The Instrument

9

CHAPTER PREVIEW

We've introduced all the essential components of the TEM. Now it's time to see how the guns, lenses, and detectors are combined to form the microscope. Just as we do for the visible-light microscope (VLM), it's convenient to divide the TEM up into three components: the illumination system, the objective lens/stage, and the

D. B. Williams et al., *Transmission Electron Microscopy*

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 for TEM imaging and diffraction, while the second is used for scanning (STEM) imaging, microanalysis, and microdiffraction.

The objective lens/stage system is the heart of the TEM. The critical region usually extends over less than 1 cm along the length of the column. Here is where all the beam–specimen interactions take place and here we create the bright-field and dark-field images and selected-area diffraction patterns (SAD) that are the fundamental TEM operations. Likewise, it is here that we manipulate a scanning beam to form STEM images and diffraction patterns. The quality of the image formed by the objective lens controls the resolution of the image that you view and record. We therefore often say that the objective lens is the most important lens. The reason is not simply because the objective lens forms the first image, but rather because the specimen must be placed so close to the center of this short-focal-length lens that it is impossible to make a perfect lens (or even a very good lens by visible-light standards).

The imaging system uses several lenses to magnify the image or the diffraction pattern produced by the objective lens and to focus these on the viewing screen. We'll refer to the magnifying lenses as the intermediate and diffraction lenses and the final lens as the projector lens (it projects an image on the viewing screen). Alternatively, an electron detector coupled to a TV/CRT can be used to display the STEM image. So all TEM operations involve observing the electrons on a viewing screen of some form, with or without a specimen in place. In many modern-day TEMs you will have a button for focus, another for magnification, and another for diffraction (or a slide on the computer screen).

The purpose of this chapter is to go through the principal functions of each of the three components and give you some feel for what is happening in the microscope when you "press the button." The more you understand the operation of the TEM, the better you can be sure that you are getting the most out of the instrument.

The Instrument 9

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. We can think of these two cases as the equivalent of wide-field illumination 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 illumination system which consists of two or three condenser lenses (which we'll call C1, etc.). We will discuss the two different ways to use the illumination system: we'll refer to these as forming a parallel beam (it is almost never truly parallel) or a convergent beam.

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×–100,000×). As shown in Figure 9.1, the C1 lens first forms a demagnified image of the gun crossover. In the case of a thermionic source, the original crossover may be several tens of micrometers across, and this is demagnified by an order of magnitude or more: in the case of an FEG, the source size may be less than the desired illumination area on the specimen so it may be necessary to magnify the crossover—the condenser lenses don't always condense! To produce a parallel beam you adjust the C2 lens to produce an underfocused image of the C1 crossover.

> Remember that the convergence angles (α) are so small that the ray diagrams are drawn with highly exaggerated angles, and 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.

In TEMs used for generating the very small electron beams we need in STEM and AEM, the upper polepiece, of the objective lens is also used to control the beam hitting the specimen as shown in Figure 9.1B. Now the C2 lens is focused to produce an image (of the crossover) at the front focal plane of the upper objective polepiece, which then generates a broad parallel beam of electrons incident on the specimen.

> Consider the question: How is this argument consistent?

When the beam is parallel, it is as coherent as possible. We'll see in Chapter 18 that parallel illumination is essential to get the sharpest diffraction patterns as well as the best image contrast. (It is also usually assumed in the interpretation of our images that the beam is parallel.) Usually you underfocus C2 until the illuminated area on the specimen fills the viewing screen. A higher magnification means strengthening C2 so the beam illuminates less of the specimen (so you see, it isn't really "parallel," just not very "convergent").

In the parallel-beam TEM mode, 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 falling on 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

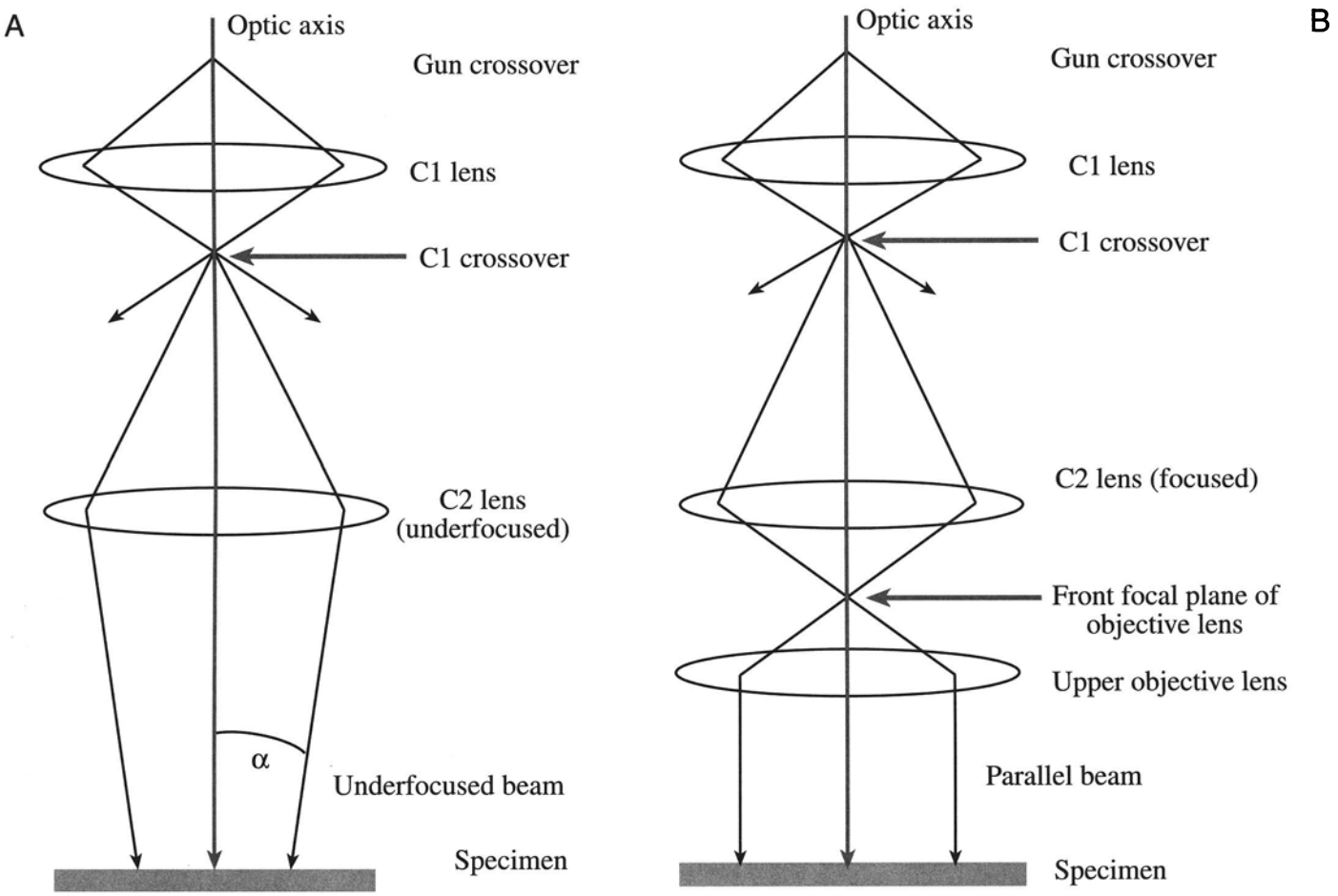


Figure 9.1. Parallel-beam operation in the TEM (A) using just the C1 and an underfocused C2 lens and (B) using the C1 and C2 lenses to image the source at the front focal plane of the upper objective lens.

focused rather than underfocused, and you form an image of the C1 crossover at the specimen, as shown in Figure 9.3. This is the condition under which you can view the source image to adjust its saturation or to measure the dimensions of the beam. 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. 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 for poor transmission through a thick specimen.

There are times when we need to deliberately create a focused convergent beam at the specimen. We then use the other principal way to operate the illumination system: the convergent-beam mode. When you use this mode you won't immediately see a useful image of your specimen; the convergence destroys the parallelism and 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.

> The convergent beam is a probe. We use such a probe when we want to localize the signals coming from the specimen, as in microanalysis or convergent-beam (also known as micro or nano) diffraction.

Now, unless you have an FEG, it isn't possible to use just the C1 and C2 lenses as in Figure 9.3 to converge the beam to as small a probe as you would like (<10 nm). This is because the C1 and C2 lenses can't demagnify the gun crossover sufficiently. So the usual solution is to con-

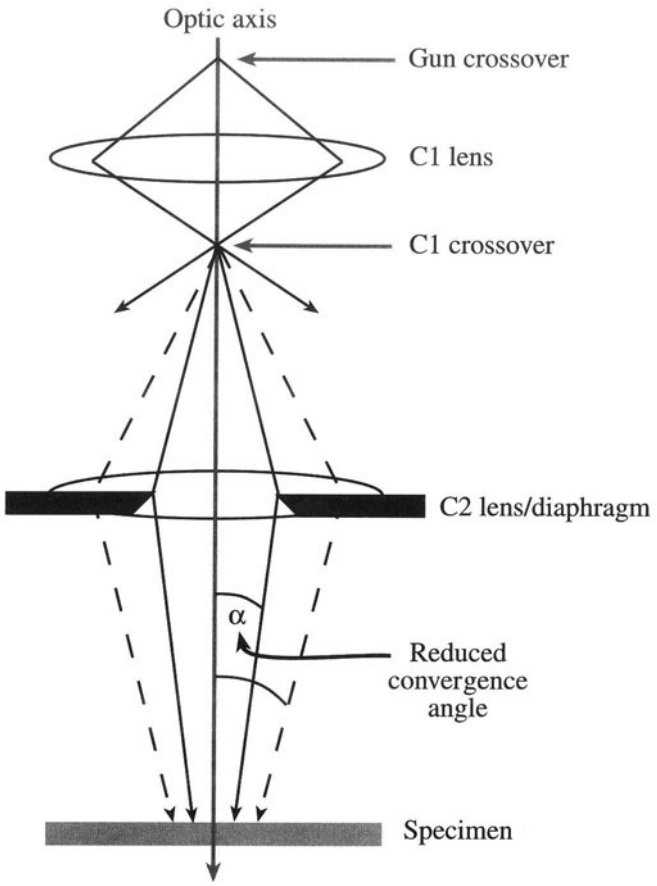


Figure 9.2. Effect of the C2 aperture on the parallel nature of the beam: a smaller aperture creates a more parallel beam.

