Diffraction Patterns

11

CHAPTER PREVIEW

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 which we will examine in Chapters 19 to 21.

We need to know how to use the information which 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 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.

D. B. Williams et al., *Transmission Electron Microscopy*

Diffraction Patterns

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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 that 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 monocrystalline? 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?

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 ~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 both 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 SAD patterns are not always the most useful DPs, since CBED (Chapters 20 and 21) can give you other useful information. Nevertheless, we are emphasizing SADs 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

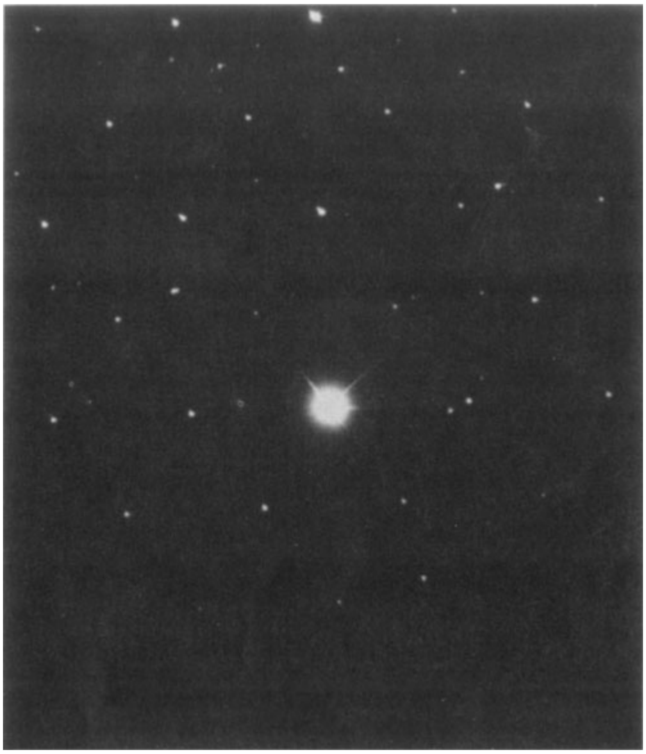

Figure 11.1. An experimentally observed diffraction pattern showing the central, intense, direct beam and an array of diffraction spots from different atomic planes. Such a pattern, with sharply focused spots, is best obtained by underfocusing the beam.

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 usually 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 the television, but remember that in TEM we are using very high energy electrons (≥100 keV rather than 20 keV used in a TV). References to 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 it takes to record a DP on a photographic plate; XRD takes minutes or hours unless you have a synchrotron or a position-sensitive detector to count every photon, while electron diffraction patterns can be recorded in <1 second although, in practice, several seconds to a minute should usually be used.

Much of our discussion of electron diffraction follows directly from the analysis of XRD. This has advantages and disadvantages, depending on whether or not you are familiar with XRD. Several references to XRD are given at the end of the chapter. When considering diffraction, remember that there are important differences between electrons and X-rays:

- Electrons have a much shorter wavelength than the X-rays commonly encountered in the research lab.
- Electrons are scattered more strongly because they interact with both the nucleus and the electrons of the scattering atoms through Coulomb forces.
- Electron beams are easily directed because electrons are charged particles.

It is particularly important that the electron beam can be deflected off the optic axis a short distance above the specimen, and then pass through the specimen; this process of tilting the beam was described in Section 9.1.C. The most obvious effect of this deflection on the DP is that the whole DP is translated relative to the viewing screen. The more subtle effect results from the change in the direction of the incident beam with respect to the crystal lattice, as we will discuss in subsequent chapters.

11.3. SCATTERING FROM A PLANE OF ATOMS

If you go back to Chapter 3 on elastic scattering you'll see that we introduced the two different ways of thinking about diffraction: the Laue conditions and the Bragg Law. In this chapter we'll derive the Bragg Law again, introducing a vector notation that we'll use throughout the rest of the book. In Chapter 12, we'll do the same with the Laue conditions.

The simple diagram in Figure 11.2 shows an *initial* wavefront, W_I, being scattered by two planes of atoms to produce a *diffracted* wavefront, W_D. Whether or not W_D corresponds to a diffracted beam will depend on whether the atoms are scattering in phase, which itself is determined by the angles between the incident beam, the diffracted beam, and the diffracting planes. The conditions for the individual waves being in phase are known as the Laue conditions, which we introduced in Section 3.9.B. To analyze the situation we first simplify the diagram as shown in Figures 11.3 and 11.4. These figures define the wave propagation vectors, which we will refer to simply as the wave vectors or the **k** vectors. We begin by considering scattering from only two atoms.

> Notice that we are already mixing the concepts of waves and beams.

