The Transmission Electron Microscope

1

CHAPTER PREVIEW

A typical commercial transmission electron microscope (TEM) costs about $2 for each electron volt of energy in the beam, and if you add on all the options, it can cost about $4–5 per eV. As you'll see, we use beam energies in the range from 100,000–400,000 eV, so a TEM becomes an extremely expensive piece of equipment. Consequently, there have to be very sound scientific reasons for investing such a large amount of money in one microscope. In this chapter (which is just a brief overview of many of the concepts that we'll talk about in detail throughout the book) we start by introducing you to some of the historical development of the TEM because the history is intertwined with some of the reasons why you need to use a TEM to characterize materials. Other reasons for using TEM appeared as the instrument developed. Unfortunately, coupled with the advantages are some serious drawbacks, which limit the microscope performance, and you must be just as aware of the instrument's limitations as you are of its advantages, so we summarize these also.

A TEM can appear in several different forms, all of which are described by different acronyms such as HRTEM, STEM, and AEM, and we'll introduce you to these different instruments. We'll also use the same acronym to denote both the technique (microscopy) and the instrument (microscope). We regard all of the dif-

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ferent types of TEM as simply variations on a basic theme and that is why only "TEM" is in the book title. We will describe some of the basic physical characteristics of the electron. Throughout the book you'll have to confront some physics and mathematics every now and again. The reason for this is because understanding what we can do with a TEM and why we operate it in certain ways is governed by the fundamental physics of electrons, how electrons are controlled by magnetic fields in the microscope, and how electrons interact with materials.

Finally, we will summarize some of the most popular computer software packages for TEM. We will refer to many of these throughout the text. We are including them in the first chapter to emphasize the role of the computer in today's TEM analysis.

The Transmission Electron Microscope

1

1.1. WHY USE ELECTRONS?

Why should we use an electron microscope? Historically, TEMs were developed because of the limited image resolution in light microscopes, which is imposed by the wavelength of visible light. Only after electron microscopes were developed was it realized that there are many other equally sound reasons for using electrons, most of which are utilized to some extent in a modern TEM. By way of introduction to the topic let's look at how the TEM developed and the pros and cons of using such an instrument.

1.1.A. An Extremely Brief History

Louis de Broglie (1925) first theorized that the electron had wave-like characteristics, with a wavelength substantially less than visible light. Then Davisson and Germer (1927) and Thompson and Reid (1927) independently carried out their classic electron diffraction experiments which demonstrated the wave nature of electrons. It didn't take long for the idea of an electron microscope to be proposed, and the term was first used in the paper of Knoll and Ruska (1932). In this paper they developed the idea of electron lenses into a practical reality, and demonstrated electron images taken on the instrument shown in Figure 1.1. This was a most crucial step, for which Ruska received the Nobel Prize, somewhat late, in 1986. Within a year of Knoll and Ruska's publication, the resolution limit of the light microscope was surpassed. Ruska, surprisingly, revealed that he hadn't heard of de Broglie's ideas about electron waves and thought that the wavelength limit didn't apply to electrons. TEMs were developed by commercial companies only four years later. The Metropolitan-Vickers EM1 was the first commercial TEM. It was built in the UK in 1936, but apparently it didn't work very well and regular production was really started by Siemens and Halske in Germany in 1939. TEMs became widely available from several other sources (Hitachi, JEOL, Philips and RCA, *inter alia*) after the conclusion of World War II.

For materials scientists a most important development took place in the late 1940s when Heidenreich (1949) first thinned metal foils to electron transparency. This work was followed up by Bollman in Switzerland and Hirsch and co-workers in Cambridge. Because so much of the early TEM work examined metal specimens, the word "foil" has come to be synonymous with "specimen." In addition, the Cambridge group also developed the theory of electron diffraction contrast with which we can now identify, often in a quantitative manner, all known line and planar crystal defects in TEM images. This theoretical work is summarized in a formidable but essential text often referred to as the "Bible" of TEM (Hirsch *et al.* 1977). For the materials scientist, practical applications of the TEM for the solution of materials problems were pioneered in the United States by Thomas and first clearly expounded in his text (Thomas 1962). Other materials-oriented texts followed, e.g., Edington (1976) and Thomas and Goringe (1979).

Today, TEMs constitute arguably the most efficient and versatile tools for the characterization of materials. If you want to read a history of the TEM, the book by Marton (1968) is a compact, personal monograph and that edited by Hawkes (1985) contains a series of individual reminiscences. Fujita (1986) emphasizes the contribution of Japan to the development of the instrument. The field is now at the point where many of the pioneers have put their memoirs down on paper, or Festschrifts have been organized in their honor (e.g., Cosslett 1979, Ruska 1980, and Hashimoto 1986) which detail their contributions over the decades, and compile some useful overview papers of the field. If you enjoy reading about the history of science, we strongly recommend the review of *Fifty Years of Electron Diffraction,* edited by Goodman (1981), and *Fifty Years of X-ray Diffraction,* edited by Ewald (1962). (The spelling of X-ray is discussed in the *CBE Manual,* 1994.)

Figure 1.1. The electron microscope built by Ruska and Knoll in Berlin in the early 1930s.

1.1.B. Microscopy and the Concept of Resolution

When asked what a "microscope" is, most people would answer that it is an instrument for magnifying things too small to see with the naked eye, and most likely they would be referring to the visible-light microscope. Because of the general familiarity with the concept of the light microscope, we will draw analogies between electron and visible-light microscopes wherever it's instructive.

The smallest distance between two points that we can resolve with our eyes is about 0.1–0.2 mm, depending on how good our eyes are, and assuming that there's sufficient illumination to see by. This distance is the *resolution* or *resolving power* of our eyes. So any instrument that can show us pictures (or "images" as we'll refer to them) revealing detail finer than 0.1 mm could be described as a microscope, and its highest useful magnification is governed by its resolution. A major attraction to the early developers of the TEM was that, since electrons are smaller than atoms, it would be possible, at least theoretically, to build a microscope that could "see" detail well below the atomic level. The idea of being able to "see" with electrons may be confusing to you. Our eyes are not sensitive to electrons. If a beam of high-energy electrons was aimed into your eye, you would most likely be blinded as the electrons killed the retinal cells, but you wouldn't see anything! So an integral part of any electron microscope is a viewing screen of some form, which translates electron intensity to light intensity, and which we observe or record photographically. We'll discuss these screens and other ways of recording electron images in Chapter 7.

The resolution of a TEM means different things for different functions of the instrument, and we'll discuss them in the appropriate chapters. It's easiest to think of the image resolution in TEM in terms of the classical Rayleigh criterion for light microscopy, which states that the smallest distance that can be resolved, δ, is given approximately by

$$\delta = \frac{0.61\lambda}{\mu \sin \beta} \qquad [1.1]$$

In equation 1.1, λ is the wavelength of the radiation, μ the refractive index of the viewing medium, and β is the semi-angle of collection of the magnifying lens. For the sake of simplicity we can approximate $\mu \sin \beta$ (which is sometimes called the numerical aperture) to unity and so the resolution is equal to about half the wavelength of light. For green light in the middle of the visible spectrum, λ is about 550 nm (5500 Å), and so the resolution of a good light microscope is about 300 nm. In TEMs we can approximate the resolution in equation 1.1 to $0.61\lambda/\beta$ which, as we'll see later, is very small.

