7

How to 'See' Electrons

CHAPTER PREVIEW

If we are studying the structure of a material, when all is said and done, all we have to show for learning how to operate our expensive TEM, the many hours spent in specimen preparation, etc., is an image or a DP. These images and DPs, which are just different distributions of electron intensity, have first to be viewed in some manner. After viewing, we have to decide if we want to save the results for future reference, perhaps so we can print out the data for a presentation, technical report, or scientific publication. Since, as we noted in the opening chapter, our eyes are not sensitive to electrons, we have to find ways to translate the electron-intensity distributions generated by the specimen into visible-light distributions, which we can see. This chapter will explain how we 'see' electrons.

We'll break the process down into two parts: first, detection (and display) of the image, and second, recording of the image. Both these areas are undergoing rapid change because of ongoing advances in electronic imaging and storage technology, and so this chapter will undoubtedly contain anachronisms by the time you read it. In particular, numbers are favored over photographic data; how can we quantitatively compare two photographs? Comparing two sets of numbers is routine.

7.1 ELECTRON DETECTION AND DISPLAY

As we saw back in Figure 2.1, images and DPs are different kinds of two-dimensional, electron-density distributions which are produced when a thin specimen scatters electrons. We detect and display them in different ways depending on whether we are using a TEM or STEM, as we'll explain in Chapter 9. In a conventional TEM, the images and DPs are static, because the incident beam is fixed, and so we can easily project them onto a viewing screen within the microscope column. TEM images, for example, are *analog* images of electron-density variations in the image plane of the objective lens. We cannot manipulate the image or its contrast in any way between the electrons leaving the image plane and being projected onto the viewing screen. So we will briefly discuss the properties of the viewing screen. The manufacturer controls the choice of screen materials so you might think there's not much need to understand this aspect in any depth. You might be surprised by the limitations you don't need to accept or the improvements which could be made.

When we operate our TEM as a STEM, or we use a dedicated STEM, the image is not static; it is built up over time as the small probe is scanned across the area of interest. Under these circumstances, we can detect the electron signals in several ways. If we are seeking secondary electron (SE) or backscattered electron (BSE) signals, then these detectors sit in the specimen stage area. If we are seeking the same forward-scattered electrons that we view on the TEM screen, the detectors are in the viewing chamber of the TEM. After we've detected any one of these signals, it is usually digitized and the *digital* scanning image is presented on a fluorescent screen as an analog image. You may hear this fluorescent screen referred to as the CRT, which are the initials for cathode-ray tube and a relic from the early days of electron physics. It is becoming much more common for the image or DP to be displayed on a flat-panel screen beside the main TEM column (or even on a plasma or LCD screen on the wall of the EM lab) controlled by the TEM's computer.

We should point out that the sequential or serial nature of the scanning image makes it ideal for on-line image enhancement, image processing, and subsequent image analysis. The signal from any electronic detector can be digitized and electronically manipulated prior to display on the CRT or computer screen, in a way that is impossible with analog images. We can adjust the digital signal to enhance the contrast or to reduce the noise. Alternatively, we can store the digital information and process it mathematically. The availability of cheap memory and

fast computers permits on-line image processing and the rapid extraction of quantitative data from the scanning image; we discuss all this and more in Chapter 31. Because of developments in computer technology, there is great interest in recording analog TEM images via a TV camera in order to digitize them; charge-coupled device (CCD) cameras are readily available for on-line viewing and processing, particularly of HRTEM images. CCD technology is advancing rapidly, driven largely by the digital-camera market and microscopists will continue to benefit from the availability of ever-larger CCD detectors. So we'll spend part of this chapter on CCDs which you'll have now worked out are equally sensitive to visible light and high-energy electrons.

In attempting to compare the properties of detection and recording devices we often use the concept of the 'detection quantum efficiency' or DQE. If a detector is linear in its response then the DQE is defined simply as

$$\mathrm{DQE} = \frac{\left(\frac{S_{\mathrm{out}}}{N_{\mathrm{out}}}\right)^2}{\left(\frac{S_{\mathrm{in}}}{N_{\mathrm{in}}}\right)^2} \qquad (7.1)$$

where S/N is the signal-to-noise ratio of the output or input signal. So a perfect detector has a DQE of 1 and all practical detectors have a DQE <1.

Note on terminology: We use several different terms, often imprecisely, to describe how we 'see' electrons. Since our eyes can't in fact see electrons, we have to resort to the phenomenon of cathodoluminescence (CL) (which we introduced back in Section 4.4) in order to provide an interface between electrons and our eyes. Any electron display system that we look at relies on CL at some point. The CL process converts the energy of the electrons (cathode rays) to produce light (luminescence). As a result, any electron display screen emits light in proportion to the intensity of electrons falling on it. A few definitions are in order

- *Light emission* caused by ionizing radiation is *scintillation*.
- The process of *fluorescence* implies *rapid emission*.
- *Phosphorescence* implies that the wavelength and the *delay time* are longer than for fluorescence.

All these terms are used in electron microscopy (interchangeably and often inaccurately) because the 'fluorescent' screen is coated with a long-delay phosphor (see Chapter 9).

7.2 VIEWING SCREENS

The viewing screen in a TEM is coated with a material such as ZnS, which emits light with a wavelength of ~450 nm. The ZnS is usually modified (doped) to give off green light at closer to 550 nm; hence you'll see screens of different shades of green which, being in the middle of the visible spectrum, is most relaxing for your eyes. As long as sufficient light is emitted, the main requirement of the viewing screen is that the ZnS particle (grain) size be small enough so that your eye cannot resolve individual grains. This means that grain sizes < 100 μm are acceptable (although you can see the grain size if you look at the screen through the auxiliary focusing binoculars). Typical screen coatings are made with a ZnS grain size of ~50 μm, although they may be as small as 10 μm for the highest-resolution screens.

As we've seen in Chapter 4, the cross section for inelastic interactions (and hence the emission intensity of most signals, including CL) decreases with increasing beam voltage. You would thus expect the light intensity to degrade at higher voltages, but this is offset by the increase in gun brightness. In some HVEMs the support for the small focusing screen is made of a heavy metal such as Pt to enhance backscatter and increase screen intensity, Of course, this backscattering will broaden the volume where light is generated and blur the image, so we don't gain very much. In fact most TEMs have very similar screens. Other signals are also given off by the viewing screen, such as X-rays, and whenever you look at the screen you are protected from this lethal radiation flux by lead glass, which is carefully selected to reduce transmitted radiation to levels at or below ambient background. In HVEMs this can amount to several tens of millimeters of glass and, invariably, the optical transmission capabilities are degraded as the glass gets thicker, but obviously we have no alternative if we want to view the screen directly.

