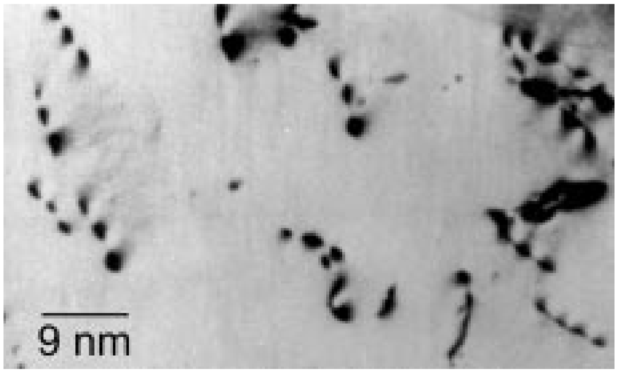
7. Diffraction Contrast in TEM Images



7.1 Contrast in TEM Images

This chapter explains the origin of features observed in many TEM images of crystalline materials. These microstructural features, having sizes from nanometers to microns, control many important properties of materials. TEM provides information about these microstructural features that is often more detailed and more direct than can be obtained by any other experimental technique. There are subtleties, however, in the interpretation of images such as those from the dislocation segments running from top to bottom of the sample in the figure above. The dislocations themselves do not have the modulations in width seen in the image, and the dislocation images are displaced horizontally from the actual positions of the cores of the dislocations. By changing the tilt of the incident beam on the Bragg planes of the crystal, i.e., changing the "diffraction condition," the images of the dislocations can shift in position, split in two, or disappear entirely.

"Contrast" is the appearance of a feature in an image. Contrast in bright-field (BF) and dark-field (DF) TEM images is usually "diffraction contrast," or the variations in intensity of diffraction across the sample. Chapter 5 discussed the Laue condition, written as $\boldsymbol{\Delta k} = \boldsymbol{g} - \boldsymbol{s}$, where the direction of

$\boldsymbol{\Delta k}$ is adjustable by tilts, and $\boldsymbol{g}$ is a reciprocal lattice vector of the crystal. Diffraction contrast and the appearance of features in BF and DF images depend sensitively on how the Laue condition is satisfied – specifically which diffraction is active, and the value of the deviation parameter, s (the magnitude of $\boldsymbol{s}$). The alternative "mass thickness contrast" is generally weaker and overshadowed by the stronger effects of electron diffraction, except in cases where there are large differences in atomic number or when diffraction is weak. "Phase contrast" and "Z-contrast" methods of high-resolution imaging, described in Chap. 10, offer better spatial resolution than conventional TEM imaging, but these HRTEM methods require considerably more sophisticated instruments, operator skill, and usually more interpretation.

The focus of this chapter is on individual "defects" in crystalline materials. The name "defect" may lack dignity, but it has come to mean the microstructural features within materials that are responsible for many of the structure-property relationships in materials science. Some aspects of diffraction from three-dimensional defects, e.g., second phase particles, were presented in Sect. 5.4.3. Their BF and DF TEM images are typically two-dimensional regions having different contrast (brightness) than the surrounding matrix. The present chapter provides a thorough discussion of images of these defects. It also discusses two-dimensional interfaces between crystals and between domains. The diffraction contrast from crystalline interfaces often contains rows of one-dimensional bands, or fringes. There are several different types of fringes, and they can often be distinguished by how their appearance changes with the tilt of the beam or with the tilt of the crystal. The important one-dimensional crystalline defect, the dislocation, has structures and strains that are reviewed in Appendix A.12. Dislocations cause severe local distortions of the surrounding crystal. It is, in fact, the strains in the crystal that provide the diffraction contrast of the dislocation, not the core of the dislocation itself. Zero-dimensional point defects, e.g., vacancies and impurities, are generally not visible in conventional TEM images, but strain effects around nanometer-scale chemical zones, such as clusters of atoms or vacancies, can be imaged and understood semi-quantitatively.

This chapter begins with a review of kinematical diffraction theory. In conventional TEM, many of the diffraction effects are dynamical in origin, but dynamical theory is not presented until Chap. 11. Nevertheless, we use the "extinction length" from Chap. 11 to justify the kinematical dependence of diffraction intensity on specimen thickness and diffraction error. The mathematical form of the kinematical intensity is the same as for dynamical theory without absorption, and the predicted contrast is often correct qualitatively. The physical origin of the diffraction contrast is often different in dynamical and kinematical theory, however.

Semi-quantitative analyses of diffraction contrast are often performed with a "phase-amplitude diagram." The phase-amplitude diagram is a graphical construction of a complex Fourier transform, so it is drawn on the complex plane. The phase-amplitude diagram provides a sum of the relative phases of the wavelets diffracted from unit cells through the thickness of a sample. This vector sum provides the amplitude of the total diffracted wave. The phase-amplitude diagram is often puzzling when encountered for the first time. Once the reader acquires the knack for using it, however, he or she can obtain quick answers to many new diffraction problems for which analytical Fourier transforms are unavailable.

There are indeed several important cases where kinematical theory proves entirely inadequate for understanding image contrast, even when it is extended formally to dynamical theory without "absorption." Results from dynamical theory with absorption are needed to analyze quantitatively the nature of stacking faults and the "Ashby-Brown contrast" from small coherent particles. Such methods are presented in this chapter, but largely without justification. A full justification is beyond the scope of this book, both in its level of detail and in its reliance on computer calculation. A qualitative discussion of dynamical theory with absorption is provided in Chap. 11.

7.2 A Review of Structure and Shape Factors

With the kinematical theory as developed so far, we can obtain simple analytical expressions for the scattered wave, ψ, for only a few geometries, such as a perfect crystal with flat surfaces. This chapter presents a graphical method that is a powerful and intuitive tool for evaluating ψ in an approximate way. Most importantly, we show how this method of "phase-amplitude diagrams" can predict the diffraction contrast near an individual crystalline defect.

We begin by reviewing the deviation vector $\boldsymbol{s}$ and its effect on the diffraction intensity from a crystal:

$$\boldsymbol{g} = \boldsymbol{\Delta k} + \boldsymbol{s} \ , \tag{7.1}$$

where $\boldsymbol{g}$ is a reciprocal lattice vector and $\boldsymbol{\Delta k}$ is the diffraction vector whose ends lie on the Ewald sphere ($\boldsymbol{\Delta k} \equiv \boldsymbol{k} - \boldsymbol{k}_0$). For high-energy electrons, the shortest distance between the Ewald sphere and a reciprocal lattice point, $\boldsymbol{g}$, is parallel to the $\widehat{\boldsymbol{z}}$ direction, so we often work with only the magnitude of $\boldsymbol{s}$, plus its sign along $\widehat{\boldsymbol{z}}$, known as the "deviation parameter," s:

s is the minimum distance ***from*** *the Ewald* ***sphere***
to *the reciprocal lattice* ***point****.*

We choose a sign convention for s that is convenient when we determine s by measuring the positions of Kikuchi lines. Positive s means that $\boldsymbol{s}$ points along positive z.[1] Figure 6.25 shows that s is positive when the reciprocal lattice

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[1] By convention, $\widehat{\boldsymbol{z}}$ points towards the electron gun. This is handy for diffraction patterns and stereographic projections. On the other hand, when integrating wavelet amplitudes from top to bottom of a specimen, we may want $\widehat{\boldsymbol{z}}$ to point down. In such cases it may be necessary to handle with care the sign of the phase $2\pi sz$, as in Sect. 7.9.