Chapter Three
Transmission Electron Microscope

Transmission electron microscopy

Transmission Electron Microscopy (TEM) 1.2.4

In Transmission Electron Microscopy (TEM) a thin solid specimen (≤ 200 nm thick) is bombarded in vacuum with a highly-focused, monoenergetic beam of electrons. The beam is of sufficient energy to propagate through the specimen. A series of electromagnetic lenses then magnifies this transmitted electron signal. Diffracted electrons are observed in the form of a diffraction pattern beneath the specimen. This information is used to determine the atomic structure of the material in the sample. Transmitted electrons form images from small regions of sample that contain contrast, due to several scattering mechanisms associated with interactions between electrons and the atomic constituents of the sample. Analysis of transmitted electron images yields information both about atomic structure and about defects present in the material.

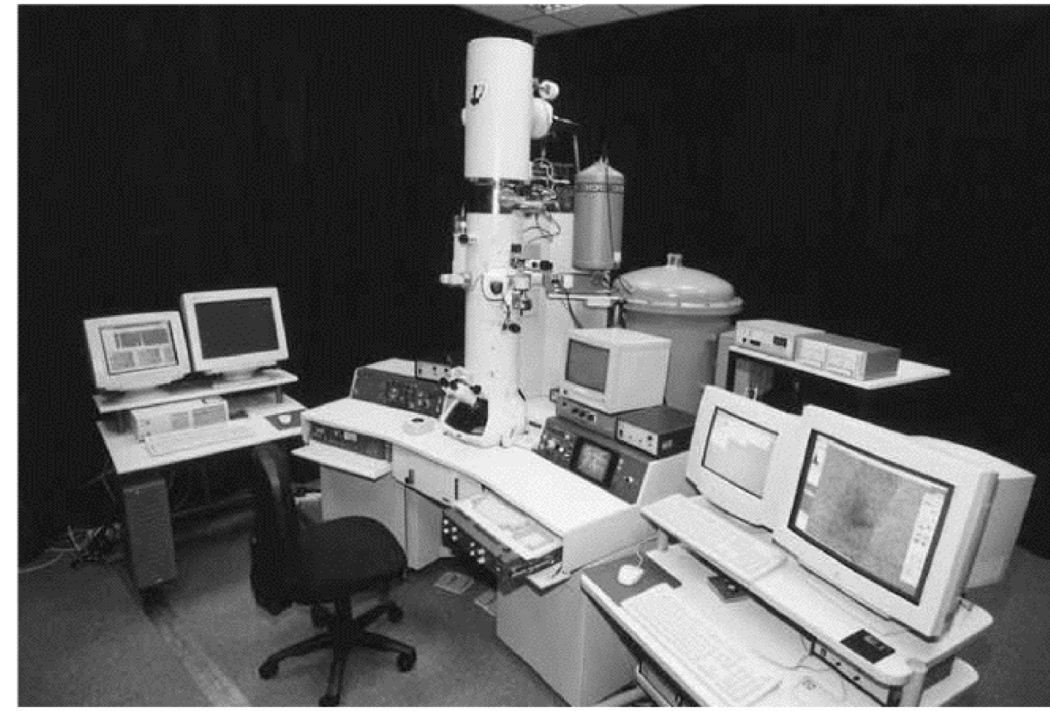

Figure 4.1 A modern transmission electron microscope. (Courtesy of Professor A. G. Cullis)

Range of elements	TEM does not specifically identify elements measured
Destructive	Yes, during specimen preparation
Chemical bonding information	Sometimes, indirectly from diffraction and image simulation
Quantification	Yes, atomic structures by diffraction; defect characterization by systematic image analysis
Accuracy	Lattice parameters to four significant figures using convergent beam diffraction
Detection limits	One monolayer for relatively high-Z materials
Depth resolution	None, except there are techniques that measure sample thickness
Lateral resolution	Better than 0.2 nm on some instruments
Imaging/mapping	Yes
Sample requirements	Solid conductors and coated insulators. Typically 3-mm diameter, < 200-nm thick in the center
Main uses	Atomic structure and Microstructural analysis of solid materials, providing high lateral resolution
Instrument cost	\$300,000–\$1,500,000
Size	100 ft.^{2} to a major lab

electrons) and electrons ejected from the specimen (such as secondary electrons) are also of interest and we will not totally neglect them (although they are of much greater interest in the SEM where they provide atomic number contrast and surface-sensitive, topographical images, respectively).

