

2.5. ELECTRON DIFFRACTION AND ELECTRON MICROSCOPY IN STRUCTURE DETERMINATION

(c) the diffraction intensity $|\Phi(U)|^2$ is a radially symmetric, smoothly varying function such as is normally produced by a sufficiently large area of the image of an amorphous material;

(d) there is no astigmatism present and no drift of the specimen; either of these factors would remove the radial symmetry.

From the form of (2.5.2.54) and a preknowledge of $|\Phi(U)|^2$, the zero crossings of $\sin \chi$ and the form of $E(U)$ may be deduced. Analysis of a through-focus series of images provides more complete and reliable information.

(2) Detail on a scale much smaller than the resolution of the electron microscope, as defined above, is commonly seen in electron micrographs, especially for crystalline samples. For example, lattice fringes, having the periodicity of the crystal lattice planes, with spacings as small as 0.6 Å in one direction, have been observed using a microscope having a resolution of about 2.5 Å (Matsuda *et al.*, 1978), and two-dimensionally periodic images showing detail on the scale of 0.5 to 1 Å have been observed with a similar microscope (Hashimoto *et al.*, 1977).

Such observations are possible because

(a) for periodic objects the diffraction amplitude $\Psi_0(uv)$ in (2.5.2.31) is a set of delta functions which may be multiplied by the corresponding values of the transfer function that will allow strong interference effects between the diffracted beams and the zero beam, or between different diffracted beams;

(b) the envelope functions for the WPOA, arising from incoherent imaging effects, do not apply for strongly scattering crystals; the more general expression (2.5.2.36) provides that the incoherent imaging factors will have much less effect on the interference of some sets of diffracted beams.

The observation of finely spaced lattice fringes provides a measure of some important factors affecting the microscope performance, such as the presence of mechanical vibrations, electrical interference or thermal drift of the specimen. A measure of the fineness of the detail observable in this type of image may therefore be taken as a measure of 'instrumental resolution'.

2.5.2.10. Electron diffraction in electron microscopes

Currently most electron-diffraction patterns are obtained in conjunction with images, in electron microscopes of one form or another, as follows.

(a) Selected-area electron-diffraction (SAED) patterns are obtained by using intermediate and projector lenses to form an image of the diffraction pattern in the back-focal plane of the objective lens (Fig. 2.5.2.2). The area of the specimen from which the diffraction pattern is obtained is defined by inserting an aperture in the image plane of the objective lens. For parallel illumination of the specimen, sharp diffraction spots are produced by perfect crystals.

A limitation to the area of the specimen from which the diffraction pattern can be obtained is imposed by the spherical aberration of the objective lens. For a diffracted beam scattered through an angle α , the spread of positions in the object for which the diffracted beam passes through a small axial aperture in the image plane is $C_s \alpha^3$, e.g. for $C_s = 1$ mm, $\alpha = 5 \times 10^{-2}$ rad (10,0,0 reflection from gold for 100 keV electrons), $C_s \alpha^3 = 1250$ Å, so that a selected-area diameter of less than about 2000 Å is not feasible. For higher voltages, the minimum selected-area diameter decreases with λ^2 if the usual assumption is made that C_s increases for higher-voltage microscopes so that $C_s \lambda$ is a constant.

(b) Convergent-beam electron-diffraction (CBED) patterns are obtained when an incident convergent beam is focused on the specimen, as in an STEM instrument or an STEM attachment for a conventional TEM instrument.

For a large, effectively incoherent source, such as a conventional hot-filament electron gun, the intensities are added for

each incident-beam direction. The resulting CBED pattern has an intensity distribution

$$I(uv) = \int |\Psi_{u_1 v_1}(uv)|^2 du_1 dv_1, \quad (2.5.2.55)$$

where $\Psi_{u_1 v_1}(uv)$ is the Fourier transform of the exit wave at the specimen for an incident-beam direction u_1, v_1 .

(c) Coherent illumination from a small bright source such as a field emission gun may be focused on the specimen to give an electron probe having an intensity distribution $|t(xy)|^2$ and a diameter equal to the STEM dark-field image resolution [equation (2.5.2.47)] of a few Å. The intensity distribution of the resulting microdiffraction pattern is then

$$|\Psi(uv)|^2 = |\Psi_0(uv) * T(uv)|^2, \quad (2.5.2.56)$$

where $\Psi_0(uv)$ is the Fourier transform of the exit wave at the specimen. Interference occurs between waves scattered from the various incident-beam directions. The diffraction pattern is thus an in-line hologram as envisaged by Gabor (1949).

(d) Diffraction patterns may be obtained by using an optical diffractometer (or computer) to produce the Fourier transform squared of a small selected region of a recorded image. The optical diffraction-pattern intensity obtained under the ideal conditions specified under equation (2.5.2.54) is given, in the case of weak phase objects, by

$$I(uv) = \delta(uv) + 4\sigma^2 |\Phi(uv)|^2 \cdot \sin^2 \chi(uv) \cdot E^2(uv) \quad (2.5.2.57)$$

or, more generally, by

$$I(uv) = c\delta(uv) + |\Psi(uv) \cdot T(uv) * \Psi^*(uv) \cdot T^*(uv)|^2,$$

where $\Psi(uv)$ is the Fourier transform of the wavefunction at the exit face of the specimen and c is a constant depending on the characteristics of the photographic recording medium.

2.5.3. Point-group and space-group determination by convergent-beam electron diffraction

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2.5.3.1. Introduction

Because the cross section for electron scattering is at least a thousand times greater than that for X-rays, and because multiple Bragg scattering preserves information on symmetry (such as the absence of inversion symmetry), electron diffraction is exquisitely sensitive to symmetry. The additional ability of modern electron-optical lenses to focus an electron probe down to nanometre dimensions, and so allow the study of nanocrystals too small for analysis by X-rays, has meant that the method of convergent-beam diffraction described here has now become the preferred method of symmetry determination for very small crystals, domains, twinned structures, quasicrystals, incommensurate structures and other imperfectly crystalline materials.

Convergent-beam electron diffraction (CBED) originated with the experiments of Kossel & Möllenstedt (1938). However, modern crystallographic investigations by CBED began with the studies performed by Goodman & Lehmpfuhl (1965) in a modified transmission electron microscope. They obtained CBED patterns by converging a conical electron beam with an angle of more than 10^{-3} rad on an ~ 30 nm diameter specimen area, which had uniform thickness and no bending. Instead of the