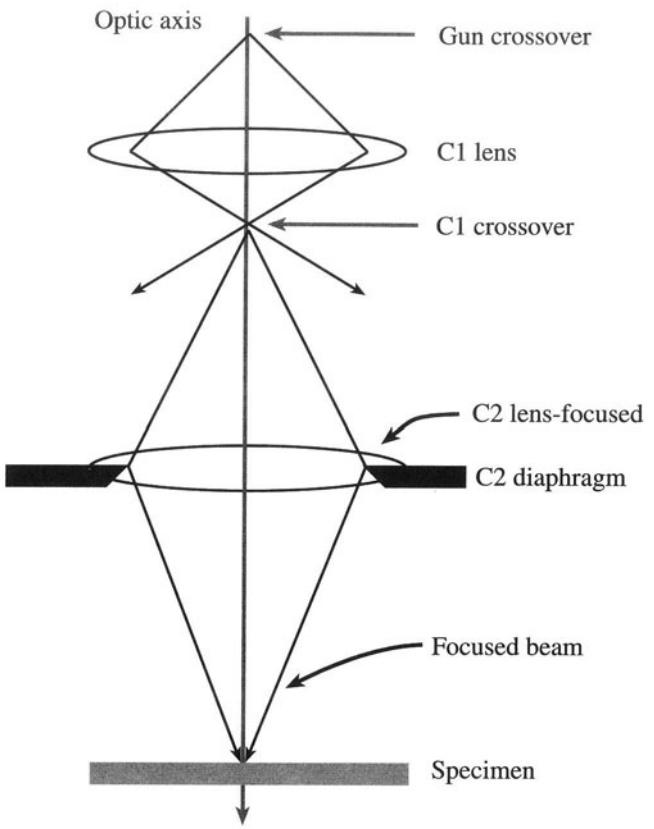


Figure 9.3. A focused C2 lens illuminates a small area of the specimen with a nonparallel beam.

vert the upper polepiece of the objective lens into a third condenser lens, which we then call a condenser-objective lens (C3). We make the upper part of the objective lens much stronger than usual and weaken C2 or turn it off, as shown in Figure 9.4. 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 (v) is much less than the object distance (u), which gives a large demagnification of the C1 crossover (see equation 6.2). From Figure 9.4 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 that the correct choice of C2 aperture is important in convergent-beam electron diffraction (CBED) and also in defining the exact dimensions of the probe for X-ray microanalysis (see Chapter 36).

The role of C1 here is fundamentally different from its role in parallel-beam TEM; now the C1 lens is used directly to form the probe because we adjust it to change the probe size at the specimen. As shown in Figure 9.5, 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 v, thus lengthening u for the probe-forming C3 lens and therefore increasing the C3 demagnification. When convergent probe TEMs were first constructed, it was not possible to design a C3 lens that would give both a parallel and a convergent beam with the same polepiece. This problem was overcome in the mid-1970s by the introduction of an extra lens between C2 and C3 (not shown in the diagrams) and this auxiliary lens is now standard on TEMs that also operate as STEMs.

9.1.C. 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 microanalysis). Similarly, there are times when we need to tilt the beam off axis so it impinges on the specimen at a specific angle (e.g., for centered dark-field imaging using a specific diffraction spot which we describe in Section 9.3.C). Ray diagrams to explain translating and tilting are shown in Figures 9.6A and B. Both operations are accomplished by varying the

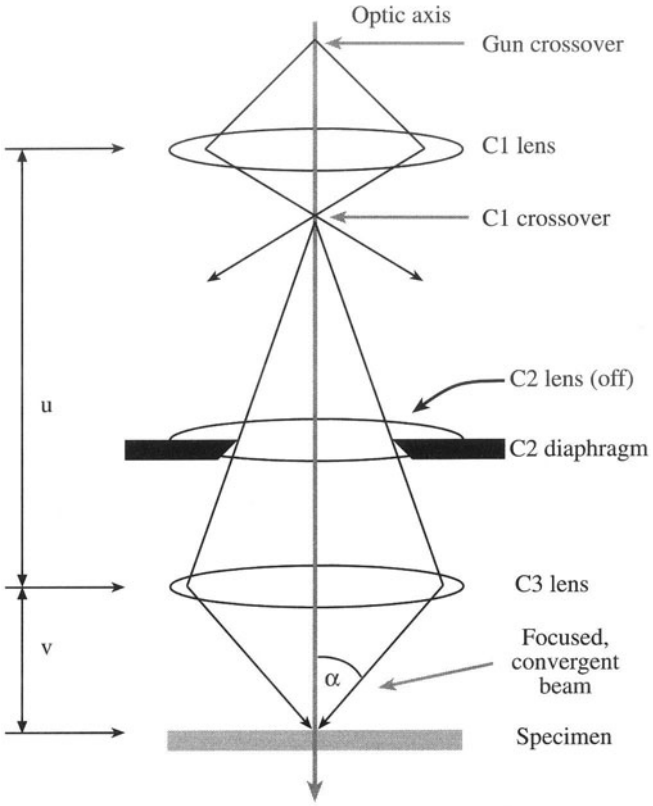


Figure 9.4. Use of the objective polepiece as a third condenser lens (also called a condenser-objective, or C3, lens) gives the smallest possible probe and large convergence angles. The large *u/v* ratio gives the maximum demagnification of the image of the gun crossover.

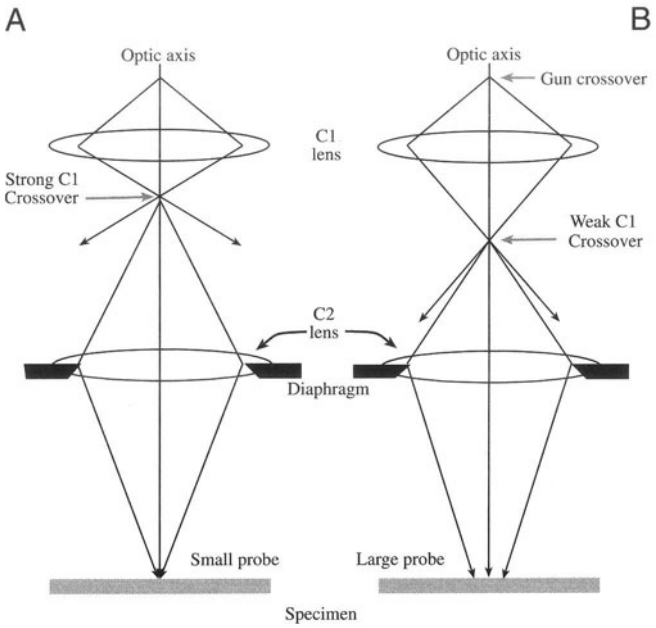


Figure 9.5. Effect of the C1 lens strength on probe size: a stronger C1 lens (A) results in greater demagnification by any subsequent lens (C2 or C3), giving a smaller electron beam at the specimen. A weaker lens (B) gives a broader probe.

current through potentiometers, which we'll call *scan coils*. We use these scan coils (of which there are several in the column) to apply a local magnetic field to deflect (rather than focus) the beam. To translate the beam we use deflector scan coils. To tilt the beam just before it reaches the specimen we use tilt scan coils situated between C2 and C3.

When we create a scanning beam for STEM imaging, the beam must always move parallel to the optic axis. 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 front focal plane of C3. Then, wherever the beam enters the C3 lens field, it is bent to follow a path parallel to the optic axis. You can see how this is done if you look ahead to Figure 9.15. This rather complex adjustment is computer-controlled. Like many other procedures on a modern TEM, this adjustment is made automatically when you select STEM mode.

9.1.D. Alignment

If you correctly align the illumination system, the gun crossover is on the optic axis and the electrons can then

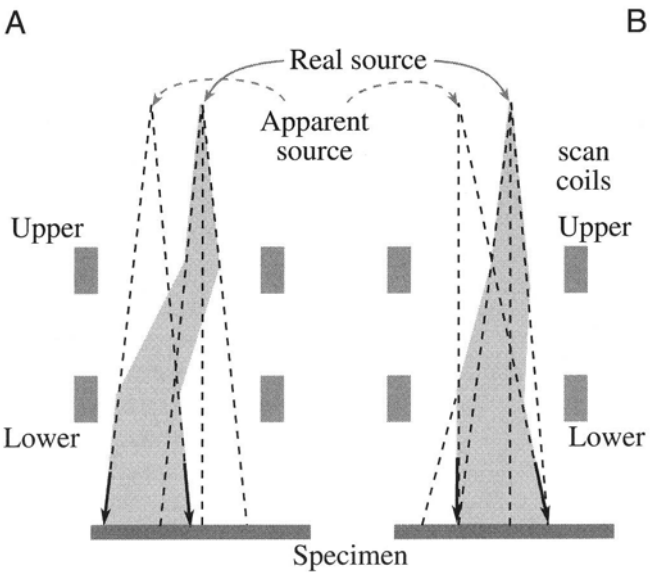


Figure 9.6. The use of pre-specimen scan coils for (A) traversing the beam and (B) tilting the beam. Traversing moves the beam to a different area of the specimen but it stays parallel to the optic axis. Conversely, tilting the beam illuminates the same area of the specimen, but from a different angle.

follow a straight line through the lenses and apertures until they hit the specimen. Alignment used to be a tedious manual affair involving tilting and translating the gun and the condenser lenses and centering the apertures on axis. Now most of the components are machined accurately enough that minor electronic adjustment is all that is needed. Nevertheless, manual centering of the C2 aperture remains a most critical step in obtaining the best performance out of the TEM, particularly if you intend to operate in scanning mode for STEM imaging and microanalysis.

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 it 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.

Gun Alignment

First, you have to undersaturate the filament so structure can be seen in the image, as shown back in Figure 5.5. If the gun is very badly misaligned, you may have to turn the condenser lenses off, before you use the gun traverses to center the filament image. Then use the gun tilts to make the source image symmetrical and repeat the whole procedure. If this alignment is very bad, then either there is a major problem with the gun or with the previous user; in either case expert help is required to correct the fault.