A FEW WORDS OF CAUTION ABOUT YOUR SCREEN

There isn't much you can do about choosing the best material for the viewing screen since the manufacturer selects it for you, but you can extend its life substantially by taking care to minimize overexposure. The greatest source of screen damage is the intense direct beam that comes through thin specimens and constitutes the central spot in DPs. Using what you'll learn about operations of the TEM in Chapter 9, you can minimize burning of the screen by (a) only going to diffraction mode with the selected-area aperture inserted, (b) only going to diffraction mode with the C2 lens overfocused, and (c) if the spot appears exceptionally intense despite these precautions, then insert the beam stop while you're observing the pattern on the screen (but not when recording it).

While it is surprising that a modern TEM still relies on an analog screen, the end is perhaps already in sight. One of the latest TEM models (go back and look at Figure 1.9) is built without an operator's viewing screen; all the information is shown on flat-panel computer displays on a console that is separate from the column. Such a design breaks away from more than 70 years of TEM design but has the distinct advantages that

- Anyone in the room (or indeed anyone connected via the Internet) can see the images and DPs, which creates a much better teaching environment.
- The lights don't have to be out to view and record the information.
- The TEM column can be placed in a room that is separated from the operator, whose presence invariably reduces the resolution capabilities of the highest-performance microscopes.

Moving to digital display and recording brings with it the possibility of processing the image or DP to enhance or suppress information prior to publication or presentation. There are obvious ethical considerations here since the scientific community expects that published data be presented with sufficient background information that others would be able to reproduce and cross-check the experiment. So if you process digital images it is wise to publish the unprocessed data at the same time so others can see what data processing has been used. We'll talk a lot more about such ethical issues and related topics when we discuss image processing in Chapter 31.

7.3 ELECTRON DETECTORS

We have several alternatives to the fluorescent screen for detecting electrons. These other electron detectors play a major role in STEMs and AEMs (as well as in SEMs). They are actually essential to the STEM image-forming process that we'll describe in Chapter 9. Such detectors are usually one of two kinds: semiconductor (Si p-n junction) detectors or scintillator-photomultiplier systems. We'll examine the pros and cons of each of these two types and end with a section on CCDs.

7.3.A Semiconductor Detectors

A full understanding of how semiconductor detectors work requires a fair knowledge of solid-state physics. We'll just give a brief outline of the principles as they affect the use of the TEM but if you want to dig deeper the place to start is the excellent text by Pierret.

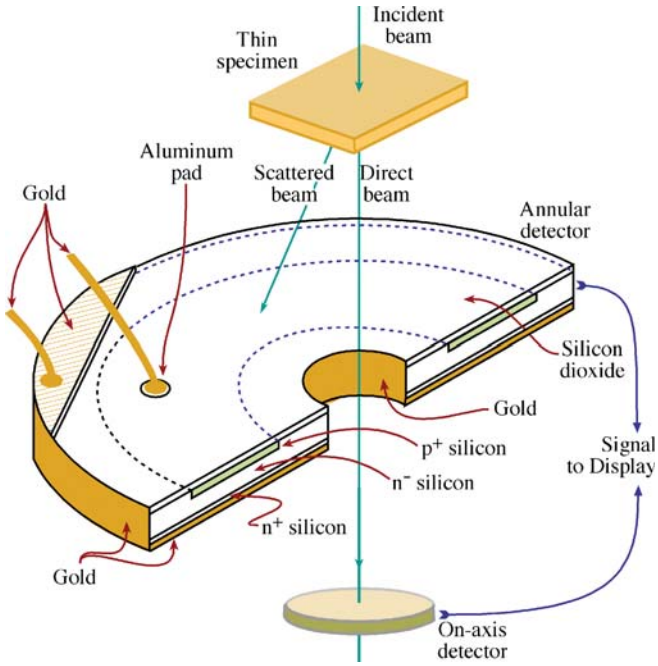

FIGURE 7.1. Semiconductor detector of the surface-barrier type, shown in a configuration where it would be used to detect high-energy, forward-scattered electrons. The direct beam is detected by a small circular detector on the optic axis of the microscope surrounded by a concentric wide-angle annular detector, which detects any scattered electrons.

The semiconductor detector, shown schematically in Figure 7.1, is a doped single-crystal sheet of Si (often inaccurately described as a solid-state detector). We make the Si into an electron-sensitive detector by creating a p-n junction beneath the Si surface in one of two ways. In one type of detector, we create the junction by doping the Si (e.g., by ion implantation of n-type impurity atoms into p-type Si or vice versa). This doping disturbs the equilibrium charge carrier concentration and creates a region across the p-n junction that is free of majority carriers which we call a 'depletion region.' A conducting metal layer is evaporated onto both surfaces to provide ohmic contacts. The alternative type of detector is called a surface-barrier detector (or sometimes a Schottky diode) and we fabricate this by evaporating a thin layer of Au on the surface of high-resistivity n-type Si, or evaporating Al onto p-type Si. This surface layer acts as an electrical contact and also creates a depletion layer and a p-n junction just inside the Si.

When we put either of these detectors into a beam of high-energy electrons, most of the beam energy is transferred to valence-band electrons in the Si which are excited across the band gap into the conduction band thus creating electron-hole pairs (see Figure 4.8). We can separate the electrons and holes most efficiently by applying an external reverse bias to the detector; that is, we put a negative bias on the p side of the junction and a positive bias on the n side. In practice, however, so many electron-hole pairs are created at TEM beam energies

that an external bias is not usually necessary, and the internal bias of the p-n junction acts to separate the electrons and holes. Because the electrons and holes move quite quickly in Si, it takes only a few nanoseconds to gather most of the carriers over an area of $\sim$1 μm^2. So the semiconductor detector is remarkably responsive to electrons. The net result is that the incoming electron signal is converted to a current in the external circuit between the surface contacts, as shown in the surface-barrier detector in Figure 7.1.

