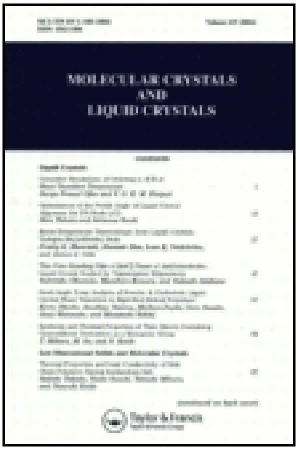

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An X-ray Study of Devices Containing a N*???SmC*Liquid Crystal Mixture

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AN X-RAY STUDY OF DEVICES CONTAINING A $N^* \rightarrow$ SmC* LIQUID CRYSTAL MIXTURE

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The structure of a ferroelectric liquid crystal mixture with an $N^* \rightarrow SmC^*$ transition in skew aligned cells has been studied by X-ray diffraction measurements of the layer normal distribution. The rubbing directions of the cells were set parallel, and skewed at ±9°, ±18° and ±40°. The cell with parallel alignment directions showed two sets of layers with normals twisted in-plane by ∼ ± 20° from the rubbing direction. The layer normal distribution of each set of layers was consistent with a chevron-like structure but with an additional untilted region, probably at the apex. It was also observed that the tilt of the chevron arms was weakly temperature dependent, with their normals tilting out of the plane of the device by an angle of 9° to 12° on cooling. The cells with alignment skewed by ±9° and ±18° also showed chevron-like structures which are probably developed in the middle of the cell with each arms tilted by ∼20° and with normals slightly twisted in-plane by ±4° with respect to the mean rubbing direction. However, there were additional components of the layer-normal distribution, which are believed to be a continuous distribution of layer orientations between the middle of the cell and layers near the substrate with normals twisted further away from the mean rubbing direction. The cell with alignment directions skewed by ±40° showed only a chevron structure with the normals to its two arms twisted with respect to each other by 2° and tilted out of plane by ∼17°.

The authors wish to thank Emma Walton and Martin Tillin from Sharp Laboratories of Europe for the use of their facilities to fabricate the X-rays cells and much helpful guidance. They also wish to thank Gunter Grössman from Daresbury Laboratory for helping us with the diffraction experiments.

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1. INTRODUCTION

The Ferroelectric liquid crystal (FLC) phase was predicted Meyer in 1975 [1] and is found in tilted smectic phases such as the smectic C*. The possibility to make fast displays using the smectic phase was proposed by Clark and Lagerwall [2]. Electro-optical displays made of FLC materials are fabricated by confining the material between substrates separated by a distance less than the pitch of its intrinsic helix. Such a configuration is known as a surface stabilized ferroelectric liquid crystal (SSFLC). Materials with a $N^* \rightarrow SmA^* \rightarrow SmC^*$ phase transition have been the first and most studied materials with this geometry [3]. Patel and Goodby [4] studied the influence of polymer alignment layers and electric field treatment on the orientation of the layers of a FLC material with a $N^* \rightarrow SmC^*$ phase transition in a surface stabilized device. It was shown that when this material is confined between substrates the layers twist with respect to the rubbing direction by an amount similar to the cone angle of the SmC. Furthermore, it was observed that while the buffing direction defined the orientation of the director, an AC electric field could change the direction of the layers. Ouchi *et al.*[5] perform X-ray scattering experiments based on the technique reported by Rieker *et al.*[6]. for a $N^* \rightarrow SmC^*$ material. A chevron structure was formed, demonstrating that this structure is closely related to the molecular tilt of the SmC*.

Patel [7] demonstrated a configuration using a twisted smectic structure. A $N^* \rightarrow SmC^*$ material with a cone angle, θ, of 45° was confined between glass plates with rubbing directions set at 90°. Consequently, the twist induced by the skewed rubbing directions could be accommodated by the rotation of molecules around the cone (without distorting the layers) producing a director configuration and switching similar to a Super Twisted Nematic devices but with the rapid switching response of the smectic phase.

In this paper we present the results of high resolution X-ray scattering from cells filled with a $N^* \rightarrow SmC^*$ material in different cell configurations with parallel and skewed rubbing directions.

2. EXPERIMENTAL

The liquid crystal material is a proprietary material sourced from Clariant with the reference name LZ972. It is characterized by $N^* \rightarrow SmC^*$ phase transition at a nominal temperature of 65°C. The spontaneous polarization was reported [8] to be 3.5 nCcm^{-2} and its pitch varies from roughly 15 μm at 30°C to roughly 25 μm at 40°C and so the SSFLC state is achieved with the 2 μm cells used in this work.

