine the direction of the tilt axis for a specimen of known geometry.

Yesterday's advice:

If you gently push on the end of your side-entry specimen holder, the image moves parallel to the principal tilt axis. But don't pull the holder in the other direction since you may inadvertently cause air to leak into the airlock.

Today's advice:

We're not letting you remove the draft-excluder (which covers the end of your specimen holder) to touch the holder after it has equilibrated. Monitor how you load the specimen instead which takes care and practice—but so do most TEM operations.

If you are looking at the DP, defocus the pattern so you can see the BF image in the direct-beam spot, as in Figure 9.26, then carry out the same exercise. If you are using a top-entry holder you will need to calibrate this tilt using a known specimen geometry, which is more challenging.

Focal increments of the objective lens: If you're going to do high-resolution, phase-contrast imaging and you don't have a C_s corrector, then you need to know the

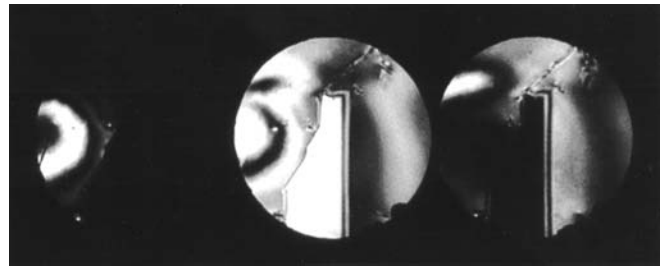


FIGURE 9.26. Defocused multiple-DF image showing how it is possible to determine simultaneously the direction of features in the image (e.g., the vertical twin boundary) and directions in the DP (e.g., the horizontal vector between the diffraction disks). If the specimen holder is moved in the direction of the principal tilt axis, the image will move and identify the relationship between that tilt axis and the crystallographic direction in the DP.

value of each defocus step of the objective lens so you can correctly calculate and interpret the image contrast. There is a simple method for determining this step value. Superimpose a focused image and an image defocused by a known number of objective-lens focal increments (Δf). The two images will be separated by a distance Δx which is related to Δf by

$$\Delta f = \frac{\Delta x}{2 M m \theta} \quad (9.4)$$

where M is the magnification, m is the number of focal increments, and θ is the Bragg angle for the reflection used to form the image. If you insert some typical values, you'll find that it is difficult to be very accurate with this method. We'll return to this topic in Chapter 28.

CHAPTER SUMMARY

We've now shown you how a TEM is put together. While the manufacturer does the best job possible, there are still some essential steps for you, the operator, to carry out. You must understand how to align the illumination system so the beam is on axis. You can then create a parallel beam for TEM and a convergent one for STEM. The C_2 aperture is a crucial part of the whole illumination system and the most easily misaligned. Astigmatism is not too much of a problem if the instrument in general and the apertures in particular are kept clean. The objective lens/stage combination controls all the useful information that is created as the beam is scattered by your specimen. *Always* start a microscope session by fixing the eucentric height and, before you do any worthwhile imaging, align the objective center of rotation, and minimize any objective astigmatism at high magnification. Diffraction and STEM operations require a centered, focused DP.

If you want to make any quantitative measurements from your images and DPs (and you really ought to do this if you have any aspirations to be a real microscopist), then calibration cannot be avoided. Your images and DPs are relatively useless unless you know their magnification and camera length, respectively, and the angular relationship between the two. So take the time to do this early in your studies. In doing so you will not only ensure that you produce quality data, but you will also learn an enormous amount about how these complex instruments work.

You'll notice that some of the data (e.g., Figure 9.12B and Table 9.1) are for older TEMs. It would be better to use data from even older machines ("Why?" you should ask) but you are unlikely to see such machines in operation.

SOME HISTORY

SAD was invented by LePoole in 1947. Some early machines used a pair of blades with a 90° bend that slid past one another to give a square aperture! See, e.g., LePoole, JB 1947 *A New Electron Microscope with Continuously Variable Magnification* Philips Tech. Rept. 9(2) 33–45. You can find further discussion of TEM calibration in Edington's text.

