ESRF	**Experiment title:** COMMENSURABILITY OF THE TWIST GRAIN BOUNDARY SMECTIC C PHASE (TGBc) IN A WEDGE	**Experiment number:** SC-280
Beamline: ID 13	**Date of Experiment:** from: 07 / 03 / 97 to: 10 / 03 / 97	**Date of Report:** 25 / 07 97
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Report:

The SC280 experiment on the microfocus beamline ID1 3 continued an investigation, started in 1996 under proposal SC132, to understand the commensurate nature (or N-fold helical symmetry) of the liquid crystalline Twist Grain Boundary smectic C phase (TGBc).

SC132 had given promising results unambiguously eliminating the possibility of coexisting commensurate and incommensurate regions. The signal to noise ratio was however of marginal quality. The main reason was that the liquid crystal film was modified for exposures to the intense microfocussed beam longer than 0.1 seconds.

The latest TGBc experiment was improved in three substantial ways over SC 132:

* (i) a more sensitive CCD camera with an image intensifier was used.
* (ii) long effective exposures were produced by summing 10 to 20 images of 0.13 secs. each separated by 60 sec. intervals.
* (iii) the liquid crystal sample was aligned in a wedged cell allowing a study of the evolution of the diffraction patterns with sample thickness.

The combination of points (i) and (ii) were crucial to significantly improving the signal to noise ratio of the experiment. We checked that 0.13 sec. exposures were safe (damage occurred for exposures longer than 0.4 sec.) and that all 10 or 20 images recorded in a row were identical provided the interval between two exposures was > = 60 sec. This relatively long time corresponds to the thermal diffusion time required to dissipate the heat produced by beam absorption in the

glass walls and liquid crystal film. The 10 summed images produced an effective 1.3 sec. exposure time with a reasonably low noise. The ID13 scientific staff provided invaluable help to synchronize the CCD camera and fast shutter and to develop software for on-line summation of the images.

The wedged cell, mentioned in point (iii), was new to SC280. The idea was to change continuously the balance between surface and volume effects (i.e. alignment constraint versus intrinsic TGB properties) by changing the sample thickness using a wedged cell. The 0.36 deg wedge was prepared from two $150\mu m$ thick glass plates. Reasonable quality diffraction patterns were recorded in regions as thin as $4\ \mu m$.

A monochromatic, 13KeV, beam was focused using the standard ID13 beamline optics to a 20 x 40 μm^2 spot. A temperature-controlled sample chamber (from CRPP) was mounted on a theta-X-Z stage. The temperature was manually set and controlled to an accuracy of 0.1 deg K.

The expected scattering pattern was a set of Bragg spots, distributed on a ring, with maximum intensity on the horizontal diameter. Diffraction patterns were recorded along a line of increasing thickness, D, of the wedge cell in 40 μm increments.

The main result of experiment SC280 was the evolution in diffraction patterns as the thickness of the wedge cell was increased. The observed patterns can be divided into three distinct groups:

* 1 * Thin region of the wedge: thickness D < 12 urn.

The TGBC phase is comprised of discrete blocks of smectic-C layers (- 500-700 Å wide) which are rotated with respect to each other by an angle $2\pi/N$. In the thin region of the wedge cell, Bragg spots corresponding to the individual smectic blocks were observed for the first time. They appear in groups of 2, 3 and 4 spots upon increasing cell thickness corresponding to regions with 2, 3 and 4 helical pitches respectively separated by Grandjean-Cano dislocation lines. Spots arising from pitch n and n+l do not superimpose, which is the signature of an incommensurate TGB structure.

* 2 * Intermediate region: $12\ \mu m < D < 18\ \mu m$.

The Bragg spots arising from individual smectic blocks are too many to distinguish any regularity. Diffraction patterns still look incommensurate.

* 3 * Thicker region: $18\ \mu m < D < 30\ \mu m$.

The scattered intensity merges into a small number of equispaced broad spots. The diffraction patterns now appear to be periodic around the ring of scattering i.e. commensurate. This periodicity is evidenced by the existence of a strong Fourier component of the intensity around the ring at a wavenumber $2\pi/N$ (N - 20). Defining the degree of commensurability as the intensity of this Fourier component normalized to the integrated intensity, the commensurability increases with thickness which suggests that its origin is intrinsic rather than induced by the confinement.

Conclusion: the two experiments run at the ESRF on the microfocus beamline, SC132 and SC280, have provided strong evidence for the commensurability of TGBC samples stimulating renewed theoretical research to understand the origin of this commensurate lock-in.

Perspective: two experiments suggested by our results include:

-l- repeat the wedge cell experiment with a TGBA material, which is known to be always incommensurate, to compare behavior.

-2- distort and unwind the TGBC helix with an external transverse electric field. Preliminary results have already been obtained using a rotating anode x-ray source, but the higher spatial resolution provided by the synchrotron is required to understand the results in quantitative detail.