We'll try to use nanometers throughout this book, but you'll find that many microscopists still insist on using Ångstroms rather than the SI units. However, the Ångstrom is close to the atomic diameter and so is a more convenient unit because it saves us using convoluted phrases like "three tenths of a nanometer."

Now although 300 nm is a small dimension to us it corresponds to about 1000 atom diameters, and therefore many of the features that control the properties of materials are on a scale well below the resolution of the light microscope. So there's a real need to image detail down to the atomic level if we want to understand the properties of materials, and that's a major reason why TEMs are so useful.

This limit of light microscopy was well understood at the turn of this century and prompted Ernst Abbe, one of the giants in the field, to complain that "it is poor comfort to hope that human ingenuity will find ways and means of overcoming this limit." (He was right to be so depressed because he died in 1905, some 20 years before de Broglie's ingenuity solved the problem.) Now de Broglie's famous equation shows that the wavelength of electrons is related to their energy, E, and if we ignore relativistic effects we can show approximately (and exactly in Section 1.4 below) that

$$\lambda \sim \frac{1.22}{E^{1/2}} \qquad [1.2]$$

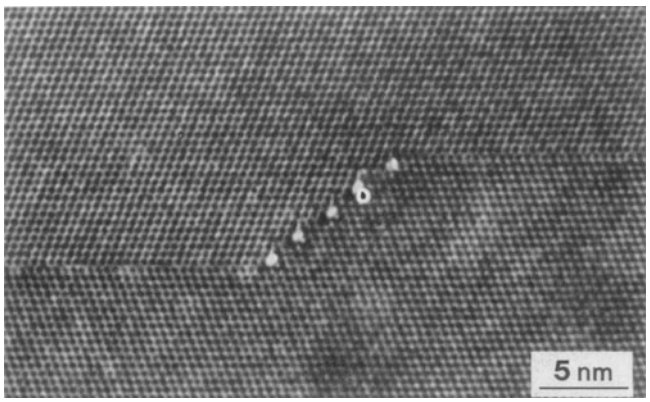


Figure 1.2. A twin boundary in spinel stepping from one {111} plane to another parallel plane. The white dots are columns of atoms. The change in atomic orientation across the twin boundary can be readily seen, even if we do not know what causes the white dots or why, indeed, they are white.

In this equation E is in electron volts (eV) and λ in nm. Remember that we should be precise in our use of the units V and eV: the former represents the *accelerating voltage* of the microscope while the latter refers to the *energy* of the electrons in the microscope. So for a 100-keV electron, we find that $\lambda \sim 4$ pm (0.004 nm), which is much smaller than the diameter of an atom.

We'll see later that we are nowhere near building TEMs that approach this wavelength limit of resolution, because we can't make perfect electron lenses (see Chapter 6). But progress was rapid after Ruska's early work on lenses and, since the mid-1970s, many commercial TEMs have been capable of resolving individual columns of atoms in crystals, creating the field of "high-resolution transmission electron microscopy," or HRTEM, which we'll discuss in Chapter 28. A typical HRTEM image is shown in Figure 1.2. The advantages of shorter wavelengths led in the 1960s to the development of high voltage electron microscopes (HVEMs), with accelerating potentials between 1 MV and 3 MV. In fact, most of these instruments were used to introduce controlled amounts of radiation damage into specimens in an attempt to simulate nuclear reactor environments, but changes in the emphasis of energy research mean there is not much call for such instruments today. While we can still improve the resolution by incremental amounts, the drive for much better resolution is now no longer paramount and the TEM is developing in other ways. In fact, only one HVEM (1 MV) for HRTEM imaging was constructed in the 1980s and three 1.25-MV machines in the 1990s. Intermediate voltage electron microscopes (IVEMs) were introduced in the 1980s. These TEMs operate at 300 or 400 kV, but still offer very high resolution, close to that achieved at 1 MV.

1.1.C. Interaction of Electrons with Matter

Electrons are one type of "ionizing radiation," which is the general term given to radiation that is capable of removing one of the tightly bound inner-shell electrons from the attractive field of the nucleus.

One of the advantages to using ionizing radiation is that it produces a wide range of secondary signals from the specimen, and some of these are summarized in Figure 1.3. Many of these signals are used in "analytical electron microscopy," or AEM, giving us chemical information and a lot of other detail about our samples. AEM uses X-ray energy dispersive spectrometry (XEDS) and electron energy-loss spectrometry (EELS). For example, Figure 1.4A is an X-ray spectrum from a very small region of a TEM specimen showing characteristic peaks which identify the elements present. We can transform such spectra into quantitative data describing elemental changes associated with inhomogeneous microstructures as also shown in Figures 1.4B and C. This aspect comprises Part IV of the book. In contrast, microscopes using nonionizing radiation such as visible light usually only generate light (but not much heat, which is good). AEMs generally offer improved performance at intermediate voltages, similar to HRTEMs.

In order to get the best signal out of our specimens we have to put the best signal in, and so the electron source is critical. We are now very accomplished in this respect as you'll see in Chapter 5, so modern TEMs are very good

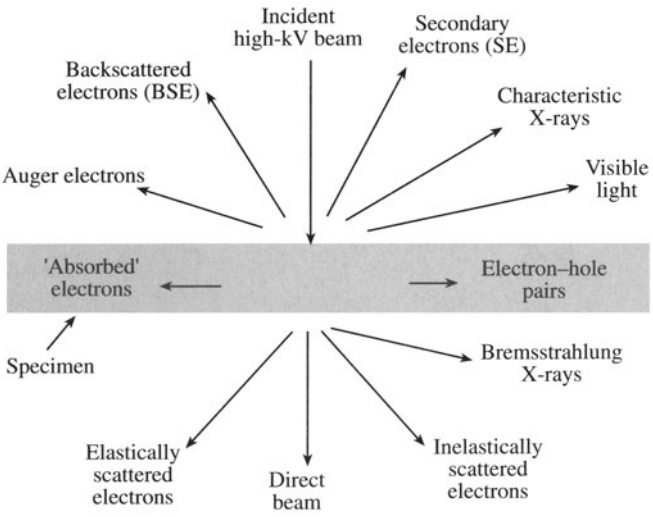
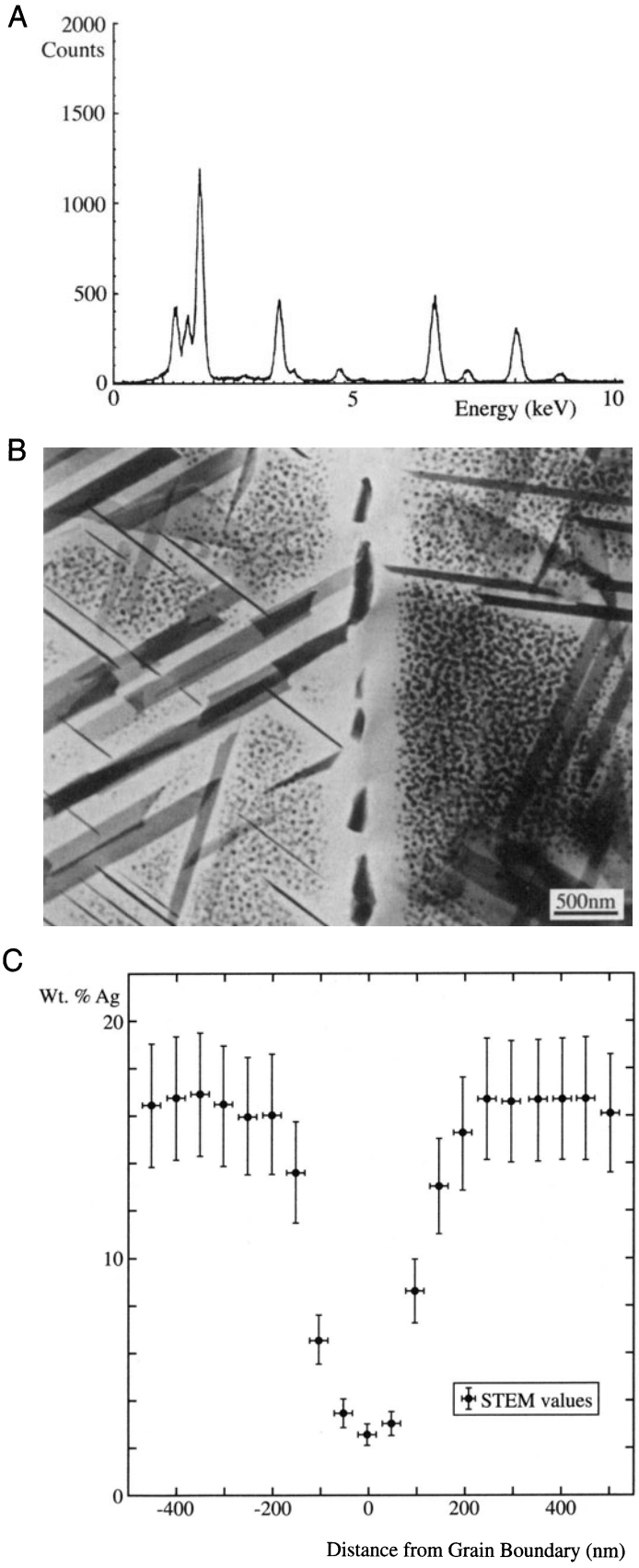