The ferroelectric liquid crystal material was confined between glass substrates whose thickness was only 100 μm to reduce the absorption of

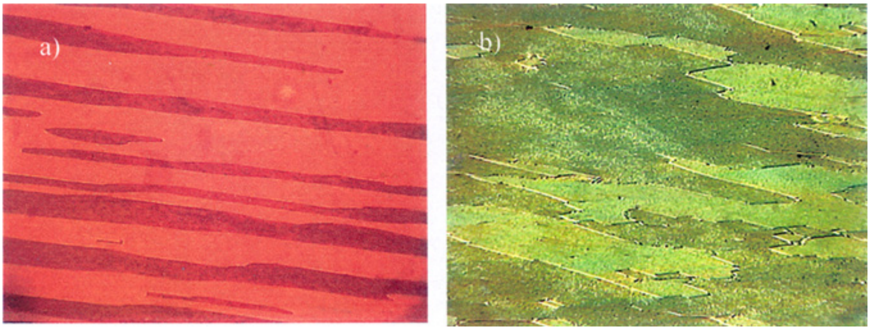

FIGURE 1 Room temperature textures of FLC in a) a parallel cell and b) a cell with rubbing directions skewed by $\pm 18^\circ$. (See COLOR PLATE XV)

X-rays. The space between the substrates was set to 2 μm, which was controlled with spacer beads. The glass substrates were coated with a polyimide alignment layer in order to promote uniform orientation of the director along the cell. The nominal pre-tilt induced by this polymer alignment layer is less than 1° [9], so planar alignment has been assumed. Cells were made with the rubbing directions of the two substrates set parallel and skewed by $\pm 9^\circ$, $\pm 18^\circ$ and $\pm 40^\circ$. Figure 1 shows the texture under the polarizing microscope of the FLC material in a parallel cell and in a cell skewed by $\pm 18^\circ$.

The X-ray experiment was performed at a small angle X-ray scattering station (2.1) at Daresbury Laboratory. The synchrotron radiation used had a wavelength of 1.5 Å and the sample detector distance was set to $\sim$1.5 m. The diffraction patterns were recorded by a two dimensional detector. The beam dimensions were set to $\sim 0.5 \times 1$ mm. The sample was housed in an electrically heated oven, which was designed to allow the sample to be rotated up to 60° from its initial orientation with the glass substrates perpendicular to the incident beam. The rocking curve was measured by rotating the sample in 0.25° steps and recording diffraction from the smectic layers at each orientation.

3. DATA ANALYSIS

Since X-rays are only reflected from the smectic layers when the Bragg conditions are satisfied, the reflected intensity can be used to probe the layer-normal distribution within a cell. The sample rotation angle at which the layer reflection is obtained is determined by the tilt of the layers with respect to the substrates. The azimuthal position of the Bragg reflection on the detector is determined by the in-plane twist of the layer-normals about the mean rubbing direction. In this work, we have transformed the measured Bragg intensity, as a function of sample angle and azimuthal angle, into a layer-normal distribution using a previously described method [10–12]. Our results for the layer-normal distribution within cells are therefore presented as two-dimensional maps of the relative number of layers with out-of-plane tilts, δ, and in-plane twists, γ of their normals. A tilt angle, δ, of zero corresponds to layers exactly perpendicular to the substrate; and a twist angle, γ, of 90° corresponds to a layer normal pointing along the rubbing direction or, in the case of the skewed cells, pointing along the bisector of the two rubbing directions.

4. RESULTS

4.1. Bulk Studies

X-rays diffraction studies of the FLC material in the bulk state were done by using a sealed X-ray tube source at Bristol. The nature of the $N^* \rightarrow SmC^*$ phase transition was studied by analyzing the change of the layer reflection versus the temperature. The material was cooled from the isotropic phase in a magnetic field of ~0.5 T, which produced a well-aligned sample with

