

The Transmission Electron Microscope

CHAPTER PREVIEW

A typical commercial transmission electron microscope (TEM) costs about \$5 for each electron volt (eV) of energy in the beam and, if you add on all available options, it can easily cost up to \$10 per eV. As you'll see, we use beam energies in the range from 100,000 to 400,000 eV, so a TEM is 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 a TEM have appeared as the instrument continues to develop, to the point where it can seriously be claimed that no other scientific instrument exists which can offer such a broad range of characterization techniques with such high spatial and analytical resolution, coupled with a *completely* quantitative understanding of the various techniques. Indeed as nanotechnology and related areas seize both the public and the technological community's imaginations, it is increasingly obvious that the TEM is the central tool for complete characterization of nanoscale materials and devices. Unfortunately, coupled with the TEM's advantages are some serious drawbacks 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 acronyms or initials (go back and read p. xxi) to denote both the technique (microscopy) and the instrument (microscope). We regard all of the different types of TEM as simply variations on a basic theme and that is why only 'TEM' is in the book title. We will also 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 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, how electrons interact with materials, and how we detect the many signals emitted from a specimen in the TEM.

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 central role of the computer in today's TEM operation and analysis. A basic lesson to take away from this chapter is not just the versatility of the TEM but the fact that it is fundamentally a signal-generating and detecting instrument rather than simply a microscope for high-resolution images and diffraction patterns (we'll call them DPs), which is how it operated for many decades.

1.1 WHAT MATERIALS SHOULD WE STUDY IN THE TEM?

The materials scientist has traditionally examined metals, alloys, ceramics, glasses, polymers, semiconductors, and composite mixtures of these materials, with

sporadic adventures into wood, textiles, and concrete. In addition to thinning them from the bulk state, particles and fibers of some of these materials are also commonly studied and, in such shapes, they are sometimes thin enough for direct TEM examination. Nanotechnology, which will feature as a common theme throughout

this book, is defined as “the ability to understand and control matter at dimensions of roughly 1 to 100 nanometers, where unique phenomena enable novel applications. Encompassing nanoscale science, engineering and technology, nanotechnology involves imaging, measuring, modeling, and manipulating matter at this length scale” (URL #1).

THE CRUCIAL WORDS

“Imaging, measuring, modeling, and manipulating matter” can be accomplished with the help of the TEM and are often thrown together as part of the emerging field of ‘nanocharacterization,’ a term which we will try not to use too often.

When we create nanoscale materials, they come with specific dimensional limits in 1D, 2D, or 3D and the TEM is well suited to observing them, precisely because of these limits. We will include examples of archetypal dimensionally limited structures throughout the book. For example, single layers (such as graphene sheets or quantum wells), nanotubes and nanowires, quantum dots, nanoparticles, and most catalyst particles can be viewed as 1D structures. We can put all of these types of specimen into the TEM without modification, since 1D is always thin enough for direct observation; 2D nanomaterials include interfaces, and complex 3D nanomaterials are typified by multilayer, semiconductor devices, functional materials, or nanoporous structures such as substrates for catalyst-particle dispersions. Lastly, we should note the rapidly growing interface between the nano- and the bio-worlds. While much of biological electron microscopy has been superseded in the last decade or more by less-damaging techniques such as confocal, two-photon, multi-photon, and near-field light microscopies, there is still a major role for TEM in biomaterials, bio/inorganic interfaces, and nano-bio/biomaterials.

1.2 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.

1.2.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 in 1927 two



FIGURE 1.1. The electron microscope built by Ruska (in the lab coat) and Knoll, in Berlin in the early 1930s.

research groups, Davisson and Germer and Thomson and Reid, 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” he was quoted as saying), in 1986, shortly before his death in 1988. 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. Some idea of the power of Ruska’s breakthrough is the fact that commercial TEMs were first developed only 4 years later. The Metropolitan-Vickers EM1 was the first such instrument and was built in the UK in 1936. Apparently it didn’t work very well and regular production of commercial TEMs 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 mid-1950s when Bollman in Switzerland and Hirsch and co-workers in Cambridge, in the UK, perfected techniques to thin metal foils to electron transparency. (In fact, because so much of the early TEM work examined metal specimens, the word ‘foil’ came to be synonymous with ‘specimen’ and we’ll often use it this way.) 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 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. Other materials-oriented texts followed, notably the first student-friendly ‘hands-on’ text by Edington.

Today TEMs constitute arguably the most efficient and versatile tools for the characterization of materials over spatial ranges from the atomic scale, through the ever-growing ‘nano’ regime (from < 1 nm to ~ 100 nm) up to the micrometer level and beyond. If you want to read a history of the TEM, the book by Marton (1968) is a compact, personal monograph and the text edited by Hawkes in 1985 contains a series of individual reminiscences. Fujita’s (1986) paper emphasizes the substantial contribution of Japanese scientists 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, Hashimoto 1986, Howie 2000, Thomas 2002, Zeitler 2003) 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). More recently, Haguenu et al. (2003) compiled an extensive list of references describing key events in the history of electron microscopy. As always, there is a wealth of information, some of it accurate, available on the Web.

1.2.B Microscopy and the Concept of Resolution

When asked “what is a microscope?,” 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 (VLM). Because of the general familiarity with the concept of the VLM, we will draw analogies between electron and 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 by which to see. This distance is the *resolution* or (more accurately) the *resolving power* of our eyes. So any instrument that can show us pictures (or *images* as we’ll often 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 should 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 your retinal cells, but you wouldn’t see anything (ever again!). So an integral part of any electron microscope is a viewing screen of some form (now usually a flat-panel computer display), which displays electron intensity as light intensity, which we first observe and then record photographically or store digitally. (We’ll discuss these screens and other ways of recording electron images in Chapter 7.)

VLM

We’ll try to avoid the phrases ‘optical microscope’ (they all are) and ‘light microscope’ (some are very heavy). ‘Visible-light microscope/y’ is simple and appropriate use of the hyphen.

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 classic Rayleigh criterion for VLM, which states that the smallest distance that can be resolved, δ , is given approximately by

$$\delta = \frac{0.61\lambda}{\mu \sin \beta} \quad (1.1)$$

In equation 1.1, λ is the wavelength of the radiation, μ the refractive index of the viewing medium, and β 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, and so the resolution of a good VLM is about 300 nm. In TEMs we can approximate the best resolution using an expression similar to equation 1.1 (actually $\sim 1.22\lambda/\beta$) which, as we’ll see later, is very small.

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 VLM. Also, 300 nm is well above the upper limit of the nano regime which we defined earlier. So there’s a real need in nano/materials science and engineering to image details, all the way down to the atomic level, if we want to understand and ultimately control 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 the last 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.) Louis 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 (ignoring the inconsistency in units)

$$\lambda = \frac{1.22}{E^{1/2}} \quad (1.2)$$

In this equation E is in electron volts (eV) and λ in nm.

So from equation 1.2 you can work out that for a 100 keV electron, $\lambda \sim 4$ pm (0.004 nm), which is much smaller than the diameter of an atom.

V AND eV

Remember that we should be precise in our use of these units: V represents the accelerating voltage of the microscope while eV refers to the energy of the electrons in the microscope (look ahead to equation 1.4 to see the relation between the two).

We’ll see later that we cannot yet build a ‘perfect’ TEM that approaches this wavelength-limited limit of resolution, because we can’t make perfect electron lenses (see Chapter 6). Until recently, a top of the line lens could rightly be compared to using the bottom of a Coca-Cola™ bottle as a lens for light microscopy. 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 1.2A.

The advantages of shorter wavelengths led in the 1960s to the development of high-voltage electron microscopes (HVEMs), with accelerating potentials between 1 and 3 MV. In fact, rather than push the resolution limits, most of these instruments were used to introduce controlled amounts of radiation damage into specimens, in an attempt to simulate nuclear-reactor environments. Three-Mile Island and Chernobyl contributed to changes in the emphasis of energy research; recently there has not been much call for HVEMs. Today, climate change is forcing a reconsideration of nuclear power. 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 200–400 kV, but still offer very high resolution, close to that achieved at 1 MV. In fact, progress is such that most IVEMs purchased these days are, effectively, HRTEMs with atomic resolution.

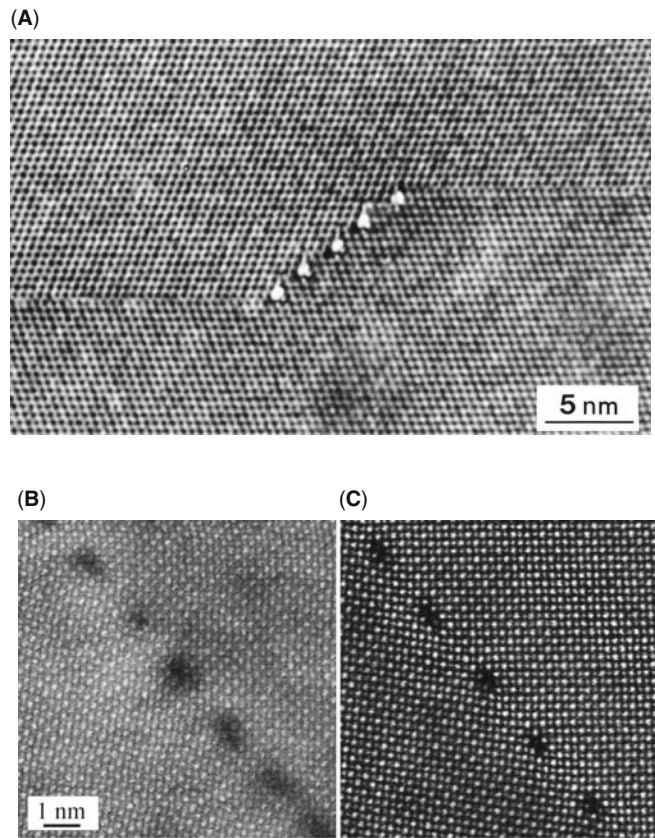


FIGURE 1.2. (A) A twin boundary in spinel stepping from one {111} plane to another parallel plane. The white dots are columns of atoms. The change in atomic orientation across the twin boundary can be readily seen even if we do not know what causes the white dots or why indeed they are white. (B) A grain boundary in SrTiO₃ imaged without C_s correction and (C) with C_s correction. As you can see, the effect is just as dramatic as putting on your reading glasses (if you need them).

