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## X-Ray Study of the Layer Structure in a High Pre-Tilt, Anti-Parallel Aligned Ferroelectric Liquid Crystal

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X-ray diffraction has been used to investigate the layer structure of a ferroelectric liquid crystal device as a function of temperature. Obliquely evaporated silicon monoxide alignment layers arranged anti-parallel to each other were used to align a 2  $\mu\text{m}$  sample of racemic SCE13. In the smectic A phase, the cell formed a uniform tilted layer with a tilt angle in good agreement with the 25° pre-tilt of the SiO. Below the transition to Smectic C there was evidence for a highly asymmetric chevron with layers oriented away from the preferred alignment plane, together with domains of uniformly tilted layers. Further cooling to 30°C caused the chevron structure to disappear, until only the uniformly tilted layer remained.

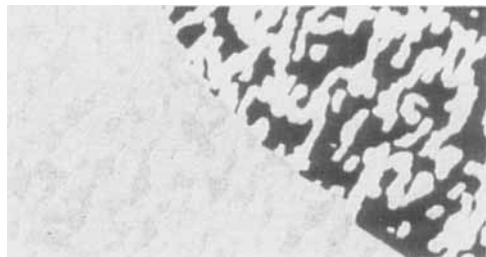
**Keywords:** X-rays; layer structure; smectic C; ferroelectric liquid crystal

### INTRODUCTION

To improve and develop ferroelectric liquid crystal display devices, it is important to understand their detailed structure in a variety of sample geometries. X-ray diffraction studies enable the direct measurement of the distribution of the smectic layers within a device. It has been shown<sup>[1,2]</sup> that, in a thin cell, the smectic C\* (SmC\*) layers tend to form a chevron structure and not the bookshelf geometry generally observed for the smectic A (SmA) phase. Further studies<sup>[3-6]</sup> have suggested that the formation of the chevron structure was due to the layer thinning associated with the director tilting from the layer normal below the SmA to SmC\* transition. Subsequent investigations<sup>[7-10]</sup> suggested that chevron formation may also occur in the SmA phase for some materials where the layer spacing decreases on cooling,

such as the commercial mixture SCE13<sup>[10]</sup>.

In the present work a detailed analysis of the structure of a high pre-tilt anti-parallel cell as a function of temperature is made. Previous work by Rieker *et al.*<sup>[11]</sup> showed that such samples form uniformly tilted layer (UTL) structures, rather than the chevron geometry. Such a geometry would be useful to measure physical properties of the SmC phase, including the three biaxial permittivities and the elastic constants. However, optical microscopy indicates that there is a different structure closer to the SmA to SmC(\*) transition, as shown in figure 1. In addition to the uniform domains associated with the UTL structure are areas of a



**Figure 1: Optical texture of a 3μm SCE13 sample with antiparallel aligned high tilt SiO at 55°C.**

“sandy” texture, where optical extinction requires

non-crossed polarisers to be used. This observation is indicative of an asymmetric director profile, wherein there is no mirror symmetry about the centre plane of the cell. In this paper we elucidate the nature of these domains.

## EXPERIMENTAL

The liquid crystal studied was the racemic version of the commercial mixture SCE13, with the phase sequence:

$$\text{SmC}^* \leftrightarrow 60.8 \leftrightarrow \text{SmA} \leftrightarrow 86.3 \leftrightarrow \text{N}^* \leftrightarrow 100.8 \leftrightarrow \text{I} \quad / ^\circ\text{C}$$

The material was contained in specially constructed glass cells with 100μm thick walls spaced at approximately 2μm. Alignment was provided by 5° evaporated SiO giving a pre-tilt of approximately 25°.