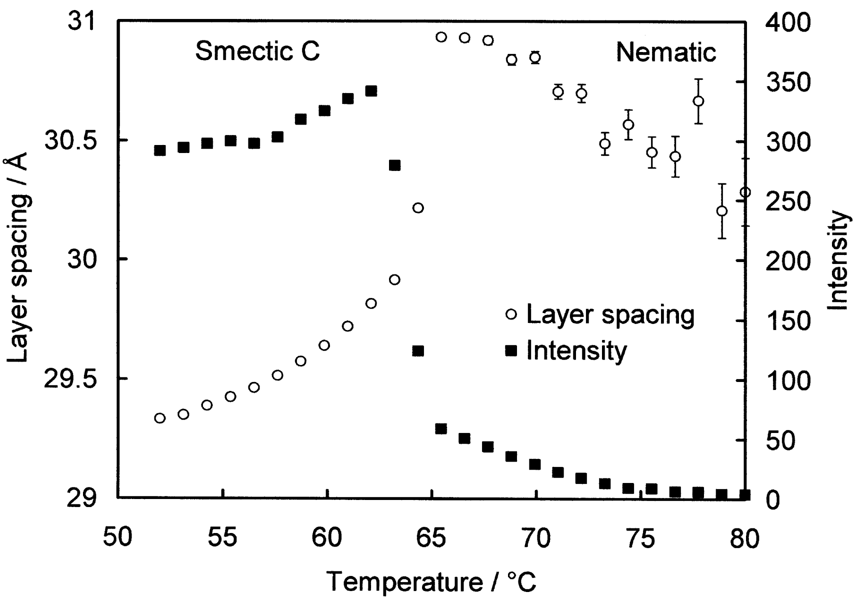

FIGURE 2 The spacing and intensity of the layer reflection as a function of temperature. In the nematic phase, the smectic-like short range order is sufficient for an observable diffraction peak.

two sharp Bragg peaks on the detector. The values of the integrated intensity and the layer spacing are given in Figure 2.

The $N^* \rightarrow SmC^*$ transition was observed as a rapid increase in the layer reflection intensity between 65°C and 63°C. The two degree range of this transition was also observed in differential scanning calorimetry results and appears to be an intrinsic feature of the material. The transition is preceded by an increase in diffracted intensity for ~10°C inside the N^* phase. This indicate pretransition formation of SmC^* like short-range order in the N^* phase. In the SmC^* phase the layers shrink by about 0.5 Å in the ~10°C region below the transition, but the layer spacing remains constant on further cooling to room temperature. It interesting to note that close to the $N^* \rightarrow SmC^*$, at 67°C, the diffraction pattern was characterized by four diffuse peaks tilted by 19° with respect to the magnetic field direction as can be seen in Figure 3. This indicates that in the nematic, the director is parallel to the magnetic field while incipient, short-range, layers are tilted away from it. The value of 19° is expected to be close to the material's cone angle, θ.

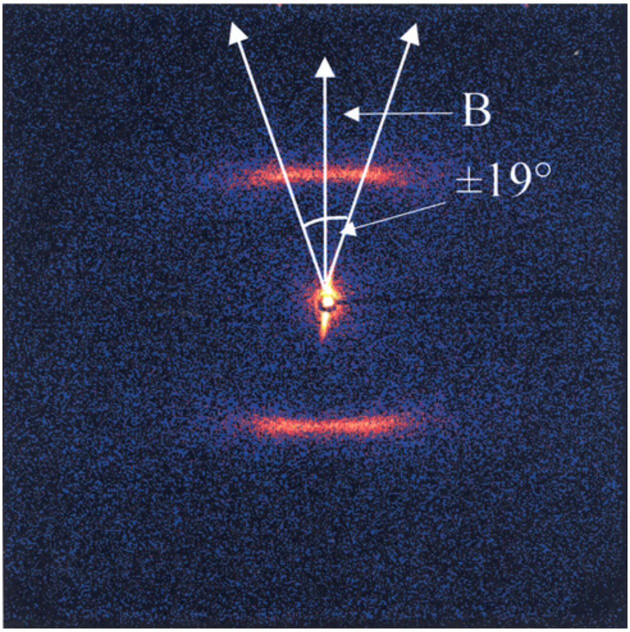

FIGURE 3 X-ray diffraction pattern at 67°C. The diffuse peaks are due to SmC like short range order in the nematic phase. The magnetic field direction is vertical and the diffuse peaks are at an angle of about 19° to it. (See COLOR PLATE XVI)