Since it takes approximately 3.6 eV to produce an electron-hole pair in Si at room temperature, a 100-keV electron can theoretically produce $\sim$28,000 electrons. This represents a maximum detector gain of close to 3×10^4 but in practice there are losses due to electron absorption in the metal contact layer and recombination of the electrons and the holes close to the Si surface (in a region called the dead layer), and we actually get a gain of closer to 2×10^4.

These semiconductor detectors are very efficient at picking up and amplifying electron signals. Unfortunately, they have an inherently large capacitance, so they are not very responsive to rapid changes in signal intensity. Such changes are quite likely to occur during the rapid scanning process of STEM imaging. In other words, the detector has a narrow bandwidth (typically 100 kHz); this is not a good property for a detector which is subject to widely varying signal intensities. We could lower the capacitance by decreasing the detector area, but if we do this, the signal-to-noise ratio will be lowered. It is the S/N ratio that ultimately limits the quality of all scanning images.

Semiconductor detectors have several advantages

- We can easily fabricate them.
- They are cheap to replace.
- They can be cut into any shape, as long as it is flat.

This latter advantage makes them ideal for squeezing into the confines of TEM stages and columns. For example, we can make the semiconductor detector in annular form so that the main electron beam goes through the hole in it, but the scattered electrons are very efficiently detected. This produces a dark-field (scattered electron) detector. We can also make detectors that are divided into halves or quadrants and each segment is insulated from the other(s). These detectors are very useful for discriminating directional signals such as those coming from magnetic specimens.

There are also some drawbacks to semiconductor detectors

- They have a large dark current (the current registered when no signal is incident on the detector). This dark current arises from thermal activation of electron-hole pairs, or from light falling on an uncoated detector. Since the detectors in a TEM invariably have a metal ohmic contact, the light problem is minimal because light can't penetrate the metal film. Now we could minimize thermal activation by cooling the detector to liquid-nitrogen temperatures but that step is impractical and introduces a cold surface into the vacuum which would simply collect contamination, so we live with noise due to the thermal activation.
- Because noise is inherent in the semiconductor detector, its DQE is poor for low-intensity signals, but rises almost to unity for high-intensity signals.
- The electron beam can damage the detector, particularly in intermediate voltage microscopes. In these circumstances, a doped p-n detector is less sensitive than a surface-barrier detector, because the depletion region is deeper in the Si.
- They are insensitive to low-energy electrons such as secondary electrons.

Despite these drawbacks, both types of Si detector are far more robust than the alternative scintillator, which we will now describe.

7.3.B Scintillator-Photomultiplier Detectors/TV Cameras

A scintillator emits visible light when struck by electrons because of the same CL process that occurs in fluorescent screens. While we are viewing a static TEM image, we want the fluorescent screen to continue emitting light for some time after the electrons hit it, so we use a long-delay scintillator. Of course, when we are using a scintillator to detect rapid changes in signal as in scanning beam imaging, we want the light emission to decay rapidly. So we don't use ZnS in scintillator detectors but rather materials such as Ce-doped yttrium-aluminum garnet (YAG) and various doped plastics and glasses. These materials have decay times on the order of nanoseconds rather than the microseconds needed for ZnS. Once we've converted the incoming electron signal to visible light, the light from the scintillator is amplified by a photomultiplier (PM) system, attached to the scintillator via a light pipe. Figure 7.2 shows a schematic diagram of a scintillator-PM detector setup to detect secondary electrons in a TEM, but the design used to detect primary scattered electrons in the STEM is essentially identical.

The scintillators that we use in STEMs or SEMs are often coated with a 100-nm-thick layer of Al to reflect any light generated in the microscope and stop it from entering the PM tube where it would add noise to the signal. If the detector is in the stage of the microscope, this light could come from the specimen itself if it is cathodoluminescent, or it could be light coming down

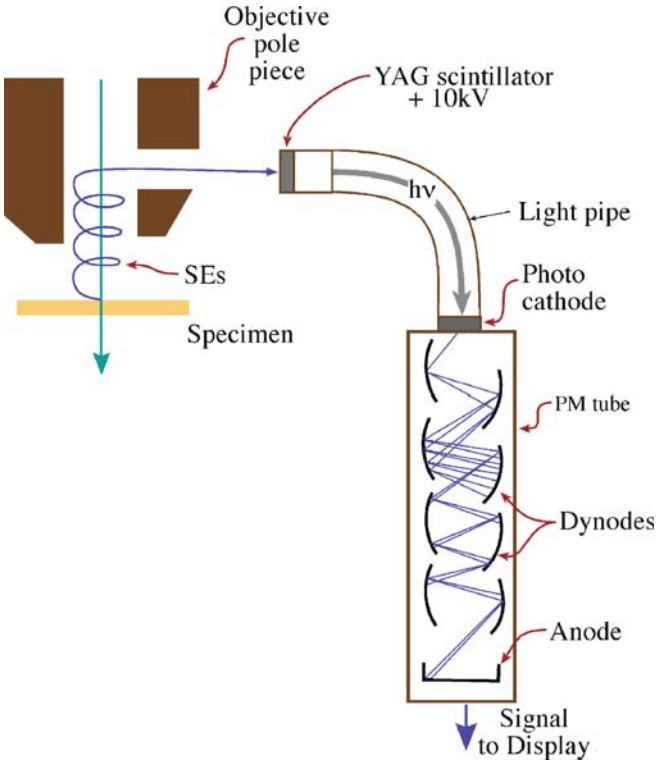

FIGURE 7.2. Scintillator-photomultiplier detector system for SE detection in a TEM. SEs from the specimen spiral back up through the objective lens polepiece and are accelerated by the high voltage onto the scintillator, generating visible light which travels via fiber optics to a photocathode. There the light is re-converted to electrons. The electron signal is then multiplied by several electrodes (dynodes) in the PM tube before being used to modulate the display screen.

the column from a thermionic source and reflected from the polished surface of the specimen. If you have an uncoated scintillator detector in the viewing chamber, then room light may also hit the detector, so you should cover the windows of the viewing chamber.

The advantages of the scintillator-PM system are

- The gain of the system is very high. The gain for the total detector system is of the order of 10^n, depending on the number (n) of electrodes (often called dynodes) in the PM. A value of 10^8 is not unusual (compare with $\sim 10^4$ for the semiconductor detector). This performance is reflected in a typical DQE of close to 0.9 for several commercial scintillators.
- The noise level in a scintillator is low compared with semiconductor detectors, and the bandwidth of the scintillator is in the MHz range. As a result, both low-intensity images and TV-rate images are easily displayed. There is a tremendous practical advantage to TV-rate imaging of digital signals, because such images, when suitably processed and displayed can be viewed, stored, and recorded under normal conditions of room illumination. So you don't have to work in the dark while operating your (S)TEM.