We are still improving the resolution, and recent breakthroughs in spherical- and chromatic-aberration corrections (see Chapters 6 and 37, respectively) are revolutionizing the TEM field. Among many advantages, corrections of spherical aberration (which, for reasons we’ll explain in Chapter 6, we abbreviate to C_s) and chromatic aberration (C_c) allow us to produce sharper atomic-resolution images. By filtering out electrons of different wavelengths we can also better image thicker specimens.

The combination of IVEMs and C_s correction has pushed TEM image resolution to well below the 0.1 nm (1 Å) barrier. Today the point has perhaps been reached where the drive for much better resolution is now no longer paramount and the TEM will develop more constructively in other ways. As we’ll illustrate many times throughout the book and elaborate in the companion text, C_s correction is perhaps the most exciting advance in TEM technology in several decades and Figure 1.2B and C shows beautifully the difference in a typical atomic-resolution image with and without C_s correction. The advantages of C_s and C_c aberration correction in

TEM are explored in depth in chapters on C_s correction and energy-filtered TEM (EFTEM) in the companion text.

C_s, C_c AND MAGNIFICATION

Having extolled the virtues of C_s correction it is worth pointing out that most TEM images are recorded at magnifications where such correction makes *no* discernible difference. Most TEM specimens are not thin enough to produce images with resolution that benefits from C_s correction. For thicker specimens C_c correction via energy filtering is much more useful.

1.2.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 the tightly bound, inner-shell electrons from the attractive field of the nucleus by transferring some of its energy to individual atoms in the specimen.

One of the advantages of 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 (AEM), giving us chemical information and a lot of other details about our specimens. AEM uses X-ray energy-dispersive spectrometry (XEDS) and electron energy-loss spectrometry (EELS). For example, Figure 1.4A shows X-ray spectra from very small regions of the TEM specimen imaged in Figure 1.4B. The spectra exhibit characteristic peaks, which identify the different elements present in different regions. We can transform such spectra into quantitative

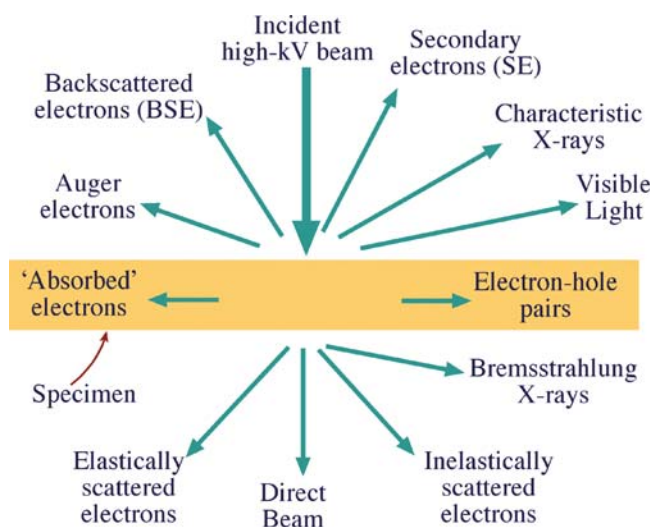


FIGURE 1.3. Signals generated when a high-energy beam of electrons interacts with a thin specimen. Most of these signals can be detected in different types of TEM. The directions shown for each signal do not always represent the physical direction of the signal, but indicate, in a relative manner, where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

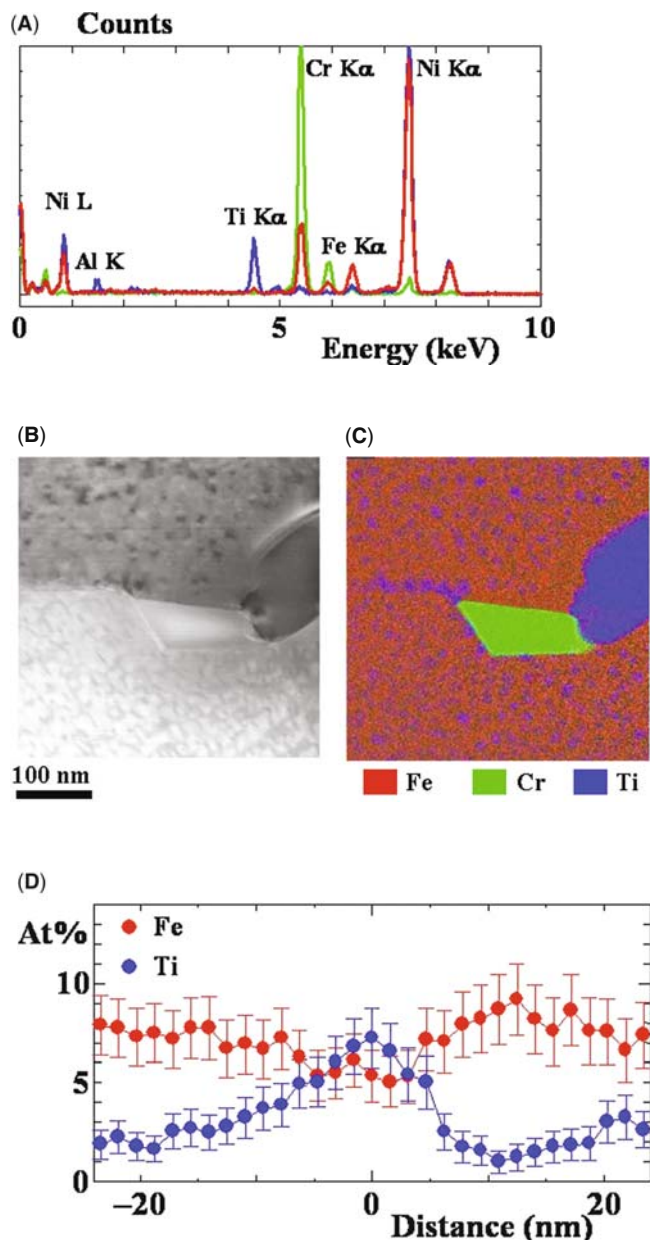


FIGURE 1.4. (A) X-ray spectra from three different regions of a specimen of Ni-base superalloy imaged in (B). The spectra are color-coded to match the different regions of the specimen highlighted in (C) which is a quantitative map showing the distribution of the elements detected in the spectra in (A) (e.g., green areas are rich in Cr, blue areas contain predominantly Ti, etc.). Quantitative composition profiles showing the localized changes in composition across one of the small matrix precipitates in (C) are shown in (D).

images of the distributions of all the elements present in the specimen (Figure 1.4C) and from such images extract quantitative data describing elemental changes associated with inhomogeneous microstructures as shown in Figure 1.4D. This and similar analyses with EELS comprise Part 4 of the book. In contrast, microscopes using non-ionizing radiation, such as visible light, usually only generate light (but not much heat, which is good).

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; modern TEMs are very good signal-generating instruments. To localize these signals we need our TEM to produce a very small electron beam (or *probe* as it is often called), typically <5 nm and at best <0.1 nm in diameter. We combine 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 (or scanned-probe) 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. AEMs offer improved analytical performance at intermediate voltages, similar to the improvement in image resolution gained in standard TEMs.

Most importantly, C_s correction permits the generation of smaller electron probes with higher currents, thus significantly improving both analytical spatial resolution and sensitivity. Chromatic-aberration correction (i.e., energy filtering) also offers the opportunity to form images of electrons with a whole range of specific energies, permitting such breakthroughs as band-gap imaging and chemical-bond imaging.

1.2.D Depth of Field and Depth of Focus

The depth of field of a microscope is a measure of how much of the *object* that we are looking at remains in focus at the same time; the term depth of focus refers to the distance over which the *image* can move relative to the object and still remain in focus. If you are confused, it may help to recall that *depth of field* and *field of view* both refer to the *object* in everyday photography. The lenses in the TEM govern these properties just as they determine the resolution. Electron lenses are not very good, as we've already mentioned, and one way to improve their performance is to insert very small limiting apertures, narrowing the beam down to a thin 'pencil' of electrons which at most is a few micrometers across. These apertures obviously cut down the intensity of the electron beam, but they also act to increase the depth of field of the specimen and depth of focus of the images that we produce, as we explain in detail in Chapter 6.

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, your specimen is usually in focus from the top to the bottom surfaces at the same time, independent of its topography, so long as it's electron transparent! Figure 1.5 shows a TEM image of some dislocations in a crystal.