4.2. SSFLC State

4.2.1. Parallel Alignment

The layer normal distribution of the cell with parallel alignment at 63°C in the smectic to nematic transition region is shown in Figure 4. It is characterized by two sets of peaks , A, B, C, at $\gamma \approx 110^\circ$ and, D, E, F at $\gamma \approx 70^\circ$. These peaks are rotated by $\pm 20^\circ$ from the rubbing direction ($\gamma \approx 90^\circ$) and are consistent with the formation of horizontal chevrons [13]. The difference in intensity between the two sets of peaks, A, B, C and D, E, F is due to the material mosaic texture: the beam illuminated more of one type of domains than the other. In the D, E, F set, the intensity is spread along δ. The set of peaks at $\gamma \approx 110^\circ$, A, B and C, have a similar feature, although the distribution is less continuous in δ. At the next temperature, 58°C, and all lower temperatures such as 48°C shown in Figure 5, the γ value of the two sets of peaks does not change, although δ increases. This increase is due to the layer shrinkage observed in the bulk material as shown Figure 2. As the cell is cooled down, the continuous intensity between A, B and C and between D, E and F disappears. Thus, the layer-normal distribution of the two domains, one at $\gamma \approx 70^\circ$ the other at $\gamma \approx 110^\circ$, is characterized by a sets of 3 peaks centered at $\delta \approx 12^\circ$, $\delta \approx 0^\circ$, $\delta \approx -12^\circ$, as shown in Figure 5. On further cooling the tilt of the layers did not change. Therefore,

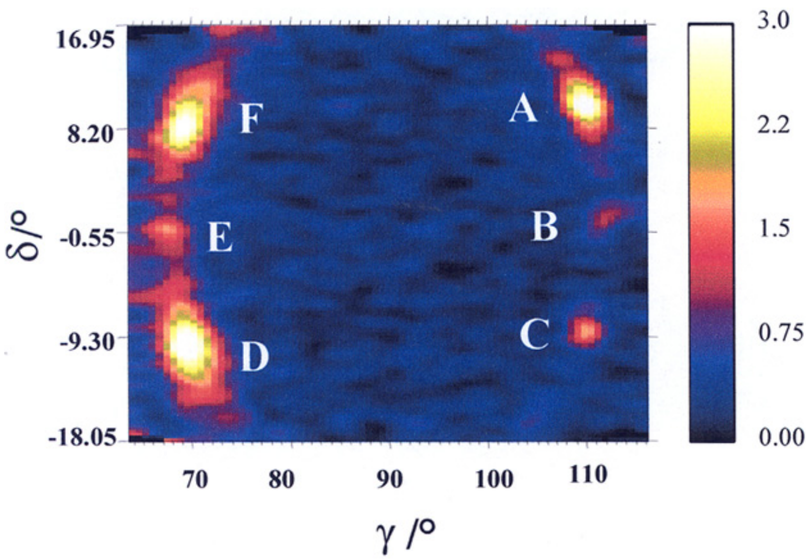

FIGURE 4 Layer normal distribution from the parallel cell at 63°C. The scale shows the relative number of layer normals corresponding to each colour. (See COLOR PLATE XVII)

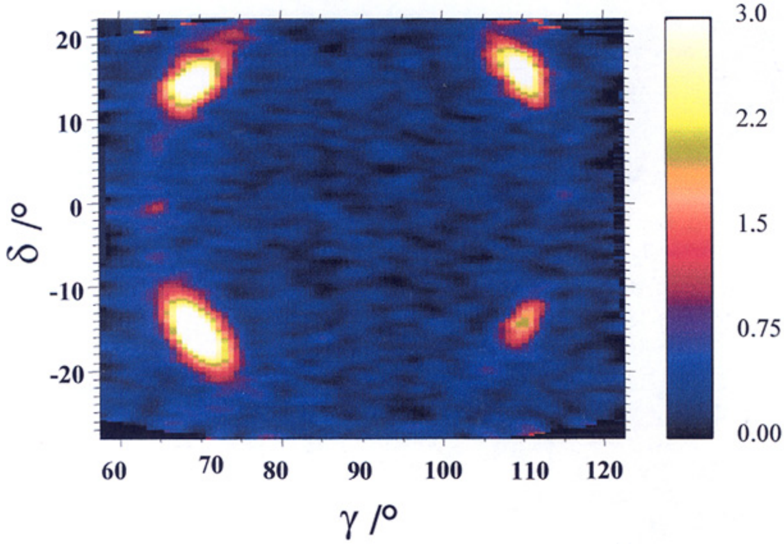

FIGURE 5 Layer normal distribution of the parallel cell at 48°C. The distribution remained very similar at all temperatures down to 30°C. (See COLOR PLATE XVIII)

the layer distribution of this cell (at and below 48°C) consists of horizontal chevrons with layers tilted by 12° with respect to the surface, however, this is not a standard vertical chevron [3] structure as the reversal of the tilt at the chevron interface is made through a region with no tilt, $\delta \approx 0^\circ$, corresponding to peaks B and D, as shown in Figure 6. The twist angle, γ, of the layer-normals remains constant with temperature. This is expected because changes in the twist of the layer-normals would imply a slip of the molecules at the surface, which is unlikely due to the anchoring introduced by the polymer alignment layer.

