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2.8. Neutron diffraction topography

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2.8.1. Introduction

Some salient differences between neutron diffraction and X-ray diffraction are that

(*a*) neutron beams are not available in standard ('home') laboratories,

(*b*) the available neutron fluxes are small even at a high-flux reactor and even when compared with laboratory X-ray generators (Scherm & Fåk, 1993),

(*c*) absorption is negligible in most materials (see Section 4.4.6), and

(*d*) magnetic scattering is a strong component (see Section 4.4.5).

All these differences have effects on the use of neutrons for diffraction imaging (hereafter called, according to standard usage, neutron topography), while the obvious similarities in scattering amplitude and geometry make such topography possible. The effect of (*a*) is that the first attempts at neutron topography occurred late, with the work of Doi, Minakawa, Motohashi & Masaki (1971), Ando & Hosoya (1972), and Schlenker & Shull (1973), and that it is practised at very few places in the world, though one of them, at Institut Laue–Langevin (ILL), is open to external users.

2.8.2. Implementation

As a result of (*b*), the resolution of neutron topography is poor. It was estimated to be no better than 60 μm in non-polarized work on the instrument installed at ILL Grenoble, for exposure times of hours, as a result of roughly equal contributions from detector resolution, geometric blurring due to beam divergence, and shot noise, *i.e.* fluctuation in the number of diffracted neutrons reaching a pixel. The same reason leads to the technique being instrumentally simple because refinements that might lead, for example, to better resolution are discouraged by the increase in exposure time they would imply. Typically, a neutron beam with divergence of the order of 10′ is monochromated by a non-perfect crystal (mosaic spread a few minutes of arc), and the monochromatic beam illuminates the sample, which can be either a single crystal or a grain in a polycrystal. It is advantageous, but not mandatory, to use a white beam delivered by a curved neutron guide tube as the divergence is already limited and high-energy parts of the spectrum, which would contribute to unwanted background, as well as γ-rays, are eliminated. After the specimen is set for a chosen Bragg reflexion with the help of a detector and counter, a neutron-sensitive photographic detector (see §7.3.1.2.3) is placed across the diffracted beam, as near the sample as possible to minimize geometric blurring effects while avoiding the direct transmitted beam. Very crude but comparatively fast exposures can be made with Polaroid film and an isotopically enriched ^{6}LiF (ZnS) phosphor screen. Better topographs are obtained with X-ray film associated with a gadolinium foil (if possible isotopically enriched in ^{157}Gd) acting as an $n \rightarrow \beta$ converter, or with a track-etch plastic foil with an ^{6}LiF or $^{10}B_4C$ foil or layer ($n \rightarrow \alpha$ converter) (Malgrange, Petroff, Sauvage, Zarka & Englander, 1976). Alternatively, an electronic position-sensitive neutron detector can be used for both setting and imaging (Davidson & Case, 1976; Sillou *et al.*, 1989).

Polarized neutrons are extremely useful in the investigation of magnetic domains. The use of a polarizing monochromator and a crude attachment providing a guide field and the possibility to flip the polarization can provide this possibility as an option because the requirements are much less stringent than in quantitative structural polarized-neutron-diffraction work.

It is also possible to use the white beam from a curved guide tube directly (Boeuf, Lagomarsino, Rustichelli, Baruchel & Schlenker, 1975), in the same way as in synchrotron-radiation X-ray topography, that is to say making a Laue diagram, each spot of which is a topograph. The technique is then instrumentally extremely simple, but background is a problem. Because the beam divergence is so much larger than for synchrotron radiation, the resolution is much worse than in the latter case, but it is not expected to differ significantly from the monochromatic beam neutron version.

The ability of neutron beams to go through furnaces or cooling devices, one of the advantages in neutron diffraction work in general, is of course retained in topography. It is, however, desirable to retain a small ($\sim$<2 cm) specimen-to-film distance.

2.8.3. Application to investigations of heavy crystals

As indicated in (*c*) in Section 2.8.1, the absorption of neutrons by most materials is very small. As a result, it is possible to investigate the defect distribution in samples that are too large and/or contain elements too heavy to be suitable for X-ray topography. Another interesting potentiality is the investigation of crystals where X-rays induce a reaction, for example polymerization (Dudley, Baruchel & Sherwood, 1990). While there is no problem except for the resolution and exposure time with thin crystals of heavy materials (Baruchel, Schlenker, Zarka & Petroff, 1978), the observation of large crystals by the standard, wide-beam technique that corresponds to standard topography implies a superposition of the contributions of sizeable portions of the crystal. It is therefore convenient to restrict the observation to a virtual slice, exactly as in Lang's method of X-ray section topography in low-absorption cases (Schlenker, Baruchel, Perrier de la Bathie & Wilson, 1975; Davidson & Case, 1976). This method is useful in particular in the process of preparation of monochromator crystals (Hustache, 1979). It has been applied in metallographic investigations of large crystals of copper-based alloys (Tomimitsu, Doi & Kamada, 1983).

2.8.4. Investigation of magnetic domains and magnetic phase transitions

The strong contribution of electronic magnetic moments in materials to neutron diffraction [item (*d*) in Section 2.8.1] makes magnetic neutron scattering a unique tool in determining magnetic structures on the unit-cell level. When a single-crystal specimen contains regions with different magnetic structures, *i.e.* magnetic domains or coexisting phases with different magnetic structures, they will be imaged because of the local variations in structure amplitude, and hence in diffracted intensity, they entail. Ferromagnetic domains can be imaged by neutron topography (Schlenker & Shull, 1973). This is of restricted value because so many other and more efficient techniques are available, albeit not for observations in the bulk. But neutron topography is the only method available to visualize antiferromagnetic domains of various kinds. The pioneering work by Ando & Hosoya (1972, 1978) and Davidson, Werner &

Arrott (1974) showed spin-density wave domains in antiferromagnetic chromium. Chirality domains (right/left-handed helix in helimagnets), as well as 180° antiferromagnetic domains, could also be observed for the first time using polarized neutron topography (Baruchel, Schlenker & Palmer, 1990). Neutron topography is also a valuable tool in the investigation of the coexistence of different magnetic phases, for example heli- and ferromagnetic, at a first-order phase transition, which can be driven either by temperature changes or by applying a magnetic field (Baruchel, 1989).

2.7 (*cont.*)

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