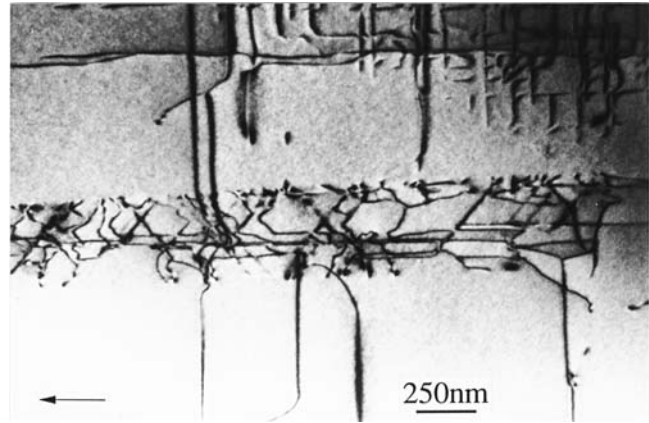


FIGURE 1.5. TEM image of dislocations (dark lines) in GaAs. The dislocations in the band across the middle of the image are on slip planes close to 90° to one another and thread through the thin specimen from the top to the bottom but remain in focus through the foil thickness.

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 surfaces, and they remain in sharp focus at all times. (By the time you finish reading this book, you should be able to work out which is the top and which is the bottom surface of the specimen.) Furthermore, you can record the final image at different positions below the final lens of the instrument and it will still be in focus (although the magnification will change). Compare these properties with the VLM where, as you probably know, unless the surface of your specimen is flat 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 VLM. You should note that, in this rare situation, C_s correction is not an advantage since it permits the use of larger apertures without degrading the resolution of the lens. But *smaller* apertures are the ones that give better depth of focus and depth of field (see Section 6.7). However, if you are using a C_s corrector, your specimen has to be so thin that it will still remain in focus except under extreme conditions. We'll see more on this topic in the companion text and also mention using TEM in a 'confocal' mode.

1.2.E Diffraction

As we've noted, Thompson, Reid, Davisson, and Germer independently showed that electrons could be diffracted when passing through thin crystals of nickel. Performing electron diffraction in TEMs was first realized by Kossel and Möllenstedt (1939). Today, electron diffraction is an indispensable part of TEM and is arguably the most useful aspect for materials scientists and nanotechnologists for whom crystal structure (and particularly crystal defects) is an essential characteristic when it comes to controlling properties. Figure 1.6

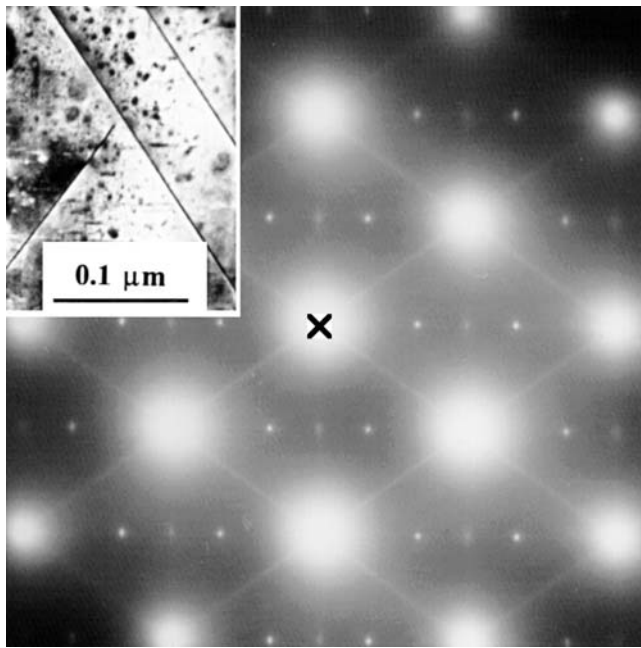


FIGURE 1.6. TEM DP from a thin foil of Al-Li-Cu containing various precipitate phases, shown in the inset image. The central spot (X) contains electrons that come directly through the foil and the other spots and lines are diffracted electrons which are scattered from the different crystal planes.

shows a TEM DP that 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 2 that, in addition to the things we just listed, if you converge the usually parallel TEM beam to a focused probe, then you can produce even more striking convergent-beam patterns (see Figure 2.13D) from which you can conduct a complete crystal-symmetry analysis of minuscule crystals, including such esoteric aspects as point-group and space-group determination. You shouldn't be surprised by now if we tell you that aberration correction can produce even better DPs, which are both sharper (by reducing chromatic aberration) and come from smaller regions of the specimen (by reducing C_s). The crystal structure produces no diffraction information in a VLM because of the relatively large wavelength of visible light.

KEY POINT TO REMEMBER

At all times the crystallographic information in the DP (and all the analytical information) can be related to the image of your specimen.

So a TEM can produce atomic-resolution images, it can generate a variety of signals telling you about your specimen 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.

1.3 LIMITATIONS OF THE TEM

1.3.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 around 1970 estimating that all TEMs, since they first became available commercially (~15 years), had only examined 0.3 mm^3 of material! Extending that calculation to the present time probably increases this volume to no more than 10^3 mm^3 . So we have an instrument that is not a good sampling tool! This sampling problem only serves to emphasize that, if you're just starting your research, 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 VLM, and the SEM. In other words, know the forest before you start looking at the veins in the leaves on the trees.

1.3.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 illustrates this problem well by showing a picture of two rhinoceros 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 (sometimes called the projection-limitation) is that generally all the TEM information that we talk about in this book (images, DPs, spectra) is averaged through the thickness of the specimen. In other words, a single TEM image has no depth sensitivity. As we noted in Figure 1.5 there often is information about the top and bottom surfaces of the thin foil, but this is not immediately apparent. So

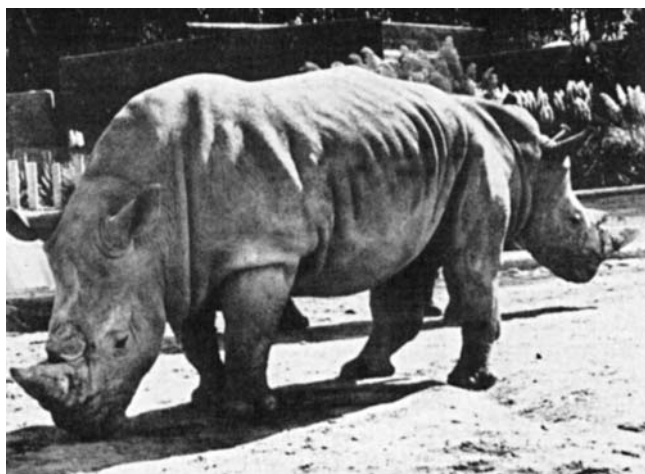


FIGURE 1.7. Photograph of two rhinos taken so that, in projection, they appear as one two-headed beast. Such projection artifacts in reflected-light images are easily discernible to the human eye but similar artifacts in TEM images are easily mistaken for 'real' features.

other techniques which are more clearly surface sensitive or depth sensitive, such as field-ion microscopy, scanning-probe microscopy, Auger spectroscopy, and Rutherford backscattering, are necessary complementary techniques if you want a full characterization of your specimen.

However, there has been progress in overcoming this limitation, which was much more of a problem for biologists interested in the shape of complex molecules, cells, and other natural structures. So they invented the technique of electron tomography, which uses a sequence of images taken at different tilts to create a 3D image, identical in principle to the more familiar medical CAT (computerized-axial tomography) scans using X-rays. Recently, there has been rapid improvement in specimen-holder design to permit full 360° rotation and, in combination with easy data storage and manipulation, nanotechnologists have begun to use this technique to look at complex 3D inorganic structures such as porous materials containing catalyst particles. This relatively new aspect of TEM for materials scientists is explored in depth in the companion text.

1.3.C Electron Beam Damage and Safety

A detrimental effect of ionizing radiation is that it can damage your specimen, particularly polymers (and most organics) or certain minerals and ceramics. Some aspects of beam damage are exacerbated at higher voltages, and with commercial instruments offering up to 400 kV, beam damage can now limit much of what we do in the TEM, even with refractory metals. The situation is even worse with more intense beams made

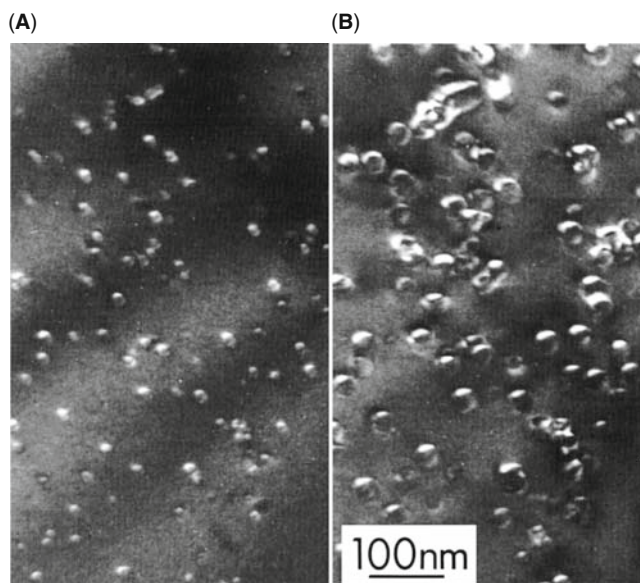


FIGURE 1.8. Beam damage (bright bubble-like regions) in quartz after bombardment with 125 keV electrons. With increasing time from (A) to (B) the damaged regions increase in size.

possible because of advances in C_s correction. Figure 1.8 shows an area of a specimen damaged by high-energy electrons.