The disadvantages of the scintillator-PM system are

- The scintillator is not as robust as the semiconductor detector, being even more susceptible to radiation damage, particularly after longtime exposure to the beam.
- The scintillator-PM combination is also substantially more expensive and bulky compared to semiconductor detectors and therefore it does not fit well within the TEM stage, nor is it easily manufactured into multi-detector configurations; it is also more expensive. However, plastic scintillators can be shaped to give large collection angle, such as the Robinson BSE detector used in many SEMs.
- The energy-conversion efficiency of a scintillator is also rather low ($\sim$ 2%–20%) compared to a semiconductor detector and, typically, we only get about 4000 photons per incident 100-keV electron, $\sim 7\times$ less than for the semiconductor detector. This low efficiency is offset by the gain in the PM tube.

On balance, the scintillator-PM detector is preferred over the semiconductor detector for most general electron detection in TEM/STEM systems. However, you must take care to minimize any high-intensity beams that may damage the detector and lower its efficiency. Therefore, you need to take more care when operating scintillator detectors.

We've already mentioned that you can view the TEM image directly through a TV camera, rather than looking at the fluorescent screen. There are real advantages to TV cameras, e.g., for on-line viewing of faint HRTEM images (see Chapter 28) or for recording of dynamic in-situ events (see Chapter 29). Also, from a teaching standpoint, anything that pulls the TEM image, in real time, out of the viewing chamber (which only the operator can see into clearly) and onto a classroom or laboratory computer screen or plasma display makes life so much easier. With the increased interest in telemicroscopy, TV cameras and webcams are becoming much more common within the TEM room (e.g., URLs #1 and #2). TV cameras attached to TEMs come in both analog and digital forms. Typically the cameras are placed below the viewing screen so you have to lift up the screen to detect the TV image. The camera may have to be offset if there is another post-column attachment such as an EELS system. Sometimes the camera is placed within the column and is then moved on axis when needed.

Analog YAG-based scintillator TV cameras may be used for applications requiring image intensification if you are dealing with faint images (e.g., if you are using low-dose techniques because your specimen would otherwise be damaged by the beam). Also, wide-angle

cameras are available that collect a much greater area of the image or DP than the standard TEM photographic plate. However, the most widely used TV cameras use only digital-detection technology, which we'll now discuss.

7.3.C Charge-Coupled Device (CCD) Detectors

Electronic technology for recording images and spectra is rapidly closing in on the more traditional analog methods. CCD cameras are becoming the norm for real-time TV recording of images and DPs. They are also being used for two–dimensional arrays for parallel-collection EELS and energy-filtered images, as we describe in Chapter 37.

CCDs are metal-insulator-silicon devices that store charge generated by light or electron beams. CCD arrays consist of several mega- (millions of) pixels which are individual capacitors electrically isolated from each other through the creation of potential wells under each CCD cell, so they can accumulate charge in proportion to the incident radiation intensity, as shown in Figure 7.3A. The largest CCD arrays, as of writing this text are giga-pixels (10^9). Because such systems are so expensive, they are typically developed for use in major astronomical telescopes for detecting very faint light sources. In fact there isn't a good reference text for CCD use in electron microscopy but there is a great one by Howell for astronomy that gives excellent background material if you are interested in digital-image recording.

The maximum size of CCD currently available for TEM use is 4k×4k but the size will only increase with time. (It is also possible to stitch together multiple CCD images using software so, if you have the time, the size of the CCD itself is not a serious issue.) The individual cells currently can be as small as 6 μm although a more typical size range is ~10–15 μm. To create a picture, we have to read out the array. We do this by changing the applied potentials to transfer the charge serially from each potential well along a line in the array into an output amplifier, as shown in Figure 7.3B. With good design of the electrodes, charge-transfer efficiencies of 99.999% can be achieved. Once all the cells are empty the array can be re-exposed. This so-called 'full-frame' design is simple and robust and offers the highest resolution and highest pixel density.

Rather than serial readout of full-frame CCDs, it is also possible to have frame transfer CCDs in which the whole frame is transferred to an adjacent storage array leaving the main array free to collect a new signal flux. This method allows for shorter frame times, and thus a faster image acquisition, but is much more complex and, since more of the device is taken up with the storage, the CCD has lower resolution and much higher cost. The typical frame times needed to record TEM images are long enough that high-speed frame transfer is not usually needed.

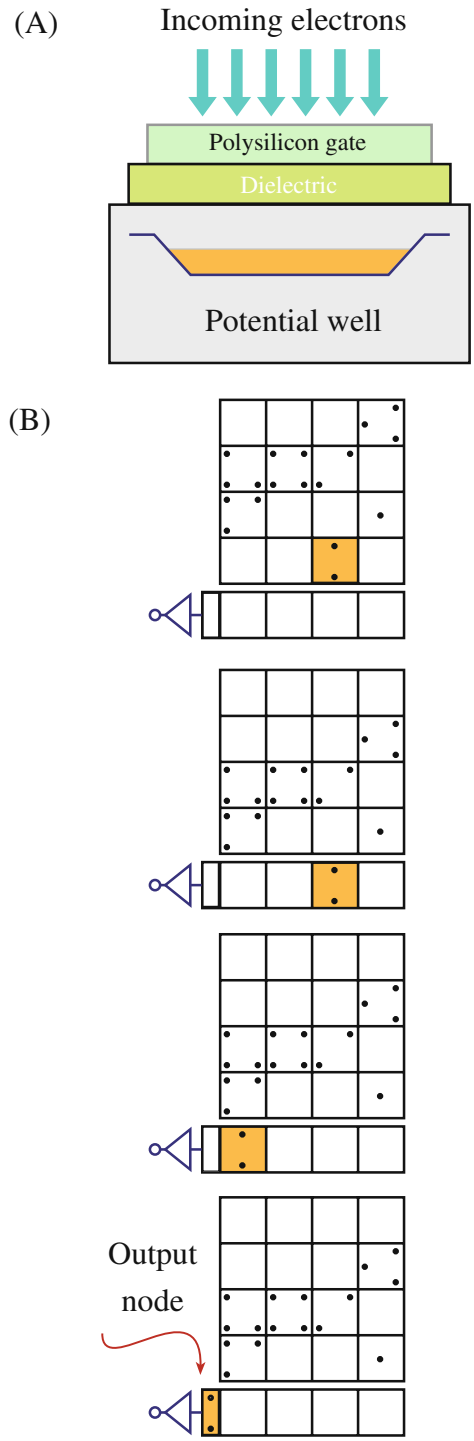

FIGURE 7.3. (A) A single cell in a CCD array showing the storage of charge in the potential well under one pixel. If we vary the applied potential to rows of pixels in sequence as in (B), one pixel row is shifted to the parallel register and is read out pixel by pixel, after which the next row is moved to the parallel register, and so on. The stored charge in each pixel is thus fed into an amplifier and digitized.