Figure 1.3. Signals generated when a high-energy beam of electrons interacts with a thin specimen. Most of these signals can be detected in different types of TEM. The directions shown for each signal do not always represent the physical direction of the signal but indicate, in a relative manner, where the signal is strongest or where it is detected.



signal-generating instruments. To localize these signals we need to get our TEM to form a very fine electron beam, typically <10 nm and at best <1 nm in diameter. We accomplish this by combining TEM and scanning electron microscope (SEM) technology to create a scanning transmission electron microscope (STEM). The STEM is both the basis for AEMs and a unique scanning imaging microscope in its own right. In fact there are instruments that are only capable of operating in scanning mode and these are sometimes referred to as "dedicated STEMs," or DSTEMs.

1.1.D. Depth of Field

The depth of field of a microscope is a measure of how much of the *object* we are looking at remains "in focus" at the same time. Like the resolution, this property is governed by the lenses in the microscope. The best electron lens is not a very good one, as we've already mentioned, and has been compared to using the bottom of a Coca-Cola bottle as a lens for light microscopy. To minimize this problem we have to use very small limiting apertures in the lenses, narrowing the beam down to a thin "pencil" of electrons which at most is a few micrometers across. These apertures cut down the intensity of the electron beam, but also act to increase the depth of focus of the images that we produce. Remember that "depth of field" refers to the specimen while "depth of focus" refers to the image.

While this large depth of field is chiefly used in the SEM to produce 3D-like images of the surfaces of specimens with large changes in topography, it is also critical in the TEM. It turns out that in the TEM, all of the specimen is usually in focus at the same time, independent of the specimen topography, as long as it's electron transparent! Figure 1.5 shows a TEM image of some dislocations in a crystal. The dislocations appear to start and finish in the specimen, but in fact they are threading their way through the specimen from the top to the bottom, and they remain in sharp focus at all times. Furthermore, we can record the final image at different positions below the final lens of the instrument and it will still be in focus. Compare this with

Figure 1.4. (A) An X-ray spectrum from a small biotite crystal showing peaks at energies that are characteristic of the elements present in the region that interacts with the electron beam. The major peaks from left to right are for Mg, Al, Si, K, Fe, and the Cu support grid. (B) A TEM image of a precipitate-free zone (PFZ) in an aged Al–16 wt% Ag alloy. (C) The Ag profile across the PFZ in (B), obtained through X-ray spectrometry in the TEM showing the depletion of Ag responsible for the PFZ formation.

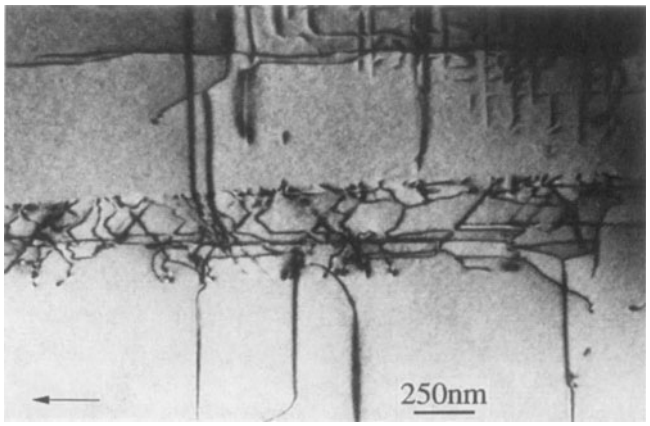


Figure 1.5. TEM image of dislocations in GaAs. A band of dislocations threads through the thin specimen from the top to the bottom but remains in focus through the foil thickness.

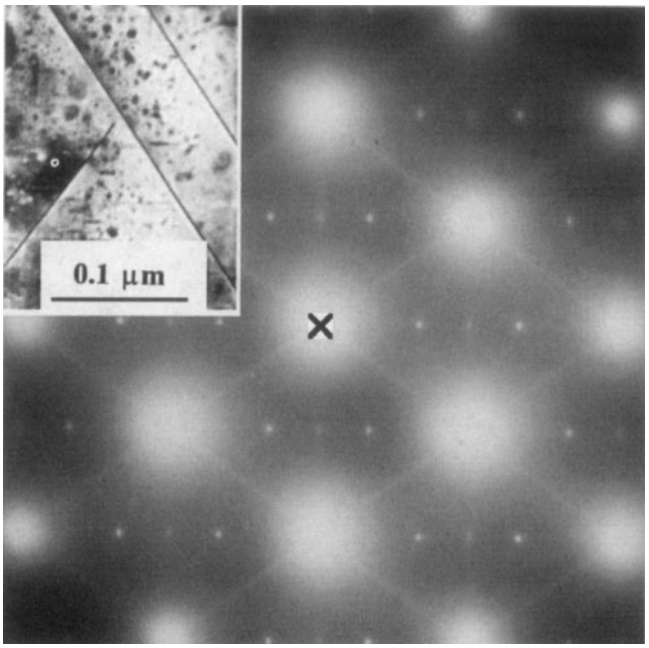


Figure 1.6. TEM diffraction pattern from a thin foil of Al-Li-Cu containing various precipitate phases, shown in the inset image. The central spot (X) contains electrons that come directly through the foil and the other spots and lines are diffracted electrons which are scattered from different crystal planes.

the visible-light microscope where, as you probably know, unless the surface of the specimen is flat to within the wavelength of light, it is not all in focus at the same time. This aspect of TEM gives us both advantages and disadvantages in comparison to the visible-light microscope.