However, all is not lost and we can combine more intense electron sources with more sensitive electron detectors and use computer enhancement of noisy images to minimize the total dose received by the specimen to levels below the damage threshold. Minimum-dose microscopy techniques, often combined with specimen cooling (cryo-microscopy) and low-noise, charge-coupled device (CCD) cameras (see Chapters 7 and 31, respectively), are standard approaches in biological TEM and permit images to be obtained even when only a few hundred electrons/ nm^2 are hitting the specimen. These approaches are finding increasing usage in TEM of materials where digital control of the beam in STEMs is another way to minimize radiation damage.

The combination of high kV beams with the intense electron sources that are available means that you can destroy almost any specimen, if you 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 will kill tissue (and managed to damage some operators in the early days of the technique). 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!

1.3.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, CCD, or photographic plate to give an interpretable image in a reasonable time. Generally this requirement is a function of the electron energy and the average atomic number (Z) of your specimen. Typically for 100-keV electrons, specimens of aluminum alloys up to $\sim 1 \mu\text{m}$ would be thin, while steel would be thin up to about several hundred nanometers. However, it is an axiom in TEM that, almost invariably, *thinner is better* and specimens $< 100 \text{ nm}$ should be used wherever possible. In extreme cases such as doing HRTEM or electron spectrometry, specimen thicknesses $< 50 \text{ nm}$ (even $< 10 \text{ nm}$) are essential. These demands become less strict as the beam voltage increases, but this is offset by the production of beam damage.

Again these words of caution are balanced by the development of a specialized specimen-preparation tool called a focused ion beam (FIB) which is essential to the semiconductor-device fabricators who use them (by the dozen) to prepare, in a matter of a few tens of minutes, thin foils of specific, individual gates or junctions from one of the many millions of such on a 12-in. VLSI wafer. The only drawback is that, to buy a FIB, you have to pay as much as it costs to buy a TEM. We'll talk about this and other methods of specimen preparation in Chapter 10 and in the companion text.

THIN SPECIMENS

A *major* limitation of the TEM is we need thin specimens. 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 specimens, changing both their structure and chemistry. So you need to be aware of the drawbacks of specimen preparation and learn to recognize the artifacts introduced by standard preparation methods.

A terminological distinction is worth noting here. The words *sample* and *specimen* are often used interchangeably but, in this text, we'll distinguish the specimen specifically as the thin piece of material that you insert into the TEM in a specimen holder and we'll assume that the specimen was thinned from a much larger, bulk sample of the material you are interested in studying. Sometimes we'll mix the terms to test your understanding.

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 VLM and SEM before venturing into TEM studies. Otherwise you may fall-foul of some of the limitations we have just listed. But you should also have got the message that we are constantly improving the technique and overcoming these limitations while, at the same time, making the positive aspects even better.

1.4 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 200 or 300 keV TEM can combine aspects of *all* the above microscope types. Figure 1.9 shows several 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- or three-story room; the operator shows the scale of this instrument. A modern machine essentially is an electron-optical 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 a flat-panel display: there is no (internal) viewing screen. This aspect is becoming a more popular design feature in TEMs because, if the screen is not in the microscope, then the operator doesn't have to be in the room or in the building or even in the same country. Removing the operator from proximity to the TEM overcomes many of the factors that limit the performance of the best instruments. Remote operation (or telepresence) is an increasingly attractive feature, which will give many more researchers access to the most sophisticated TEMs, as already happens in the world of astronomy with major telescopes.

1.5 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 that 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 GI Taylor's famous experiment in which he

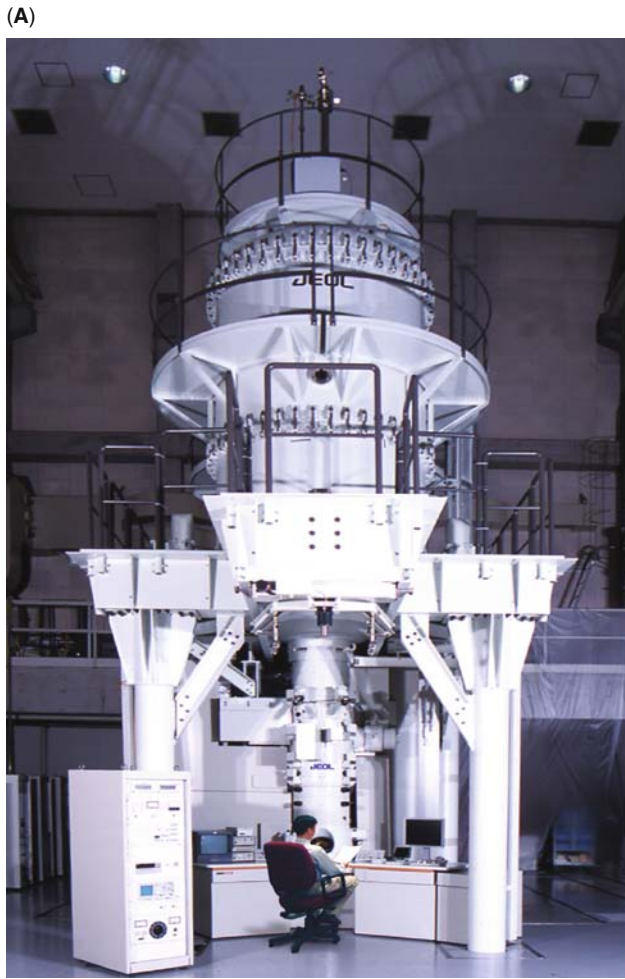
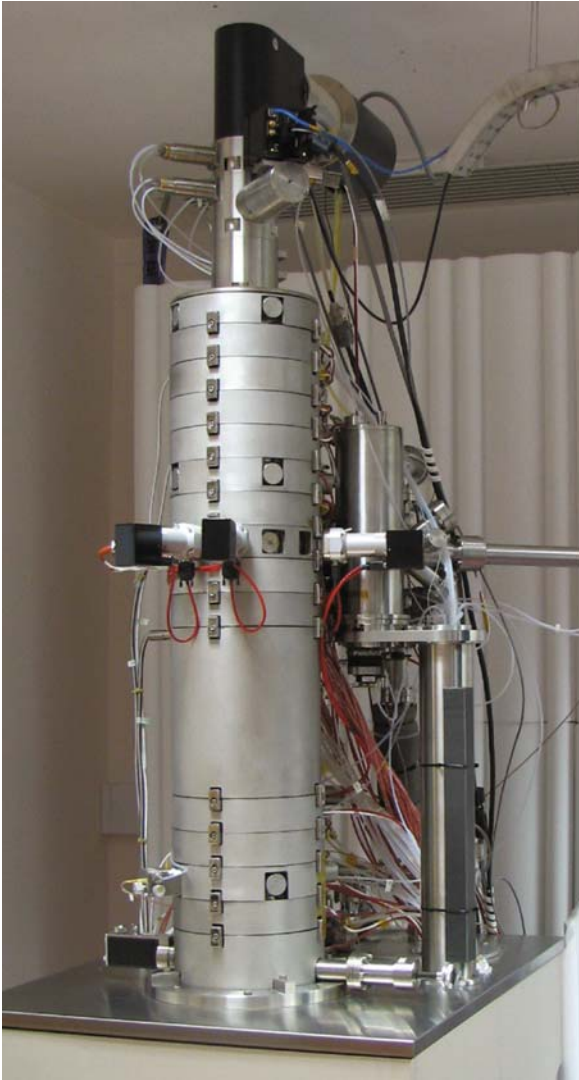


FIGURE 1.9. (Continued)

(E)



(F)