We'll only consider plane wavefronts, i.e., the wavefront is flat and **k** is normal to this wavefront. The diagram in Figure 11.3a,b defines vectors $\mathbf{k}_I$, $\mathbf{k}_D$, and **K** and gives us the following important equation (which is just vector addition)

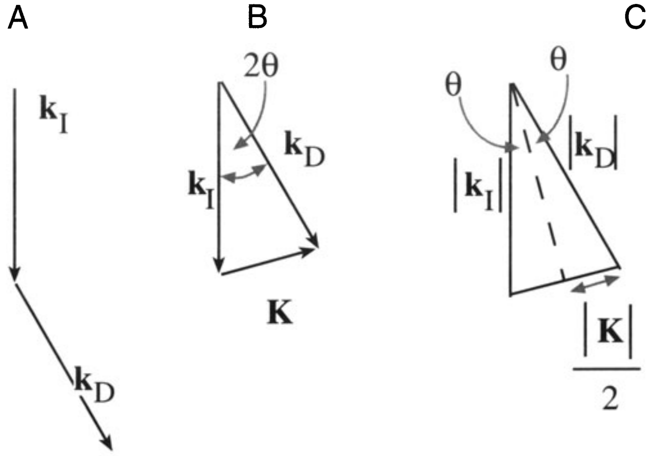

Figure 11.3. Definition of the scattering vectors: (a) the incident wavefront normal is $\mathbf{k}_I$, the diffracted wave normal is $\mathbf{k}_D$; (b) **K** is the difference vector (= $\mathbf{k}_D - \mathbf{k}_I$); (c) sin θ is defined as $K/2k_I$.

$$\mathbf{K} = \mathbf{k}_D - \mathbf{k}_I \qquad [11.1]$$

where $\mathbf{k}_I$ and $\mathbf{k}_D$ are the **k** vectors of the incident and diffracted waves, respectively. The vector **K** is thus the change in **k** due to diffraction. An important feature of this analysis is that this construction can be made for any $\mathbf{k}_D$ and thus for any value of **K**; the angle θ shown here need not be a Bragg angle.

Following our discussion in Section 3.10.B, we can always write that

$$|\mathbf{k}_I| = |\mathbf{k}_D| = \frac{1}{\lambda} = |\mathbf{k}| \qquad [11.2]$$

providing the energy of the electron is unchanged during diffraction, i.e., the scattering process is elastic. From Fig-

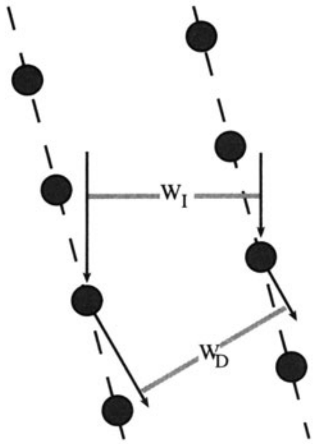

Figure 11.2. Scattering from two planes of atoms. W_I and W_D are the incident and diffracted wavefronts, respectively.

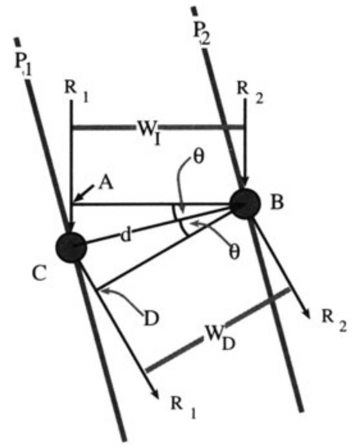

Figure 11.4. Two beams are scattered from two points, C and B, which lie on different planes, P_1 and P_2. The rays travel different distances, giving a path difference of AC + CD.

ure 11.3C we can write down an expression for θ using simple trigonometry

$$\sin\theta = \frac{|\mathbf{K}|/2}{|\mathbf{k}_I|} \qquad [11.3]$$

or

$$|\mathbf{K}| = \frac{2\sin\theta}{\lambda} \qquad [11.4]$$

> $|\mathbf{K}|$, like $|\mathbf{k}_I|$, has units Å^{-1} if λ is measured in Å. **K** and $\mathbf{k}_I$ are then referred to as *reciprocal lattice vectors*. Note that this scattering process is taking place inside the crystal and therefore the **k**-vectors are all appropriate to the electrons inside the crystal (rather than in the vacuum).

Equation 11.4 is very important; whenever you see the term (sin θ)/λ remember that it is just **K**/2 and is thus related to a change in wave vector.

If we now extend this argument to consider the interference between waves scattered from two points (which you can visualize as being atom sites) then we have the situation sketched in Figure 11.4. This figure should remind you of the idea of constructive and destructive interference, which we discussed back in Section 3.10. You will recognize that the geometry of Figure 11.4 is essentially a cross section of the two slits used by Young to demonstrate the wave nature of light (see also Section 2.10). We can then define two planes, P_1 and P_2, to be normal to the vector **CB**, which has length *d*. The distance traveled by ray R_1 is then larger than that traveled by ray R_2 by the path difference AC + CD. Simple geometry shows that

$$AC + CD = 2d\sin\theta \qquad [11.5]$$

which is the basis for the Bragg Law, as we'll now see.

11.4. SCATTERING FROM A CRYSTAL

We introduced the Bragg angle in Figure 3.9 as the most important scattering angle in TEM; at the Bragg angle the electron waves interfere constructively. If we now analyze Figure 11.4 further, we see that in the special case when θ equals the Bragg angle, θ_B, equation 11.4 becomes

$$|\mathbf{K}| = \frac{2\sin\theta_B}{\lambda} \qquad [11.6]$$

When θ is θ_B, the path difference in equation 11.5 is $n\lambda$, where *n* is any integer, and the equation becomes

$$n\lambda = 2d\sin\theta_B \qquad [11.7]$$

which is Bragg's Law (equation 3.22). If *n* is 1

$$2\sin\theta_B = \frac{\lambda}{d} \qquad [11.8]$$

but we already know from equation 11.6 that, at the Bragg angle,

$$2\sin\theta_B = \lambda\,|\mathbf{K}| \qquad [11.9]$$

so when we are at the Bragg angle, the magnitude of the vector **K** has a special value, K_B,