The frame time for reading the CCD depends on the size of the image and the specific technology used to readout the detected signal. Ultrahigh-speed CCD cameras are available with $>10^5$ frames/second, but such high speeds are not essential in standard TEM. It is worth noting, however, that time-resolved TEM is an area of growing importance and, in such dedicated instruments, ultra-fast recording is required. Routinely, frame times of <0.001 seconds, well below standard TV rates of 0.033 seconds, are available for in-situ recording of fast events. But the frame time can equally well be several minutes (e.g., for picking up diffuse scattering in faint DPs). Obviously, the longer the exposure, the more the image is susceptible to external vibration, drift, etc.; so long exposures, e.g., for HRTEM images, are not good.

CCD detectors have several advantages

- When cooled, they have very low noise and a good DQE (>0.5) even at low input signal levels.
- The dynamic range is high, making them ideal for recording DPs which can span an enormous intensity range.
- They respond linearly to changes in input signal and show a fairly uniform response across many pixels.

There are some drawbacks to CCDs, not least of which is their cost, but that is always decreasing, as with any Moore's-law-based technology. However, 'blooming,' which occurs when too much signal fills up the pixel and the signal overflows into surrounding pixels, can be a problem. This problem can be overcome substantially by building anti-blooming or overflow drain structures within the device. Apart from these minor factors it is clear that, in the end, CCDs, or other electronic technology, will eventually record and store all TEM images, DPs, and spectra.

7.3.D Faraday Cup

In conventional TEM there isn't much need to know the beam current, but for X-ray analysis in the AEM, it is essential, since there is often a need to compare analytical results obtained under identical beam current conditions. A Faraday cup is a detector that simply measures the total electron current in the beam. We don't use it for any imaging process, but rather as a way of characterizing the performance of the electron source as we saw in Chapter 5. Once the electrons enter the Faraday cup, they cannot leave except by flowing to ground through an attached picoammeter that measures the electron current.

You can easily construct a Faraday cup to go in an SEM, but it is more difficult to design one that fits in the stage of a TEM. A dedicated Faraday cup holder is shown in Figure 7.4A. The entrance aperture is small and the chamber is relatively deep and lined with a low-Z material to minimize backscatter. If you tilt it slightly, the electrons have little chance of being scattered directly back. With such a holder you can only find the hole if you can image the upper surface with SE or BSE detectors, and if these are not available then you must have a cup with a hole in the lower surface too. When the cup is not tilted, the electrons go straight through; if you tilt the cup, then all the electrons are trapped as shown in Figure 7.4A. The way to ensure that you are measuring the maximum current is to look at the picoammeter reading as you tilt the cup. Some manufacturers now incorporate a Faraday cup in the specimen holder. You can measure the current by deflecting the beam into the cup or partially extracting the holder so the beam falls into the cup (Figure 7.4B).

FARADAY CUP

Remember: A Faraday Cup is a Black Hole for Electrons.

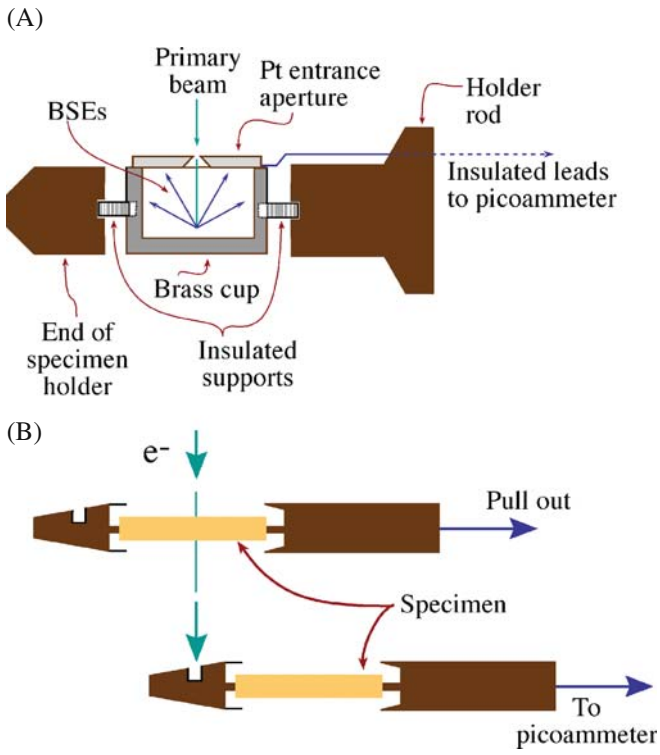

FIGURE 7.4. (A) Schematic diagram of a dedicated Faraday cup in the end of a side-entry specimen holder (more details about these holders in Chapter 8). The entrance aperture has to be found by imaging the top surface using SEs or BSEs. In (B) the specimen holder is retracted so the electrons fall into a cup on the tip of the holder (in which position of course the TEM image of your specimen cannot be seen). In either case, the electron current is measured as it goes to ground through a picoammeter attached to the outside of the holder.

If you don't have a Faraday cup, it is possible to get an approximate reading of the current by just measuring the current through an insulated line from a bulk region of your specimen and correcting for electron backscatter. Backscattering is independent of the accelerating voltage and approximately linear with atomic number up to about $Z = 30$. For example, the backscatter coefficient for Cu is about 0.3 and for Al is about 0.15. It is also possible to deflect the beam onto the last beam-defining diaphragm (see Chapters 6 and 9) and measure the current via an insulated feed-through (also correcting for backscatter).