4.2.2. Skew Alignment

Optical studies of the FLC material reveals that the cone angle of the material is between $18^\circ \sim 20^\circ$. The skew angle between the rubbing directions of the substrates was set at values of $\pm 9^\circ$, $\pm 18^\circ$ and $\pm 40^\circ$ to span the material's cone angle. The objective was to investigate whether the director moved around the cone to accommodate the twist induced by the skew alignment or whether the layers twist or bend in order to preserve the twisted director structure of the nematic phase.

The layer-normal distribution of the cell with the alignment skewed at $\pm 9^\circ$ is characterized by 4 peaks, A, B, C and D, as is shown in Figure 7. The peaks A and C are centered at $\delta \approx 20^\circ$ and $\delta \approx -20^\circ$ respectively,

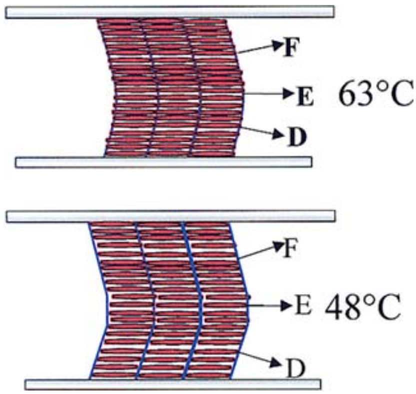

FIGURE 6 Schematic cross section of layer structure in the parallel cell. It shows the flattened apex which was found at temperatures of 58°C and below. (See COLOR PLATE XIX)

and are shifted in γ by 4° with respect to each other. The peaks B and D are centered at $\delta \approx 14°$, $\gamma \approx 110°$ and $\delta \approx -14°$, $\gamma \approx 70°$ respectively. It is interesting to notice the arc-like shape of the peaks B and D: δ increases as the twist angle, γ, approaches 90°. The peaks A and C are consistent with a chevron-like structure, tilted with respect to the surface by 20° but with the layer-normals of each arm twisted in-plane by ±2° with respect to each other. The shape of the peaks B and D shows that the layer-normals tilt out-of-plane more as they twist toward the bisector of the skew angle (i.e. $\gamma \approx 90°$). We propose a model in which the layers related to the peaks B and D are close to the substrates of the cell while the layers related to the peaks A and C are situated in the bulk of the cell. Peaks A and C suggest that there is a chevron pointing along the bisector in the middle of the cell. This is suggested by the observation that regions of the cell were found where the layer normal distribution was inverted suggesting that these regions contain a chevron formation in the opposite direction. The peaks B and D come from layers nucleated at the surface of the device, where the director is pointing along the rubbing directions and so the layer normals are twisted at an angle of 9° to the bisector as well as tilting out of the plane by about 15°. The continuous distribution between peaks A and B and between D and C suggests that there are layers continuously twisted between the substrate and the bulk.

The layer normal distribution of the cell with the alignment skewed by ±18° is similar to the cell with the alignment skewed by ±9°. It is characterized by 4 peaks, A, B, C and D, as shown in Figure 8. The peaks A and C are at $\delta \approx 20°$ and $\delta \approx -20°$ respectively, but spread more in γ. Although, the satellite peaks B and D are similar to those observed in the ±9° cell, the