Alignment of the C2 Aperture

You must 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 align the aperture on axis, you need first to overfocus C2 so the image of the beam is spread and the outline of the C2 aperture is visible on the screen (make sure any other apertures in the imaging system are out of the column). Then use the external drives to center the aperture on the screen. Next, you must adjust C2 so the image of the beam is focused. Then, center the beam with the deflector controls. Now underfocus the C2 lens until you can again see the aperture and center it again with the external drives. 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. 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 saves you from manually underfocusing and overfocusing the lens.

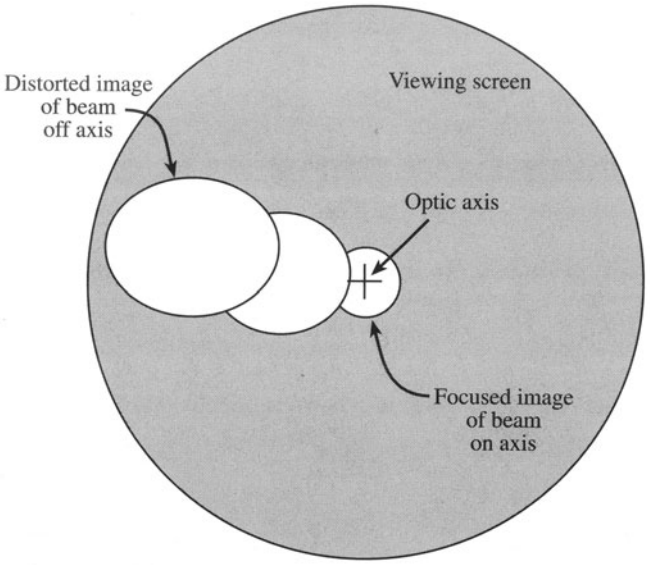


Figure 9.7. If the C2 aperture is misaligned, underfocusing or overfocusing the C2 lens causes the image of the beam to sweep off axis (i.e., across the viewing screen) and to become distorted.

9.1.E. Condenser Lens Defects

The illumination system lenses suffer from the standard lens defects, such as aberrations and astigmatism. These defects don't really limit the operation of the TEM in paral-

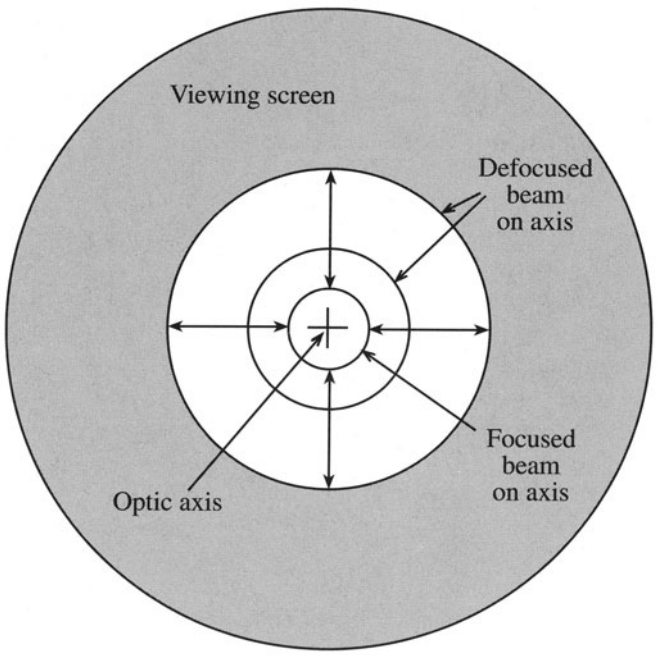


Figure 9.8. If the C2 aperture is aligned, the image of the beam remains circular and expands or contracts about the optic axis as the lens is underfocused or overfocused.

lel-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 6.23) of $r_{min} \sim 0.91(C_s\lambda^3)^{1/4}$. This is why 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 6.22) for the selected probe size $\alpha_{opt} = 0.77\ \lambda^{1/4}/C_s^{1/4}$. 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.

Chromatic Aberration

Remember this aberration depends on the energy spread of the electrons. Since the electrons in the illumination system have such a small energy spread, you can regard them as monochromatic and there is no detectable degeneration of the probe dimensions.

Astigmatism

This is the most common defect in the TEM illumination system and arises either because the final C2 limiting aperture is misaligned or it is contaminated and charging up, thus deflecting the beam. Let's assume you've centered the C2 aperture as we just described and talk about correcting any residual astigmatism due to contaminated apertures.

You can discern astigmatism in the illumination system if you look at an image of the electron source on the screen; focus C2 so the beam is a minimum diameter and the image of the beam is circular, as you did when aligning the aperture. If you then wobble the C2 lens either side of the focal setting, 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. The condenser stigmators introduce a compensating field which you use to correct this distortion. You first overfocus the beam so you can see the effect of

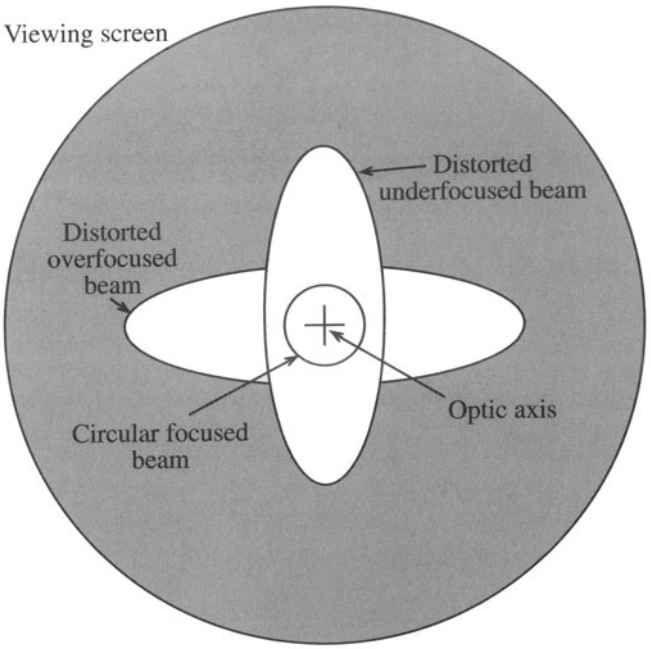


Figure 9.9. The effect of astigmatism in the illumination system is to distort the image of the beam elliptically as the C2 lens is underfocused or overfocused. Correction of this astigmatism results in an image that remains circular as the C2 lens is defocused.

the astigmatism (i.e., the beam appears elliptical). 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.

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 you need to remove the source of the astigmatism by flame cleaning the condenser diaphragms, as we described in Chapter 6.

9.1.F. Calibration

We've already seen what it takes to calibrate the performance of the electron gun and optimize the brightness so that the maximum beam current goes into the minimum beam size. All that's left is to calibrate the condenser system. The major variables are the probe size 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. Figure 9.10 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

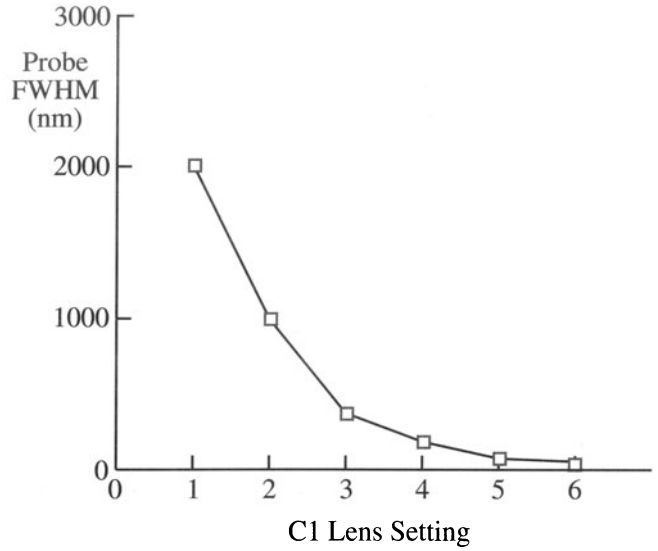


Figure 9.10. Calibration of the illumination system requires determining the variation of the probe size with C1 lens strength.

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 C2 aperture size governs the convergence semiangle 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 2α by increasing the C2 aperture size (Figure 9.11).

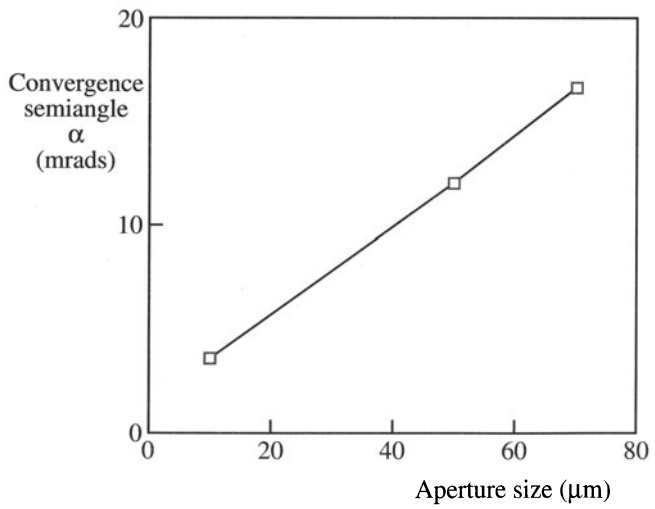


Figure 9.11. Variation of the beam-convergence semiangle, α, with the C2 aperture dimensions.

Different TEMs will have different responses. On some instruments the C2 aperture is virtual (so you have an effective aperture size), which makes it rather difficult to measure 2α. (See Goldstein *et al.* 1992 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 as a function of both 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 diffraction patterns 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 with which you have to make adjustments. Our discussion will emphasize the side-entry holder since this is becoming the standard, but top-entry holders require the same adjustment of the z-control or specimen height.

> We need to fix the height of the specimen on the optic axis. This will allow us to work at the same objective lens current and thus at a fixed objective lens magnification.

As a practical consideration, you would like to be able to tilt the sample without changing its height on the optic axis. Otherwise, you would be continuously using the z-control when you tilt the sample. 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.