$$|\mathbf{K}_B| = \frac{1}{d} \qquad [11.10]$$

and we define this vector, $\mathbf{K}_B$, to be **g** so that

$$\mathbf{K}_B = \mathbf{g} \qquad [11.11]$$

This sequence of steps may seem rather pedantic but the conclusion is extremely important. Bragg's Law and the geometry used to "prove" it will be used so frequently in our discussions that it is worthwhile to delve a little into what it really tells us. Although it is not really a valid treatment of the phenomenon we are seeing, Bragg's Law gives us a very useful physical picture of the diffraction process because the diffracting planes appear to behave as mirrors for the incident electron beam. Therefore, the diffracted beams, or the spots in the DP, are often called "reflections" and we sometimes refer to the vector **g** as the diffraction vector. This derivation is simply geometry. In Section 12.3 we will derive the Laue equations and hence deduce Bragg's Law from first principles.

> Don't forget: we are really dealing with diffraction, not reflection, and we derived Bragg's Law by considering just two atoms. The reason that this derivation of Bragg's Law is not valid is that it really applies to scattering at a glancing angle where the beam exits the same surface as it enters, not transmission.

We mentioned earlier that the angles shown in all of our figures are exaggerated for the case of diffraction in the TEM. For example, for 111 planes in Cu, *d* is 0.21 nm; λ is 3.35 pm (0.00335 nm or 0.0335 Å) for 120-kV electrons;

equation 11.8 then gives $\theta = 7.97$ mrads (0.46°) for $n = 1$. As a rule of thumb, the Bragg angles of interest are usually no more than 1° when we are forming images, although important information may be present in DPs at much larger (10° to 20°) angles; you will find it useful to remember the order of magnitude of these numbers.

Remember that 10 mrads is 0.573°, i.e., about 0.5°.

We can now generalize from single atoms to planes of atoms. Let's imagine that Figure 11.4 shows two "planes of atoms," P_1 and P_2, and that the points B and C are not necessarily atoms but are simply points on these planes, and that *d* is the shortest distance between the two planes. How is the "in-phase" nature changed if we move atom B but keep it on plane P_2?

Consider scattering from a single plane as shown in Figure 11.5. Geometry shows that while ray R_1 travels a distance EJ, ray R_2 travels a distance HF and that these two distances are equal. Thus there is no path difference for scattering from atoms located anywhere on a particular plane. This seemingly trivial result means that we can generalize our conclusions from Figure 11.4.

It does not matter how the atoms (scattering centers) are distributed on these two planes; the scattering from any two points on planes P_1 and P_2 will produce the same path difference $2d \sin\theta$.

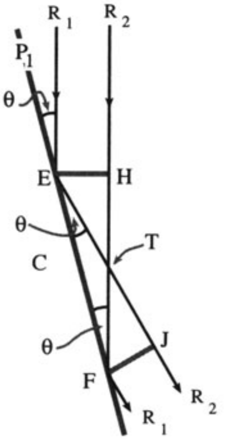

Figure 11.5. Two beams are scattered from two points, E and F, which lie on the same plane P_1. This simple diagram shows that the two beams travel the same distance since triangles EHF and FJE are congruent.

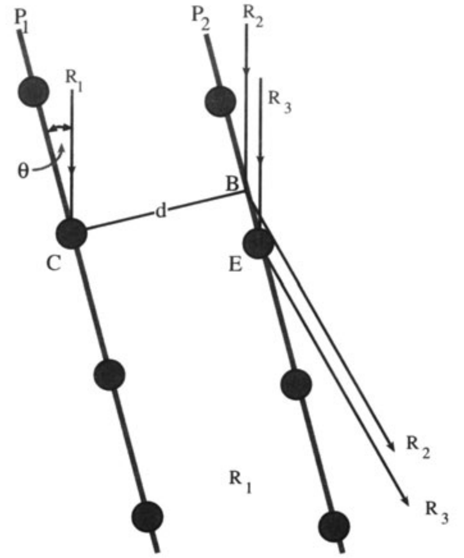

Figure 11.6. Scattering from three points on two planes. The path difference for scattering from points B and C is $2d \sin\theta$, so the path difference for scattering from points C and E is also $2d \sin\theta$. Hence scattering in the direction of the diffracted beam from all points shown will be in phase if $2d \sin\theta = n\lambda$.

This result is summarized in Figure 11.6. Rays R_1, R_2, and R_3 all scatter in phase, if $\theta = \theta_B$.

Next, we extend this analysis to include many parallel planes each a distance *d* from its neighbors, as is shown in Figure 11.7.