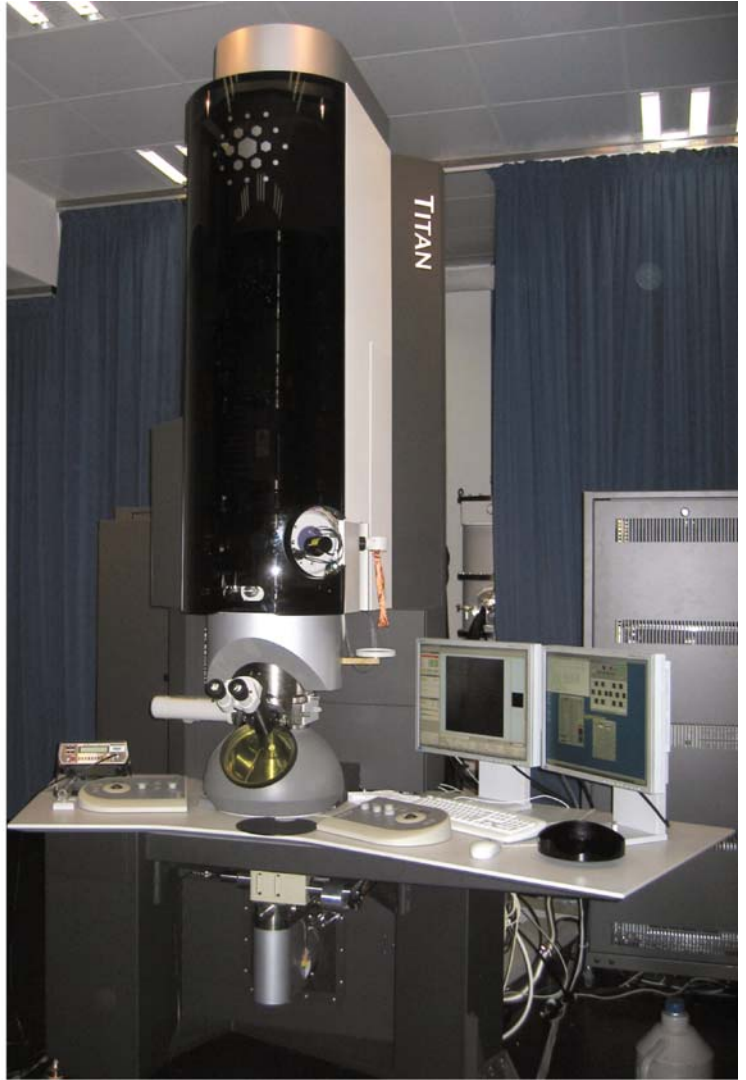


FIGURE 1.9. A selection of different commercial TEMs: (A) JEM 1.25 MeV HVEM. Note the size of the instrument; often the high-voltage tank is in another room above the column. (B) Zeiss HRTEM with a C_s corrector and an in-column energy filter. Note the large frame to provide high mechanical stability for the highest-resolution performance. (C) Hitachi 200 keV dedicated STEM; note the absence of a viewing chamber. Such instruments are often designed to aid failure analysis for the semiconductor device manufacturers. Specimens thinned from wafers on the production line can be easily transferred and examined. (D) JEOL 200 keV TEM/STEM; note also the absence of a viewing chamber. (E) Nion 200 keV ultrahigh vacuum SuperSTEM; the only US-manufactured (S)TEM and current holder of the world record image resolution (F) FEI Titan. Comparison with Ruska's instrument (Figure 1.1), which is 70–80 years older than these instruments, is instructive.

demonstrated Young's slits interference patterns despite using such a weak light source that only one photon passed through either slit at any one time. The electron beam current in a TEM can be as high as $\sim 0.1\text{--}1\ \mu\text{A}$, 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 $0.5c$ (actually $\sim 1.6 \times 10^8\ \text{m/s}$), so they are separated by $\sim 1.6\ \text{mm}$ 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

require interaction between different electron waves. 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, along with some relevant physical constants.

There are a few important equations that 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

$$\lambda = \frac{h}{p} \quad (1.3)$$

TABLE 1.1 Fundamental Constants and Definitions

Charge (e)	(-) 1.602×10^{-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 \times voltage)	1.602×10^{-19} N m (for 1 volt potential) = J
Planck's constant (h)	6.626×10^{-34} N m s
1 A	1 C/s
Speed of light in vacuum (c)	2.998×10^8 m/s

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, thus

$$eV = \frac{m_0 v^2}{2} \tag{1.4}$$

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

$$p = m_0 v = (2m_0 eV)^{1/2} \tag{1.5}$$

These three simple equations define the relationship between the electron wavelength λ and the accelerating voltage of the electron microscope, V

$$\lambda = \frac{h}{(2m_0 eV)^{1/2}} \tag{1.6}$$

If you look back, this equation is equivalent to equation 1.2. The inverse relationship between λ and V introduces a very important concept: by increasing the accelerating voltage we decrease the wavelength of the electrons. So you, the operator, can do this whenever you wish!

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 non-relativistic 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 energies $> \sim 100$ keV because the velocity of the electrons (as particles) becomes greater than half the speed of light! So to be exact we must modify equation 1.6 to give

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

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 greater for higher accelerating voltages as shown in Table 1.2 which comprises all the commercial TEM accelerating voltages.

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) or in the electronic version of the almost 100-year-old standard source, Kaye and Laby (1986) (URL #2), or on the NIST database from which you can quickly find any number or constant that you need (URL #3).

TABLE 1.2 Electron Properties as a Function of Accelerating Voltage

Accelerating voltage (kV)	Non-relativistic wavelength (nm)	Relativistic wavelength (nm)	Mass ($\times m_0$)	Velocity ($\times 10^8$ 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

1.6 MICROSCOPY ON THE INTERNET/ WORLD WIDE WEB

TEM users are well integrated into the Internet and the World Wide Web (WWW) and this is a source of useful information (and also some useful knowledge!) about what's going on in the field. You can view research TEMs in real time over the Internet and, as we've seen, you may not only see other instruments but you may be able to operate them remotely (a quick search on tele-microscopy or telepresence microscopy will get you some useful hits). So rather than spend a week in sunny Storrs operating an advanced TEM you can (regretfully) do it from the comfort of your own office or lab (or even from the beach in Aruba if there's a good broadband connection). As Internet2 and the National Lambda Rail further penetrate research labs in the United States and equivalent systems spread throughout the world, the rapid exchange of experimental information and the parallel simulation of data via access to high-end computational resources will continue to expand the options available to microscopists and analysts.

In addition, specialized software packages are also available on the Web which allow you to carry out many of the advanced analyses that we will introduce in this text (e.g., DP analysis and image/diffraction/spectral simulation). In many cases access to this software is limited (i.e., you have to pay for it) but any serious microscopy operator should have access to such software on site. Sometimes it is useful to explore the possibilities before you purchase.

We have already referenced a few important URLs in this chapter and we will continue to do so throughout the book and give lists at the end of each chapter. We have tried to keep the referenced URLs to sites associated with longstanding, reputable organizations such as national labs, professional societies, and major publishers. A list of broadly useful sites is included below and, not surprisingly, much of this list is very different from the one that we gave more than a decade ago when the first edition of this text was published. Nothing much has changed in this respect and so we cannot guarantee that these same URLs will still be active when you get round to searching for them. Such is life in the age of the Web.

1.6.A Microscopy and Analysis-Related Web Sites

<http://www.amc.anl.gov> This is the best source for TEM information on the Web in the United States and it is run by NJ 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 to access Software/Image Libraries. Other useful connections through this site include:

<http://microscopy.com/MicroscopyListserver/> This is an email-based discussion forum giving members of the EM community a centralized Internet address to which questions/comments/answers in the various fields of microscopy or analysis can be rapidly distributed to a list of (subscribed) individuals by electronic mail.

<http://www.microscopy.com/MMMeetingCalendar.pl> Listing of currently planned meetings, symposia, and courses.

<http://zaluzec.com/cgi-bin/ANLWWWListingSQL.pl?SearchOrg=society> This is a listing of the Web sites of various national and international microscopy and analysis societies. There are also connections to university, government, and individual microscopy-related sites.

<http://cimewww.epfl.ch/EMYP/emyp.html> This is the home of the Electron Microscopy Yellow Pages which is a similar operation to the ANL site, but based at the Ecole Polytechnique Fédérale de Lausanne in Switzerland, run by P Stadelmann. The Yellow Pages contain electron microscopy laboratories, software for electron microscopy, learned societies, instruments, equipment and consulting education in electron microscopy, data and databases, news and publications-related sources of information, conferences, workshops and schools, and getting somewhere else on the Web.

<http://cimesg1.epfl.ch/CIOL/> and <http://cimesg1.epfl.ch/CIOL/summary.html> Stadelmann also offers access to very sophisticated EMS software for high-resolution image analysis and diffraction simulation and much more detail about this is covered in Chapter 30 and in the companion text.

<http://iucr.org/resources/commissions> A great resource operated for the International Union of Crystallography by JCH Spence at Arizona State University giving listings of electron diffraction and microscopy-related software, etc.

http://www.numis.northwestern.edu/IUCR_CED/ L Marks resource site for electron microscopy and diffraction

<http://tem.msae.wisc.edu/emdb/index.html> NSF supported TEM data base maintained by P. Voyles.

1.6.B Microscopy and Analysis Software

There is a lot of software available on the WWW and 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 analysis. 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.

- **Cross sections for electron scattering:** The NIST version 3.0 of this database provides values of differential elastic-scattering cross sections, total elastic-scattering cross sections, phase shifts, and transport cross sections for elements with atomic numbers from 1 to 96 and for electron energies between 50 eV and 300 keV (in steps of 1 eV). Free downloads at <http://www.nist.gov/srd/nist64.htm>.
- **CRISP:** It is a commercial package running under Windows on a PC. It is designed for imaging process 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). www.calidris-em.com/crisp.htm—the site does not change very often.
- **DigitalMicrograph™ (DM):** It is a complete system for the acquisition, control, and processing of digital images from any electron microscope and is the central software for the Gatan Microscopy Suite™ (GMS) (see below). Gatan is to TEM as Microsoft is to PC.
- **DTSA (Desk-Top Spectrum Analyzer):** The NIST/NIH Desktop Spectrum Analyzer generates, interprets, and analyzes X-ray spectra from specimens under electron bombardment. This remarkable software/database package simulates the experimental environment and emulates specimen properties to generate spectra reflecting the relevant physics, chemistry, and statistics of a real-world application. DTSA incorporates many widely accepted X-ray data analysis procedures. Technical Contact: johnhenry.scott@nist, (301) 975-4981; <http://www.cstl.nist.gov/div837/Division/outputs/DTSA/DTSA.htm>. Really essential for the X-ray analyst but currently only available from NIST for Macintosh users. A PC version has been developed by Masashi Watanabe at Lehigh University.
- **ELD:** It is a commercial package from the producers of CRISP running under Windows on a PC. It is intended for quantitative analysis of DPs and is available from Calidris, Manhemsvägen 4, S-191 46 Solltuna, Sweden (46 8 625 00 41). www.calidris-em.com/eld.htm—still not changing often.
- **ELP:** The original Gatan energy-loss acquisition, processing, and analysis program now incorporated as part of the Gatan Microscopy Suite™ (GMS) (see below).
- **EMS and jEMS:** Image simulation program; see the listing of its capabilities in Section 1.6.A.
- **Gatan Microscopy Suite™ (GMS):** It permits data acquisition, processing, and analysis for a wide range of TEM applications including EELS analysis, and energy-filtered compositional mapping, DP analysis,

and 3D tomography acquisition. Open to individual scripting for user-specific needs. Widely used by many microscopists and analysts for acquiring, analyzing, and processing any kind of image data. Use with a CCD camera that provides digital images from the TEM or interface to any STEM system. From Gatan Inc., 5933 Coronado Lane, Pleasanton, CA 94588, (925) 463-0200; info@gatan.com.