WAVE AND PARTICLE

The electron is treated in two different ways throughout this book: in electron scattering it is a succession of particles, while in electron diffraction it is treated by wave theory. The analogy to X-rays or visible light would be to compare a beam of photons and an electromagnetic wave. However, you must always remember that electrons are charged particles and that Coulomb forces are very strong.

In this chapter we introduce the fundamental ideas of electron scattering; then, in the next two chapters, we discuss the two principal forms of scattering, namely, elastic and inelastic. Both forms are useful to us, but you'll see that the latter has the unfortunate side effect of being responsible for specimen damage and ultimately limits what we can do with a TEM.

To give you some feel for the importance of electron scattering, it is worth illustrating at this stage the basic principles of the TEM. You will see in due course that in a TEM we illuminate a thin specimen with a broad beam of electrons in which the intensity is uniform over the illuminated area.

We will often refer to incident and scattered electrons as beams of electrons, because we are dealing with many electrons, not an individual electron; these electrons are usually confined to well-defined paths in the microscope. So the electrons that hit the specimen are often called the incident beam and those scattered by the specimen are called scattered (or sometimes specifically, diffracted) beams. Electrons coming through a thin specimen are separated into those that suffer no angular deviation and those scattered though measurable angles. We call the undeviated electrons the 'direct beam' (in contrast to most texts that describe this as the 'transmitted beam' despite the fact that all electrons coming through the specimen have been 'transmitted'). As the electrons travel through the specimen they are either scattered by a variety of processes or they may remain unaffected. The end result, however, is that a non-uniform distribution of electrons emerges from the exit surface of the specimen, as shown schematically in Figure 2.1. It is this non-uniform distribution that contains all the structural, chemical, and other information about our specimen. So everything we learn about our specimen using TEM can be attributed to some form of electron scattering.

DIRECT BEAM

The beam that comes through the specimen, but remains parallel to the direction of the incident electrons is a very important beam, which we will term the **direct beam**.

We'll see in Chapter 9 that the TEM is constructed to display this non-uniform distribution of electrons in two different ways. First the *spatial distribution* (Figure 2.1A) of scattering can be observed as contrast in *images* of the specimen, and the *angular* distribution of scattering (Figure 2.1B) can be viewed in the form of scattering patterns, usually called *diffraction* patterns. A simple (and fundamental) operational step in the TEM is to use a restricting aperture, or an electron detector, of a size such that it only selects electrons that have suffered more or less than a certain angular deviation. So you as the operator have the ability to choose which electrons you want to use and thus you control what information

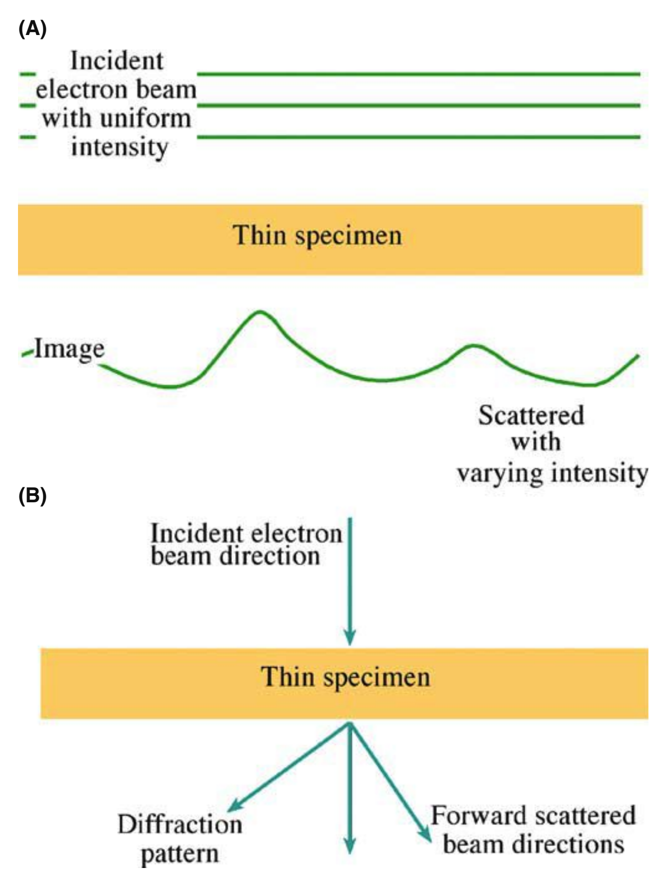

FIGURE 2.1. (A) A uniform intensity of electrons, represented by the horizontal lines, falls on a thin specimen. Scattering within the specimen changes both the spatial and angular distributions of the emerging electrons. The spatial distribution (intensity) is indicated by the wavy line. (B) The change in angular distribution is shown by an incident beam of electrons being transformed into several forward-scattered beams.

will be present in the image. Therefore, to comprehend these images, you have to understand what causes electrons to scatter in the first place. The same is true for DPs since you can also control (to a lesser extent) the angular-scattering distribution, e.g., by tilting your specimen.