1.1.E. Diffraction

Thompson and Reid showed that electrons could be diffracted when passing through thin crystals of nickel, and the possibility of combining electron diffraction into TEMs was realized by Kossel and Möllenstedt (1939). Today, electron diffraction is an indispensable part of TEM and is arguably the most useful aspect of TEM for materials scientists. Figure 1.6 shows a TEM diffraction pattern which contains information on the crystal structure, lattice repeat distance, and specimen shape, as well as being a most striking pattern. We'll see that the pattern can always be related to the image of the area of the specimen from which it came, in this case shown in the inset. You will also see in Part II that, in addition to the things we just listed, you can conduct a complete crystallographic symmetry analysis of minuscule crystals, including such esoteric aspects as point-group and space-group determination, and at all times the crystallography can be related to the image of your specimen. There is no similar capability on a light microscope because of the relatively large wavelength of visible light.

So an electron microscope can produce atomic level images, can generate a variety of signals telling you about your sample chemistry and crystallography, and you can always produce images that are in focus. There are many other good reasons why you should use electron microscopes. We hope they will become evident as you read through this book. At the same time there are many reasons why you should *not* always seek to solve your problems with the TEM, and it is most important that you realize what the instrument *cannot* do, as well as knowing its capabilities.

1.2. LIMITATIONS OF THE TEM

1.2.A. Sampling

All the above advantages of the TEM bring accompanying drawbacks. First of all, the price to pay for any high-resolution imaging technique is that you only look at a small part of your specimen at any one time. The higher the resolution, therefore, the worse the sampling abilities of the instrument. Von Heimendahl (1980) reported a calculation by Swann in around 1970 estimating that all TEMs, since

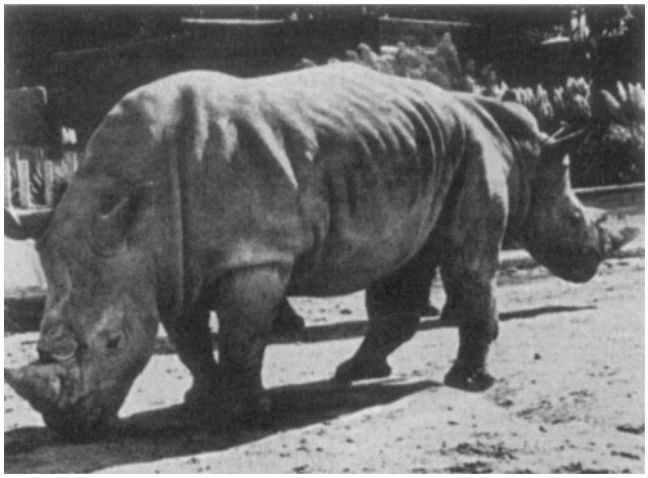


Figure 1.7. Photograph of two rhinos taken so that, in projection, they appear as one two-headed beast. Such projection artifacts in reflected-light images are easily discernible to the human eye but similar artifacts in TEM images are easily mistaken for "real" features.

they first became available commercially, had only examined 0.3 mm^3 of material! Extending that calculation to the present time at best doubles the volume to 0.6 mm^3. So we have an instrument that is a terrible sampling tool. This only serves to emphasize that before you put your specimen in the TEM you must have examined it with techniques that offer poorer resolution but better sampling, such as your eyes, the visible-light microscope, and the scanning electron microscope. In other words, know the forest before you start looking at the leaves on the trees.

1.2.B. Interpreting Transmission Images

Another problem is that the TEM presents us with 2D images of 3D specimens, viewed in transmission. Our eyes and brain routinely understand reflected light images but are ill-equipped to interpret TEM images, and so we must be cautious. Hayes (1980) illustrates this problem well by showing a picture of two rhinos, side by side such that the head of one appears attached to the rear of the other (see Figure 1.7). As Hayes puts it: "when we see this image we laugh" (because we understand its true nature in 3D) "but when we see equivalent (but more misleading) images in the TEM, we publish!" So beware of artifacts, which abound in TEM images.

One aspect of this particular drawback is that, generally, all the TEM information that we talk about in this book (images, diffraction patterns, spectra) is *averaged through the thickness of the specimen.* In other words, a single TEM image has no depth sensitivity, as is apparent from Figure 1.5. So other techniques which are surface-sensitive or depth-sensitive, such as field ion microscopy, scanning-probe microscopy, Auger spectroscopy, Rutherford backscattering, etc., are necessary complementary techniques if you want a full characterization of your specimen.

1.2.C. Electron Beam Damage and Safety

A side effect of ionizing radiation is that it can damage your specimen, particularly in materials such as polymers and some ceramics. Some aspects of beam damage are exacerbated at higher voltages and, with commercial instruments offering up to 400 kV, beam damage now limits much of what we can do in the TEM, even with refractory metals. Figure 1.8 shows an area of a specimen damaged by high-energy electrons. The combination of high-kV beams with the intense electron sources that are available means that we can destroy almost any specimen, if we are not careful. At the same time comes the danger that should *never* be forgotten, that of exposing yourself to ionizing radiation. Modern TEMs are remarkably well engineered and designed with safety as a primary concern, but *never* forget that you are dealing with a potentially dangerous instrument that generates radiation levels that could kill you. So *never* modify your microscope in any way without consulting the manufacturer and without carrying out routine radiation leak tests. If in doubt, don't do it!

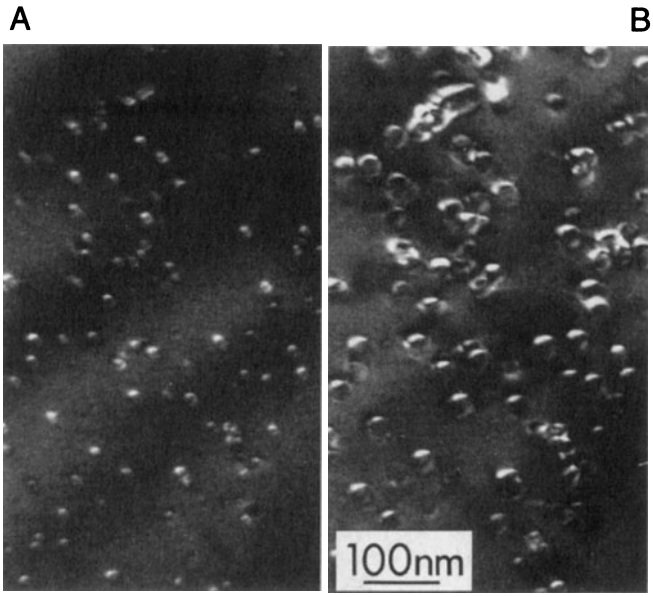


Figure 1.8. Beam damage in quartz after bombardment with 125-keV electrons. With increasing time, from (A) to (B), the damaged regions increase in size.