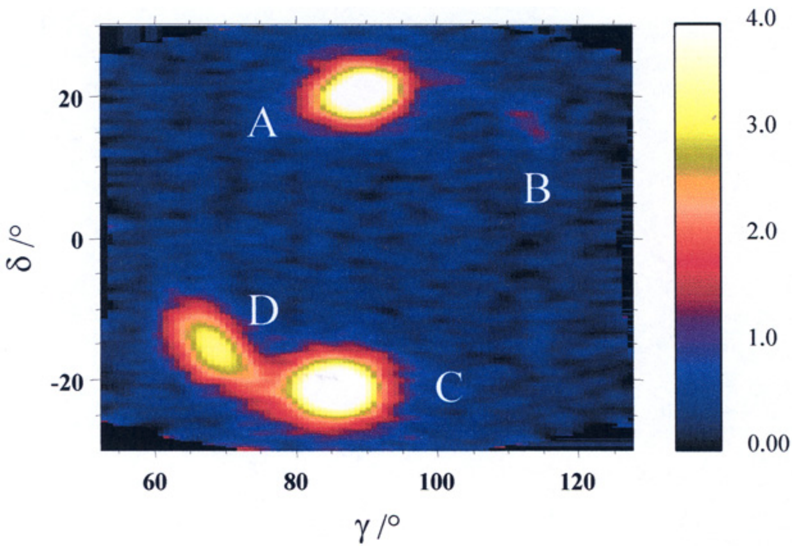

FIGURE 7 Layer normal distribution of the cell with skew alignment set at $\pm 9^{\circ}$. (See COLOR PLATE XX)

intensity is now more uniform and there is a no well-defined maximum. The layer distribution proposed for this device is characterized by a chevron-like structure tilted 20°, corresponding to peaks A and C. However, the spreading of these peaks suggests that there may be domains with layer-normals slightly twisted with respect to the bisector of the rubbing directions. The peaks B and D come from layers nucleated at the surface of the device, where the director is pointing along the rubbing directions and so the layer-normals are twisted at an angle of 18° to the bisector. As for the $\pm 9^{\circ}$ cell, there is a continuous distribution between peaks suggesting that the layer-normals are continuously twisted between the substrate and the bulk.

The cell with the alignment skewed by $\pm 40^{\circ}$, Figure 9, shows two peaks, A and B, at $\delta = 17^{\circ}$ and $\delta = -17^{\circ}$ respectively shifted in γ by 2° respect to each other. Therefore, the layer normal distribution of this cell is characterized by a chevron-like structure tilted by 17° with its arms shifted by $\pm 1^{\circ}$.

5. DISCUSSION

The layer normal distribution of a SSFLC cell is expected to be influenced by the director profile in the previous phase. Thus, the layer structure of a $N^* \rightarrow SmA \rightarrow SmC^*$ material is more predictable than a $N^* \rightarrow SmC^*$, as the

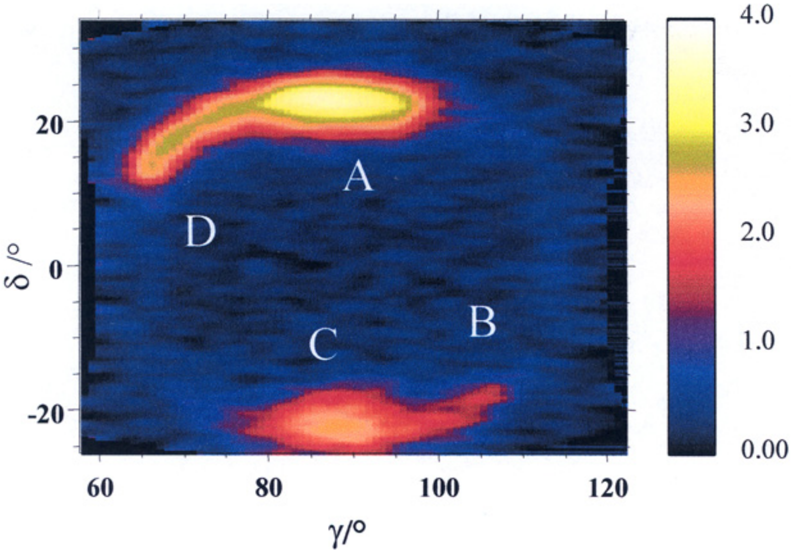

FIGURE 8 Layer normal distribution of the cell with skew alignment set at ±18°. (See COLOR PLATE XXI)

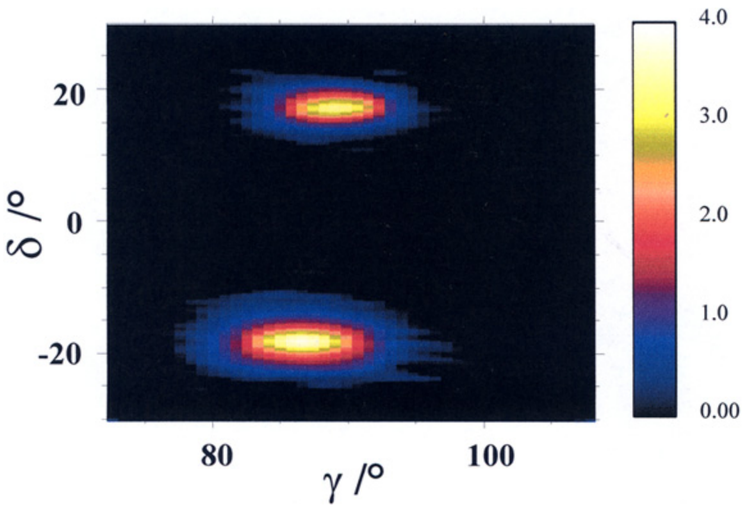

FIGURE 9 Layer normal distribution of the cell with skew alignment set at ±40°. (See COLOR PLATE XXII)

