ESRF

Experiment title:

COMMENSURABILITY OF THE TWIST GRAIN BOUNDARY SMECTIC C PHASE (TGBC).

Experiment number:

SC 132

Beamline: BL1 / ID13

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Report:

The microfocus beamline was fitted with the 30~mcollimator and the CCD camera. The selected energy was 13 keV. The temperature controller (from CRPP) was mounted on a theta-X-Z stage. The liquid crystal sample was a 25 pm film oriented by treatment of the glass surfaces. Total thickness of the glass was 50~m. Temperature of the sample was manually set and controlled by the two stage oven (+lOmK). Excellent alignment of the liquid crystal was checked at ESRF with apolarizing microscope.

The liquid crystalline phase of interest was the Smectic C Twist Grain Boundary phase (TGBC). The polymorphism was Cholesteric-(103.2 °C)-TGBC-(102.2)-Smectic C* on cooling. The reciprocal structures of the TGBC phase is recalled on fig. 1.

The helical axis of the TGBC was first set parallel to the x ray beam with the help of a laser. The theta angle was then rotated by 13 degrees to bring a pair of reciprocal vectors as close as possible to the Ewalds sphere on the horizontal plane. The expected scattering pattern (simulated before the experiment) was a set of Bragg spots, distributed on a ring, with maximum intensity on the horizontal diameter.

Three main pieces of information were extracted from experiment SC 132:

•1• Resistance of a liquid crystal sample to a very strong x rav flux:

We first investigated the effect of exposure time on the diffraction patterns. For 0. 1s exposure, a typical TGBC diffraction pattern was observed (fig. 2). For exposures longer than 0.2s, the diffraction pattern was clearly affected by the beam until it was totally destroyed above 1s. The diffraction was then similar to the diffuse ring of the cholesteric mesophase. The TGBC pattern could not however be recovered at lower temperature. Chemical damage is suspected.

Better statistics could be obtained by splitting longer exposures into 0.1s shots (with 60s interval between the shots). No visible damage was then detected up to ten shots (1s cumulated exposure). We tried to write “macro” commands to pilot exposures this way but we did not succeed (this will be useful for future experiments). Note that TGB’s are certainly among the most “fragile” soft materials: they are very sensitive to impurities and their complex structural parameter change every 200mK or so! The nice conclusion is that the rnicrofocus beamline can be used successfully even with such fragile samples.

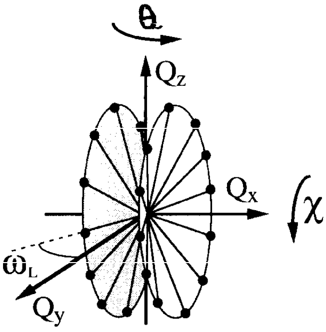

Figure 1

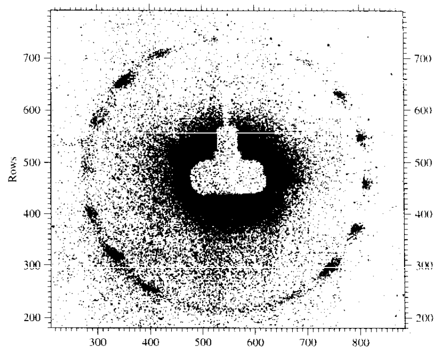

Figure 2

• 2 • Alignment of the helical axis:

We performed a series of weak theta rotations about the estimated Bragg position (by steps of 0.4 degree) to optimize the incident angle. We found that the diffraction patterns changed with theta in a way which is consistent with the simulations of the intersection of Ewald's sphere with a perfect single TGBC crystal: the maxima of scattering for instance are not symmetrical on the two branches of the image. This simple effect, due to the curvature of the Ewald’s sphere, shows that the crystallographic axes of the soft mesophase are defined within less than the Bragg angle (0.67 degrees). We thus conclude that the distortions of the helical axis of the TGBC phase (splay or bend) are remarkably weak across the sample.

.3 • Commensurabilitv of the TGBC phase:

Two types of TGBC diffraction patterns are expected: incommensurate (uniform ring of diffraction) or commensurate (a finite number of Bragg spots are visible). The spatial distribution of the commensurate zones was the main goal of this experiment. This objective has been reached successfully: almost all the diffraction patterns recorded all over the sample area look commensurate. In order to get this information, we moved the sample in the XZ plane perpendicular to the beam by steps of 60~m (0.6 x 0.6mm grid) and 200~m (4 x 3mm grid). More than 400 images were recorded. This tremendous amount of data has been our main problem at CRPP ! Format conversion and data storage have been solved only recently. The study of spatial variations of the orientation of the crystallographic axes is still in progress.

Anyway, the main result is that the TGBC sample is truly commensurate. The next step is to ask what is responsible for this commensurability? Is it a surface effect or a bulk property? Our suggestion is to work on a wedged sample to study the influence of thickness on commensurability.

In conclusion, we believe that experiment SC 132 has been a real success: the question of the commensurability of the TGBC mesophase is now unambiguously answered. Note that this result justifies the use of a wider beam to study TGBC samples of uniform thickness. In addition, two more pieces of information have been obtained: the resistance of a soft liquid crystal to the beam and its excellent (or rigid) orientation accross the thickness of the film. A publication on this work is currently in preparation. At last, it is a pleasure to acknowledge the great assistance of the ESRF staff on beamline BL 1.