1.2.D. Specimen Preparation

Your specimens have to be thin if you're going to get any information using transmitted electrons in the TEM. "Thin" is a relative term, but in this context it means "electron transparent." For a specimen to be transparent to electrons it must be thin enough to transmit sufficient electrons such that enough intensity falls on the screen or photographic film to give us an interpretable image in a reasonable time. Generally this requirement is a function of the electron energy and the average atomic number of the specimen. Typically for 100-keV electrons, specimens of aluminum alloys almost up to 1 μm would be thin, while steel would be thin up to about several hundred nm. However, it is an axiom in TEM that thinner is better, and specimens below 100 nm should be used wherever possible, and in extreme cases, such as when doing HRTEM or electron spectrometry, specimen thicknesses <50 nm are essential. These demands become less strict as the beam voltage increases, but this is offset by the danger of beam damage.

The requirement for thin specimens is a *major* limitation of the TEM. Methods to prepare thin specimens exist for almost all materials, and we talk about them in Chapter 10. But as a general rule the thinning processes that we use do affect the specimen, changing both its structure and its chemistry. So you need to be aware of the dangers of specimen preparation and learn to recognize the artifacts introduced by standard preparation methods.

So it should be obvious to you by now that while TEM and associated techniques are tremendously powerful characterization tools when used properly, they should *never* be used in isolation to solve a materials problem. You must understand your material at low magnification with your eyes and with visible-light microscopy and scanning electron microscopy (SEM) before venturing into TEM studies. Otherwise you may fall foul of some of the limitations we have just listed.

1.3. DIFFERENT KINDS OF TEMs

As you read through the previous sections you will have seen that TEMs come in a wide variety of types: HRTEMs, HVEMs, IVEMs, STEMs, and AEMs. Complete books have been written on each of these instruments, but it is our philosophy that all these are simply different forms of the basic TEM. So in this book we intend to treat them as such. Indeed a current 300 or 400 keV TEM can combine aspects of *all* the above microscope types. Figure 1.9 shows four of the different kinds of TEMs we have mentioned. It is instructive to consider some of the features of the instruments shown here. An HVEM usually requires a two-story room; the scale of each instrument can be judged from the common height of the operator's console. A modern machine essentially is an electron optic column in which we can maintain a good vacuum but the lenses and most other functions can be controlled by one or more computers. Note that the DSTEM only has CRT displays. There is no viewing screen. Furthermore, the electron source is at the base of the column rather than at the top, as we will assume in all of our discussions.

1.4. SOME FUNDAMENTAL PROPERTIES OF ELECTRONS

Many times in the book we'll have to refer to some of the basic properties of electrons. You know that electrons show both particle and wave characteristics, illustrating one of the great puzzles of quantum physics, which we all seem to accept without too much trouble. In fact the TEM routinely demonstrates both the particle and wave characteristics of the electron, repeating the electron analog of G. I. Taylor's famous experiment in which he demonstrated Young's slits interference patterns despite using such a weak light source that only one photon passed through the apparatus at any one time. A typical electron beam current in a TEM is about 0.1–1 μA, which corresponds to about 10^{12} electrons passing through the specimen plane. But as we'll see below, with 100-keV energy, these electrons travel at about $0.5c$ (1.6×10^8 m/s), so they are separated by 0.16 cm and this means that there is never more than one electron in the specimen at any one time. Nevertheless, electron diffraction and interference occur, both of which are wave phenomena, and imply interaction between the different electron beams. Despite this dilemma, we know a lot about the electron and its behavior and some of the basic characteristics are summarized in Table 1.1.

There are a few important equations which you should know. First of all, based on de Broglie's ideas of the wave-particle duality, we can relate the particle momentum p to its wavelength λ through Planck's constant; thus

$$\lambda = \frac{h}{p} \qquad [1.3]$$

In the TEM we impart momentum to the electron by accelerating it through a potential drop, V, giving it a kinetic energy eV. This potential energy must equal the kinetic energy, so

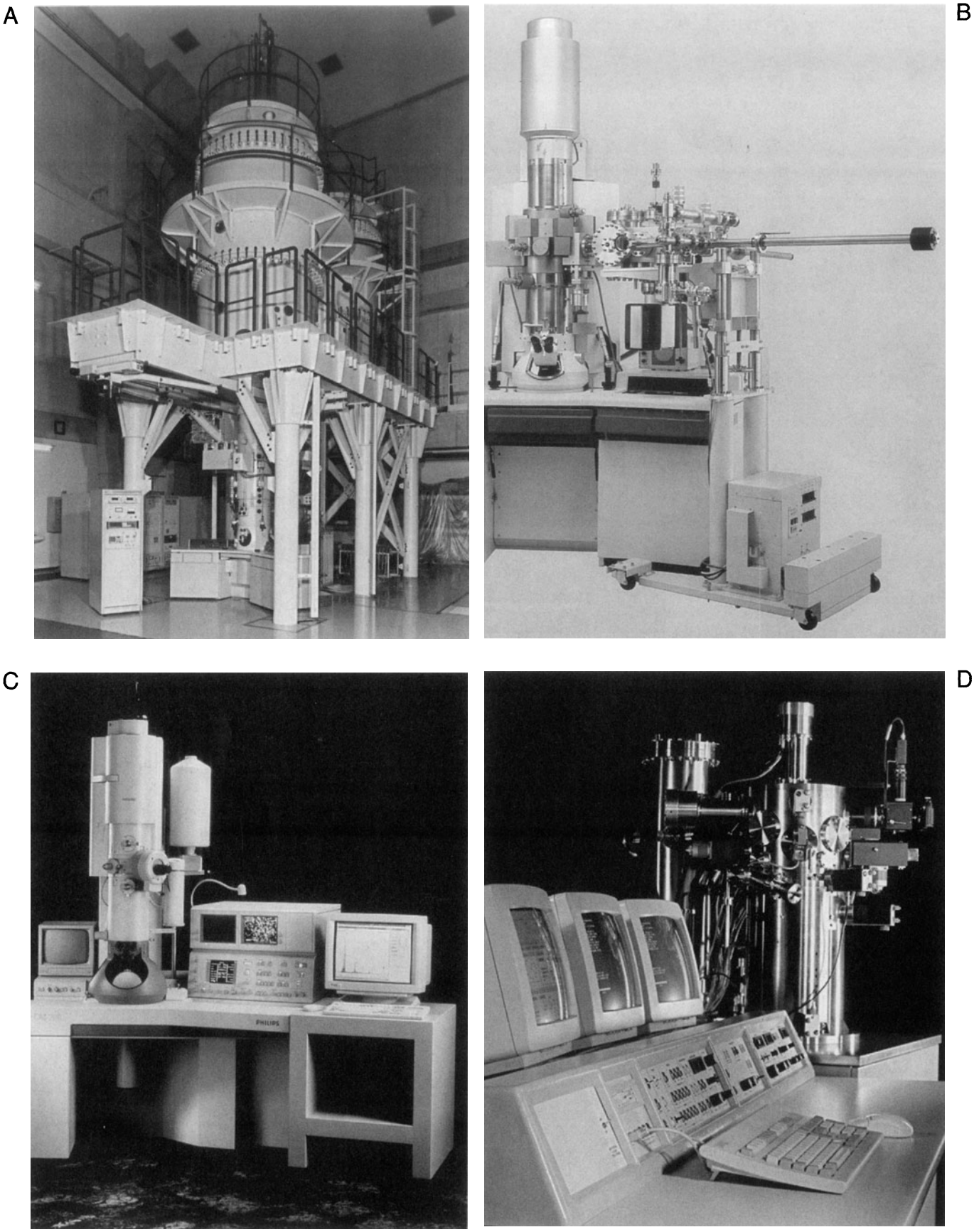


Figure 1.9. Different TEMs: (A) a JEOL 1.25-MV high voltage microscope, used for high-resolution imaging; (B) a Hitachi specialized ultrahigh vacuum TEM for high-resolution surface imaging; (C) a Philips 200-kV analytical microscope with an X-ray spectrometer attached to the stage (the liquid-N_2 dewar cools the detector); and (D) a VG dedicated 100-kV ultrahigh vacuum scanning transmission microscope. Comparison with Ruska's instrument (Figure 1.1) which is 50–60 years older is instructive.