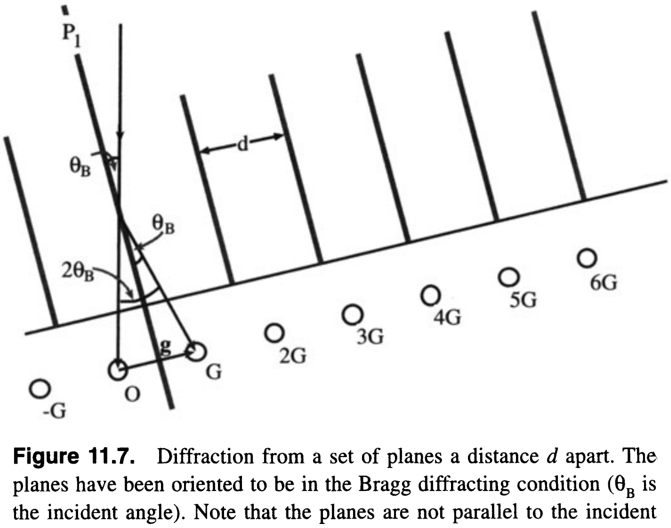

Figure 11.7. Diffraction from a set of planes a distance *d* apart. The planes have been oriented to be in the Bragg diffracting condition (θ_B is the incident angle). Note that the planes are not parallel to the incident beam. The resultant diffraction spots (reciprocal lattice points) are labeled G, 2G, etc. The vector **g** from the origin (O) to the first diffraction spot G is normal to the diffracting plane.

Notation: The zone axis, [*UVW*], is a direction which is common to all the planes of the zone. So [*UVW*] is perpendicular to the normal to the plane ($hk\ell$) if the plane is in the [*UVW*] zone. Later, we will see that [*UVW*] is defined as the incident beam direction. This result applies to all crystal systems and gives the Weiss zone law: $hU + kV + \ell W = 0$.

The Bragg reflection, **g**, is then perpendicular to the set of planes. Clearly this is just another way of expressing equation 11.11. Figures 11.2 and 11.7 remind us that Bragg diffraction occurs when **K** has the value **g**.

11.5. MEANING OF *n* IN BRAGG'S LAW

As is shown in Figure 11.7, and in the DP in Figure 11.1, in practice there will not just be one Bragg reflection but a series of reflections which are periodically spaced along a line; these are known as a *systematic row* of reflections, -G, O, G, 2G, 3G, etc., with corresponding diffraction vectors, $\bar{\mathbf{g}}$, **0**, **g**, 2**g**, 3**g**, etc.

Notation: When discussing beams in diffraction patterns, the letter O will refer to the "direct" beam which is present even when there is no specimen, the letter G (not bold—it's not a vector) will refer to any single diffracted beam; the number **0** (bold) will refer to the diffraction vector for beam O (it is a vector of zero length), and the letter **g** (always bold to remind us that it is a vector) will denote the diffraction vector (in the DP) for beam G. Having said that, many microscopists use G and **g** interchangeably, so beware.

The vector $\bar{\mathbf{g}}$ is pronounced "bar g" and is -G, pronounced "minus g" (!); you will also hear $\bar{\mathbf{g}}$ pronounced "g bar."

These other reflections ($n\mathbf{g}$, where $n \neq 1$), called higher-order reflections, are particularly important in TEM. Pictorially, you can imagine them as arising from the interference from planes which are a distance *nd* apart, where *n* is a rational fraction. To understand the physical meaning of this statement, put a plane P_3 halfway between P_1 and P_2, as shown in Figure 11.8.

Now planes P_1, P_2, and P_3 will scatter in phase when

$$2\left(\frac{d}{2}\right)\sin\theta = \lambda \qquad [11.12]$$

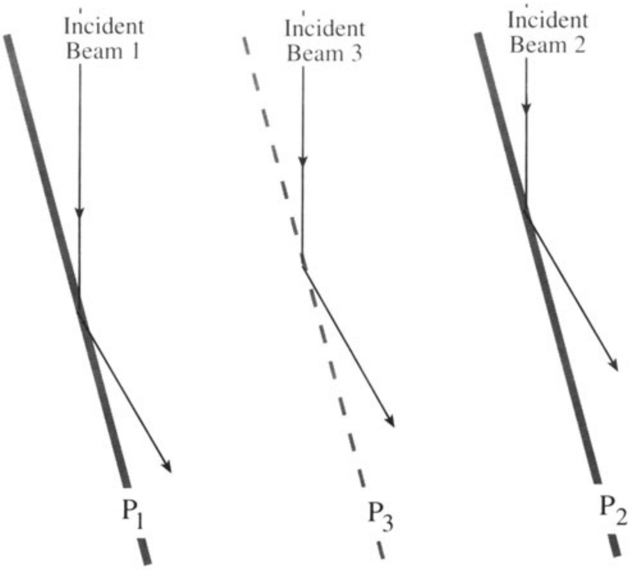

Figure 11.8. Scattering from three planes with plane P_3 positioned exactly halfway between planes P_1 and P_2.

because the new "*d*" is *d*/2. Thus coherent scattering will occur when

$$\left|\mathbf{g}_2\right| = \frac{2}{d} \qquad [11.13]$$

i.e., when

$$\left|\mathbf{g}_2\right| = 2\,|\mathbf{g}| \qquad [11.14]$$

As we noted in the discussion of Figure 11.3, this scattering from plane P_3 will occur no matter how the atoms (scattering centers) are distributed on this plane—even if there are no atoms on the plane! Thus we will always see $\mathbf{g}_2 = 2\mathbf{g}$ and similarly $\mathbf{g}_3 = 3\mathbf{g}$, etc. So we can generalize equation 11.12 to be

$$2\left(\frac{d}{n}\right)\sin\theta = \lambda \qquad [11.15]$$

or rewrite this as

$$2d\sin\theta = n\lambda \qquad [11.16]$$

which gives a physical explanation for the *n* in equation 11.7.

