

Short Communications

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Neutron diffraction from a smectic *A* monodomain. By H. HERVET, *Collège de France, Laboratoire de Physique de la Matière Condensée, Paris, France*, S. LAGOMARSINO and F. RUSTICHELLI, *Institut Max von Laue-Paul Langevin, Grenoble, France*, and Physics Division, J.R.C. EURATOM, *Ispra, Italy* and F. VOLINO, *Institut Max von Laue-Paul Langevin, Grenoble, France*

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It is shown by neutron diffraction that a relatively weak magnetic field (1.75 kG), is able to induce an order in a large terephthal-bis-(4*n*-butylaniline) (TBBA) sample in the smectic *A* phase. A monodomain is obtained with the planes perpendicular to the magnetic field. Rocking curves are performed whose full widths at half maximum give a value of a few degrees for the misorientations of the planes.

The present paper reports some results on a neutron diffraction experiment on a large sample of TBBA (Taylor, Arora & Ferguson, 1970) in the smectic *A* phase. The aim was to check if, by cooling a liquid crystal from the nematic phase to the smectic *A* phase under a relatively weak magnetic field (~ 1.7 kG), it was possible to obtain a good alignment of the planes of the smectic *A* phase, even in a large, flat sample.

We used the partially deuterated derivative of TBBA, previously called DTBBA (Hervet, Volino, Dianoux & Lechner, 1974, 1975; Volino, Dianoux, Lechner & Hervet, 1975), placed between two 25 mm diameter Al plates separated by 0.8 mm, heated by a surrounding wire, yielding a temperature homogeneity and stability better than 1°C. The experiment was performed on the multi-detector powder diffractometer DIB (*ILL Neutron Beam Facilities at the NFR*, 1974) installed at the Institut Laue-Langevin at Grenoble. The wavelength was 2.4 Å, the monochromator being a pyrolytic graphite crystal. The second-order contamination was eliminated by a graphite filter. The divergence of the neutron beam at the monochromator was that of the neutron guide, *i.e.* for this wavelength, nearly 28°. The diffraction patterns were corrected for detector efficiency and sample-holder contribution and normalized to the same number of incident neutrons. Fig. 1(a) shows the diffraction pattern of the non-aligned sample in the smectic *A* phase at 180°C. At small angles, the two peaks on the left and right sides of the zero position (incident beam) are the intersections of a Debye-Scherrer cone with the multidetector circle. These two peaks correspond to an interplanar spacing of the smectic planes of $d = 27.6 \pm 1.1$ Å, and the weak broad peak at large angle ($\sim 30^\circ$) corresponds to an intermolecular distance in the smectic planes of ~ 4.5 Å. The results are in agreement with X-ray data (Doucet, 1972).

The sample was then heated to 209°C in the nematic phase and placed in a vertical magnetic field of ~ 1.75 kG, produced by a permanent magnet, for about 20 min. The sample was then cooled to 180°C in the smectic *A* phase under the magnetic field, which was removed, and the diffraction pattern shown in Fig. 1(b) recorded. The low-angle peaks have disappeared. The sample was then turned by 90° around the incident beam direction and the dif-

fraction pattern shown in Fig. 1(c) was obtained. The two low-angle peaks are now strongly enhanced with respect to those of Fig. 1(a), indicating that a certain order has been induced in the sample by the magnetic field. These results can be interpreted by assuming an alignment of the molecules in the direction of the field ($\chi_a > 0$) since in Fig. 1(b) the lattice vector of the smectic layer is perpendicular to the scattering plane (zero intensity), and in Fig. 1(c) it lies in the scattering plane (enhanced intensity). However, the simultaneous existence of the two peaks in Fig. 1(c) shows that a 'misalignment' of the smectic layers equal to at least twice the Bragg angle exists. In the geometry of Fig. 1(c), the sample was then rotated about a vertical axis by several values of the rotation angle ω . Fig. 2 shows the maximum intensities of the left and right low-angle peaks

