

## 2. DIFFRACTION GEOMETRY AND ITS PRACTICAL REALIZATION

sample is detectable by neutron low- $Q$  scattering. The neutron spins need not be oriented themselves, although important contributions can be expected from measuring the difference between the scattering of neutron beams with opposite spin orientation. At present, several low- $Q$  instruments are being planned or even built including neutron polarization and polarization analysis.

Studies of magnetic SANS without (and rarely with) neutron polarization include dislocations in magnetic crystals and amorphous ferromagnets [see the review of Kostorz (1988)].

Janot & George (1985) have pointed out that it is important to apply contrast variation for suppressing surface-roughness scattering and/or volume scattering in order to isolate magnetic scattering contributions by matching the scattering-length density of the material with that of a mixture of heavy and light water or oil, *etc.*

## 2.6.2.3.1. Spin-contrast variation

For a long time, the magnetic properties of the neutron have been neglected as far as 'nonmagnetic' matter is concerned. Spin-contrast variation, proposed by Stuhrmann (Stuhrmann *et al.*, 1986; Knop *et al.*, 1986), takes advantage of the different scattering lengths of the hydrogen atoms in its spin-up and spin-down states. Normally, these two states are mixed, and the cross section of unpolarized neutrons with the undirected spins gives rise to the usual value of the scattering amplitude of hydrogen. If, however, one is able to orient the spins of a given atom, and especially hydrogen, then the interaction of *polarized* neutrons with the two different oriented states offers an important contribution to the scattering amplitude:

$$A = b + 2BI \cdot s, \quad (2.6.2.5)$$

where  $b$  is the isotropic nuclear scattering amplitude,  $B$  is the spin-dependent scattering amplitude,  $s$  is the neutron spin, and  $I$  the nuclear spin. For hydrogen,  $b = -0.374 \times 10^{-12}$  cm,  $B = 2.9 \times 10^{-12}$  cm.

The sample protons are polarized at very low temperatures (order of mK) and high magnetic fields (several tesla) by dynamic nuclear polarization, *i.e.* by spin-spin coupling with the electron spins of a paramagnetic metallo-organic compound present in the sample, which are polarized by a resonant microwave frequency. It is clear that the principles mentioned above also apply to other than biological and chemical material.

## 2.6.2.4. Long wavelengths

An important aspect of neutron scattering is the ease of using long wavelengths: Long-wavelength X-rays are produced efficiently only by synchrotrons, and therefore their cost is similar to that of neutrons. Unlike neutrons, however, they suffer from their strong interaction with matter. This disadvantage, which is acceptable with the commonly used Cu  $K\alpha$  radiation, is in most cases prohibitive for wavelengths of the order of 1 nm.

Very low  $Q$  values are more easily obtained with long wavelengths than with very small angles, as is necessary with X-rays, since the same  $Q$  value can be observed further away from the direct beam. Objects of linear dimensions of several 100 nm, *e.g.* opals, where spherical particles of amorphous silica form a close-packed lattice with cell dimensions of up to several hundreds of nm, can still be investigated easily with neutrons. X-ray double-crystal diffractometers (Bonse & Hart, 1966), which may also reach very low  $Q$ , are subject to transmission problems, and neutron DCD's again perform better.

## 2.6.2.5. Sample environment

Important new fields of low- $Q$  scattering, such as dynamic studies of polymers in a shear gradient and time-resolved studies of samples under periodic stress or under high pressure, have become accessible by neutron scattering because the weak interaction of neutrons with (homogeneous) matter permits the use of relatively thick (several mm) sample container walls, for example of cryostats, Couette-type shearing apparatus (Lindner & Oberthür, 1985, 1988), and ovens. Air scattering is not prohibitive, and easy-to-handle standard quartz cells serve as sample containers rather than very thin ones with mica windows in the case of X-rays.

Unlike with X-rays, samples can be relatively thick, and nevertheless be studied to low  $Q$  values. This is particularly evident for metals, where X-rays are usually restricted to thin foils, but neutrons can easily accept samples 1–10 mm thick.

## 2.6.2.6. Incoherent scattering

Incoherent scattering is produced by the interaction of neutrons with nuclei that are not in a fixed phase relation with that of other nuclei. It arises, for example, when molecules do not all contain the same isotope of an element (isotopic incoherent scattering). The most important source of incoherent scattering in SANS, however, is the spin-incoherent scattering from protons. It results from the fact that only protons and neutrons with identical spin directions can form an intermediate compound nucleus. The statistical probabilities of the parallel and antiparallel spin orientations, the similarity in size of the scattering lengths for spin up and spin down and their opposite sign result in an extremely large incoherent scattering cross section for  $^1\text{H}$ , together with a coherent cross section of normal magnitude (but negative sign). Incoherent scattering contributes a background that can be by orders of magnitude more important than the coherent signal, especially at larger  $Q$ . On the other hand, it can be used for the calibration of the incoming intensity and of the detector efficiency (see below).

## 2.6.2.6.1. Absolute scaling

Wignall & Bates (1987) compare many different methods of absolute calibration of SANS data. Since the scattering from a thin water sample is frequently already being used for correcting the detector response [see §2.6.2.6.2], there is an evident advantage for performing the absolute calibration by  $\text{H}_2\text{O}$  scattering.

For a purely isotropic scatterer, the intensity scattered into a detector element of surface  $\Delta A$  spanning a solid angle  $\delta\Omega = \Delta A/4\pi L^2$  can be expressed as

$$\Delta I = I_0(1 - T_i)\delta\Omega g/4\pi, \quad (2.6.2.6)$$

with  $T_i$  the transmission of the isotropic scatterer, *i.e.* the relation of the number of neutrons in the primary beam measured within a time interval  $\Delta t$  after having passed through the sample,  $I_T$ , and the number of neutrons  $I_0$  observed within  $\Delta t$  without the sample. In practice,  $T_i$  is measured with an attenuated beam; typical attenuation factors are about 100 to 1000.  $g$  is a geometrical factor taking into account the sample surface and the solid angle subtended by the apparent source, *i.e.* the cross section of the neutron guide exit.

Vanadium is an incoherent scatterer frequently used for absolute scaling. Its scattering cross section, however, is more than an order of magnitude lower than that of protons. Moreover, the surface of vanadium samples has to be handled with much care in order to avoid important contributions from