We devote the whole of Part 2 to diffraction phenomena and Part 3 to images. Lastly, Part 4 deals with ways in which we use inelastic scattering for analytical electron microscopy (AEM) to study, e.g., the chemistry and the bonding of the atoms in our specimen.

2.2 TERMINOLOGY OF SCATTERING AND DIFFRACTION

Electron-scattering phenomena can be grouped in different ways. We've already used the most important terms: *elastic* and *inelastic* scattering. These terms, respectively, describe scattering that results in no loss of energy or in some measurable loss of energy (usually very small with respect to the beam energy). In either case, we can consider the beam electrons and specimen atoms as particles, and scattering of the incident electrons by the atoms in the specimen can often be approximated to something like billiard balls colliding. The billiard-ball analogy will be good through Section 2.7 after which we'll be talking about waves.

ELECTRON SCATTERING
This theme permeates the whole text and connects ALL aspects of TEM.

However, we can also separate scattered electrons into *coherent* and *incoherent*, which refers, of course, to their wave nature. These distinctions are related since elastically scattered electrons are usually coherent and inelastic electrons are usually incoherent (note the modifier 'usually'). Let's assume that the incident electron waves are coherent, that is, they are essentially in step (in phase) with one another and of a fixed wavelength, governed by the accelerating voltage. You'll see that this isn't a bad assumption in most circumstances. Then, coherently scattered electrons are those that remain in step and incoherently scattered electrons have no phase relationship, after interacting with the specimen.

The nature of the scattering can result in different angular distributions. Scattering can be either *forward scattering* or *back scattering* (usually written as one word) wherein the terms refer to the angle of scattering with respect to the incident beam and a specimen that is normal to that beam. (Note: you will sometimes see the term 'forward scattering' used in another sense.) If an electron is scattered through $< 90^\circ$, then it is forward scattered and $> 90^\circ$ it is backscattered. These various terms are related by the following general principles, summarized in Figure 2.2.

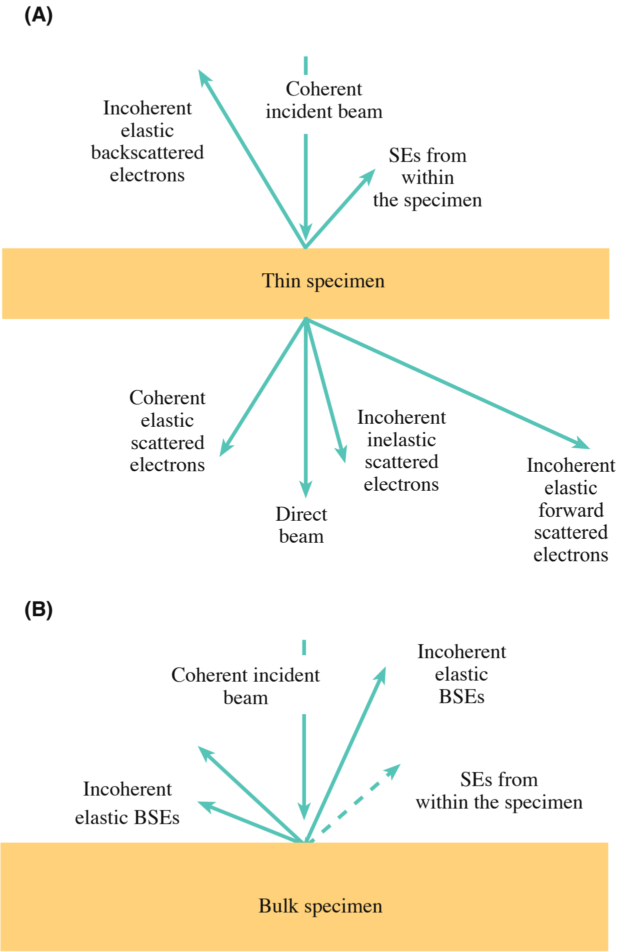

FIGURE 2.2. Different kinds of electron scattering from (A) a thin specimen and (B) a bulk specimen: a thin specimen permits electrons to be scattered in both the forward and back directions while a bulk specimen only backscatters the incident-beam electrons.