Table 1.1. Fundamental Constants and Definitions

Charge (e)	$(-)\ 1.602 \times 10^{-19}$ C
1 eV	1.602×10^{-19} J
Rest mass (m_0)	9.109×10^{-31} kg
Rest energy (m_0c^2)	511 keV
Kinetic energy (charge × voltage)	1.602×10^{-19} N m (for 1 volt potential)
Planck's constant (h)	6.626×10^{-34} N m s
1 ampere	1 C/sec
Speed of light in vacuum (c)	2.998×10^{8} m/sec

$$eV = \frac{m_0 v^2}{2} \qquad [1.4]$$

Now we can equate the momentum p to the electron mass (m_0) times the velocity (v), and substituting for v from equation 1.4 we obtain

$$p = m_0 v = \left(2m_0 eV\right)^{1/2} \qquad [1.5]$$

What all this leads to is the relationship between the electron wavelength λ and the accelerating voltage of the electron microscope, V

$$\lambda = \frac{h}{\left(2m_0 eV\right)^{1/2}} \qquad [1.6]$$

This expression is identical to equation 1.2. This relationship between λ and the accelerating voltage introduces a very important concept: by increasing the accelerating voltage we decrease the wavelength of the electrons.

Equations 1.2 and 1.6 are useful expressions for deducing ballpark estimates, but be careful to note the differences. We can use equation 1.6 to calculate the nonrelativistic electron wavelength for typical commercial TEM operating voltages as listed in Table 1.2.

The simple treatment we just went through neglects relativistic effects and, unfortunately for electron microscopists, relativistic effects cannot be ignored at 100-keV energies and above because the velocity of the electrons (as particles) becomes greater than half the speed of light! (The speed of light in vacuum is 2.998×10^8 m/s.) So to be exact we must modify equation 1.6 to give

$$\lambda = \frac{h}{\left[2m_0 eV\left(1 + \frac{eV}{2m_0 c^2}\right)\right]^{1/2}} \qquad [1.7]$$

A full listing for many more voltages can easily be generated by putting equations 1.6 and 1.7 into a spreadsheet. The effect of relativity is greatest for higher accelerating voltages, as shown in Table 1.2.

There will be many times when it's useful to refer back to these numbers, especially when we consider the resolution of the microscope and when we need to make calculations about the way electrons interact with matter.

A word about units. As we noted above, we should all be using SI units. We don't for two reasons: first, some special units are ideal for the purpose at hand; second, we forget to include special conversion factors in some formulas. The difference between, e.g., the Gaussian system of units and SI units is summarized in the invaluable reference by Fischbeck and Fischbeck (1987).

1.5. MICROSCOPY ON THE INTERNET/WORLD WIDE WEB

TEM users are well integrated into the Internet/WWW and this is a source of useful information (and also some useful knowledge!) about what's going on in the field. Already

Table 1.2. Electron Properties as a Function of Accelerating Voltage

Accelerating voltage (kV)	Nonrelativistic wavelength (nm)	Relativistic wavelength (nm)	Mass (× m_0)	Velocity (×10^8 m/s)
100	0.00386	0.00370	1.196	1.644
120	0.00352	0.00335	1.235	1.759
200	0.00273	0.00251	1.391	2.086
300	0.00223	0.00197	1.587	2.330
400	0.00193	0.00164	1.783	2.484
1000	0.00122	0.00087	2.957	2.823

you can view research TEMs in real time on the Internet and in due course you'll not only see other instruments but be able to operate them remotely. Such "telepresence microscopy" will represent an extraordinary leap in our ability to characterize materials, since advanced instruments will effectively be available to you in your own laboratories without the need to travel to special sites.

In addition, specialized software packages that allow you to carry out many of the advanced analyses that we will introduce in this text (e.g., diffraction pattern analysis and image/diffraction/spectral simulation) are also available through the Web. In many cases access to this software is limited and any serious microscopy operation should have the software on site, but sometimes it is useful to see the possibilities before you purchase. A list of useful sites is included below but, as with all aspects of the Web, this list is already out of date and the number of actual sites is growing daily.

1.5.A. Microscopy and Microanalysis-Related WWW Sites

http://www.amc.anl.gov

This is the best source for TEM information on the Web in the United States. It is run by N.J. Zaluzec at Argonne National Laboratory (ANL). Through it you can get access to the Microscopy ListServer and a Software Library. There is a connection to the Microscopy & Microanalysis FTP Site and access to Software/Image Libraries. Other useful connections through this site include

http://146.139.72.10/Docs/nonanl/Meetings.html

List of Meetings/Conferences on Microscopy/Microanalysis

http://146.139.72.10/Docs/nonanl/ShortCourses.html

List of Short Courses/Workshops on Microscopy/Microanalysis

http://146.139.72.10/Docs/nonanl/msa/MSA.html

Microscopy Society of America information

http://146.139.72.10/Docs/nonanl/aust/aust.html

Australian Microscopy Societies information

http://146.139.72.10/Docs/nonanl//msc/MSc.html

Microscopical Society of Canada information

http://146.139.72.10/Docs/nonanl/rms/RMS.html

Royal Microscopical Society information

http://146.139.72.10/Docs/nonanl/mas/MAS.html

Microbeam Analysis Society information

http://146.139.72.10/Docs/NonAnl/EduSites.html

University/Educational Sites

http://www.amc.anl.gov/Docs/NonAnl/GovSites.html

Governmental Microscopy Sites

http://146.139.72.10/Docs/NonAnl/ComSites.html

Commercial Sites—microscopy-related manufacturers/suppliers

http://cimewww.epfl.ch/Welcometext.html

A similar operation to the ANL site, but based at the Ecole Polytechnique Fédérale de Lausanne in Switzerland, run by P.-H. Jouneau and P. Stadelmann. A very useful array of software is available including Electron Microscopy Image Simulation (EMS) software, which allows you to perform the following tasks:

> Draw a crystal in perspective view or in projection, stereographic projections, list $(hk\ell)$ distances, structure factors, and extinction distances, draw the microscope contrast transfer function, with $(hk\ell)$ crystal planes, draw kinematical, dynamical, or powder diffraction patterns, draw the amplitude and phase of diffracted beams as a function of specimen thickness, do auto indexing of diffraction patterns, draw Kikuchi patterns, draw high-order Laue zone line patterns, draw convergent beam diffraction patterns (Bloch-wave calculation), draw HRTEM image maps of the crystal (Bloch-wave calculation), do conventional image calculation of dislocations for a cubic crystal

http://cimewww.epfl.ch/emyp/

Another operation based at the Ecole Polytechnique Fédérale de Lausanne in Switzerland, also run by Jouneau and Stadelmann; EM Yellow pages. Contents include:

> Software for Electron Microscopy, Professional Societies, Instruments, Equipment and Consulting, Education in Electron Microscopy, Data and Databases, News and Publications, Related Sources of Information, Conferences, Workshops and Schools, Getting Somewhere Else on the Web

http://www-personal.engin.umich.edu/~jfmjfm/newsgroup.html

Microscopy users newsgroup run by J.F. Mansfield at the University of Michigan

http://www.bocklabs.wisc.edu/imr/microscopists.html

Directory of microscopists on the net

1.5.B. Microscopy and Microanalysis Software

While there is a lot of software available on the WWW, much of it freeware or shareware, you sometimes get what you pay for, so as a serious microscopist you should have access to the best commercial programs, which are not free. Again, this is an aspect of TEM which is changing on a rapid basis, but you can now buy excellent software packages for all the fundamental aspects of microscopy—diffraction, imaging, and microanalysis. Many of these programs will be referenced throughout the text, but here is a brief summary of the best that are currently used, with an

indication of the source of the software—some of which are still free! There are many more packages than we have listed here but these are the ones with which we are familiar.

- Comis runs on a UNIX system. A version of the program is being implemented on a Macintosh computer. The graphics are currently supported for Tektronix 4010 and 4100 compatible terminals, but the program may be run from any terminal (without the menu-interface). A version based on the X-window system is also being developed. The interface to the framestore is based on a small set of routines, similar to those used by the SEMPER image processing software (see below). Output to laser printers is supported through the PostScript language. MaComis is available from ComiSoft at 70404.1710@CompuServe.com. Contact the authors for information on the UNIX version
- CRISP is a commercial package running under Windows on a PC. It is designed for image processing of HRTEM images. It can be combined with ELD (see below) and is available from Calidris, Manhemsvägen 4, S-191 46 Solltuna, Sweden (46 8 625 00 41)
- Desktop Microscopist: software for the Macintosh which allows you to calculate diffraction patterns. Available from Virtual Laboratories at http://www. Rt66.com/~virtlabs/ (505 828 1640)
- Differential Hysteresis Imaging: software that enables you to extract the full range of contrast information out of any digitized TEM (or SEM) image. From Klaus Peters at the University of Connecticut, on the Web at http://panda.uchc.edu/htklaus/index.html
- Digital Micrograph: a complete system for the acquisition, control, and processing of digital images from any electron microscope. In principle it can convert an old analog instrument to a digital one, if beam scan coils are available, but it's not worth doing this for a TEM. Use with a CCD camera that provides digital images from the TEM or interface to any STEM system. From Gatan Inc., 6678 Owens Drive, Pleasanton, CA 94588 (510 463 0200)
- DTSA: (Desk-Top Spectrum Analyzer) simulates energy (and wavelength) dispersive X-ray spectra; can also be used as a multichannel analyzer to acquire, interpret, and process spectra. Absolutely essential for the X-ray microanalyst. From NIST, Standard Reference Data Program, 221/A123 Gaithersburg, MD 20899 (301 975 2208)
- ELD is a commercial package from the producers of CRISP running under Windows on a PC. It is intended for quantitative analysis of diffraction patterns and is available from Calidris, Manhemsvägen 4, S-191 46 Solltuna, Sweden (46 8 625 00 41)
- ELP: the energy loss program that runs all Gatan EELS systems. You can't do EELS without it. From Gatan Inc., 6678 Owens Drive, Pleasanton, CA 94588 (510 463 0200)
- EMS: image simulation program, diffraction analysis, basic crystallographic data, and much more developed by P. Stadelmann at Ecole Polytechnique Fédérale de Lausanne and available from him through the Web (http://cimewww.epfl.ch/Welcometext.html). Some would argue that if you have this software, there is little else you need, which is evident from the listing of its capabilities in Section 1.5.A
- Head *et al.* (1973): The book includes a listing of the original source code for the simulation of diffraction contrast images. You can download the source code via the MSA Web page
- Maclispix: a Macintosh-based image processing program, which works in conjunction with NIH-Image (see below). Developed by David Bright at NIST and can be downloaded (free) from the WWW at http://www-sims.nist.gov/WWW/Internet/InternetResources. The FTP command is ftp://enh.nist. gov/mac/mx30a.bin
- MacTempas and CrystalKit: a Macintosh-based image analysis program from Roar Kilaas for the simulation of high-resolution images, diffraction patterns, and crystal structures. Total Resolution, 20 Florida Avenue, Berkeley, CA 94707. roar@totalresolution.com
- Monte Carlo Simulations: software to simulate electron beam trajectories through materials for estimating the spatial resolution of X-ray microanalysis or the backscattered electron yield. Available from David Joy at the University of Tennessee. A full listing is given in his textbook (Joy 1995)
- NCEMSS: (NCEM Simulation System) for HRTEM image simulations, symmetry operators for all 230 space groups, scattering factors for 98 elements. From the National Center for Electron Microscopy, Lawrence Berkeley Laboratories, University of California, Berkeley, CA 94729, or on the Web at http://ncem.lbl.gov/ncem.html
- NIH-Image: public domain software from NIH, developed by Wayne Rasband, for general image manipulation with a limited set of image processing tools. It is useful for grayscale enhancing and Fourier filtering. It can acquire, display, edit, enhance, analyze, print, and animate images. It

reads and writes TIFF, PICT, PICS, and MacPaint files, including programs for scanning, processing, editing, publishing, and analyzing images. It supports many standard image processing functions, including contrast enhancement, density profiling, smoothing, sharpening, edge detection, median filtering, and spatial convolution with user-defined kernels up to 63×63. Available from the Internet by anonymous ftp from zippy.nimh.nih.gov or on floppy disk from NTIS, 5285 Port Royal Rd., Springfield, VA 22161, part number PB93-504868. Details can be found on the WWW at http://rsb.info.nih.gov/nih-image/

- (Adobe) Photoshop and page layout programs for presentations and labeling your figures
- SEMPER (Synoptics, Ltd.): a general-purpose image processing package. It includes many operations suitable for processing of electron microscope images, and has been extended locally. It can be used for Fourier-space filtering of experimental images (including the background-subtraction method for small particles supported on amorphous substrates), for cross-correlation and lattice averaging, and for spatial-frequency enhancement. Image Processing Systems, Synoptics, Ltd., 271 Cambridge Science Park, Milton Road, Cambridge CB4 4WE, UK. Fax: (1223) 420020
- SHRLI: (Simulated High-Resolution Lattice Images) simulations from models up to 3 nm × 3 nm in area (perfect crystal or small defect structures). From the National Center for Electron Microscopy, Lawrence Berkeley Laboratories, University of California, Berkeley, CA 94729, or on the Web at http://ncem.lbl.gov/ncem.html
- Microdiffraction Programs: the listings for several programs are included in Appendix 5 of the book by Spence and Zuo (1992).