OPERATING THE TEM

- Chapman, SK 1980 *Understanding and Optimizing Electron Microscope Performance 1. Transmission Microscopy* Science Reviews Ltd. London. An old book but the principles are so clearly outlined that, if you are a serious user, this must be in your laboratory.
- Chapman, SK 1986 *Maintaining and Monitoring the TEM* Royal Microscopical Society Handbook No. 8 Oxford University Press New York. If you can't find this on the shelf in the office of the EM technical staff, be sure to buy it for them for Christmas. Your small investment will be repaid many times over.
- Chescoe, D and Goodhew, PJ 1990 *The Operation of Transmission and Scanning Microscopes* Royal Microscopical Society Handbook No. 20 Oxford University Press New York. A great in-depth description of the operational principles described briefly in this chapter.
- Christenson, KK and Eades, JA 1988 *Skew Thoughts on Parallelism*, *Ultramicroscopy* **26** 113–132.
- Edington, JW 1976 *Practical Electron Microscopy in Materials Science* Van Nostrand Reinhold New York. Now very dated, but still a valuable text for the details of hands-on, diffraction-contrast imaging and SAD; it's especially good on calibrations. However, this text was written before c/o lenses were introduced which, as we've noted, introduce complications into hitherto simple operations like forming parallel beams.
- Goldstein, JI, Newbury, DE, Joy, DC, Lyman, CE, Echlin, P, Lifshin, E, Sawyer, LC and Michael, JR 2003 *Scanning Electron Microscopy and X-ray Microanalysis* 3rd Ed, Kluwer New York. Dealing with a virtual C2 aperture, scanning imaging and much more.
- Keyse, RJ, Garratt-Reed, AJ, Goodhew, PJ and Lorimer, GW 1997 *Introduction to Scanning Transmission Electron Microscopy* Royal Microscopical Society Handbook No. 39 Bios Scientific Publishers Oxford. Basic introductory level but the only book available that emphasizes the operation of dedicated STEMs which historically were made by one company (VG) but are now available through at least three manufacturers (Hitachi, JEOL, and Nion). The DSTEM may well come to dominate the semiconductor industry in the way the SEMs did in the 1980s. If so, these instruments will become fixtures in any self-respecting EM Center. If, by now, you are getting the idea that the RMS handbook series is useful then you are right (see <http://www.rms.org.uk/other-publications.shtml>).
- McCaffrey, JP and Baribeau, JM 1995 *A Transmission Electron Microscope (TEM) Calibration Standard Sample for All Magnification, Camera Constant, and Image/Diffraction Pattern Rotation Calibrations* *Microsc. Res. Tech.* **32** 449–454.
- Watt, IM, 1997 *The Principles and Practice of Electron Microscopy* 2nd Ed. Cambridge University Press New York. Basic, hands-on, easy-to-read book, full of instructive images and diagrams, and easily understood explanations.

URLs

- 1) <http://www.emsdiasum.com/microscopy/products/catalog.aspx>
- 2) http://www.tedpella.com/calibrat_html/TEM6.htm
- 3) <http://www.2spi.com/catalog/stand.html>

COMPANION TEXT

C_c correction will be discussed in the chapters on HRTEM and EELS. The C_c monochromator takes on several forms but each decreases the total beam current.

SELF-ASSESSMENT QUESTIONS

- Q9.1 Name the three principal components of the TEM.
- Q9.2 How is the beam tilted or translated?
- Q9.3 How do you form a parallel beam; why would you want to do this and why is it not exactly parallel?
- Q9.4 How do you form a convergent beam; why would you want to do this and why is it sometimes divergent?
- Q9.5 How do you align the gun and why should you bother?
- Q9.6 How can you tell when your C2 aperture is misaligned and how would you correct it?
- Q9.7 How does the probe size change with the C1 setting and why might you want to change the size?