To summarize: electrons are diffracting from a set of planes of spacing *d* such that we have both constructive and destructive interference. We can consider *n* in equation 11.12 as indicating that electrons are diffracting from a set of planes with spacing *d*/*n* rather than *d*. This equation can then be applied to planes which are occupied by different atoms. Although this treatment is not rigorous, it will prove to be very useful in practice. The alternative, but equivalent, view is obtained by considering the Laue equations, which we will do in Chapter 12. You might consider why we don't have diffraction from planes which are *nd* apart instead of *d*/*n*.

11.6. A PICTORIAL INTRODUCTION TO DYNAMICAL EFFECTS

Dynamical diffraction traditionally strikes fear into the heart of the nonmathematician. Unfortunately, in TEM most practical imaging situations involve dynamical scattering. The terminology derives from X-ray theory (where it is not nearly so important). The reason it is very important in electron diffraction is that the electron beam interacts so strongly with the atoms in the crystal. For most purposes, it can be thought of in quite a simple manner. As you can see in Figure 11.9, the beam which has been strongly Bragg-diffracted once is necessarily in the perfect Bragg orientation to be diffracted back into the direct beam by the same set of planes. This beam is then said to be a rediffracted beam. The likelihood of this process occurring will increase as the thickness of the specimen increases. Clearly, the rediffracted beam is also perfectly oriented to be diffracted again, and so on. The two beams in Figure 11.9 are said to be dynamically coupled.

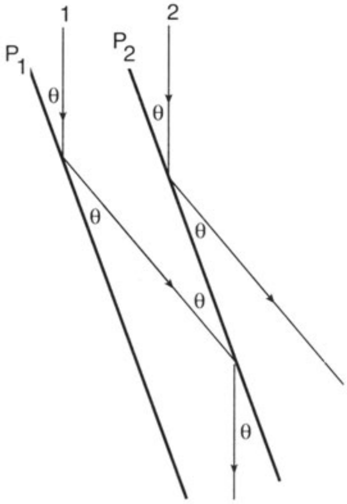

Figure 11.9. The beam can be scattered more than once. Any beam which is oriented so as to be Bragg-diffracted once is automatically in the ideal orientation to be rediffracted. This gives rise to the phenomenon of dynamical scattering.

11.7. USE OF INDICES IN DIFFRACTION PATTERNS

In Chapter 18 we'll teach you how to index diffraction patterns, i.e., how to associate a spot in the diffraction pattern with a diffracting plane in the specimen. For the time being it will be useful if we just introduce the conventions, rather than the methods, of indexing patterns.

First remember that a set of parallel crystal planes is defined by the Miller indices ($hk\ell$) and a set of such planes is $\{hk\ell\}$. We define the direct beam as the 000 reflection and each diffracted beam as a reflection with different $hk\ell$ indices. It is a crystallographic convention to refer to the diffraction spot from a specific ($hk\ell$) plane as $hk\ell$, i.e., without the parentheses. If we assign $hk\ell$ to **g**, then the second-order (2**g**) spot is $2h\ 2k\ 2\ell$, the 3**g** spot is $3h\ 3k\ 3\ell$, etc. Similarly, the $\bar{\mathbf{g}}$ reflection is $\bar{h}\bar{k}\bar{\ell}$. We'll discuss these points further in Section 12.3.

Now we can explain why we see so many spots in the DP. If we look along a zone axis in a crystal, we will see sets of planes in the edge-on orientation. Remember that a zone axis is the direction along the intersection of two or more planes.

> *Notation:* The zone axis, [*UVW*], is a direction which is common to all the planes of the zone. So [*UVW*] is perpendicular to the normal to the plane ($hk\ell$) if the plane is in the [*UVW*] zone. Later, we will see that [*UVW*] is defined as the incident beam direction. This result applies to all crystal systems and gives the Weiss zone law: $hU + kV + \ell W = 0$.

If there are many planes close to the Bragg orientation, then we will see spots from many different planes. We still have not explained why we can see the 200 spot and the 400 spot in the same pattern (they clearly can't both satisfy the Bragg condition at the same time). This results from the physical shape of the TEM specimen and will be discussed in Chapters 12 and 17.

11.8. PRACTICAL ASPECTS OF DIFFRACTION-PATTERN FORMATION

Remember from Chapter 9, we can form diffraction patterns in the TEM in two complementary ways, SAD and CBED patterns.

SAD patterns are sharply focused spot patterns, which we use to select reflections for all imaging modes. We can easily associate the sharp spots with our diffraction vectors, **g**.

CBED patterns are arrays of disks. We can associate a **g** vector with each disk but the location of **g** requires more extensive consideration. For this reason, we'll delay more detailed discussion of CBED patterns while we develop diffraction theory and then devote two chapters to the topic, because it is very important.

11.9. MORE ON SELECTED-AREA DIFFRACTION PATTERNS

We discussed how you form a DP in the SAD mode in Chapter 9. Now we will discuss some of the practical implications and drawbacks of the method.

Why do we want to select a specific area to contribute to the DP? All foils are distorted to some extent so that diffraction conditions change as we cross the specimen, so we need to select areas of constant orientation. Also, we may wish to determine the orientation relationship between two different crystals, which we can do by selecting the interfacial region. Alternatively, we may want to study the DP from a small particle within the foil. Figure 11.10 is a reminder that the DP is formed at the back focal plane (BFP) of the objective lens. A similar diagram was shown in Figure 9.13.