CHAPTER SUMMARY

TEMs comprise a range of different instruments which make use of the properties of electrons, both as particles and as waves. The TEM offers a tremendous range of signals from which we can obtain images, diffraction patterns, and several different kinds of spectra from the same small region of the specimen. In the rest of this book we'll take you through the fundamental aspects of electron microscopy, trying to explain at all times *why* we do certain things in certain ways. We'll also explain to some degree *how* we carry out certain operations. Since many different commercial TEMs exist, there's no point in being specific in how to operate the TEM, but we can explain in a generic sense, in many cases, what you have to do to get your microscope to deliver the enormous amounts of information that it generates. Not least of course, we also describe what you need to know to *interpret* the images, diffraction patterns, and spectra that you obtain.

In addition to the WWW, there is a wealth of other sources of information about TEM and, in the general reference list below, we give a selection of appropriate books that emphasize materials science, most of which remain in print, as well as some standard journals and regular conference proceedings.

REFERENCES

General References for TEM

In the reference sections throughout the book, we will list general references that amplify the overall theme of the chapter, as well as specific references that are the source of information referenced in the chapter. If a general reference is referred to specifically in the chapter, we will *not* duplicate it in the specific references.

Books

Amelinckx, S., Gevers, R., and Van Landuyt, J., Eds. (1978) *Diffraction and Imaging Techniques in Material Science,* **1** and **2**, 2nd edition, North-Holland, New York. A collection of excellent individual review articles.

Cowley, J.M., Ed. (1992) *Electron Diffraction Techniques,* **1** and **2**, Oxford University Press, New York. Another collection of excellent individual review articles.

Edington, J.W. (1976) *Practical Electron Microscopy in Materials Science,* Van Nostrand Reinhold, New York. The original out-of-print 1976 edition has been reprinted by TechBooks, 4012 Williamsburg Court, Fairfax, Virginia 22032. It is an essential text, if somewhat outdated.

Goodhew, P.J. and Humphreys, F.J. (1988) *Electron Microscopy and Analysis,* 2nd edition, Taylor and Francis, New York. A succinct summary of SEM, TEM, and AEM.

Hall, C.E. (1953) *Introduction to Electron Microscopy,* McGraw-Hill, New York. A wonderful but nowadays neglected book. The level is

very close to this text. Historically minded students will enjoy the Preface.

Hawkes, P.W. and Kasper, E. (1989, 1994) *Principles of Electron Optics,* **1–3**, Academic Press, New York. 1900 pages, comprehensive but advanced. The third volume deals with many aspects of imaging in the TEM, simulation, and processing with ~118 pages of TEM references. An exceptional modern resource.

Heidenreich, R.D. (1964) *Fundamentals of Transmission Electron Microscopy,* Interscience Publisher, New York. Another wonderful but sometimes forgotten classic.

Hirsch, P. B., Howie, A., Nicholson, R.B., Pashley, D.W., and Whelan, M.J. (1977) *Electron Microscopy of Thin Crystals,* 2nd edition, Krieger, Huntington, New York. For many years, the "Bible" for TEM users!

Loretto, M.H. (1994) *Electron Beam Analysis of Materials,* 2nd edition, Chapman and Hall, New York. A concise overview of the subject.

McLaren, A.C. (1991) *Transmission Electron Microscopy of Minerals and Rocks,* Cambridge University Press, New York. Invaluable for the geologist or ceramist.

Reimer, L. (1993) *Transmission Electron Microscopy; Physics of Image Formation and Microanalysis,* 3rd edition, Springer-Verlag, New York. Essential reference text. Strong physics background required.

Sarikaya, M., Ed. (1992) *Resolution in the Microscope, Ultramicroscopy,* **47**. Actually, a collection of reviews which all concern resolution in different instruments.

Sawyer, L.C. and Grubb, D.T. (1987) *Polymer Microscopy,* Chapman and Hall, New York. A broad-based qualitative introduction to TEM and SEM of polymers.

Thomas, G. (1962) *Transmission Electron Microscopy of Metals,* Wiley, New York. A historical volume—the first hands-on book for the materials scientist.

Thomas, G. and Goringe, M.J. (1979) *Transmission Electron Microscopy of Metals,* Wiley, New York. Invaluable for classical imaging and diffraction topics. The original out-of-print 1979 edition has been reprinted by TechBooks, 4012 Williamsburg Court, Fairfax, Virginia 22032.

von Heimendahl, M. (1980) *Electron Microscopy of Materials,* Academic Press, New York. An introductory-level text, no significant AEM or HRTEM component.

Watt, I.M. (1985) *The Principles and Practice of Electron Microscopy,* Cambridge University Press, New York. A basic, practical introduction to SEM and TEM.

Wenk, H.-R. (1976) *Electron Microscopy in Mineralogy,* Springer-Verlag, New York. Required reading for microscopy of geological or ceramic materials.

Williams, D.B. (1987) *Practical Analytical Electron Microscopy in Materials Science,* 2nd edition, Philips Electron Optics Publishing Group, Mahwah, New Jersey. A basic introduction to AEM. The original out-of-print 1987 edition has been reprinted by TechBooks, 4102 Williamsburg Court, Fairfax, Virginia 22032.

Journals

Advances in Imaging and Electron Physics, Academic Press, New York; formerly *Advances in Optical and Electron Microscopy.*

Journal of Microscopy, Blackwell Science, Oxford, United Kingdom.

Microscopy and Microanalysis, Springer, New York (formerly *Journal of the Microscopy Society of America,* Jones and Begell Publishing, Boston.)

Microscopy, Microanalysis, Microstructure (formerly *Journal de Microscopie et Spectroscopie Electronique*), Les Editions de Physique, Les Ulis Cedex A, France.

Microscopy Research and Technique (formerly *Journal of Electron Microscopy Technique*), Wiley-Liss, New York.

Ultramicroscopy, Elsevier Science Publishers, Amsterdam, the Netherlands.

Conference Proceedings

International Congress for Electron Microscopy—every four years (1994).

European Electron Microscopy Congress—every four years (1996).

Microscopy Society of America, San Francisco Press, San Francisco—annual.

Microbeam Analysis Society, VCH, Deerfield Beach, Florida. From 1996, San Francisco Press, San Francisco—annual.

Electron Microscopy and Analysis, Institute of Physics, Bristol, United Kingdom—odd years (1995).

Scanning Electron Microscopy, Scanning Microscopy International, AMF O'Hare, Illinois—annual.

Useful Sources of Numerical Data and Constants

Fischbeck, H.J. and Fischbeck, K.H. (1987) *Formulas, Facts and Constants,* 2nd edition, Springer-Verlag, New York. An invaluable reference. SI units are described in Chapter 2. Relevant equations in Gaussian units are related to SI units on page 127.

Jackson, A.G. (1991) *Handbook for Crystallography for Electron Microscopists and Others,* Springer-Verlag, New York. Ideal for the microscopist, but see the review by A. Eades (*Microsc. Res. Technique* **21**, 368).

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Kossel, W. and Möllenstedt, G. (1939) *Ann. Phys.* **36**, 113.

Marton, L. (1994) *Early History of the Electron Microscope,* 2nd edition, San Francisco Press, San Francisco.

Ruska, E. (1980) *The Early Development of Electron Lenses and Electron Microscopy* (Trans. T. Mulvey), S. Hirzel Verlag, Stuttgart.

Spence, J.C.H. and Zuo, J.M. (1992) *Electron Microdiffraction,* Plenum Press, New York.

Thompson, G.P. and Reid, A. (1927) *Nature* **119**, 890.