- Elastic scattering is usually coherent, if the specimen is thin and crystalline (think in terms of waves).
- Elastic scattering usually occurs at relatively low angles (1–10°), i.e., it is strongly peaked in the forward direction (waves).
- At higher angles ($>\sim 10^\circ$) elastic scattering becomes more incoherent (now think of particles).
- Inelastic scattering is almost always incoherent and is very low angle ($<1^\circ$) scattering (think particles).
- As the specimen gets thicker, fewer electrons are forward scattered and more are backscattered. Incoherent, backscattered electrons are the only remnants of the incident beam that emerge from bulk, non-transparent specimens (think particles).

define diffraction

The notion that electrons can be scattered through different angles is related to the fact that an electron can also be scattered more than once. Generally, the more scattering events, the greater the angle of scattering (although sometimes a second scattering event can redirect the electron back into the direct beam, so it appears to have undergone no scattering).

The simplest scattering process is *single scattering* and we often approximate all scattering within a TEM specimen to this process (i.e., an electron either undergoes a single-scattering event or it suffers no scattering). We'll see that this can be a very reasonable assumption if the specimen is very thin (something you can control). If the electron is scattered more than once, we use the term *plural scattering* and if it is scattered >20 times, we say *multiple scattering*. It is generally safe to assume that, unless you have a particularly thick specimen (through which you probably can't see anything anyhow), multiple scattering will not occur in the TEM. The greater the number of scattering events, the more difficult it is to predict what will happen to the electron and the more difficult it is to interpret the images, DPs, and spectra that we gather. So, once again, we emphasize the importance of the 'thinner is better' criterion, i.e., if you create thin enough specimens so that the single-scattering assumption is plausible, your TEM research will be easier.

Diffraction is a very special form of elastic scattering and the terminology used can be confusing. Collins' Dictionary defines *diffraction* as 'a deviation in the direction of a wave at the edge of an obstacle in its path' while *scattering* is defined as 'the process in which particles, atoms, etc., are deflected as a result of collision.' The word scatter can also be a noun denoting the act of scattering. So scattering might best apply to particles and diffraction to waves; both terms thus apply to electrons! You should also note that the term diffraction is not limited to Bragg diffraction which we'll emphasize in TEM; it refers to *any* interaction involving a wave, but many texts are not consistent in this respect.

DEFINE DIFFRACTION
An interaction between a wave of any kind and an object of any kind (Taylor 1987).

In the TEM we utilize the electrons that go through a specimen; it is important to note that such electrons are not simply 'transmitted' in the sense of visible light through window glass. Electrons are scattered mainly in the forward direction, i.e., parallel to the incident beam direction (and we've already noted the confusion between 'direct' and 'transmitted'). We'll tell you in a short while what fraction of the electrons are forward scattered and how this varies with the thickness of the specimen and atomic number of the 'target' atom. This scattering is a direct consequence of the fact that there is such a strong interaction between electrons and matter.

Forward scattering includes the direct beam, most elastic scattering, diffraction, particularly Bragg diffraction (see Chapter 3), refraction, and inelastic scattering (see Chapter 4). Because of forward scattering through our thin specimen, we see a DP or an image on the viewing screen, and detect an X-ray spectrum or an electron energy-loss spectrum outside the TEM column. But don't neglect backscattering; it is an important imaging mode in the SEM.

FORWARD SCATTERING
The cause of most of the signals used in the TEM.

When physicists consider the theory of electron interactions within a solid, they usually consider scattering of electrons by a single, isolated atom, then progress to agglomerations of atoms, first in amorphous solids and then in crystalline solids and we'll follow a similar path.

2.3 THE ANGLE OF SCATTERING

When an electron encounters a single, isolated atom it can be scattered in several ways which we will cover in the next two chapters. For the time being, let's imagine simply that, as shown in Figure 2.3, the electron is scattered through an angle θ (radians) into some solid angle ω, measured in steradians (sr). We have to define this angle first because you'll see that it plays an important role in the subsequent discussion of cross sections.

SEMI-ANGLE
Note that the scattering angle θ is in fact a semi-angle, not a total angle of scattering. Henceforth, whenever we say "scattering angle" we mean "scattering semi-angle."

Often we assume that θ is small enough such that $\sin\theta \approx \tan\theta \approx \theta$. When θ is this small, it is often convenient to use milliradians or mrads; 1 mrad is 0.0573°, 10 mrads is ~0.5°.

SMALL ANGLE
A convenient upper limit is <10 mrads.