- **ImageJ:** It is a versatile and extremely powerful image-processing and analysis open-source freeware available from NIH (has largely replaced the popular NIH Image). Developed by Wayne Rasband and aided by input from more than 1400 users round the world, ImageJ runs on Linux, Mac OS 9, Mac OS X, Windows. It is the world's fastest pure Java image-processing program. It can filter a 2048 × 2048 image in 0.1 s (40 million pixels per second!). Open and save all supported data types as TIFF (uncompressed) or as raw data. Open and save GIF, JPEG, BMP, PNG, PGM, FITS, and ASCII. Open DICOM. Open TIFFs, GIFs, JPEGs, DICOMs, and raw data using a URL. Open and save many other formats using plugins. Supports smoothing, sharpening, edge detection, median filtering, and thresholding on both 8-bit grayscale and RGB color images. Measure area, mean, standard deviation, min, and max of selection or entire image. Measure lengths and angles. Use real-world measurement units such as millimeters. Calibrate using density standards. Generate histograms and profile plots and much more. Available from <http://rsb.info.nih.gov/ij/>. Also look for Image SXM, a version of NIH Image extended to handle the loading, display, and analysis of scanning images: <http://www.liv.ac.uk/~sdb/ImageSXM/>.
- **MacIspix:** A Macintosh-based image-processing program, which works in conjunction with NIH Image (see below) or ImageJ (see above); permits analysis of Stacks: Movies, Depth profiles, Cropping and saving of large data sets. Groups: Coordinated measurements, Color overlays, Scatter diagrams. Pixels: bit, byte, integer, RGB, real, complex. Statistical measurements. Signal/noise determinations. Also diffraction analysis, segmentation (blobbing and measurement), registration, principal component analysis. Developed by David Bright at NIST (micro@nist.gov) and can be downloaded (free) from <http://www.nist.gov/lispix/MLxDoc/mlx.html>.
- **MacTempas and CrystaKit:** MacTempas is a Macintosh-based image analysis program for the simulation of high-resolution images, DPs, and crystal structures. Features include

Full multislice calculation of HRTEM Images and dynamical DPs.

Automatic calculation of the correct unit cell for any given beam orientation.

Automatic selection of correct array size and shape for the multislice calculation.

Display of atomistic models from any direction in both color and shades of gray.

Display of projected potentials, Fourier coefficients, diffractogram of images.

Plotting of amplitudes/phases of diffracted beams vs. thickness, contrast transfer functions.

Database on the 230 space groups; calculates the associated symmetry operators and atomic coordinates.

<http://www.totalresolution.com/MacTempas.html>.

CrystalKit: It works within MacTempas or EMS and builds models of crystalline defects, from point defects to grain boundaries and precipitates. A geometric grain boundary involving several thousand atoms can be generated in a matter of minutes by specifying the orientation relation between the grains, the interface plane, and the zone axis. CrystalKit allows the user to freely rotate the crystal, identify planes, measure angles and distances between atoms, visually move atoms, delete atoms, and add new atoms or create an arbitrary path interface.

<http://www.totalresolution.com/CrystalKit.html>.

- **Monte Carlo Simulations:** Software to simulate electron-beam trajectories through materials for

estimating the spatial resolution of X-ray analysis or the backscattered electron yield. The best source of information is NIST's Web site <http://www.cstl.nist.gov/div837/837.02/epq/index.html>.

For thin specimens you should use David Joy's Monte Carlo program described in his book (Joy 1995) and available at the University of Tennessee Web site <http://web.utk.edu/~srcutk/htm/simulati.htm>.

- **NIH-Image** (and **ImageJ**): It is a public domain software from NIH, developed by Wayne Rasband for general image manipulation with a limited set of image-processing tools. Details can be found at <http://rsb.info.nih.gov/nih-image/about.html> but it has been largely replaced by ImageJ (see above).
- **Adobe Photoshop:** Professional page layout programs for presentations and labeling your figures. Still the standard and available for a nominal fee at <http://www.adobe.com/products/photoshop/>.
- **Diffraction-Pattern Indexing:** Start with <http://emaps.mrl.uiuc.edu> JM Zuo's excellent free site at the University of Illinois. After this you should check the EM Yellow Pages and the EMS software and also the Web site for the International Union of Crystallography (both already referenced in Section 1.6.A). Also try SingleCrystalTM; part of the extensive CrystalMaker[®] suite of DP and crystal structure software. Free demo download at: <http://www.crystallmaker.co.uk/singlecrystal/index.html>.

CHAPTER SUMMARY

TEMs comprise a range of different instruments that make use of the properties of electrons, both as particles and as waves. The TEM generates a tremendous range of signals so we can obtain images, DPs, 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 TEM, 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 a particular TEM, but we can explain in a generic sense, in many cases, what you have to do to get your particular one to deliver the enormous amounts of information that it can generate. Not least of course, we also describe what you need to know to *interpret* the images, DPs, and spectra that you obtain.

If you count up the different imaging, diffraction, and spectroscopic operations that are available in a TEM there are almost 40 different modes of forming an image, DP, or spectrum, each of which produces different information about your specimen. (Your last homework, when you've finished the book, is to validate this claim!) Each of these information planes can be understood in a *quantitative* manner, to the extent that we can simulate all TEM images, DPs, and spectra in a computer. *No* other characterization technique comes close to the combination of versatility and quantification that is produced by this remarkable instrument, particularly when you consider the enormous range of magnifications over which the information is obtainable.

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 and

nanotechnology (most of which remain in print) as well as some standard journals and regular conference proceedings. We also encourage you to get on the WWW and see what's out there, but be careful about the validity of the content on any particular site. It is probably reasonable to trust the Web sites from reputable institutions such as government labs, commercial TEM and related equipment manufacturers organizations where, once you get past any sales pitch, there's often great educational material, and of course the Web pages of professional electron microscopists such as that associated with this text (URL #4).

REFERENCES PHILOSOPHY

In the reference sections throughout the book, we will annotate the references but we won't necessarily spell them out in the text (we'll give clues); we don't want the chapters to look like journal papers themselves and we want to encourage you to look at these reference sections. (This chapter is the exception.) In addition, don't forget the references for the figures right at the end of the textbook where you'll find much more than is in the figure captions. We do encourage you to explore the literature—you really have no choice if you want to understand TEM.

GENERAL TEM BOOKS

- Amelinckx, S, van Dyck, D, van Landuyt, J and van Tendeloo, G (Eds.) 1997 *Electron Microscopy: Principles and Fundamentals* VCH Weinheim Germany. An expensive text containing review articles by leading microscopists covering TEM and much more. A good overview to put TEM in context, but make sure your library buys this along with the even more expensive *Handbook of Microscopy: Applications in Materials Science, Solid State Physics and Chemistry* edited by the same authors and published by VCH in 1997.
- DeGraef, M 2003 *Introduction to Conventional Transmission Microscopy* Cambridge University Press New York. A complementary text to this one, in many ways. It uses different materials specimens as a thread to introduce different techniques. Supported by an excellent Web site (<http://ctem.web.cmu.edu/>) but not really an introduction!
- Edington, JW 1976 *Practical Electron Microscopy in Materials Science* Van Nostrand-Reinhold New York. The original out-of-print 1976 edition has been reprinted by TechBooks, 2600 Seskey Glen Court, Herndon, VA 22071. A very helpful, if outdated, text full of examples and hands-on operations; no AEM or HREM, just diffraction-based imaging.
- Egerton, RF 2006 *Physical Principles of Electron Microscopy; An Introduction to TEM, SEM, and AEM* Springer New York. If you need a general introduction to EM, this is a good choice.
- Ernst, F and Rühle, M (Eds.) 2003 *High-Resolution Imaging and Spectrometry of Materials* Springer Series in Materials Science 50 Springer Berlin. A collection of review articles covering some aspects of TEM and other high-resolution techniques.
- Fultz, B and Howe, JM 2002 *Transmission Electron Microscopy and Diffractometry of Materials* 2nd Ed. Springer New York. A broad-based text emphasizing diffraction-based imaging and crystallography via studies with X-ray and electrons.
- Goodhew, PJ, Humphreys, FJ and Beanland, R 2001 *Electron Microscopy and Analysis* 3rd Ed. Taylor & Francis New York. A succinct summary of SEM, TEM, and AEM.
- Hall, CE 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, PW and Spence, JCH (Eds.) 2007 *Science of Microscopy* Springer New York. Comprehensive, up-to-date, multi-author review of many forms of microscopy. Get your library to buy it.
- Heidenreich, RD 1964 *Fundamentals of Transmission Electron Microscopy* Interscience Publisher New York NY. Another wonderful, but sometimes forgotten, classic.
- Hirsch, PB, Howie, A, Nicholson, RB, Pashley, DW and Whelan, MJ 1977 *Electron Microscopy of Thin Crystals* 2nd Ed. Krieger Huntington NY. For many years, the 'Bible' for TEM users and still required reading for true TEM converts!
- Marton, L 1968 *Early History of the Electron Microscope* San Francisco Press San Francisco.
- McLaren, AC 1991 *Transmission Electron Microscopy of Minerals and Rocks* Cambridge University Press New York. Invaluable for the geologist or ceramist.
- Reimer, L 1997 *Transmission Electron Microscopy; Physics of Image Formation and Microanalysis* 4th Ed. Springer New York. Essential reference text. Strong physics background required; never uses a few words where a triple integral will do.
- Ruska, E 1980 *The Early Development of Electron Lenses and Electron Microscopy* (translated by T Mulvey) S Hirzel Verlag Stuttgart.