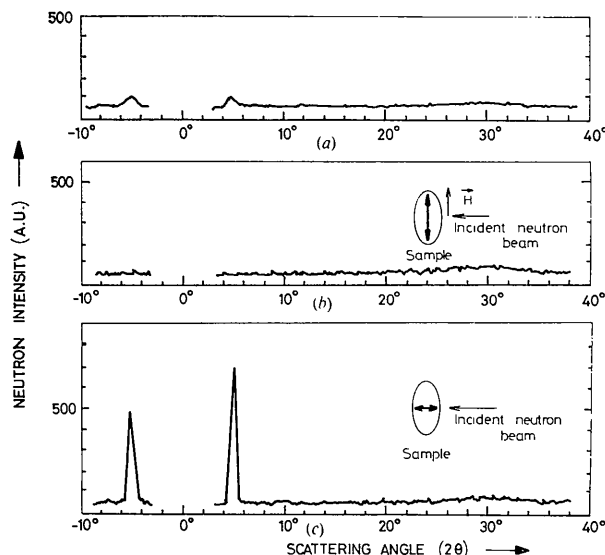


Fig. 1. Neutron diffraction patterns of smectic *A* TBBA ($T = 180^\circ\text{C}$). (a) Non-aligned sample. (b) After application of the magnetic field. (c) After application of the magnetic field. The sample is turned by 90° round the direction of the neutron incident beam.

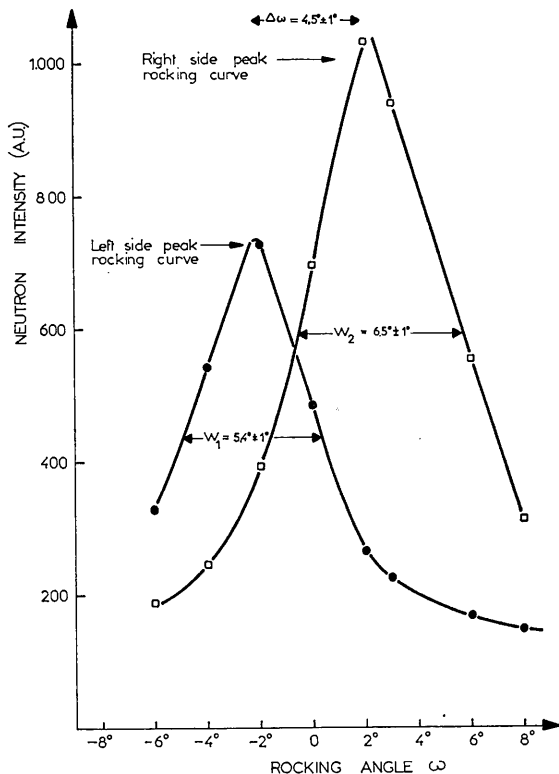


Fig. 2. Intensities of the left and right low-angle peaks *vs.* the rotation angle ω of the sample around a vertical axis. The rocking curves refer to the geometry of Fig. 1(c).

versus the 'rocking angle' ω . The two curves are quite regular and similar to the rocking curves obtained with solid mosaic crystals. The full widths at half maximum are $5.4 \pm 1^\circ$ and $6.5 \pm 1^\circ$ for the left- and right-peak rocking curves respectively, and give a quantitative estimate of the order induced. These values are of the same order of magnitude of those observed in smectic *B* monodomains (Levelut, Doucet & Lambert, 1974). The different heights of the two rocking curves are due to imperfect positioning of the sample with respect to the monochromator and the multidetector, which is irrelevant for the kind of information obtained.

We have talked here of misalignment of the smectic *A* planes instead of mosaicity, since mosaic texture only appears in the smectic *B* phase, whereas in smectic *A* phases, focal conic textures are generally involved (Friedel, 1922; Bouligand, 1972). Another point to be discussed is

the influence of the container walls. A competition may exist between the alignment effects of the container walls and the external magnetic field. This effect has been investigated theoretically by Bidaux, Boccara, Sarma, de Seze, de Gennes & Parodi (1973) who assumed that the walls tend to align the molecules perpendicular both to themselves and to the magnetic field in limited regions near the walls. In our case, we can expect that the fraction of the volume of such perturbed regions to the whole sample volume is given by the ratio of the intensities of the low-angle peaks of Figs. 1(b) and 1(c). If it is assumed that the height of the low-angle peaks in Fig. 1(b) is given by the statistical error, this fraction is smaller or equal to 4%, corresponding to a penetration depth L of $33 \mu\text{m}$. With the above-mentioned theory (Bidaux *et al.*, 1973), a value of L can be estimated from

$$L = 2 \left(\frac{K}{\chi_a} \right)^{1/2} \frac{1}{H},$$

where K is the Frank constant, χ_a the diamagnetic anisotropy and H the external field. With $K \sim 10^{-6}$ c.g.s., $\chi_a \sim 10^{-7}$ c.g.s. and $H = 1.7$ kG, we get $L \sim 35 \mu\text{m}$ in agreement with the above value (exact values of K and χ_a for TBBA have not yet been published). An evident interest of the present results is the possibility of performing inelastic and elastic neutron scattering experiments in large oriented samples.

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