- Q9.8 What is SAD and how is it different from CBED?
- Q9.9 What is the difference between a BF image and a DF image?
- Q9.10 What is the advantage of forming images using STEM rather than TEM?
- Q9.11 What causes lens astigmatism? How do you correct for it (a) in the illumination and (b) in the imaging system?
- Q9.12 Why does the objective lens rotation need to be calibrated?
- Q9.13 Why would you want to form hollow-cone DPs and images?
- Q9.14 Explain CDF, DADF, HCDF, SADP, SDP, HCDP, FFP, BFP? If you can't find all these acronyms/initials in the text, suggest what they mean.
- Q9.15 What is the most important aperture in the TEM and why?
- Q9.16 How do you change the image magnification in a TEM?
- Q9.17 How do you change the image magnification in STEM? Explain why this method confers advantages over the answer to the previous question.
- Q9.18 Why do we need to do the magnification calibration?
- Q9.19 What are the reasons you might need to translate the beam?
- Q9.20 How should you always start a microscope session (apart from quiet prayer)?
- Q9.21 How should you always finish a microscope session (apart from a loud prayer)?

TEXT-SPECIFIC QUESTIONS

- T9.1 To converge the beam, which lenses do you use and how does this differ depending on the electron source? (Hint: go back to Chapter 5 and also look at Figures 9.3–9.5.)
- T9.2 Explain the differences between the various methods used to create a parallel beam at the specimen plane in Figures 9.1 and 9.2.
- T9.3 A smaller probe has a smaller current in it. Use Figure 9.4 to explain why this is so and state some consequences of reducing the probe current.
- T9.4 Why use SAD, instead of just forming DPs without a selecting aperture?
- T9.5 What illumination-system operation is essential to form Fresnel fringes for astigmatism correction?
- T9.6 What is the most common problem with TEM illumination systems (apart from the previous user of the microscope)?
- T9.7 How can you tell when your specimen is in the eucentric plane and why do you want it to be there?
- T9.8 Typically, how accurate is the magnification of images of particles in TEM? What do you have to do to get better accuracy?
- T9.9 List the operational steps necessary to see a SADP if you have a BF image on the TEM screen.
- T9.10 List the operational steps necessary to see a CDF image if you have a DP on the TEM screen.
- T9.11 Explain the principle shown in Figure 9.13 of using a virtual rather than a real aperture in the specimen plane for selected-area diffraction.
- T9.12 Use Figures 9.16 and 9.19 to explain why we can't change the direction of the incident beam in STEM imaging.
- T9.13 Why might we say that STEM images don't suffer from the defects in the imaging lenses and would we be correct?
- T9.14 Is Figure 9.21 taken with a thermionic or FEG source? Explain your answer.
- T9.15 Discuss the pros and cons of a c/o lens system using Figure 9.5 as a basis for your discussion. Justify why every materials-research TEM must have a c/o system.
- T9.16 Distinguish traversing and tilting a beam and, using specific figures in the text, explain how these operations can be used for DADF operation, hollow-cone imaging and diffraction, stationary-DP formation, and scanning-image formation.
- T9.17 When might you want to use a wobbler? Sketch a ray diagram explaining how a wobbler works. Sketch an image of how an astigmatic beam changes as it is wobbled and how a misaligned beam changes as it is wobbled.
- T9.18 Use the references to find an equation governing the relationship between probe current and probe size.
- T9.19 Estimate the relationship between the final aperture radius in the illumination system and the convergence angle of the beam and explain how this is relevant to Figure 9.11.
- T9.20 Why does converging the beam expand the spots in the DP and why is there different contrast information (images) in different expanded spots, as in Figure 9.26?
- T9.21 Why is it important to calibrate the rotation of your DP with respect to your image?
- T9.22 Estimate the accuracy of the magnification and rotation calibrations.
- T9.23 Look up the lattice parameters of α -MoO₃, make some reasonable assumptions about the TEM, and calculate an approximate camera length for the DP in Figure 9.24.