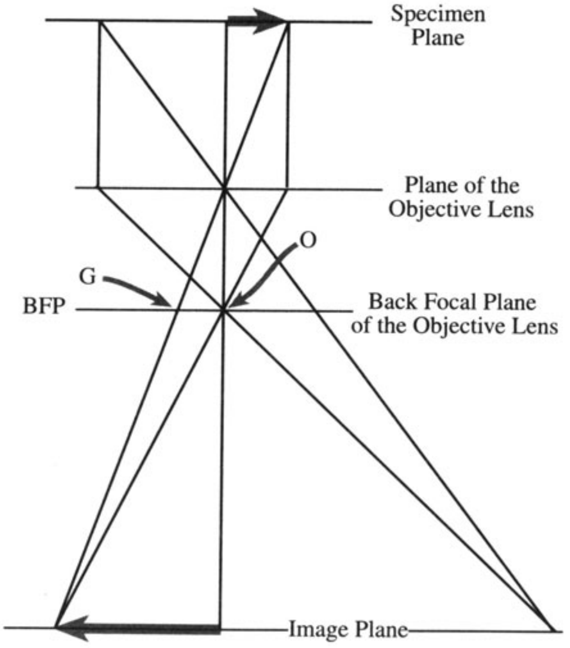

Figure 11.10. The diffraction pattern is formed at or close to the back focal plane of the objective lens. O is the direct beam and G is a diffracted beam.

The SAD method for selecting an area is to place an aperture in the first image plane below the objective lens. In this case we really are selecting an area, which is the area in an image; but we always refer back to the volume of the diffracting specimen. Since we are working at an image plane we do not need to focus the condenser lens, in fact we generally weaken (underfocus) this lens to give more parallel illumination so that all the rays are focused at the same plane, i.e., the BFP. The spots in the DP then become sharper. In practice you will generally need to "fine-tune" the focus of the DP since its focus depends on the excitation of the condenser lens.

The key practical steps in forming an SAD pattern are:

- Be sure that you are at the eucentric focus position, with an image of the area of interest focused on the screen.
- Insert the SAD aperture.
- Remove the objective aperture.
- Focus the SAD aperture.
- Switch to diffraction mode.
- Spread the beam using C2, within the limits imposed by your specimen.
- Focus the DP with the intermediate lens (diffraction focus).

Remember that using an aperture to select an area in the image plane gives an additional advantage: the area has already been magnified, typically 25×. Thus a 50-μm aperture will select a 2-μm area on the specimen.

You might ask: why can't we just use a smaller SAD aperture to select a smaller area? We can provide the answer by looking at Figure 11.11, which shows the "real" version of Figure 11.10 since the objective lens is not perfect. As we saw in Chapter 6, the beams which are further away from the optic axis are bent more strongly as they pass through the objective lens. For rays entering the lens at an angle β to the optic axis, the image formed at magnification M, is translated a distance r_M given by

$$r_{\mathrm{M}} = M\, C_{\mathrm{s}}\, \beta^3 \qquad [11.17]$$

So the area we select using the SAD aperture corresponds to the area PP_1 in the object plane *only* for the direct beam. The error increases as β increases, so that it's larger for a larger Bragg angle or for a larger **g**. The result is illustrated schematically in Figure 11.12 with values given in Table 11.1. (Note that we divide r_M by M to give the distance at the specimen.) The values in the middle column were calculated for a C_s of 3.3 mm and 100-keV electrons. If you use a smaller aperture, selecting an area of less than 1-μm diameter, even the fourth-order 111 reflection, i.e., the 444

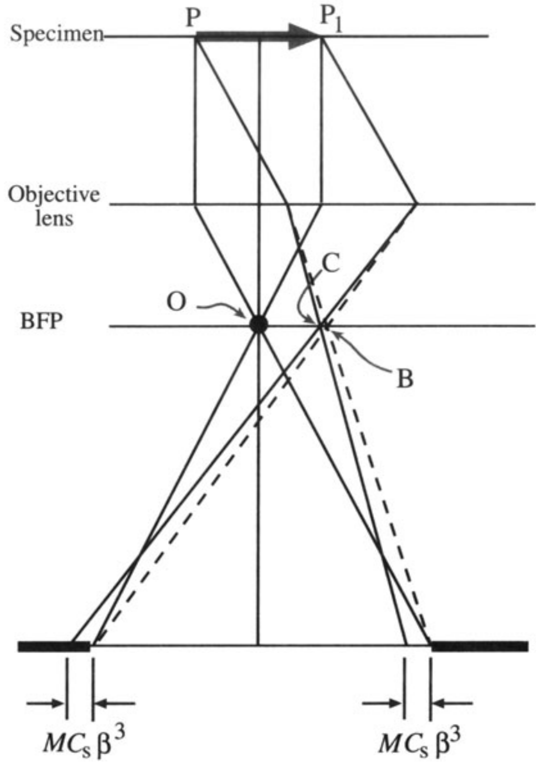

Figure 11.11. Formation of an SAD pattern showing that there is an error in selecting the area if the beams do not travel at the same angle to the optic axis. This difference is due to spherical aberration in the objective lens. B is the diffraction spot position for a perfect lens and C is the spot position with spherical aberration.

reflection, from this area would not contribute to the SAD pattern. Instead, a different area, possibly even an adjacent crystal, would contribute.