- Sawyer, LC, Grubb, DT and Meyers, GF 2008 *Polymer Microscopy* 3rd Ed. Springer New York. An expensive but useful qualitative introduction to TEM and SEM of polymers.
- Thomas, G and Goringe, MJ 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, 2600 Seskey Glen Court, Herndon, VA 22071.
- Watt, IM 1997 *The Principles and Practice of Electron Microscopy* 2nd Ed. CUP New York NY. A basic, practical introduction to SEM and TEM.
- Wenk, H-R 1976 *Electron Microscopy in Mineralogy* Springer New York NY. Required reading for microscopy of geological or ceramic materials. From the library.
- Yao, N and Wang, ZL 2005 *Microscopy for Nanotechnology* Kluwer New York. In-depth review articles on techniques for nano-characterization. Half of the 22 chapters are devoted to EM methods and more than 80% of those are TEM.

SPECIALIZED TEM BOOKS

- Ahn, CC (Ed.) 2004 *Transmission Electron Energy Loss Spectrometry in Materials Science and the EELS Atlas* 2nd Ed. Wiley-VCH Berlin. An excellent, in-depth review of EELS and the best database for ionization-edge energies.
- Brydson, R 2001 *Electron Energy Loss Spectroscopy* Bios (Royal Microsc. Soc.) Oxford UK. You must read this before going on to Egerton's classic.
- Champness, PE 2001 *Electron Diffraction in the TEM* Bios (Royal Microsc. Soc.) Oxford UK. Outstanding, essential introductory text by a crystallographer who also knows TEM.
- Cowley, JM (Ed.) 1992 *Electron Diffraction Techniques* Vols. 1 and 2 Oxford University Press New York. Another collection of excellent individual review articles.
- Egerton, RF 1996 *Electron Energy Loss Spectroscopy in the Electron Microscope* 2nd Ed. Plenum Press New York. The quintessential text for this aspect of AEM.
- Frank, J 1992 *Electron Tomography* Plenum Press New York. A biological EM text but the basis for much of what's going to happen in TEM of materials.
- Garratt-Reed, AJ and Bell, DC 2002 *Energy-Dispersive X-ray Analysis in the Electron Microscope* Bios (Royal Microsc. Soc.) Oxford, UK. Basic introduction pitched at a similar level to this text.
- Hawkes, PW and Kasper, E 1989, 1994 *Principles of Electron Optics* Vols. 1–3 Academic Press New York, 1900 pp. 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 resource.
- Head, AK, Humble, P, Clarebrough, LM, Morton, AJ and Forwood, CT 1973 *Computed Electron Micrographs and Defect Identification* North-Holland New York NY. Long out of print but often referenced.
- Horiuchi, S 1994 *Fundamentals of High-Resolution Transmission Electron Microscopy* North-Holland Amsterdam. As it says; a one topic book.
- Kirkland, EJ 1998 *Advanced Computing in Electron Microscopy* Plenum Press New York. Required reading for the mathematically inclined microscopist.
- Jones, IP 1992 *Chemical Microanalysis Using Electron Beams* Institute of Materials London. Great introduction to AEM with lots of calculations to introduce the principles of quantitative analysis.
- Loretto, MH 1994 *Electron Beam Analysis of Materials* 2nd Ed. Chapman and Hall New York. A concise overview of the subject.
- Royal Microscopical Society Handbook series: a broad range of introductory texts covering many aspects of TEM but also SEM and visible-light microscopy. Easy reading and not expensive (URL #5).
- Shindo, D and Oikawa, T 2002 *Analytical Electron Microscopy for Materials Science* Springer New York. Brief summary of X-ray and electron spectrometry.
- Spence, JCH 2003 *High Resolution Electron Microscopy* 3rd Ed. Oxford University Press New York. Practical HREM combined with lots of sound theory.
- Spence, JCH and Zuo, JM 1992 *Electron Microdiffraction* Plenum Press New York. Quantitative convergent-beam diffraction in great depth.
- Tonomura, A 1999 *Electron Holography* Springer New York. A good introduction.
- von Heimendahl, M 1980 *Electron Microscopy of Materials* Academic Press New York NY. An introductory-level text, no AEM or HRTEM component. From the library.
- Wang, ZL 1995 *Elastic and Inelastic Scattering in Electron Diffraction and Imaging* Plenum Press New York. Everything you ever need to know about scattering of electrons in the TEM.

THE COMPANION TEXT

Throughout this chapter and the rest of the text we will refer to the companion text. This new textbook is not a required reading for all students but does contain chapters on special topics that are only covered briefly in the present text.

JOURNALS

Advances in Imaging and Electron Physics Ed. PW Hawkes. Peter Hawkes is an editor, author, and historian without peer in the field of electron microscopy. Any text he edits or book he writes is worth reading. This journal merges two long-running serial journals *Advances in Electronics and Electron Physics* and *Advances in Optical and Electron Microscopy*. Reviews on particle optics at high and low energies, image science and digital image processing, electromagnetic-wave propagation, electron microscopy, and computing methods associated with all these topics. http://www.elsevier.com/wps/find/bookdescription.cws_home/711044/description#description.

Journal of Electron Microscopy Official Journal of the Japanese Society for Microscopy, becoming a more widely appreciated resource now that the papers are all in English. Oxford University Press Oxford. <http://jmicro.oxfordjournals.org/archive/>.

Journal of Microscopy Official Journal of the Royal Microscopical Society, the International Society for Stereology, the Microscopical Society of Ireland, the Polish Society for Microscopy, and the Austrian Society for Electron Microscopy. Many groundbreaking papers in the TEM of materials have appeared in this journal despite the strong biological theme of many of the papers. Blackwell Publishing Ltd Oxford, UK. <http://www.rms.org.uk/journal.shtml>.

Micron The International Research and Review Journal for Microscopy. Elsevier Amsterdam The Netherlands. http://www.elsevier.com/wps/find/journaldescription.cws_home/475/description#description.

Microscopy and Microanalysis Official Journal of the Microscopy Society of America, Microbeam Analysis Society (USA), Microscopical Society of Canada/Société de Microscopie du Canada, Mexican Microscopy Society, Brazilian Society for Microscopy and Microanalysis, Venezuelan Society for Electron Microscopy, European Microbeam Analysis Society, Australian Microscopy and Microanalysis Society. Has the largest circulation of any EM journal. Cambridge University Press New York NY. <http://www.msa.microscopy.org/MSAUnits/Journal/MscopyManalysis.html>.

Microscopy, Microanalysis, Microstructure Official Journal of the Société Française des Microscopies. Publication continued by The European Physical Journal (Applied Physics) in 1998 since when it has lost much visibility, but there are key papers in here before 1998.

Microscopy Research and Technique A place to publish your new techniques and methods for microscopy, specimen preparation, or any related aspect of TEM. John Wiley & Sons Hoboken NJ. <http://www3.interscience.wiley.com/cgi-bin/jhome/38527>.

Ultramicroscopy An international journal affiliated with multiple national societies, committed to the advancement of new methods, tools, and theories in microscopy, where much cutting-edge TEM research is published. Look out for Peter Hawkes' occasional reviews of the state of the microscopy literature. Elsevier Amsterdam The Netherlands http://www.elsevier.com/wps/find/journaldescription.cws_home/505679/description#description.

SELECTED CONFERENCE PROCEEDINGS

Asia-Pacific Electron Microscopy Conference organized by the Committee of Asia-Pacific Societies for Electron Microscopy (CAPSEM) every 4 years (2012).

Australian Microscopy and Microanalysis Society every 2 years (2010).

Electron Microscopy and Analysis Group (EMAG), Institute of Physics, London, UK every 2 years (2011).

European Microbeam Analysis Society every 2 years (2011).

European Microscopy Congress organized by the European Microscopy Society, every 4 years (2012).

Inter-American Congress for Electron Microscopy organized by Comité Interamericano De Sociedades De Microscopía Electrónica (CIASEM), every 2 years (2011).

International Congress for Microscopy organized by the International Federation of Societies for Microscopy (IFSM), every 4 years (2010 in Rio de Janeiro). The world gathers here.