The experimental X-ray scattering geometry has been described previously<sup>[10]</sup>. All the rocking curve data were collected at the Daresbury

Laboratory using Stations 8.2 & 2.1, which had beam dimensions of 2mm horizontally by 0.8mm vertically and the wavelength of radiation used was 1.5Å. The intensity of the Bragg reflection from the layers was measured using a two dimensional position sensitive detector as a function of the sample tilt  $\phi$ , and azimuthal  $A$  angles, measured with respect to the incident beam as shown in figure 2a). Measurements were made on cooling from the N\* phase. At each temperature, the sample tilt angle was scanned from  $-50^\circ$  to  $50^\circ$ , taking approximately 20 minutes. A plot of the layer normal distribution can be made by integrating the intensity of the Bragg reflection as a function of  $\phi$  and  $A$  and then correcting the angles for the curvature of the Ewald sphere<sup>[13]</sup>. The relationship between the sample and laboratory reference frames is given by:

$$\sin \delta = \sin \theta_B \cos \phi - \cos \theta_B \sin \phi \sin A \quad (1)$$

$$\tan \gamma = \frac{\tan \theta_B \sin \phi}{\cos A} + \cos \phi \tan(A - A_0) \quad (2)$$

where  $\delta$  is the tilt of the smectic layer normal from the cell plane,  $\gamma$  the in plane layer orientation with respect to the preferred alignment (i.e. evaporation) direction  $A_0$  and  $\theta_B$  is the Bragg angle.

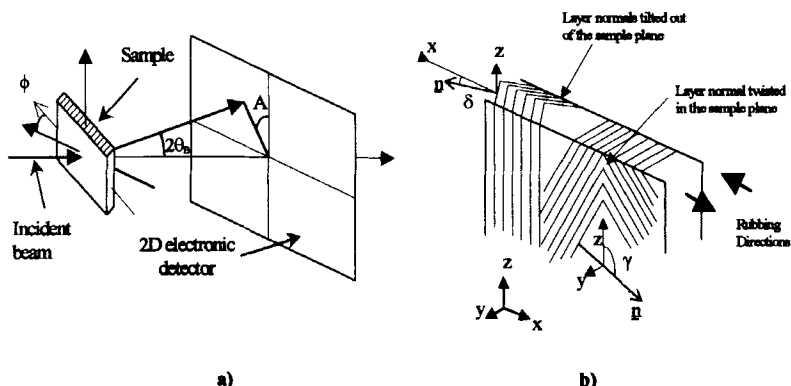
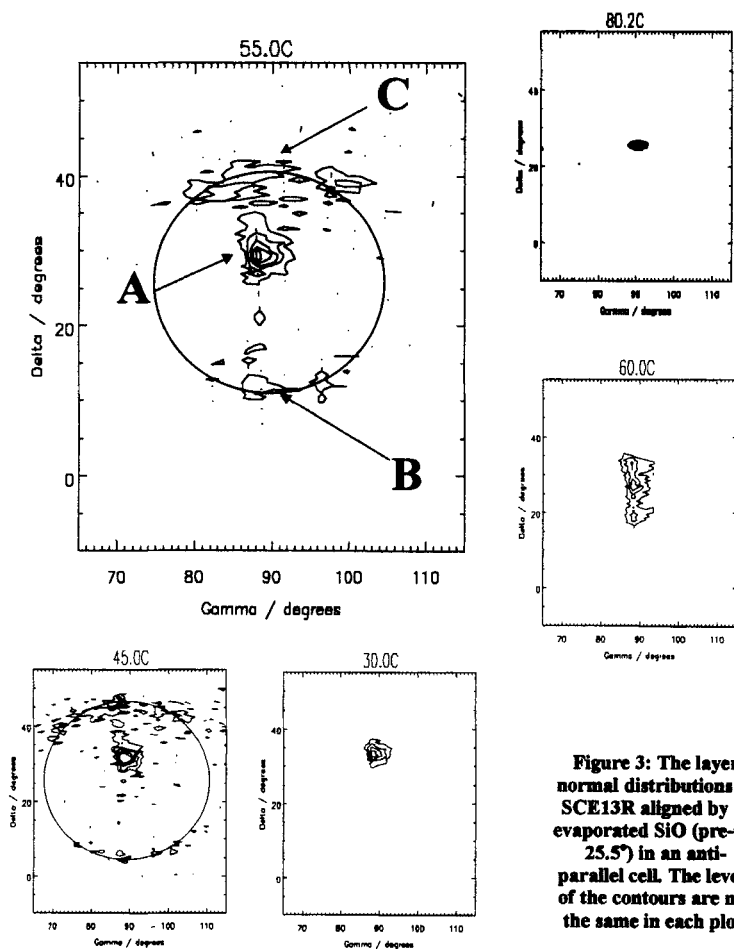