The characteristics of the scattering event are controlled by factors such as the incident-electron energy and the atomic number/weight of the scattering atom. When we consider a specimen rather than a single

Fundamentals of electron scattering

angle of scattering

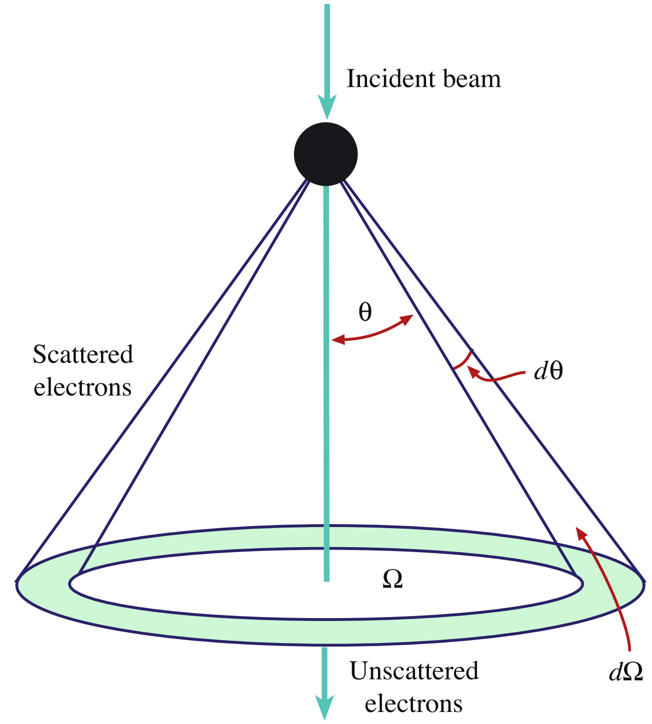

FIGURE 2.3. Electron scattering by a single isolated atom. The electrons are scattered through an angle θ and the total solid angle of scattering is Ω. An incremental increase in scattering angle $d\theta$ gives an incremental increase in a solid angle $d\Omega$, which is the basis for determining the differential scattering cross section.

atom, factors such as the thickness, density, crystallinity, and angle of the specimen to the incident beam also become important. To understand these variables, we need to examine the physics of scattering in more detail. Of necessity, we'll be rather brief and often imprecise since we're trying to condense much of Mott and Massey's substantial and classic textbook into just a few pages.

2.4 THE INTERACTION CROSS SECTION AND ITS DIFFERENTIAL

The chance of a particular electron undergoing any kind of interaction with an atom is determined by an interaction *cross section*. The concept of a cross section is well described by the following analogy given by Rudolf Peierls (Rhodes 1986)

> "If I throw a ball at a glass window one square foot in area, there may be one chance in ten that the window will break and nine chances in ten that the ball will just bounce. In the physicist's language this particular window, for a ball thrown in this particular way, has a disintegration (inelastic!) cross section of 0.1 square feet and an elastic cross section of 0.9 square feet."

So each possible interaction has a different cross section which depends on the energy of the particle, in our case the beam energy. The cross section (for which we'll use the Greek letter σ) has units of area (not square feet as used in Peierls' analogy, but a tiny fraction of the area of an atom termed a 'barn'). One barn is 10^{-28} m^2 (that's $(10^{-5}$ nm$)^2$) and the name arises because of the perverse sense of humor of some of the early atomic physicists who considered that this unimaginably small area is 'as big as a barn door.' The cross section does *not* represent a physical area but, when divided by the actual area of the atom, it represents a *probability* that a scattering event will occur.

2.4.A Scattering from an Isolated Atom

First of all we'll consider the scattering cross section for a single isolated atom, then extend the concept to a specimen with many atoms. We'll use a generalized form to start with in this chapter and then break down the concept of a total cross section into cross sections for individual processes such as elastic scattering and the various inelastic processes in the next two chapters.

SCATTERING PROBABILITY

The larger the cross section, the better the chances of scattering.

Following Heidenreich (1964), we can define the cross section (an area) in terms of the *effective radius* of a single, isolated atom, r

$$\sigma_{\text{atom}} = \pi r^2 \tag{2.1}$$

where r has a different value for each scattering process as we'll see in the next chapter. What interests us in the TEM is whether or not the scattering process deviates the incident-beam electrons outside a particular scattering angle θ such that, e.g., they do not go through the aperture in the lens or they miss the electron detector. So we have to know the *differential cross section* $(d\sigma/d\Omega)$ which describes the angular distribution of scattering from an atom. As shown in Figure 2.3 electrons are scattered through an angle θ into a solid angle Ω and there is a simple geometrical relationship between θ and Ω

$$\Omega = 2\pi(1 - \cos\theta) \tag{2.2}$$

and therefore

$$d\Omega - 2\pi \sin\theta d\theta \tag{2.3}$$