We will produce another selection error if the aperture is not located at the image plane. This effect can be seen clearly in Figure 11.13, where the objective lens is focused on plane P_f rather than on the specimen. The effect is seen by simple geometry if you extend the diffracted rays back to the specimen plane. The displacement at the first image plane (where the SAD aperture is located) corresponds to a distance y at the specimen plane, where y is given by

$$y = D\beta \tag{11.18}$$

On some older machines a "click" on the medium image focus control (i.e., of the objective lens) corresponded to a change in focus, D ($0.5D_{\mathrm{Ob}}$), of ~3 μm (see Figure 6.14). You will still find on many TEMs that the aperture in the

Table 11.1. The Displacement Distance of the Image Formed by "reflection G" Due to Spherical Aberration

Reflection in SAD pattern	$C_s\beta^3$ (nm) old TEM	$C_s\beta^3$ (nm) modern IVEM
111	13	1.2
222	100	9.1
333	350	31.9
444	760	69.3
555	1620	150
666	2800	250

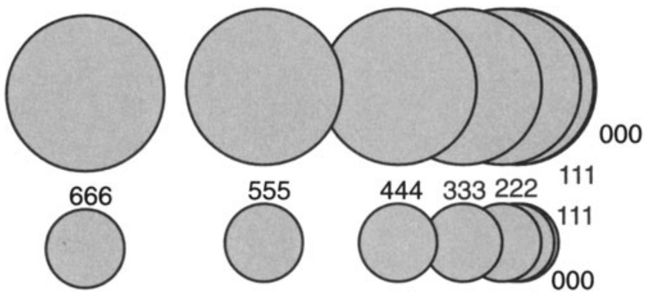

Figure 11.12. Schematic diagram showing the effective error in area selection, due to spherical aberration, for different reflections in the 111 systematic row for Al (a_0 = 4.04 Å) assuming 100-keV electrons and C_s = 3 mm. The 000 and 111 disks almost exactly overlap (the translation is 13 nm). The diameter of each disk in the top row is 1 μm, and the diameter of each disk in the bottom row is 0.5 μm.

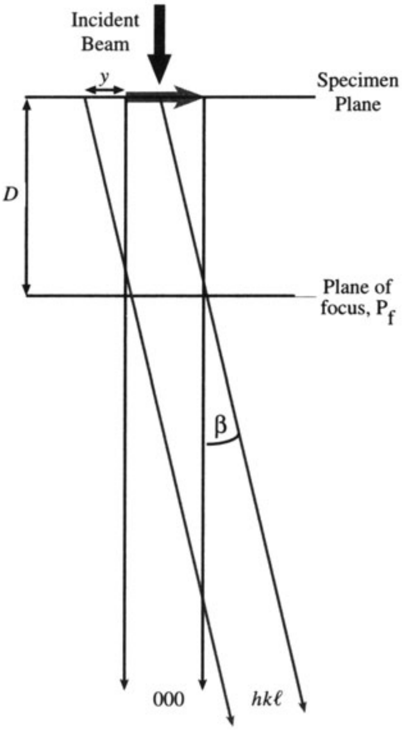

Figure 11.13. If the lens is not focused on the SAD plane, images associated with the different **g** vectors will be shifted with respect to one another. D is the defocus. The shift in the selected area is given by $y = D\beta$.

SAD plane is not always in focus when the DP is in focus. You might also consider the implications when we study very thick specimens. Remember that these two sources of error may be additive and therefore quite substantial.

You may still sometimes want to use an aperture which conventional wisdom tells you is "too small for SAD." Perhaps the best advice when this is the case is, if possible, use CBED. However, you should remember that "conventional wisdom" is based on the middle column in Table 11.1, which was first given by Hirsch *et al.* (1977) and applied to a machine built in the 1950s! A modern 300-kV machine may have a C_s of ~1 mm and a λ (at 300 kV) of 0.1968 nm. The values for $C_s\beta^3$ then become much smaller, as shown in the right-hand column in Table 11.1. Clearly you could now use a much smaller SAD aperture, and 10 μm is about the smallest that can be manufactured.

One question which is often asked is: if the SAD aperture is placed at the first image plane, how can it affect the DP which is formed above it? The relationship between the SAD pattern and the image(s) can be illustrated by forming a multiple dark-field image of the type illustrated in Figure 11.14A. To do this, you must first form the SAD pattern in the usual way. Then increase the strength of the intermediate lens so that it's focused below the BFP in Figure 11.14B. Instead of a point we then see a disk, because the beam is convergent at the BFP. To understand what is happening we must realize that the magnification of the specimen at the BFP is zero (i.e., when "X" in Figure 11.14B is in the BFP plane)! As we increase the strength of the intermediate lens, staying in diffraction mode, we increase the magnification of these images (one bright-field image and many dark-field images). Of course, these images are not in focus but this can be corrected by adjusting the strength of the objective lens, which is just conventional focusing.

Now you can appreciate directly that each disk corresponds to a reflection in the SAD pattern. The reflections that were bright now correspond to bright disks; the area was close to the Bragg condition for that reflection. It is at first surprising to realize that none of the disks is uniformly bright. Conversely, most of the disks are partly bright! We'll examine the reasons for this variation in Chapter 13.