Figure 2: Schematic diagrams of a) the diffractometer frame of reference and b) the definition of the layer tilt and in plane orientation angles  $\delta$  and  $\gamma$ .

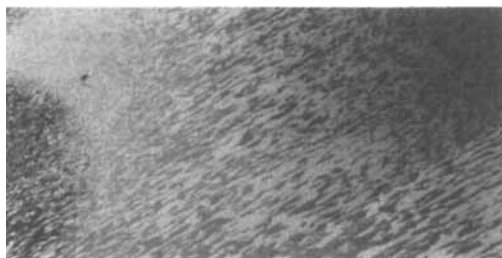
## RESULTS

Figure 3 shows the layer normal distributions of the SCE13 cell as a function of temperature. It is clear that there is a marked change in layer behaviour below the SmA→SmC\* phase transition (60.8°C). In the SmA phase, the layers are in a uniform tilted layer (UTL) structure tilted at the pre-tilt angle,  $\theta_p$  = 25.5°. Any change of the layer tilt angle that occurs on cooling through the



**Figure 3:** The layer normal distributions of SCE13R aligned by 5° evaporated SiO (pre-tilt 25.5°) in an anti-parallel cell. The levels of the contours are not the same in each plot.

transition temperature, there are two different contributions to the layer normal distribution. The major contribution is a single broad peak located at  $\gamma = 90^\circ$ ,  $\delta = 30.2^\circ$  at  $55^\circ\text{C}$  (labelled A in figure 3). It corresponds to layers that are tilted slightly more than in the SmA, the tilt angle  $\delta$  increases from  $25.5^\circ$  to  $34.8^\circ$  as the sample is cooled from  $65^\circ\text{C}$  to  $30^\circ\text{C}$ . The second contribution consists of two arcs, B and C, on a circle that remains centred about the position of the SmA peak throughout the SmC phase. As the sample cooled, this contribution weakens whilst spreading to higher in-plane “twist” angles of the smectic layers. It had disappeared completely before  $30^\circ\text{C}$  was reached, and the sample



**Figure 4: Photomicrograph of the X-ray cell at  $30^\circ\text{C}$ , immediately after the measurements of figure 3 were taken.**

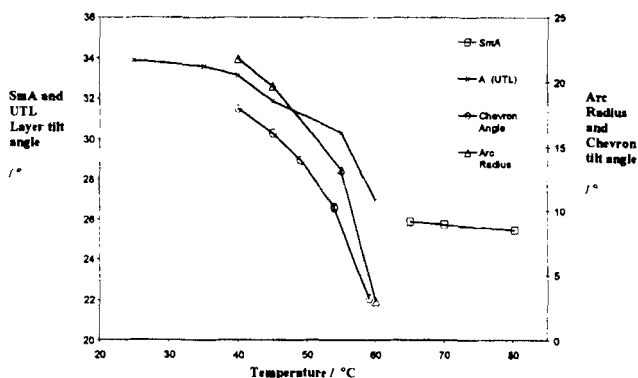
consisted of the UTL structure with domains that extinguish light at  $\pm 30^\circ$  to the evaporation direction when viewed between crossed polarisers, figure 4. This suggests that the arcs of B and C correspond to

the sandy texture.