7.4 WHICH DETECTOR DO WE USE FOR WHICH SIGNAL?

As we mentioned at the start of the chapter, the principal electron signals that we can detect are the forward-scattered electrons (which as we'll see in Chapter 9 form the most common TEM images) and the BSE and SE signals from the beam-entry surface of the specimen.

Semiconductor detectors are only sensitive to electrons with sufficient energy (>5 keV) to penetrate the metal contact layer. So we use these detectors mainly for *high-energy* forward-scattered imaging and *high-energy* BSE imaging. Because of the surface contact layer we don't use semiconductor detectors for *low-energy* SEs but instead use a scintillator-PM system. Remember that the scintillator may also be coated with Al to prevent visible light from generating noise. This coating would also prevent low-energy SEs from being detected. So for SE detection, either there must be no coating or the electrons must be accelerated to an energy high enough to penetrate the coating; we achieve the latter by applying a high kV (>10 kV) positive bias to the scintillator.

The capacitance is relatively high for semiconductor detectors so they are not the detector of choice in dedicated STEMs where high scan-rate TV images are the normal viewing mode, i.e., you need a quick response. The scintillator-PM system is again preferred under these circumstances. As most microscopes move toward TV-rate display of scanning images it is likely that the scintillator-PM will be used increasingly for forward-scattered TEM imaging. Semiconductor detectors may only be used for BSEs, which is not a major imaging mode in TEMs. A summary of all the various electron detectors in a TEM/STEM is given in Figure 7.5. We'll talk more about the methods of imaging in Chapter 9 and later in Part 3 but the primary detectors in (S)TEM pick up the forward-scattered electrons on-axis (called the bright-field (BF) detector), forward-scattered through small angles $<\sim 3^\circ$ (called the annular dark-field (ADF) detector), or scattered out to higher angles (called the high-angle annular dark-field (HAADF) detector).

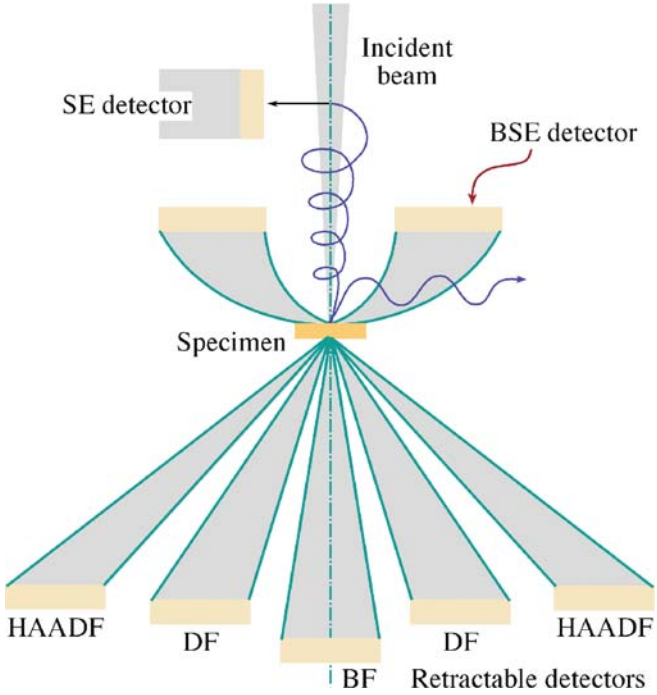

FIGURE 7.5. The various electron detectors in a STEM. Scintillator-PM detectors are invariably used for SE detection and semiconductor detectors for the BSE. The on-axis and annular forward-scattered and high-angle dark field detectors may be either type, depending on the microscope. The SE detector is rare, the BSE detector is a waste of time: only the forward-scattered electron detectors are standard.

Sometimes we examine specimens which themselves exhibit cathodoluminescence under electron bombardment. We discussed CL back in Chapter 4 and we'll give an example of why CL imaging might be useful in Chapter 29. A mirror is used to focus the light from the specimen into a scintillator-PM system; one design is shown in Figure 7.6. In this setup, the specimen must be moved and tilted until, in combination with the collimating lens, the maximum signal is detected by the PMT. This setup effectively prevents detection of most other signals, including X-rays, because the mirror occupies all the free space in the TEM stage. So you have to dedicate the TEM to CL detection alone and ignore other signals. There are only a few such CL-TEMs in the world.

7.5 IMAGE RECORDING

7.5.A Photographic Emulsions

Although photographic film is the oldest recording medium, it still retains sufficient advantages that we continue to use it in some TEMs (and most that are >10 years old). Photographic emulsions are suspensions of silver halide grains in a gel. Electrons strike the