The central requirement 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 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 the specimen is located at this plane and the image is in focus, the objective lens current is an optimum 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. Of course, if you tilt your specimen normal to the holder axis, or rotate it off axis, then the point you're examining almost invariably moves out of the eucentric plane.

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, you tilt the specimen and adjust the height of the specimen holder until the image of the specimen remains stationary when you tilt the sample through ±30° either side of zero.

With computer control and auto-focusing techniques becoming common, this operation can be automated. As a result we now see completely eucentric stages in which 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 a computer-controlled stage, be cautious.

The eucentric plane should also be coincident with the plane that is symmetrically positioned with respect to the upper and lower objective polepiece fields. This means that 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 diffraction patterns 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 diffraction pattern or an image in TEM and STEM modes. You should not have to refocus the image or diffraction pattern with the objective lens when you change from one mode to the other.

9.3. FORMING DIFFRACTION PATTERNS AND IMAGES: THE TEM IMAGING SYSTEM

You know that the objective lens takes the electrons emerging from the exit surface of the specimen, disperses them to create a diffraction pattern (DP) in the back focal plane, 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 diffraction patterns 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 will 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. The first operation that you need to master when using the TEM is viewing the diffraction pattern. In all the subsequent imaging, we'll use this pattern to select electrons that have suffered particular angles of scatter to form our images.

- To see the diffraction pattern you have to adjust the imaging system lenses so that the back focal plane of the objective lens acts as the object plane for the intermediate lens. Then the diffraction pattern is projected onto the viewing screen, as shown in Figure 9.12A.
- 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, 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 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 diffraction pattern 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. So we perform a basic TEM operation both to select a specific area of the specimen to contribute to the diffraction pattern and to reduce the intensity of the pattern falling 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 diffraction pattern.

- We could make the beam smaller.
- We could somehow 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 diffraction pattern with a parallel beam of electrons, the standard way is to use a selecting aperture. This operation is called se-

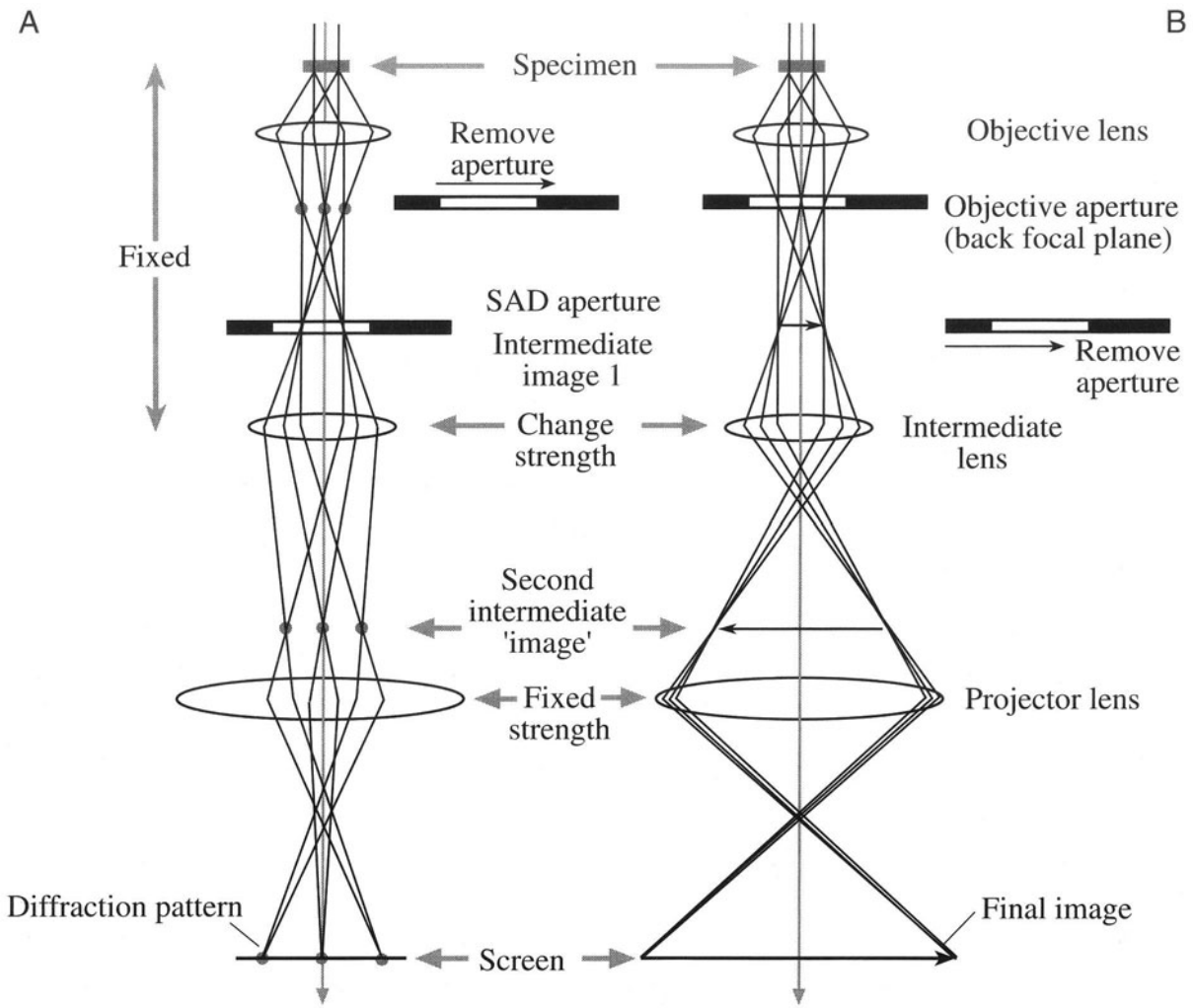


Figure 9.12. The two basic operations of the TEM imaging system involve (A) projecting the diffraction pattern on the viewing screen and (B) projecting the image onto the screen. In each case the intermediate lens selects either the back focal plane or the image plane of the objective lens as its object.

lected-area diffraction, or SAD, and was invented by LePoole (1947). Now, we can't insert an aperture at the specimen plane, because the specimen is already there! If we insert an aperture in a plane conjugate with the specimen, i.e., in one of the image planes, then it creates a virtual aperture at the plane of the specimen. This is exactly what we do.

The conjugate plane that we choose is the image plane of the objective lens, as shown in Figure 9.13. We insert the *SAD aperture* into the image plane of the objective lens and center the aperture on the optic axis in the middle of the viewing screen. You can see the image of this aperture on the viewing screen. It must be focused 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. Then any electron that hits the specimen outside the area defined by the virtual aperture will hit the real diaphragm when it travels on to the image plane. It will thus be excluded from contributing to the diffraction pattern that is projected onto the viewing screen. In practice, we can't make apertures smaller than about 10 μm, and the demagnification back to the plane of the specimen is only about 25×, which gives a minimum selected area of ~0.4 μm—which isn't as small as we'd like. We'll discuss in Chapter 11 whether or not smaller values would be useful.

The SAD pattern is often displayed on the viewing screen at a fixed magnification.

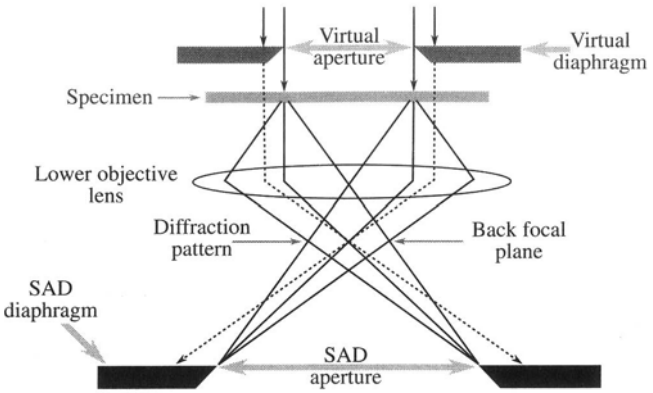


Figure 9.13. Ray diagram showing SAD pattern formation: the insertion of an aperture in the image plane results in the creation of a virtual aperture in the plane of the specimen. Only electrons falling inside the dimensions of the virtual aperture at the specimen will be allowed through into the imaging system. All other electrons will hit the SAD diaphragm.

By analogy with the hand-held camera we define a distance called the "camera length" (*L*).

This distance corresponds to the distance of the "film" from the diffraction pattern. We choose the value of *L* such that the spacings in the diffraction pattern are easily discernible on the screen and on the photographic plate. This magnification can be changed by adjusting the intermediate lenses. We'll describe how we calibrate this magnification later.

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

You can see this aperture if you want to change it or center it, by projecting the image plane onto the viewing screen, which we'll now discuss.

Beware: In most TEM books, SAD is the only standard diffraction technique. As a result, some microscopists use only SAD to obtain diffraction information. However, you should know that CBED, which we discuss in Chapters 20 and 21, can provide complementary diffraction information and must also be used by all TEM operators in the materials field. There are still certain times when you'll need to form an SAD pattern.

- 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 diffraction pattern, such as streaks (see Chapter 17).

On all other occasions, when the diffraction maxima provide the most important information in the pattern, then we strongly recommend that you use CBED.

9.3.B. Bright-Field and Dark-Field Imaging

When the SAD pattern is projected onto the viewing screen, we can use the pattern to perform the two most basic imaging operations in the TEM. No matter what kind of specimen you're are looking at, the SAD pattern will contain a bright central spot which contains the direct electrons and some scattered electrons (as shown in Figures 2.11a–c). 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 to insert an aperture into the back focal plane of the objective lens, thus blocking out most of the diffraction pattern except that which is visible through the aperture. We use the external drives to move the aperture so that either the direct electrons or some scattered electrons go through it. If the direct beam is selected as shown in Figure 9.14A, we call the resultant image a bright-field (BF) image, and if we select scattered electrons of any form, we call it a dark-field (DF) image, as shown in Figure 9.14B. Typical magnification ranges will be 25,000× – 100,000×.

