3.6. Specimens for neutron diffraction

By B. T. M. Willis

Specimen preparation for neutron diffraction presents few of the problems encountered in electron diffraction and electron microscopy (Chapter 3.5). This is because – with the exception of a few isotopes – the atomic absorption cross section for slow neutrons is several orders of magnitude less than that for electrons. Whereas thin sections must be prepared from bulk samples for examination by electron microscopy, the bulk samples themselves are used in neutron diffraction.

For structural studies with single crystals, the size of crystal required depends on the magnitude of the incident neutron flux. The fluxes available worldwide from different sources are tabulated by Bacon (1987). If a flux of 10^{14} neutrons cm^{-2} s^{-1} is assumed, a crystal of linear dimension about 1 mm is necessary. Corrections for extinction, absorption, and multiple scattering are easier to apply if the crystal is in the form of a flat plate or sphere. Crystals containing hydrogen give rise to a uniform background from incoherent scattering. This background can be removed by deuteration, but for measuring Bragg intensities this is rarely essential. The sample can be examined *in vacuo* or in an inert atmosphere by sealing it inside a silica tube, which causes very little attenuation of the neutron beam for a wall thickness of 0.5 mm.

Structural studies on polycrystalline samples are often undertaken using a cylindrical volume of material, enclosed in a holder of aluminium or vanadium. Apart from its cost, vanadium is an ideal container because its atomic coherent scattering cross section (3.3×10^{-30} m^2) is negligible compared with its incoherent cross section (530×10^{-30} m^2); consequently, the container contributes significantly to the background, but gives no diffraction lines. Aluminium has negligible absorption and incoherent scattering, and weak coherent scattering. It therefore produces no background scattering, but it does give rise to diffraction peaks, which may be superimposed on those of the sample. Unlike in single-crystal work, it is usually necessary to replace hydrogen atoms by deuterium. By using a cylindrical sample, effects due to preferred orientation can be reduced by rotating the cylinder about its vertical axis.

Coherent inelastic scattering studies, used to investigate the lattice dynamics of crystalline solids [see Section 4.1.1 in *IT* B (1992)], require single crystals of high purity and crystalline perfection (mosaic spread less than 0.3°). Counting rates are, perhaps, one thousand times less than for structural studies, so that the crystal size is measured in centimetres rather than millimetres. Crystals up to 50 cm^3 in volume may be used. For such large crystals, there is an upper limit of about 10×10^{-30} m^2 to the atomic absorption cross section. This is considerably less than the effective absorption cross section of hydrogen (arising from incoherent scattering), so that hydrogenous compounds must be deuterated for neutron work in which coherent inelastic scattering is measured.

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