previous layered phase adds a constraint to the system. In the cell with parallel alignment, filled with the $N^* \rightarrow SmC^*$ material the layer-normals twist with respect to the rubbing direction by an amount similar to the cone angle in order to maintain the director profile induced by the previous phase and fulfill the symmetry of the SmC^*. The symmetric twist of the layer-normals with respect to the rubbing direction (i.e. $\gamma = 90^\circ$) is known as "horizontal chevron" formation. [13] This structure is characterized by two types of domain with different in-plane orientations of the layer normals. One domain has its polarization up and the other down. Figure. 1a shows the texture of a cell with rubbing directions set parallel, where the director remains parallel to the rubbing direction. The optical domains correspond to the two directions of the horizontal chevrons. A. Findon *et al.* [14] studied a similar system where the layer structure of an achiral $N \rightarrow SmC$ material in a cell of thickness of 5 µm was determined by X-ray diffraction. The optical texture of the material was similar to the system studied here, although the layer normal distribution was different: layers tilted at the surfaces that bend continuously towards a bookshelf geometry on moving towards the center of the cell at room temperature.

The cells with skewed alignment showed a different layer-normal distribution from the parallel ones. It was expected that the skewed alignment would shift the in-plane component of the layer normal, γ, by an amount similar to half of the angle between the two rubbing directions whilst keeping the tilt of the layers, δ constant. The layer normal distribution of the cells with the rubbing directions of the substrates set to $\pm 9^\circ$ and $\pm 18^\circ$ shown similar features: both of them have a chevron like structure which most probably resides in the bulk of the cell and two sets of layers with normals twisted in opposite directions close to the substrates. There are two mechanisms to accommodate the twist induced by the skewed alignment of the substrates in the N^*:

1. The layer-normals are orientated along the bisector of the skew angle and through the thickness of the cell, the director rotates around the SmC cone in order to accommodate the twist (bookshelf geometry).
2. The twisted structure is maintained in the SmC phase by the elastic distortion of the layers or through the formation of defects.

We propose a model that is a combination of these extremes. In materials with an $N^* \rightarrow SmA$ phase transition the twist and bend are expelled since the director must be parallel to the layer-normal. Whereas if the material has an $N^* \rightarrow SmC^*$ phase transition, the twist and bend induced in the nematic phase can preserved through elastic distortions of the layers in the SmC^*. The peaks A and C in Figures 7 and 8 come from layers that have a tilt with respect to the surface similar to the cone angle which implies that the directors lie parallel to the surface and along the bisector of the rubbing directions. The satellites peaks B and D are better defined in the cell with the rubbing direction set at $\pm 9^{\circ}$ than in the cell with $\pm 18^{\circ}$. This can be explained in terms of the twist induced by the skewed substrates in the nematic phase. The cell with a smaller skew angle introduces less twist in the nematic phase and therefore can better accommodate the distortion close to the surface. In our model when the system goes through the $N^{*} \rightarrow SmC^{*}$ phase transition the molecules in the bulk of the cell rotate towards the bisector of the skew angle forming the tilted layers related to the peaks A and C, whereas the molecules close to the surface remain parallel to the rubbing direction forming the layer related to the peaks B and D. It seems that the system compresses the twist close to the surfaces and that the molecules in the bulk rotate towards the bisector of the skew angle to reach a minimum energy configuration. The reason for the arc-like shape of the satellites peaks, B and C, is due to the fact that when the layers twist and bend in the SmC^{*} phase, they have to keep the layer spacing constant. Therefore, the chevron structure related to peaks A and C is a consequence of the change of orientation of the molecular polarization on going from one of the substrates to the other. Figure 10 shows schematic diagrams of the layer normal distribution proposed for the cells with the rubbing direction set to $\pm 9^{\circ}$ and $\pm 18^{\circ}$. The cross section of the cell parallel to the bisector of the rubbing direction indicates the director profile and