The BF and DF images can be viewed at any magnification simply by adjusting the intermediate lenses of the microscope. It is necessary to calibrate the actual magnification and also to be able to relate directions in the image at any magnification to directions in the diffraction pattern at a fixed camera length. These are the two basic calibrations required for any TEM.

It is another 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 into the *back focal plane* of the objective lens. This is called the *objective aperture* and is most important in the TEM, since its size controls the collection angle (β) and hence determines the effect of all the aberrations and resolution of the most important lens in the instrument.

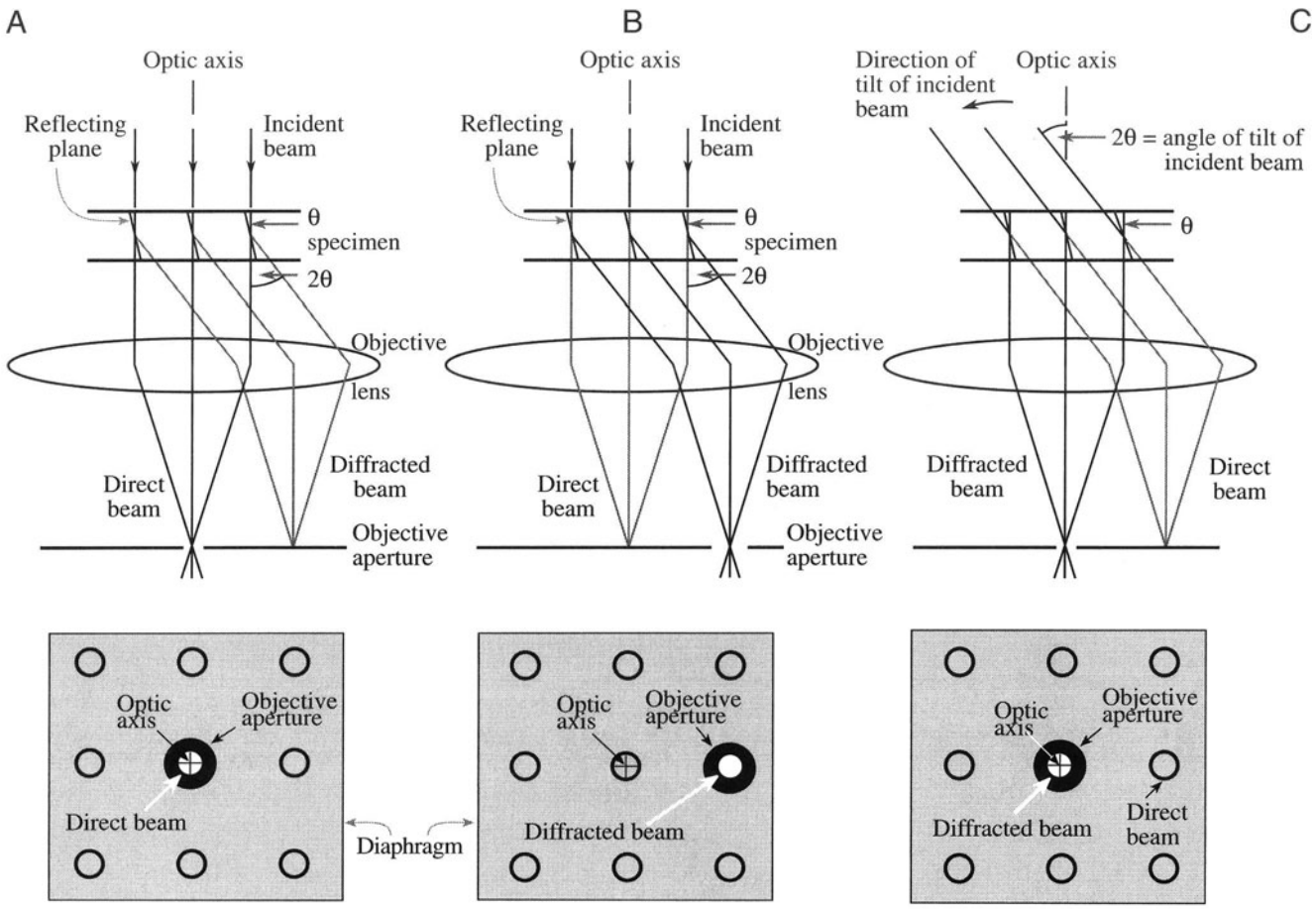


Figure 9.14. Ray diagrams showing how the objective lens/aperture are used in combination to produce (A) a BF image formed from the direct beam, (B) a displaced-aperture DF image formed with a specific off-axis scattered beam, and (C) a CDF image where the incident beam is tilted so that the scattered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

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 SAD patterns 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 back focal plane, 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 diffraction pattern, 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.

9.3.C. Centered DF 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. These off-axis electrons suffer aberrations and astigmatism and the DF image is difficult to focus, since it will move on the screen as you adjust the objective lens strength. To avoid this you have to adjust the beam tilt potentiometers above the objective lens so that the incident beam hits 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. This operation is called centered dark-field (CDF) imaging and is the way to do DF imaging in the TEM, if you want to record the best, focused image. However, there are situations where you will want to form a displaced-aperture DF image. You do this by physically moving the aperture rather than by tilting the incident beam.

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

9.4. FORMING DIFFRACTION PATTERNS 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.

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

As we show in Figure 9.15, the way we do this is to use two pairs of scan coils to pivot the beam about the front focal plane 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, then a stationary diffraction pattern is formed in the back focal plane (so called because this pattern does not move even though the beam is scanning, since it is conjugate with the front focal plane, as shown in Figure 9.16). If we stop the beam from scanning, then we have a CBED pattern in the back focal plane and we can project that onto the TEM screen if we wish. Now let's discuss how to form STEM images.

One potentially very big advantage of forming images this way is that we don't use lenses, as in an SEM. So defects in the *imaging lenses* do not affect your image resolution, which is controlled by the beam only. Hence chromatic aberration, which can limit TEM images, is absent in STEM images, which is advantageous if you're dealing with a thick specimen. However, there are drawbacks also, as we'll discuss below, and STEM images aren't widely used, particularly for crystalline specimens.

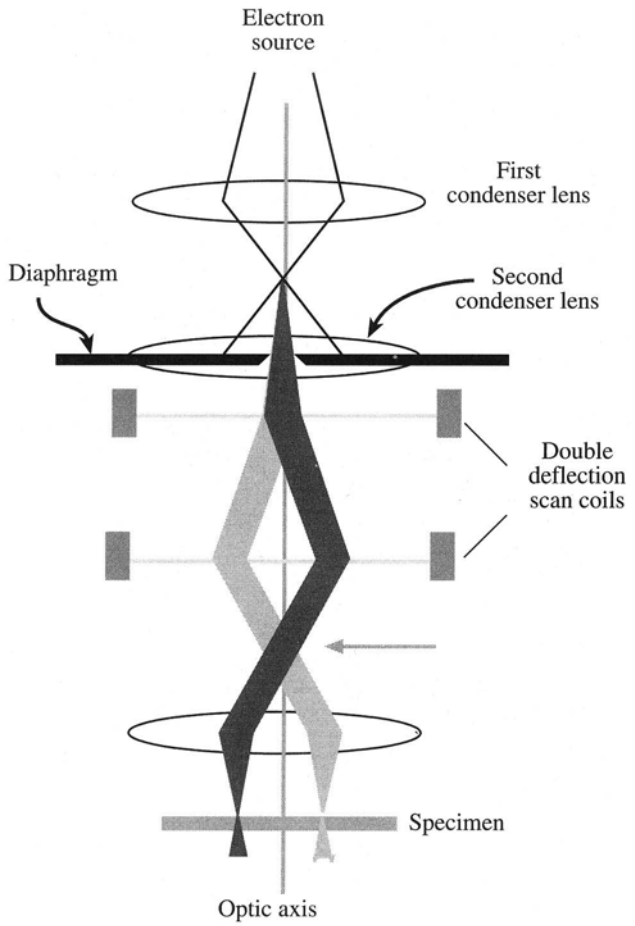


Figure 9.15. Scanning the convergent probe for STEM image formation using two pairs of scan coils between the C2 lens (usually switched off) and the upper objective polepiece. The probe remains parallel to the optic axis as it scans.

9.4.A. Bright-Field STEM Images

The basic principle of image formation in the scanning mode is fundamentally different from that for a static-beam TEM image. As you've just seen, in the TEM we se-

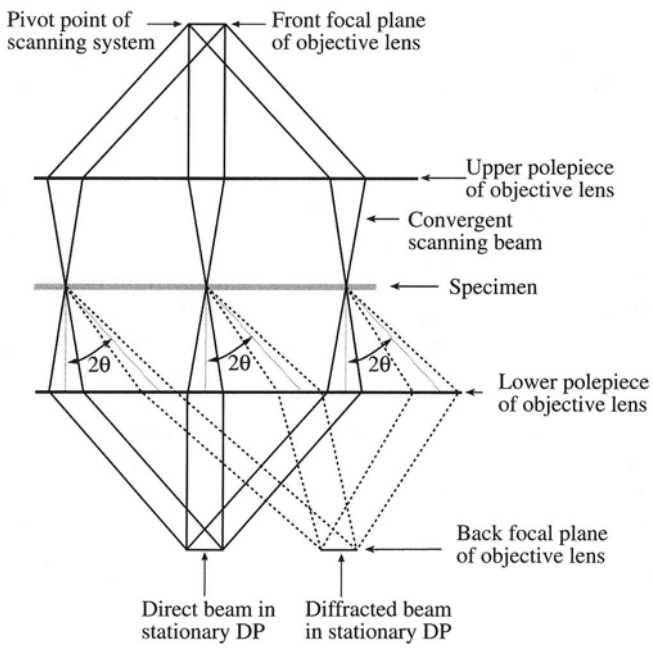


Figure 9.16. The creation of a stationary (convergent-beam) diffraction pattern in the back focal plane of the objective lens is a necessary prerequisite for STEM imaging. Note that electrons scattered through 2θ at different points in the specimen are focused at the same point in the focal plane.

lect 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 9.17. Simply stated, the beam is scanned on the specimen by adjusting the scan coils; these same coils are used to scan the CRT synchronously. The electron detector acts as the interface between the electrons coming from the specimen and the image viewed on the CRT. Since it takes up to 2048 scan lines to build up an image on the CRT, the whole process of creating a STEM image is much slower than TEM imaging: it's serial recording instead of parallel recording.