The temperature dependence of the layer orientation is shown in figure 5, together with the SCE13 chevron layer tilt angle measured in a low pre-tilt parallel aligned cell. The radius of the arcs B and C increases on cooling and is slightly greater than the chevron angle as shown in figure 5.

## DISCUSSION

To understand the changes of layer orientation during cooling it is important to consider the shrinking of the smectic layers together with the constraints imposed by the surface of the cell. Clark *et al* <sup>[1, 2]</sup> argue that the layers are



**Figure 5:** Summary of the temperature dependence of the X-ray diffraction data. The left-hand vertical axis shows the layer tilt of contribution A and the smectic A UTL, whereas the right hand axis shows the radius of the circle on which arcs B & C lie and the X-Ray chevron layer tilt measured using a low tilt parallel cell.

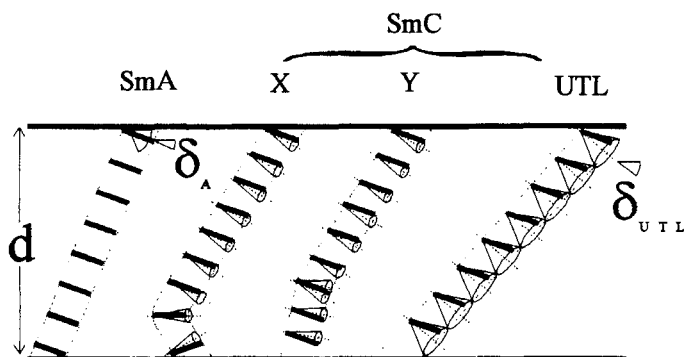
effectively pinned at both surfaces, leading to the symmetric chevron structure as the layers shrink on cooling from the smectic A phase with bookshelf layers. In the anti-parallel cell with high pre-tilt the situation is far more complicated. For the smectic layers to remain pinned at the surface, the resulting chevron must be highly asymmetric, as shown by structure X in figure 6. Continuity of the director at the chevron interface requires a significant bend of the smectic layers and corresponding increase of the smectic C cone angle. Such changes are only possible close to the phase transition, below which the layer pinning is overcome and the uniform tilted structure forms. Packing of the layers then leads to a layer tilt given by <sup>[12]</sup>:

$$\cos \delta_{\text{utl}} = \cos \delta_{\text{chevron}} \cdot \cos \theta_p \quad (3)$$

The UTL results of figure 5 (region A from figure 3) fit equation (3) to within experimental uncertainty.

The occurrence of the arcs B and C at high temperatures, however, cannot be explained using these arguments, since the layers are on a locus centred about the SmA orientation (rather than  $\delta=0^\circ$ ). These results are better





**Figure 6:** Schematic of the conflict that occurs between layer pinning, surface orientation of the director and continuity of the director at the chevron interface on cooling into the smectic C phase from a uniformly tilted smectic A sample, ( $\delta_A = \theta_p$ ).

explained by structure Y as shown in figure 6. The director remains continuous across the chevron interface, despite the discontinuity of the layers (in a similar fashion to low tilt chevron samples). However, if the layer orientation in the cell plane remains parallel to the evaporation direction ( $\gamma = 90^\circ$ ), the component of the layer spacing intersecting the lower surface will increase, producing stress in the cell. This leads to the observed layer reorientation away from the evaporation direction. With the Y structure, the chevron interface is not parallel to the cell plane, and so layer packing requires that the chevron interface moves from one surface to the other with a pitch given by  $2d / \tan \delta_A$ . As it does so,  $\gamma$  for the two chevron arms varies, and the layer normals form the arc observed in figure 3. This is what gives the texture its sandy appearance.

## CONCLUSION

Two-dimensional plots of the layer normal distribution has proven most useful for determining the evolution of the uniform tilted layer structure of the SmC phase in anti-parallel cells with high pre-tilts. It has enabled the structure of the

“sandy texture” to be clarified. Future studies will investigate the relationship between the magnitude of the surface pre-tilt and the layer structure.

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