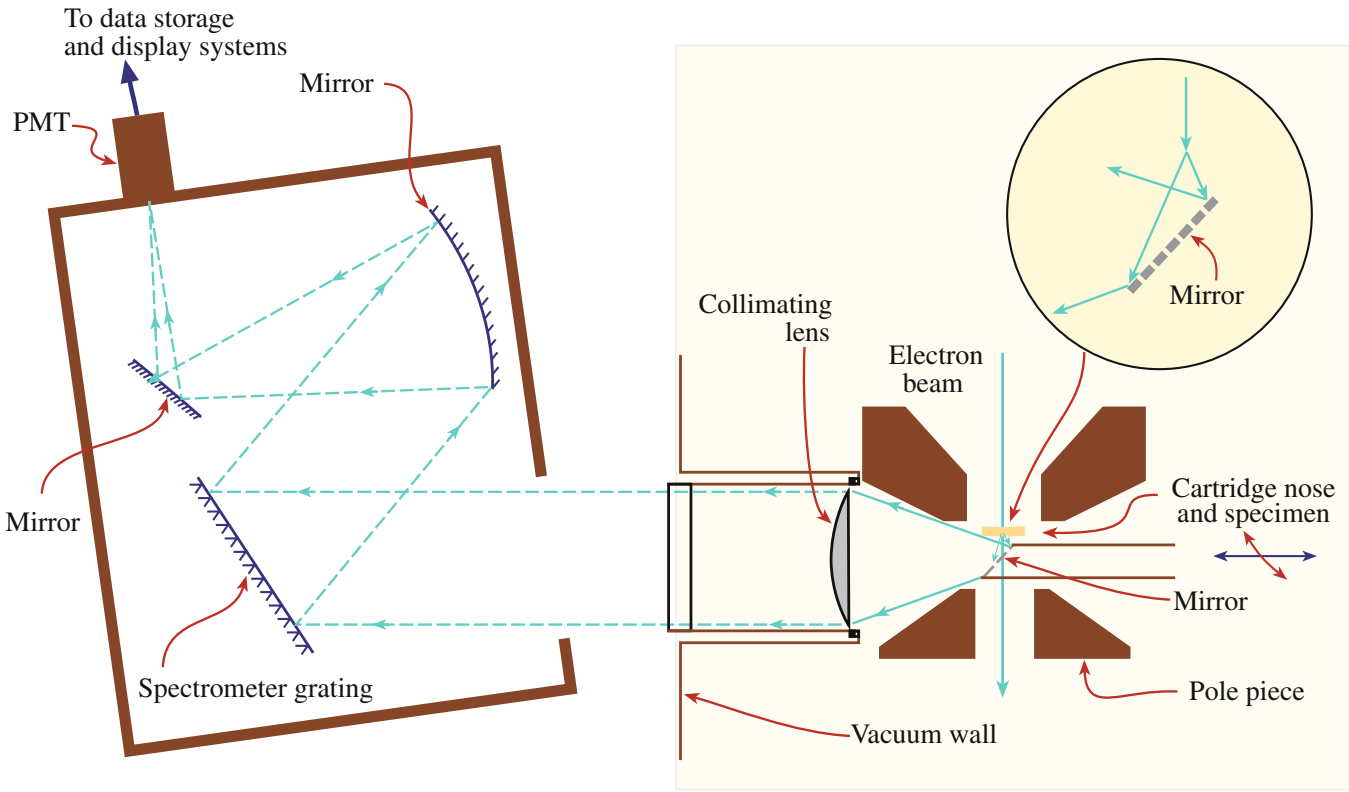

FIGURE 7.6. Cross-sectional diagram of a mirror detector below a thin cathodoluminescent specimen that collects a small fraction of the emitted light and focuses it via a collimating lens into a spectrometer-PM system. The CL signal is usually very weak to start with and so the detector has to be as large as possible, and it takes up much of the free volume in the TEM stage making it impossible to have any other detectors in the stage.

halide, ionize it, and transform it to silver. The emulsions are supported on a polymer film which, unlike the earlier glass plates, outgas and shrink during pre-pumping, or processing. However, glass plates were heavy, occupied an enormous volume compared to film, and Murphy's law meant that your best plates invariably broke because you spent more time handling them. Many aging microscopists still call their EM films 'plates.'

If you are still using film, you have a choice of photographic emulsions, just as you do for your camera (since you'll probably still have a film camera if you're using TEM film). Different speed emulsions are available, with the usual compromise that faster film means a larger grain size and therefore less resolution.

- In principle, for the highest-resolution images, the slowest (finest grain) film is best.
- In practice, we usually minimize the exposure time and go for the fastest film.

We usually want to minimize beam damage and blurring due to movement (drift) of the specimen/stage, so we keep the exposure short. In fact grain sizes for the faster film are about 5 μm compared to about 4 μm for the slowest film, so we don't lose much resolution. The loss of resolution is more than offset by the shorter exposure times which minimize the overall dose to the specimen. The only time you may need to use slower film is if you have a problem with poor image contrast. This problem is more common when imaging amorphous, biological, or polymer specimens.

Although the grain size of the emulsion may be as small as a few micrometers, the actual resolution of the recorded image is worse than this because of electron spreading in the emulsion. The practical resolution may only be about 20–50 μm. Despite this degradation we still have more than 10^7 picture elements or pixels available to store information in a standard 100 mm × 100 mm image (Kodachrome™ film has the silver halide equivalent of 1.8×10^7 pixels). Film has a high DQE, although its dynamic range is rather limited. What this means is that you can easily saturate the film (change all the halide to silver) and you lose any linear relationship between electron intensity and gray scale in the film. As we've already noted, CCDs have a high dynamic range, are linear in response, uniform in output, have anti-blooming technology and the latest CCDs boast $>10^9$ pixels. Perhaps the end is indeed in sight!

Removing photographic film from the TEM will be a major operational improvement, because the absorbed water degrades the vacuum.

PHOTOGRAPHIC FILM

A photographic emulsion on a polymer support is one of the worst things you can put into a high-vacuum instrument.

Both the emulsion and the support outgas, which is a major contribution to the residual high pressure of hydrocarbons and water vapor in the instrument which, in turn, cause contamination of your specimen.

We used to use Polaroid instant film for recording scanning images from the CRT display in STEM mode and there may be a few older instruments around that still require such. But it will generally be good to see the end of any kind of film because they were analog, expensive, used nasty chemicals, and created a lot of mess around the TEM lab. Also there was always the chance that someone before you had loaded the film incorrectly, thus rendering your precious time on the microscope useless and subsequently provoking occasional melees in graduate-student hangouts.

7.5.B Other Image-Recording Methods

Digital images can be stored and retrieved, for example, magnetically on hard drives, or optically on compact discs, DVDs, or, most recently, holographic storage disks. These devices are cheaper and easier to use than photographic recording and images on an optical disc will not degrade with time even after years of storage. However, the usual drawback to digital-storage technology is the question of how long will the technology be around to read any particular stored image. How many images do you still have on a 3.5" floppy? Probably not many! Or a zip drive? Perhaps lots more but you can't read them anymore.

An alternative is the image plate, manufactured by Fuji, which is a kind of re-usable, digital photographic plate. The plates can be used, read, and re-used and this capability permits on-line image processing of the data. While this technology is seeing a lot of use in medical X-ray laboratories, it has not caught on in the TEM field, mainly because it is so expensive.

To present a stored image for publication you still have to print it in some way; photographic methods are still occasionally used. However, laser printers now have the quality required for publishing the highest resolution images (1200 dpi or 48/mm) means that the dot size is well below the resolving power of human eye. Likewise the advent of artificial coloring of electron images, DPs, and spectral images over the last decade or so means that photographic methods of printing are becoming completely obsolete because color photography was never a satisfactory individual lab process.