> The STEM signal generated at any point on the specimen is detected, amplified, and a proportional signal is displayed at an equivalent point on the CRT. The image builds up over several seconds or even minutes.

This process is exactly the same principle as used in any scanned-probe microscope, 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 diffraction pattern 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 put a BF detector (either a semiconductor or scintillator detector) into the direct beam in the diffraction pattern in Figure 9.18. Thus, only direct electrons hit that detector from wherever the beam is scanning on the specimen. This variable signal travels from the detector via an amplification system to modulate the signal on a CRT, thus creating a BF image as also shown in Figure 9.18.

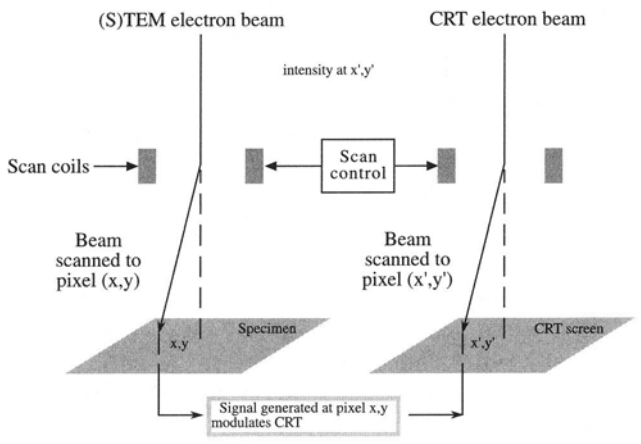


Figure 9.17. The principle of forming a scanning image, showing how the same scan coils in the microscope control the beam-scan on the specimen and the beam-scan on the CRT. Thus no lenses are required to form the image.

> The variable signal which emerges from the BF detector depends on the intensity in the direct beam from that point on the specimen.

Now, in a TEM we can't physically put the detector in the back focal plane 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 diffraction pattern, below the projector lens. 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 the screen. The stationary diffraction pattern falls on the detector and the signal goes to the CRT. In a DSTEM, there may not be any imaging system lenses, in which case the detector is 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 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 of the scattered electrons, rather than the direct electrons. Remember, in a TEM we tilt the incident beam so the scattered electrons that 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.

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

It's simple to do this with the diffraction pattern 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 DF 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 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.

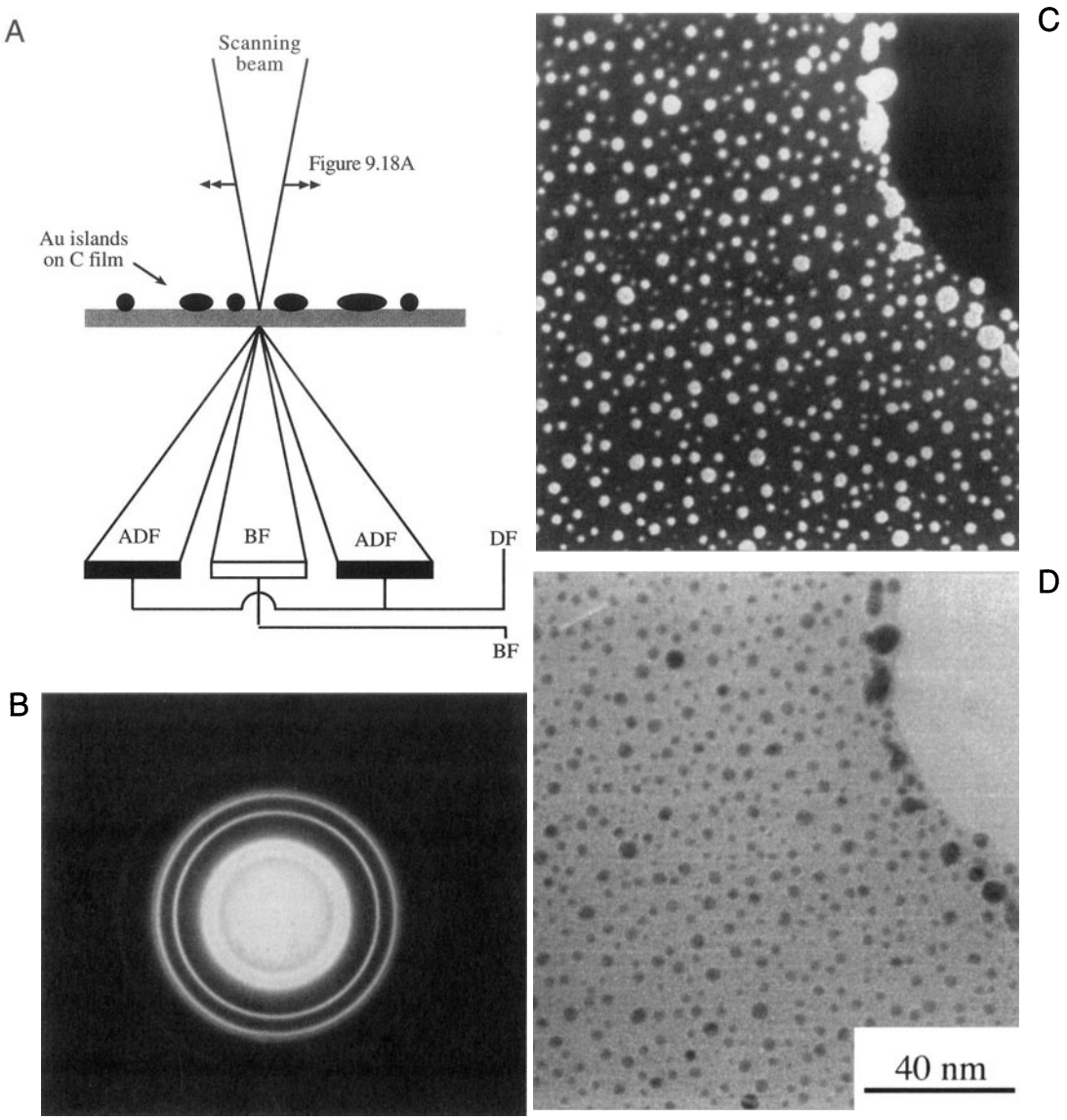


Figure 9.18. STEM image formation: A BF detector is placed in a conjugate plane to the back focal plane to intercept the direct beam (A) and a concentric annular DF detector intercepts the diffracted electrons (B). The signals from either detector are amplified and modulate the STEM CRT. The specimen (Au islands on a C film) gives complementary ADF (C) and BF (D) images.

As is shown in Figure 9.18, 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 is complementary to the BF image.

Now, of course, you can make the detector any size or shape you wish. For example, you can split the annulus into two halves or four quadrants and electrically isolate each part of the detector. Then you can form 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. 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

Any of the STEM images that we have just described appear on the CRT 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. Scanning images are *not* magnified by lenses (and are thus not affected by aberrations in the imaging lenses as we stated at the start of this

section). If the scanned area on the specimen is 1 cm × 1 cm, and the resultant image is displayed on a CRT with an area 10 cm × 10 cm (which is the standard size of the *record* CRT, though rarely the size of the *viewing* CRT screen) then the magnification is 10×. If the scan dimension is reduced to 1 mm, the magnification is 100×, and so on, up to magnifications in excess of 10^6×, which are common in dedicated STEMs. As with the TEM, we have to calibrate the STEM magnification and the camera length of the diffraction pattern 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 diffraction pattern on the optic axis. To get the best out of your TEM, you must master these two fundamental alignments.

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 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 and your image will rotate about a position off axis as you change the objective lens (focus), as shown schematically in Figure 9.19.

To center the objective rotation, you start at a relatively low magnification (say 10,000×), select an obvious reference point in the image, and observe the way the point rotates as you wobble the objective lens over and under focus. Then use the beam tilts to move the center of rotation to the middle of the screen and repeat the process at higher magnifications. Above ~100,000× 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."

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.

The diffraction center is aligned by adjusting the projector lens until the central spot in the diffraction pattern is on axis. If you change the diffraction pattern magnification (the camera length) the pattern will move off axis, which can easily be compensated for in a computer-controlled column.

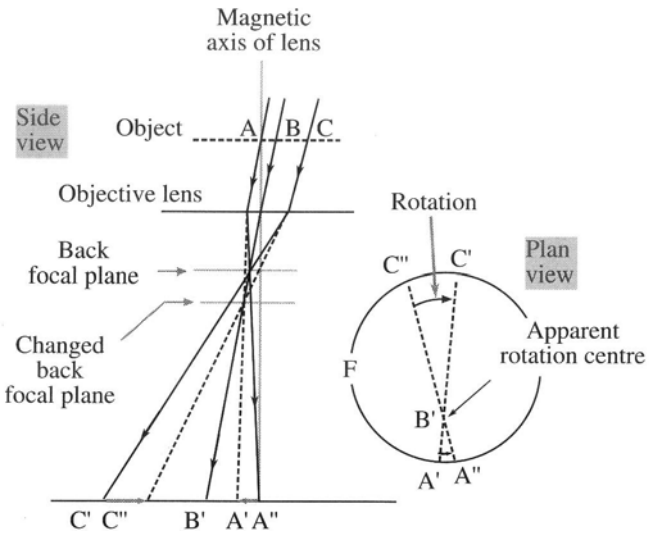


Figure 9.19. When the objective lens center of rotation is misaligned, the image appears to rotate about a point away from the center of the viewing screen when the lens is wobbled about focus.

Centering the diffraction pattern is useful in STEM image formation, since you use it to center the diffraction pattern on the STEM detector 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 diffraction pattern, the main cause of problems in the imaging system is objective and intermediate lens astigmatism.

Objective lens astigmatism occurs mainly if the objective aperture is misaligned, so you must carefully center the aperture on the optic axis, symmetrically around the electron beam used to form the BF or DF image. Despite careful centering, however, residual contamination may 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. Often you can only see objective astigmatism at the highest magnifications, where it manifests itself as a streaking in the image that rotates through 90° as you alternately underfocus and overfocus the objective lens. So again, you have to wobble the objective lens, but if the magnification is too high then manual wobbling is required.

