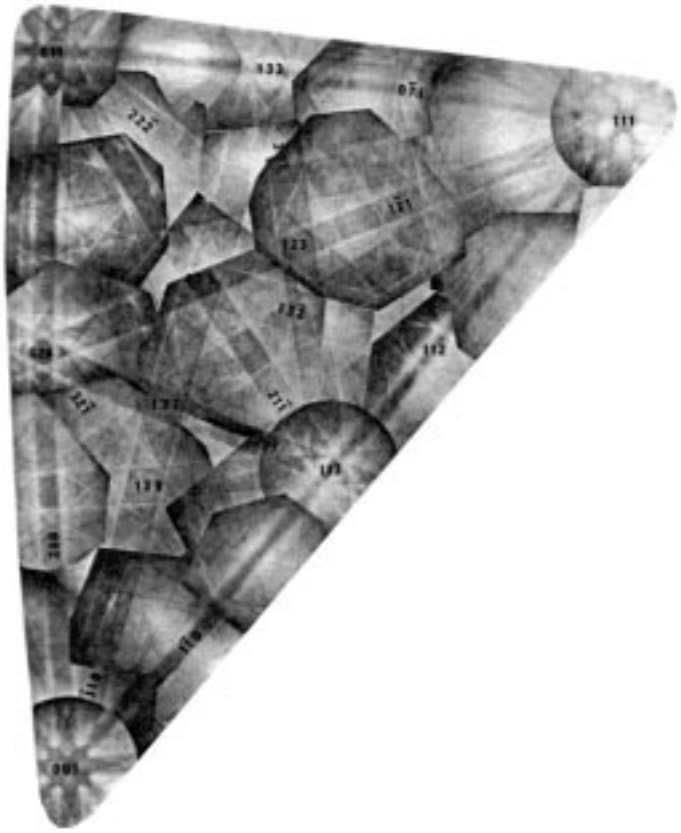
6. Electron Diffraction and Crystallography



6.1 Indexing Diffraction Patterns

Reciprocal lattices of materials are spanned by three reciprocal-lattice vectors, so the diffraction patterns of materials are inherently three-dimensional. To obtain all available diffraction information, the diffraction intensity should be measured for all magnitudes and orientations of the three-dimensional

diffraction vector, $\boldsymbol{\Delta k}$. Appropriate spherical coordinates are Δk, θ, and ϕ. A practical approach to this fairly complicated problem is to separate the control over the magnitude of $\boldsymbol{\Delta k}$ from its orientation with respect to the sample. The θ–θ or θ–2θ goniometers described in Sect. 1.3.2 provide the required control over the magnitude Δk, while maintaining a constant direction $\widehat{\boldsymbol{\Delta k}}$ on the sample. For isotropic polycrystalline samples, a single powder-diffraction pattern provides representative diffraction data because all crystal orientations are sampled. For specimens that are single crystals, however, it is also necessary to provide for the orientational degrees of freedom of the specimen (latitude and longitude angles, for example). A diffraction pattern (varying Δk) should then be obtained for each orientation within the selected solid angle ($\sin\theta\,\mathrm{d}\theta\mathrm{d}\phi$) of reciprocal space. Diffraction experiments with single crystals require additional equipment for specimen orientation, and software to relate these data to the reciprocal space structure of the three-dimensional crystal. For publication and display of these data, however, it is typical to present the diffraction intensities as planar sections through the three-dimensional data.

Diffraction data from the TEM are obtained as near-planar sections through k-space. The large electron wavevector provides an Ewald sphere that is nearly flat, allowing the handy approximation that a diffraction pattern from a single crystal is a picture of a plane in its reciprocal space.[1] The magnitude of the diffraction vector, Δk, is obtained from the angle between the transmitted and diffracted beams. Two degrees of orientational freedom are required for the sample in a TEM. They are typically provided by a "double-tilt specimen holder," which has two perpendicular tilt axes oriented perpendicular to the incident electron beam.

A modern TEM provides two modes for obtaining diffraction patterns from individual crystallites. The oldest is selected area diffraction (SAD), which is useful for obtaining diffraction patterns from regions as small as 0.5 μm in diameter (see Problem 2.16). The second method is nanodiffraction, or convergent-beam electron diffraction (CBED), in which a focused electron probe beam is used to obtain diffraction patterns from regions as small as 10 Å. Both techniques provide a two-dimensional pattern of diffraction spots, which can be highly symmetrical when a single crystal is oriented precisely along a crystallographic direction. The additional three-dimensional information available in CBED patterns is discussed in Sect. 6.5.

6.1.1 Issues in Indexing

We now describe how to "index" the planar sections of single crystal diffraction patterns; i.e., label the individual diffraction spots with their appropriate

[1] It is not quite the full picture, however, because the diffraction pattern measures the intensity of the diffracted wave and not the wave itself.