7.6 COMPARISON OF SCANNING IMAGES AND STATIC IMAGES

We have a choice of creating analog static images in conventional TEM mode or digital scanning images via electronic detection and display. Which is best? While we can only form BSE and SE images in a scanning mode, the answer is not clear for conventional BF and DF images, and the answer depends somewhat on the contrast mechanism that is operating in the specimen, as we'll see in Chapter 22. Regardless of which detector you use, scanning images are always displayed on a computer screen, and this limits the amount of information in the image. Typically the viewing screen will have up to 10^3 lines with a maximum of 10^3 pixels per line, giving a total of 10^6 pixels in each frame. Currently, high-definition TV displays offer at least 1920 pixels per line and 1080 lines per frame giving a total of $2\times\ 10^6$ pixels. In contrast, as we just noted, a TEM image recorded directly onto photographic emulsion will have a higher information density, with $>10^7$ pixels of information available in a 100 mm $\times$ 100 mm image. Furthermore, if a scanning image is to be recorded in a reasonable time, the electron beam can only stay on each point in the image (i.e., each pixel on the display) for a very short time. Typical dwell times per pixel are $<<1$ ms and this means that the signal-to-noise ratio in a scanning image is liable to be quite low. The combination of the lower pixel density compared to a photographic emulsion and the short dwell times means that, almost invariably, STEM images are poorer in quality than static TEM images. However, with the increasing availability of FEG (S)TEMs, increased probe current through C_s correction and improved detection and display technology, STEM digital picture quality compares quite well with analog TEM images. The former technology will continue to improve with the main challenge being our ability to read/access data; the latter will continue to fade away, slowly turning to shades of sepia with increasing time.

CHAPTER SUMMARY

Although it might seem surprising for such a high-end scientific instrument, the TEM is still in the age of analog images. We look at fluorescent screens and computer displays and we still record some of our pictures on photographic film. But darkrooms have disappeared from many labs and the whole area of electron detection is in a state of rapid flux as new electronic technology develops. Semiconductor detectors, scintillators, and CCDs all bring with them the advantage of digital signal collection and therefore the images can be processed and subsequently stored either magnetically or optically. As we said back in 1996, "anything we say about this technology will probably be obsolete before it is published." It is probably safe to speculate that most analog detection, recording, and storage of images and DPs will eventually be replaced by digital methods and the CCD manufacturers are already pronouncing 'the end of film.' So, TEM will produce numbers, but remember that we can all interpret images on film from the 1880s. Can you read data from computer punchcards from the 1970s or from zip disks of the 1990s? A final thought: we encourage you to read the original papers on TEM. All those images were recorded using a photographic emulsion.

REFERENCES

The general references for SEM are the standard book by Goldstein et al. (3rd Ed.) and Reimer's SEM text. The other references here are interesting (often challenging) reading.

Chapman, JN, Craven, AJ and Scott, CP 1989 *Electron Detection in the Analytical Electron Microscope* Ultramicroscopy **28** 108–117.

Howell, SB 2006 *Handbook of CCD Astronomy* 2nd Ed. Cambridge University Press NY.

Knoll, GF 2000 *Radiation Detection and Measurement* 3rd Ed. John Wiley & Sons NY.

Pierret, RF 1996 *Semiconductor Device Fundamentals* Addison-Wesley Boston MA

Reimer, L 1985 *Scanning Electron Microscopy* Springer Verlag New York.

URLs

1) http://tpm.amc.anl.gov/
2) http://telescience.ucsd.edu/gts.shtml

SELF-ASSESSMENT QUESTIONS

Q7.1 How do we 'see' electrons?
Q7.2 What are viewing screens usually coated with?
Q7.3 How do you prolong the lifetime of your screen?
Q7.4 Why is green chosen as the color of the light emitted by the screen?
Q7.5 What is DQE?
Q7.6 What is CL?
Q7.7 How do semiconductor detectors work?
Q7.8 What are the advantages and disadvantages of semiconductor detectors?
Q7.9 How does a scintillator-photomultiplier detector work?
Q7.10 What are the advantages and disadvantages of scintillator-photomultiplier detectors?
Q7.11 What are the advantages of CCD cameras?
Q7.12 What is a Faraday cup?
Q7.13 Which detector should be used for which signal in the TEM?
Q7.14 What is a photographic emulsion, and how does the chemistry work?
Q7.15 What is the difference between scintillation, fluorescence, and phosphorescence?
Q7.16 Why are ZnS-based viewing screens doped?
Q7.17 Why aren't semiconductor devices cooled with LN_2 to reduce the large dark current as they are when used as X-ray detectors (see Chapter 32)?
Q7.18 Give a major disadvantage of photographic film when used in a TEM.
Q7.19 How do you deal with the problem of recording images of specimens with poor image contrast?
Q7.20 What does a 'static' TEM image mean, as opposed to a 'scanning' image?

TEXT-SPECIFIC QUESTIONS

T7.1 Why do we still use analog viewing screens in nearly all TEMs rather than viewing all images and DPs digitally on a computer screen? What would be the biggest advantages of removing the viewing screen?

T7.2 Why do we still sometimes record TEM images and DPs on photographic plates rather than always capturing and storing them digitally?

T7.3 Examine Figure 7.1 and explain why this kind of detector is good for imaging high-angle elastically scattered electrons?

T7.4 Why is this kind of detector (Figure 7.1) not very good at imaging DPs such as those in Figure 2.13B and D?

T7.5 Why would you not use the scintillator-PM detector for the on-axis detector to pick up the direct beam signal in Figure 7.1?

T7.6 Why is the scintillator-PM detector even better than the semiconductor detector for imaging very high-angle, elastically scattered electrons?

T7.7 Go on the Web and find the smallest pixel size available in a commercial CCD camera. How does this dimension compare with the emulsion size in a typical high-resolution photographic film?

T7.8 Go on the Web and find the largest number of pixels available in a commercial CCD camera. How does this number compare with the effective number of pixels in a typical high-resolution photographic film used in a TEM?

T7.9 A problem with recording DPs on CCD cameras is that the intense direct beam can cause 'blooming.' What is blooming and how can it be corrected? (Hint: go on the Web and look for non/anti-blooming cameras.)

T7.10 Why are CL detectors much less common in TEMs than in SEMs? (Hint: take a look at Figure 7.6 and consider the relative sizes of the CL source and the CL detector.)

T7.11 Even if you can, why should you not buy (or bother to use) a BSE detector in your TEM?

T7.12 Go online and see how many TEMs are available for you to watch in someone else's lab? Can you think of specific advantages that could be gained by this accessibility? Can you think of experiments that you could do on remotely accessible TEMs that you could not do in your own laboratory?

T7.13 Why is CL imaging a relatively common imaging method in the SEM but rarely used in the TEM?