If you then overfocus C2 to ensure a parallel beam, and also overfocus the objective lens, then a Fresnel fringe will be visible at the thin edge of the specimen.

Either look for a small hole in the specimen or look at the edge of the specimen. Ideally, you might use a holey carbon film to correct residual astigmatism *before* you put in your specimen, especially while learning this procedure. In practice, you have to check your astigmatism throughout your TEM session so you use the same approach on a thin curved edge of your specimen.

As shown in Figure 9.20A, when you underfocus the objective lens, there is a bright 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 9.20D, then the focus of the lens is changing around the hole because of astigmatism. So you then 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 9.20B). At exact focus, there is no fringe and the image contrast is minimized (Figure 9.20C).

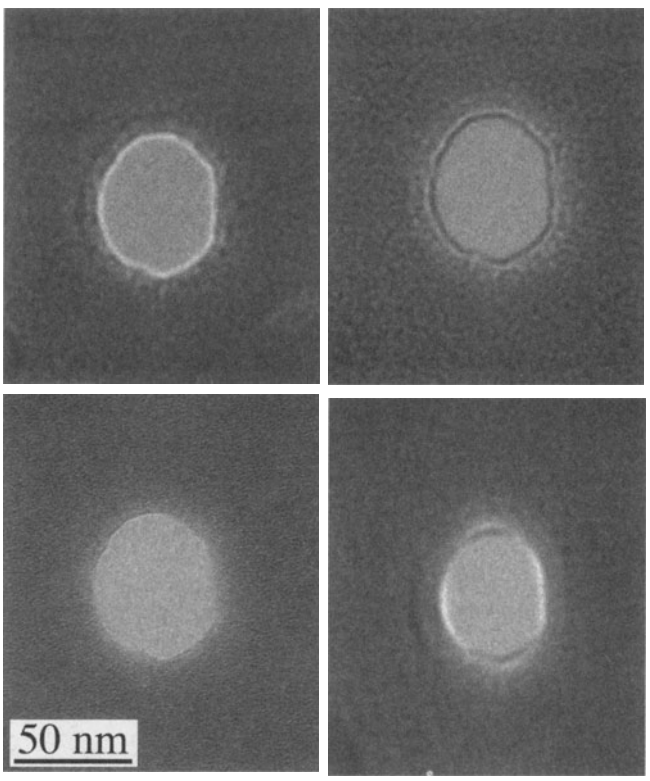


Figure 9.20. The image of a hole in an amorphous carbon film illuminated with a parallel beam showing that (A) with the beam underfocused, a bright Fresnel fringe is visible; (B) with the beam overfocused a dark fringe is visible; (C) at exact focus there is no fringe; and (D) residual astigmatism distorts the fringe.

This method of correcting the astigmatism is reasonable at magnifications up to several hundred thousand times. For high-resolution imaging at magnifications of >300,000×, we actually 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 overfocus 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. CALIBRATION OF THE IMAGING SYSTEM

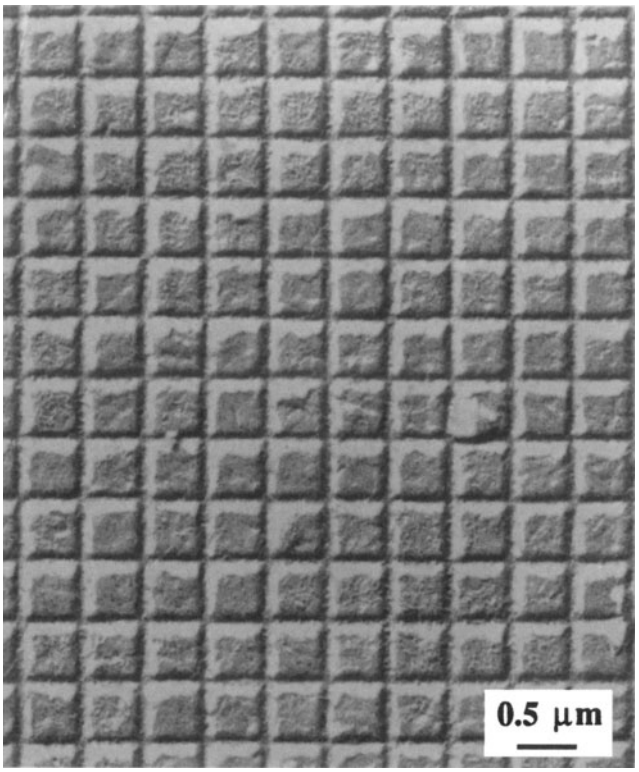
Your TEM should be calibrated when it is first installed and then periodically throughout its life, especially if you wish to carry out accurate measurements from images or diffraction patterns. If the instrument is modified substantially, then it must be recalibrated. In all cases you must specify a set of standard conditions under which the calibrations are carried out (e.g., objective lens current and other lens settings, eucentric height, etc.).

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 specimens we use are thin carbon film replicas of optical diffraction gratings of known spacing, such as shown in Figure 9.21A. The typical linear density

A



B

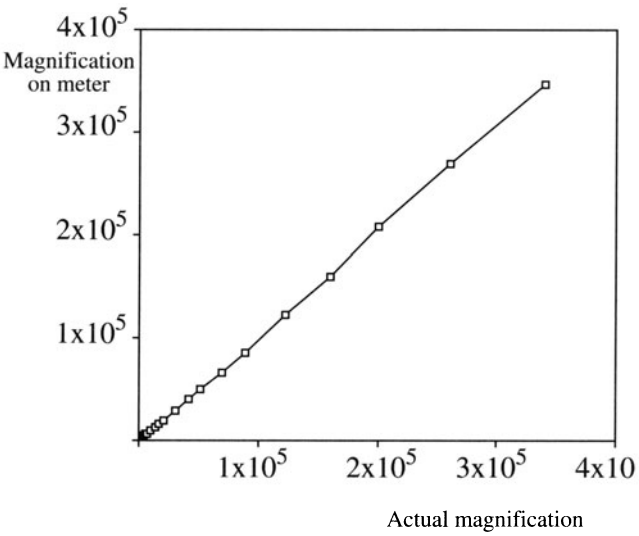


Figure 9.21. (A) An image of a diffraction grating replica in which the actual spacing of the grating is known. (B) The TEM magnification can thus be calibrated, relating specific magnification settings to be assigned specific magnifications.

of lines in the replica is 2160 lines per mm (giving a line spacing of 0.463 µm), which enables calibration up to magnifications of about 200,000×. Above this magnification, individual grating spacings are wider than the film. So we 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, the images of known crystal spacings, such as the 0002 spacing in the graphite structure (0.344 nm), can be used. 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 later in the text.

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 on the same negative.

Basically, you set the TEM to its standard conditions with the specimen at the eucentric height and focus the image. Next, record images of the diffraction grating at all magnification settings and calculate the magnification experimentally from the image. Figure 9.21B shows the magnification calibration for a Philips CM30 TEM, using both a diffraction grating and latex spheres.

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, with the efficiency of the cooling system of the lenses, and with lens hysteresis. Therefore, if you want to make accurate measurements from 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 distortions in the image, particularly at low magnification (<5000×). You can find a full description of all the details concerned with TEM calibration in Edington (1976).

Because of the magnification error in the TEM, it is not the best instrument for absolute measurement of parti-

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

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

cle sizes, etc. However, relative measurement is easily done with reasonable accuracy (±5%), so long as you note the precautions we've just described. Without a calibration the digital readout is probably no better than ±10% accurate, and so it is unwise to state magnifications to better than ±10%. You should be suspicious of any micrographs that you see in the literature with a magnification that is more precise than this (e.g., 52,700×). It may indicate that the microscopist does not understand the instrument's limitations and the work should be interpreted with due caution.

Remember that the electromagnetic lenses have hysteresis and the area of the specimen you are working on must be at exactly the right "height" in the column.

You can use an identical procedure to calibrate the STEM CRT image magnification. This is equally important 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 readout of the STEM magnification and the experimentally determined magnification using a diffraction grating replica.

9.6.B. Camera-Length Calibration

We describe the magnification of the diffraction pattern 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 these cameras magnification is increased by moving the recording film further away from the specimen. This principle can be applied in the TEM, as shown in Figure 9.22. This figure represents the imaging system, but without the lenses drawn in.

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 in the TEM is thus 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), then the separation of the direct and diffracted beams as measured on the screen (R) is determined by L since

$$\frac{R}{L} = \tan 2\theta \sim 2\theta \qquad [9.1]$$

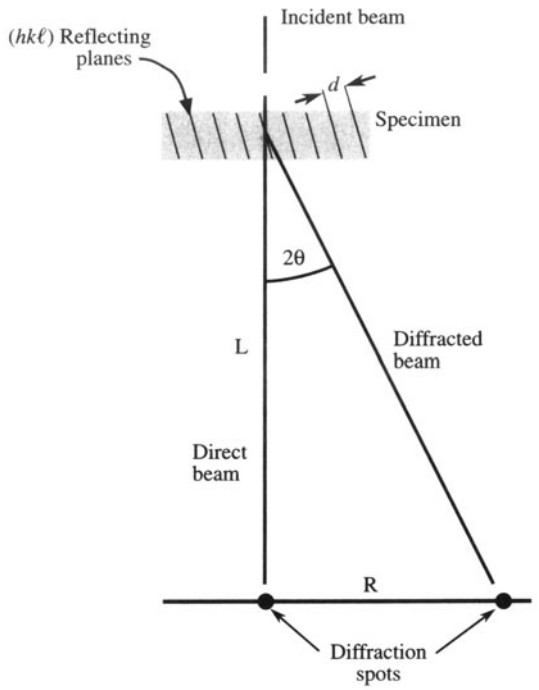


Figure 9.22. The relationship between the spacing R of diffraction maxima and the camera length, L. Increased magnification corresponds to effectively increasing L, although in practice this is accomplished with lenses.

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

$$Rd = \lambda L \qquad [9.2]$$

Thus to calibrate the magnification of the diffraction pattern we need to record patterns from a specimen with known crystal spacing (d), such as a thin film of a polycrystalline Au or Al. This gives a ring pattern (see Figure 2.11). We know the lattice parameter of the specimen, we can measure the ring radius R on the photographic film for any plane that is diffracting (see Chapter 18 to find out exactly how we do this), and since we know λ we can determine L. A typical TEM camera length calibration is shown in Table 9.2. The STEM camera length calibration may be different than 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.