This uncertainty in the area of selection of SAD patterns is one reason that CBED patterns can have some advantage whenever you want to get crystallographic information about specific regions of your specimen.

We'll end with some more practical points.

You can change the detail present in your DP simply by changing the C2 lens setting and the exposure time.

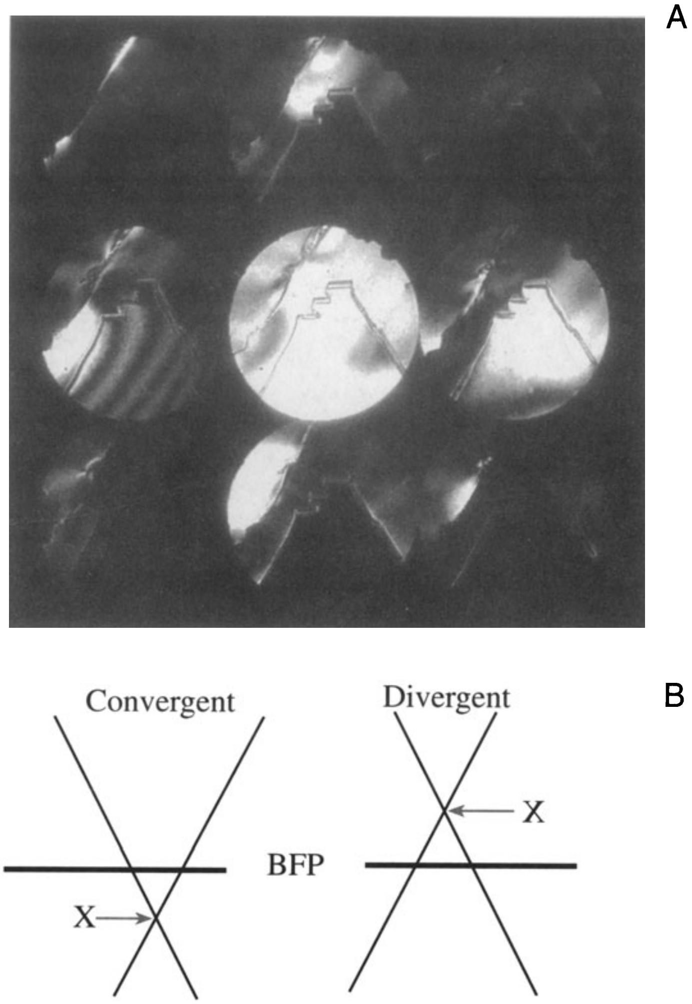

Figure 11.14. (A) Multiple dark-field images formed by defocusing the SAD pattern revealing dark-field images in each diffraction disk. Close inspection reveals that each image (of a twin boundary) is slightly shifted from the adjacent images, reflecting the increased error in area selection for higher-order reflections; (B) formation of a disk occurs because a defocused beam is either convergent or divergent at the BFP. An underfocused convergent beam is preferred, since it is more parallel than an overfocused divergent beam (see Figure 6.5).

To record the SAD pattern you should never use an exposure of <10 s. You don't need to use a 1-second exposure to limit drift! If you're interested in the details in the diffraction pattern you should take as many as three exposures, say 10 s, 30 s, and 100 s. So spread the beam with C2 and remove that beam stop (better still, don't use it; you're damaging your specimen if the beam is that intense). Correct the astigmatism in the intermediate lens after you've spread the beam; this astigmatism becomes noticeable when your spots are small (not all microscopes allow you to do this). Focus the spots to sharp points with the diffraction (intermediate) lens; now you've focused the diffraction pattern. Just for the exercise, focus the spots in the

SAD pattern as you generally view it with the beam condensed down to a minimum diameter. Now spread the beam with C2 and refocus the spots; you will see quite a difference in how sharp the spots are. Use the binoculars for focusing the spots after you've spread the beam. Unless the pattern is well focused, you will miss many of the fine details that make electron diffraction so useful.

Deciding which pattern is best really depends on what information you require. If you would like to see fine detail in your SAD pattern, you will probably need to underfocus the beam using C2. If the beam of interest is of low intensity, you may need to increase the exposure time at the risk of broadening the more intense spots. In fact, it's good practice to record patterns with a range of exposures, from a few seconds to 100 s if necessary. DPs can be recorded on video or sent directly to the computer using a video camera. The use of a CCD camera can give a much greater range of intensities than the photographic film; this will become the preferred method of recording diffraction patterns in the future.

Cooling the specimen can reduce the thermal diffuse scattering and thus reduce the background intensity considerably. Changes in the lattice parameter will not usually be a problem in SAD since we are not looking for that level of accuracy, but they will be noticeable in the HOLZ-line patterns (see Chapter 21).

Finally, if your specimen charges, you'll probably have to coat it with a thin film of carbon. Do practice this. Repeat several thin coatings if necessary and be sure that the charging is not due to a problem in the specimen contacting the specimen holder or the holder contacting the ground.

CHAPTER SUMMARY

Diffraction patterns are the basis of all image formation in the TEM as well as all crystallographic analysis and defect characterization. We can understand DPs in terms of Bragg reflection from planes of atoms in the specimen, and we can define the diffraction vector **g** associated with each Bragg reflection and associate each **g** with a crystal plane $hk\ell$. The diffracting planes are all in a specific zone axis *UVW*, which we can define as parallel to the incident beam direction.

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