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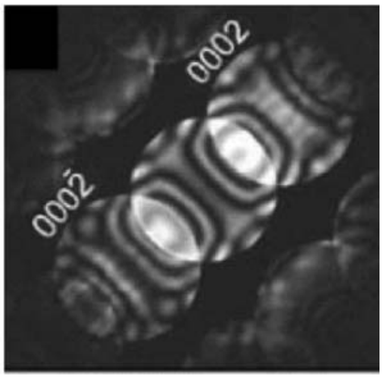

Figure 1.6.6.1
Polarity determination by convergent-beam electron diffraction. A CBED pattern from ZnO with the beam normal to the *c* axis is shown. The intensity distribution along **c** does not have inversion symmetry, reflecting the noncentrocentrosymmetric nature of the structure. Reproduced with permission from Wang *et al.* (2003). Copyright (2003) by The American Physical Society.

and recorded. The entire pattern thus consists of many independent 'point' diffraction patterns (each for a slightly different incident beam direction) laid beside each other. Fig. 1.6.6.1 shows a CBED pattern from the wurtzite structure of ZnO, with the beam normal to the *c* axis (Wang *et al.*, 2003). The intensity variation along a line running through the centres of these discs (along the *c* axis) is not an even function, strongly violating Friedel's law for this elastic scattering. At higher scattering angles, curvature of the Ewald sphere allows three-dimensional symmetry elements to be determinated by taking account of 'out-of-zone' intensities in the outer higher-order Laue zone (HOLZ) rings near the edge of the detector. Since sub-ångstrom-diameter electron probes and nanometre X-ray laser probes (Spence *et al.*, 2012) are now being used, the effect of the inevitable coherent interference between overlapping convergent-beam orders on space-group determination must be considered (Spence & Zuo, 1992).

A systematic approach to space-group determination by CBED has been developed by several groups. In general, one would determine the symmetry of the projection diffraction group first (ignoring diffraction components along the beam direction *z*), then add the *z*-dependent information seen in HOLZ lines, allowing one to finally identify the point group from tables, by combining all this information. After indexing the pattern, in order to determine a unit cell the Bravais lattice is next determined. The form of the three-dimensional reciprocal lattice and its centring can usually be determined by noting the registry of Bragg spots in a HOLZ ring against those in the zero-order (ZOLZ) ring. Finally, by setting up certain special orientations, tests are applied for the presence of screw and glide elements, which are revealed by a characteristic dark line or cross within the CBED discs. Tables can again then be used to combine these translational symmetry elements with the previously determined point group, to find the space group. As a general experimental strategy, one first seeks mirror lines (perhaps seen in Kikuchi patterns), then follows these around using the two-axis goniometer fitted to modern TEM instruments in a systematic search for other symmetry elements. Reviews of the CBED method can be found in Steeds & Vincent (1983), in Goodman (1975), and in the texts by Tanaka *et al.* (1988). A textbook-level worked example of space-group determination by CBED can be found in Spence & Zuo (1992) and in the chapter by A. Eades in Williams & Carter (2009).

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