As a general rule, you should *always* do the calibrations yourself, and not rely on any factory calibrations, because the conditions you use in your laboratory may differ from those of the manufacturer.

Table 9.2. Comparison of Experimentally Measured Camera Length (and Camera Constant) with the Digital Readout for a Philips EM400T Operating at 120 kV (λ = 0.0335Å)

Camera length Setting	Digital readout (mm)	Measured camera length, L (mm)	Camera constant λL (mm Å)
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

9.6.C. Rotation of the Image Relative to the Diffraction Pattern

Anyone studying crystalline materials must determine the angle between directions in the image and directions in the diffraction pattern. At a fixed camera length, the diffraction pattern 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 diffraction pattern. (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 common directions.)

To determine this rotation, we use a specimen of α-MoO_3, because it forms thin asymmetric crystals with a long edge known to be parallel to the 001 direction in the crystal. You must take care to ensure that, as usual, the image is focused with the specimen at the eucentric plane. Then insert the SAD aperture and ensure that it is focused using the intermediate lenses to coincide with the image plane. Finally, switch to diffraction mode with the beam underfocused and adjust the diffraction focus to give sharp diffraction maxima. Then, take a double exposure of the diffraction pattern and the image as shown in Figure 9.23A. Repeat the whole exercise for different magnifications and plot out the variation of the angle Φ as shown in Figure 9.23B. You can do the same if necessary for different values of L, which introduce a systematic change in Φ. It is recommended that you carry out all your SAD work at a standard value of L; 500–1000 mm is usually optimum.

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 diffraction pattern 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 9.24.

9.6.D. Analysis of TEM Images and Diffraction Patterns

If you don't use a double exposure when comparing images and diffraction patterns (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.

> Whenever you're comparing images and diffraction patterns, 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 diffraction patterns.

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

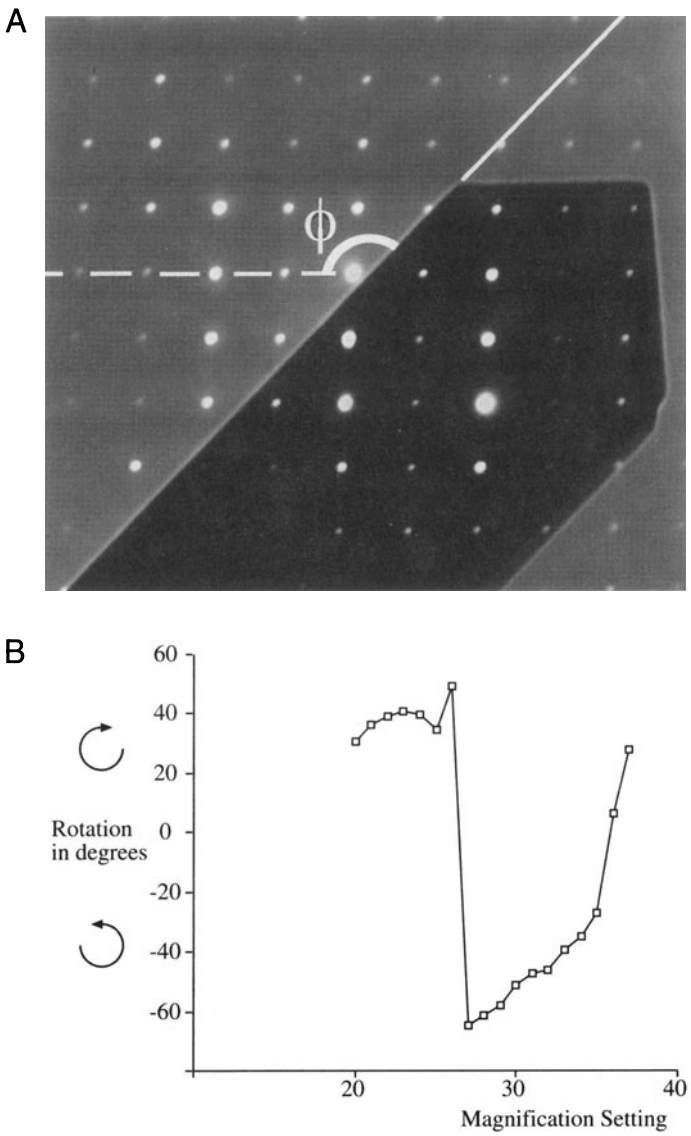


Figure 9.23. (A) A double exposure showing the superposition of an image of a MoO_3 crystal on a diffraction pattern from the same crystal, defining the rotation angle Φ. (B) The rotation calibration gives the angle Φ between equivalent directions in the image and the diffraction pattern as the magnification is varied. The calibration assumes a constant camera length.

measure the electron wavelength λ by measuring the angle ϕ between two Kikuchi line pairs (see Chapter 19) which intersect at a distance R from the direct beam

$$\tan\phi = \frac{R}{L} = \frac{\lambda}{d} \qquad [9.3]$$

Alternatively, you can match simulations of higher-order Laue-zone lines to experimental lines in CBED patterns (see Chapter 21) and determine which λ gives the best match. Finally, if you can get your X-ray computer system

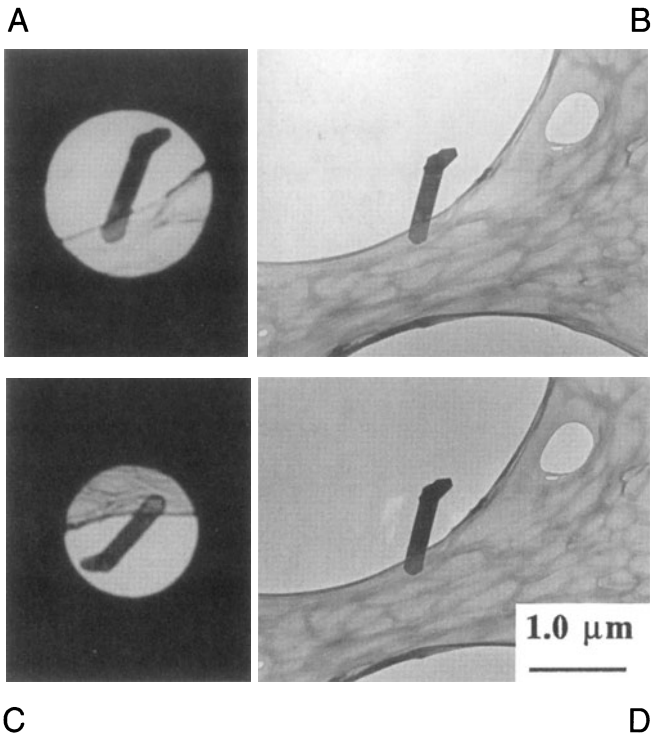


Figure 9.24. Defocused direct beam in a diffraction pattern from MoO_3 compared with a BF image, showing how to determine if a 180° inversion exists or not. If the image in the spot is rotated with respect to the image on the screen, as in (C) and (D), then the 180° inversion is required. In (A) and (B), no rotation occurs between the DP and BF image.

to display the X-ray spectrum (Chapter 34) out to E_0, the beam energy, the bremsstrahlung intensity vanishes to zero at the exact beam energy (this is called the Duane-Hunt limit).

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 often rotated relative to the specimen, how can you locate the rotation axis? Move the specimen. From this movement, you can determine the sense of tilt for a specimen of known geometry.

> If you gently push on the end of your side-entry specimen holder, the image moves parallel to the principal tilt axis.

If you are looking at the diffraction pattern, defocus the pattern so you can see the BF image in the zero spot, as in Figure 9.25, 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.

Focal increments of the objective lens: If you're going to do high-resolution phase-contrast imaging, then

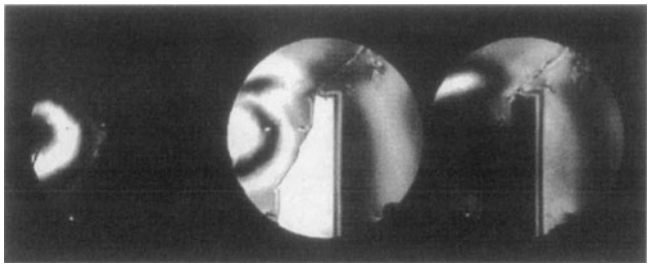


Figure 9.25. Defocused multiple DF image showing how it is possible to determine simultaneously the direction of features in the image (e.g., the vertical twin boundary) and directions in the DP (e.g., the horizontal vector between the diffraction disks). If the specimen holder is moved in the direction of the principal tilt axis, the image will move and identify the relationship between that tilt axis and the DP.

you need to know the value of each defocus step of the objective lens. 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

$$\Delta f = \frac{\Delta x}{2Mm\theta} \qquad [9.4]$$

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 use 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 a pretty good job, 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 C2 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 is 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 the astigmatism at high magnification. Diffraction and STEM operation require a centered diffraction pattern.

If you want to make any quantitative measurements from your images and diffraction patterns (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 diffraction patterns are relatively useless unless you know their magnification (camera length) 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.

REFERENCES

General References

Chapman, S.K. (1980) *Understanding and Optimizing Electron Microscope Performance 1. Transmission Microscopy,* Science Reviews Ltd., London.

Chapman, S.K. (1986) *Maintaining and Monitoring the TEM,* Royal Microscopical Society Handbook, No. 8, Oxford University Press, New York.

Chescoe, D. and Goodhew, P.J. (1990) *The Operation of Transmission and Scanning Microscopes,* Royal Microscopical Society Handbook, No. 20, Oxford University Press, New York.

Specific References

Edington, J.W. (1976) *Practical Electron Microscopy in Materials Science,* Van Nostrand Reinhold, New York.

Goldstein, J.I., Newbury, D.E., Echlin, P., Joy, D.C., Romig, A.D. Jr., Lyman, C.E., Fiori, C.E., and Lifshin, E. (1992) *Scanning Electron Microscopy and X-ray Microanalysis,* Plenum Press, New York.

LePoole, J.B. (1947) *Philips Tech. Rundsch.* **9**, 93.