International Union of Microbeam Analysis Societies every 4 years (2012).

Japanese Society for Microscopy annually.

Microscopy & Microanalysis combined meeting of the Microscopy Society of America, the Microbeam Analysis Society and occasionally others (including the Canadians); annually.

USEFUL SOURCES OF NUMERICAL DATA AND CONSTANTS

- Fischbeck, HJ and Fischbeck, KH 1987 *Formulas, Facts and Constants* 2nd Ed. Springer 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, AG 1991 *Handbook for Crystallography for Electron Microscopists and Others* Springer New York. Ideal for the microscopist but see the review by JA Eades *Microsc. Res. Tech.* **21** 368.
- Kaye, GWC and Laby, TH 1986 *Tables of Physical and Chemical Constants* 15th Ed. This invaluable resource was first published in 1911 and is now online courtesy of the National Physical Laboratory (UK) (URL #2).

SPECIFIC REFERENCES IN CHAPTER 1

- Usually we will group these into topics but this time the topic is 'The Introduction.'
- CBE (Council of Biology Editors) 1994 *Scientific Style and Format* 6th Ed. Cambridge University Press New York.
- Cosslett, VE 1979 *The Cosslett Festschrift* *J. Microsc.* **117** 1–184.
- Davison, CJ and Germer, LH 1927 *Diffraction of Electrons by a Crystal of Nickel* *Phys. Rev.* **30** 705–740. Early work by Clinton J. Germer and Lester H. German; read the introduction for encouragement.
- de Broglie, L 1925 *Recherches sur la Theorie des Quanta (Researches on the Quantum Theory)* *Ann. Phys.* **3** 22–128.
- Ewald, PP 1962 *Fifty Years of X-ray Diffraction* International Union of Crystallography D. Reidel Dordrecht.
- Fujita, H 1986 *History of Electron Microscopes* Business Center for Academic Societies Japan.
- Goodman, P 1981 *Fifty Years of Electron Diffraction* International Union of Crystallography Utrecht.
- Haguenau, F, Hawkes, PW, Hutchison JL, Satiat-Jeunemaitre, B, Simon, G and Williams, DB 2003 *Key Events in the History of Electron Microscopy* *Microsc. Microanal.* **9** 96–138.
- Hashimoto, H 1986 *High Resolution and High Voltage Electron Microscopy* *J. Elec. Microsc. Tech.* **3** 1.
- Hawkes, PW (Ed.) 1985 *The Beginnings of Electron Microscopy, Advances in Electronics and Electron Physics* Academic Press New York NY.
- Hayes, TL 1980 *Biophysical Aspects of Scanning Electron Microscopy SEM-1980* **1** 1–10 Ed. O Johari SEM Inc. AMF O'Hare IL.
- Howie, A 2000 *A Symposium in Honor of Professor Archie Howie's 65th Birthday* Eds. PL Gai, ED Boyes, CB Carter, DJH Cockayne, LD Marks and SJ Pennycook. *Microsc. Microanal.* **6** 281–284.
- Joy, DC 1995 *Monte Carlo Modeling for Electron Microscopy and Microanalysis* Oxford University Press New York.
- Knoll, M and Ruska, E 1932 *Das Elektronenmikroskop (Electron Microscope)* *Z. Phys.* **78** 318–339.
- Kossel, W and Möllenstedt, G 1939 *Electroneninterferenzen im Konvergenten* *Ann. Phys.* **36** 113–140.
- Thomas, G 2002 *A Symposium in Honor of Gareth Thomas' 70th Birthday* Eds. DG Howitt, CB Carter, U Dahmen, R Gronsky, DB Williams and R Sinclair *Microsc. Microanal.* **8** 237–364.
- Thomson, GP 1928 *Experiments on the Diffraction of Cathode Rays* *Proc. Roy. Soc. Lond.* **117** 600–609. George Paget Thomson was the son of J. J. Thomson; he shared the 1937 Nobel Prize for physics with Davison. A good read.
- Zeitler, E 2003 *Zeitler Festschrift* Eds. GA Botton, K Moore and D Su *Micron* **34** 119–260.

SPECIFIC URLS

1. <http://www.nano.gov/html/facts/whatIsNano.html>
2. <http://www.kayelaby.npl.co.uk/>
3. <http://www.physics.nist.gov/cuu/Constants/archive1998.html>
4. <http://www.TEMbook.com>
5. <http://www.rms.org.uk/other-publications.shtml>

SELF-ASSESSMENT QUESTIONS

- Q1.1 Define ionizing radiation and explain why it's useful.
- Q1.2 List four signals generated by electrons interacting with the atoms in your specimen.
- Q1.3 What makes electrons interact strongly with matter?
- Q1.4 What is the resolution or the resolving power of a microscope?
- Q1.5 What limits the resolution of VLMs?
- Q1.6 What limits the resolution of TEMs?
- Q1.7 How can we get high resolution in a TEM?
- Q1.8 Define depth of field for a specimen in the TEM.
- Q1.9 Explain why sampling is a problem with TEMs and how to combat this limitation.

- Q1.10 List three benefits of using a TEM rather than any other instrument to characterize nanoparticles.
- Q1.11 What is the major difficulty with interpreting all TEM images?
- Q1.12 Why is TEM so powerful a characterization tool?
- Q1.13 What signals are most commonly used in the conventional TEM?
- Q1.14 Define the acronyms TEM, AEM, STEM, HREM, HVEM. Are there any other kinds of electron microscopes?
- Q1.15 Which two signals produced when electrons interact with matter are used in AEM?
- Q1.16 What happens to the wavelength of the electron beam when the accelerating voltage of the TEM is increased?
- Q1.17 What does 'thin' mean when referring to a TEM specimen?
- Q1.18 Give a ballpark figure for a suitably 'thin' TEM specimen.
- Q1.19 Why is TEM such an important experimental tool for materials scientists and nanotechnologists?
- Q1.20 What is a typical electron beam current in a TEM?
- Q1.21 What are the typical causes of specimen damage in the TEM?
- Q1.22 What effect should we consider in any calculations when the energy of the electrons is 100 keV or higher and why?

TEXT-SPECIFIC QUESTIONS

- T1.1 If you were a physicist in the 1920s why would you think that the fact that electrons can be diffracted by crystals is a good reason to develop an electron microscope?
- T1.2 Calculate an approximate wavelength for 50 kV electrons via equations 1.1 and 1.2 and then estimate the Rayleigh-defined resolving power of a microscope using such electrons. We tell you to assume $\mu \sin \beta \sim \beta$ so what does this tell you about what numbers might be reasonable for the refractive index and numerical aperture in a TEM?
- T1.3 Examine Figure 1.2A; draw a schematic diagram to summarize all that you can deduce about the crystallography of the specimen with the information given in the text.
- T1.4 Look at the arrows in Figure 1.3. From what little you know at this stage about how electrons interact with matter, can you work out which signals are most likely to be used in a TEM and which are less likely to be used? (Hint: relate the diagram to the actual construction of the TEM.)
- T1.5 Examine Figure 1.4; as a materials scientist, do you expect there to be more of the Ti (blue) alloying element present in the smaller precipitates in the matrix, the larger precipitates on the boundary or the large particle on the boundary, the big dark lumps up the middle of the image or in the matrix? Are there reasons to be concerned about trying to estimate compositions from the color in the image only?
- T1.6 In Figure 1.5 some dislocations appear to begin or end in the material but you will recall that dislocation lines cannot begin or end in a crystal. (Refer, e.g., to Hull, D and Bacon, DJ Introduction to Dislocations, Butterworth-Heinemann, 2001 ISBN 0750646810 or Weertman, J and Weertman, J, Elementary Dislocation Theory, Oxford University Press; 1992 ISBN 0195069005.) So why do the lines appear to do just this?
- T1.7 Why do you think it is useful to be able to relate the directions in the DP in Figure 1.6 to the directions of the plate-like precipitates in the inset in the same figure?
- T1.8 Can you think of an analogy to Figure 1.7 involving images viewed in transmission using visible light? (Hint: think about looking at images of objects at varying distance apart viewed through a transparent medium, e.g., air or water).
- T1.9 Examine Figure 1.8; what information in the images would lead you to infer that electron-beam damage was occurring and what could you do to try and avoid further damage? (Hint: what is the difference between the two images and what is causing it?)
- T1.10 Would you expect a higher-voltage TEM to be capable of more damage to your specimen than a lower-voltage instrument?
- T1.11 Use equations 1.6 and 1.7 in a spreadsheet to reproduce Table 1.2 and add in lines for electrons accelerated to 50 and 250 kV.
- T1.12 Compare the usefulness of Google searches for 'TEM' and 'transmission electron microscopy,' 'AEM', and 'analytical electron microscopy.' What does this tell you about acronyms (TMBA?)
- T1.13 See how many of the URLs listed in Section 1.6 are still active. Make your own list of useful URLs related to your own interests in electron microscopy.
- T1.14 Confirm for yourself the calculation referred to in Section 1.4 that there is never more than one electron inside a typical thin specimen in a 100 keV TEM.
- T1.15 Looking at Figure 1.9, you'll notice that high-voltage microscope is bigger than other ones. Can you think of any reasons why this is so?
- T1.16 Why do some TEMs have viewing screens and some only have computer displays?
- T1.17 Why might you want to build an ultrahigh vacuum TEM and why are there so few available?