This chapter will set the stage for our discussion of imaging using diffraction contrast. Put simply, diffraction contrast arises because the intensity of the diffracted beams is different in different regions of the specimen. These variations may arise because of changing diffracting conditions or because of differences in specimen thickness. In our study of diffraction in the TEM, we will see spots—lots of them. Sometimes the ‘spots’ will be small faint points and other times they will be large disks, which themselves contain ‘structure’ and more information. Other patterns will contain lines that we will examine in Chapters 19–21.

We need to know how to use the information that these spot patterns (diffraction patterns or DPs) contain. We will discuss the practical question of how we can best record the DPs, so that we can maximize the information they contain, but we will not try to give a rigorous proof of every equation used. These DPs give direct crystallographic information about small areas of the specimen. This capability is one of the most important features of the TEM, because we can relate the crystallography to the images we see.

In reading this chapter you should remember our discussion of the scattering of waves using an array of slits (Chapter 2). Much of the analysis is geometrically the same as we found for visible light—it’s essentially the same physical optics. The big differences are that we have ‘modulated’ holes which are located in 3D space and both our wavelengths and the spacing of the ‘holes’ are very small.

A note on history: spot patterns in DPs have always been recorded using a photographic emulsion but many TEMs no longer have photographic plates. The CCD has a much greater dynamic range but you may need to record the DP using several different camera lengths. Plus—burning a hole in photographic film is one thing, doing the same to the CCD is another.

11.1 WHY USE DIFFRACTION IN THE TEM?

Let’s begin by looking at an experimental DP. The pattern shown in Figure 11.1, like those we introduced in Chapter 2, was recorded from a thin specimen, in this case silicon. The main features to note are there are many spots and the spots vary in intensity and size (these are related effects).

We can list some of the questions you might ask on first seeing such a DP.

- What is it?
- What can we learn from it?
- Why do we see it?
- What determines the scale? What determines the distances between the spots or the positions of the lines?

What do we want to know about our specimen? To a materials scientist, perfect crystals are often pretty boring and can usually be better studied using such techniques as X-ray diffraction (for structural characterization), the electron microprobe (for chemical characterization), etc., although new EM techniques may change this situation. The TEM is the instrument of choice when the specimen is not perfect, particularly when the feature of interest is what makes the material imperfect or, paradoxically, useful!

The questions that we can address using DPs obtained in the TEM include the following

- Is the specimen crystalline? Crystalline and amorphous materials have very different properties.
- If it is crystalline, then what are the crystallographic characteristics (lattice parameter, symmetry, etc.) of the specimen?
- Is the specimen monocristalline? If not, what is the grain morphology, how large are the grains, what is the grain-size distribution, etc.?
What is the orientation of the specimen or of individual grains with respect to the electron beam?

Is more than one phase present in the specimen? If so, how are they oriented?

In general, if we see spots then the specimen is at least partly crystalline. (We’ll discuss quasicrystals later.) The ability to determine crystallographic orientations locally (down to the nm level) gives TEM its great advantage over SEM and visible-light microscopes. Later on we can make this determination even more precise (to an accuracy of \( \pm 0.001 \)) using convergent-beam patterns, as we’ll see in Chapter 21.

In this chapter we will restrict the discussion to the geometry of the spot patterns. These are necessarily associated with crystalline materials. We’ll see that spot patterns provide a great deal of information themselves; they also provide the basis for understanding other DPs. We will find that standard DPs which are common to a group of materials allow us quickly to recognize particular orientations and even certain grain boundaries and twin boundaries, etc., without having to index the pattern from scratch. For example, in a particular orientation, all cubic crystals give the same array of spots although some of the spots may have no intensity! We will consider the intensity of the spots in Chapter 12.

Remember, however, that SADPs patterns are not always the most useful DPs, since CBED (Chapters 20 and 21) can give you other useful information. Nevertheless, we are emphasizing SADPs here, since we use them to explain the contrast in TEM images, in Part III.

### 11.2 THE TEM, DIFFRACTION CAMERAS, AND THE TV

The use of electron diffraction for materials studies began around 1930 using diffraction cameras which very much resembled X-ray tubes in their physical appearance. Later on, if you pursue TEM in depth, you will find many of the earlier texts on electron diffraction useful for gaining a deeper understanding of TEM. It will be helpful to bear in mind some of the historical circumstances behind these developments when reading some of these texts. For example, many articles show ray diagrams with the optic axis horizontal. One reason for this is that much of the early theoretical analysis was developed as an extension of X-ray diffraction (XRD) or by researchers who were actively using either X-ray or electron-diffraction cameras. In each case, the optic axis of the instrument was horizontal as is still the case for visible-light optical benches. The optic axis of all electron microscopes is now vertical although the beam may originate at either the top or the bottom of the column. Actually, more than one of the early TEMs, e.g., the Philips EM100, was built with the optic axis horizontal and the electron beam directed at the observer. This arrangement is similar to that used for television, but remember that in TEM we are using very high energy electrons (\( \geq 100 \text{ keV} \)) rather than 20 keV used in a TV).

References to some of the early texts, and their historical significance, are given at the end of this chapter. When you are reading early texts on TEM remember that many were written at a time when most TEMs operated at 100 kV. This fact may easily be overlooked but it affects many features of diffraction including the camera length.

We will be talking about positions of spots and not their intensities for most of the time in this book. This type of analysis differs from many X-ray studies. The reason that beam intensities are not measured in TEM is that the electron beams are diffracted many times in a typical TEM specimen. A similar, but not identical, situation actually occurs when producing powder patterns by X-ray diffraction (XRD); diffraction then occurs in many different grains at the same time. We can compare the electron-diffraction pattern with that encountered in XRD. In the X-ray case, if you have a single crystal, then you either have to rotate the crystal to ‘see’ all the beams or use ‘white’ radiation (i.e., essentially use a range of wavelengths). Electron diffraction is very different. We can use a single wavelength and still see many diffracted beams. The techniques differ also with respect to the time.