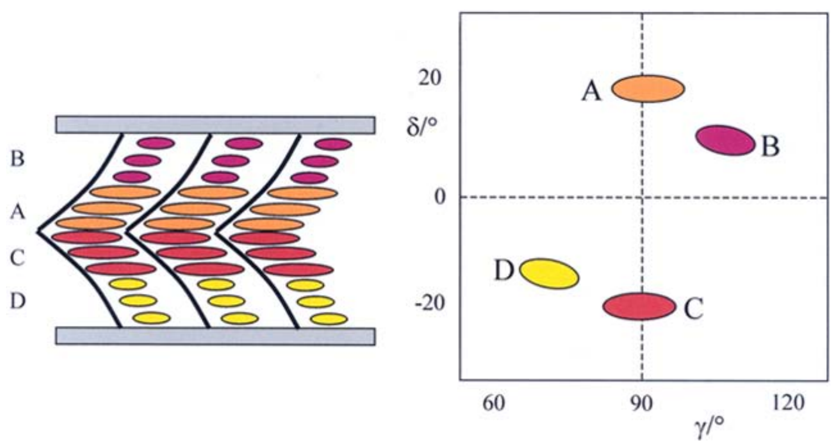

FIGURE 10 Schematic diagram of model for the cells with rubbing directions skewed by $\pm 9^{\circ}$ and $\pm 18^{\circ}$. It is a cross section of the cell parallel to the bisector. The orientation of the director and the layers in four zones and the corresponding peaks in the layer normal distribution are shown for the four zones in the cells. The colours correspond to the four zones in Figure 11. (See COLOR PLATE XXIII)

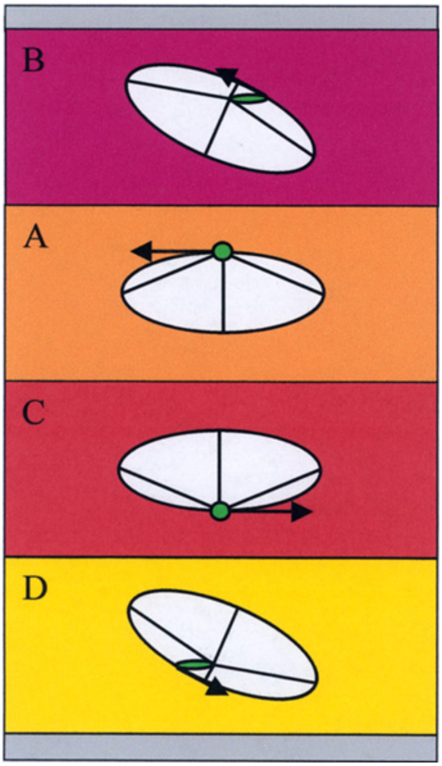

FIGURE 11 Idealized diagram of model for the cells with rubbing directions skewed by $\pm 9^\circ$ and $\pm 18^\circ$. It is a cross section of the cell perpendicular to the bisector. It shows the orientation of the smectic C cones. The layer-normals are parallel to the cone axes. They are pointing out of the page but tilted right and down in zone B, left and up in zone D, down in zone A and up in zone C. The green ellipses represents the molecules (or director) and are pointing exactly out of the page in zones A and C but are tilted slightly left or right in B and D. The arrow indicates the polarization. The measured layer normal distributions indicated a more continuous gradation between the layer-normals in A and B and between the layer-normals in C and D than is shown on this schematic. The structure of the cell with parallel alignment resembles zones B and D only and the structure of the $\pm 40^\circ$ cell resembles zones A and C only. (See COLOR PLATE XXIV)

the change of orientation of the layers between the substrates. An interesting consequence of this arrangement is the different domains of polarization that arise from the different orientation of the layers. The domains close to the surface have a component of polarization pointing perpendicular to the surfaces (purple and yellow), whereas the domains in the bulk of the cell have a polarization pointing parallel to the surfaces (orange and red) as shown in Figure 11.

The layer normal distribution for the cell with the rubbing directions set at $\pm 40^\circ$ is characterized by the lack of satellites peaks. This can be explained in term of amount twist induced in the nematic phase by the substrates. In this cell, the elastic distortion of the material cannot sustain the twist of the previous phase and so only the chevron like structure form. It has to be noticed that tilt of the chevron of this cell is different from the cells with smaller skew angle. This could be due to the lack of interaction between the layers nucleated at the surface with those nucleated in the bulk of the cell.

It is also possible to rationalize layer-normal distribution of the $\pm 40^\circ$ cell using this model. Since the rubbing direction is now 40° away from the director in the middle of the cell, it is no longer possible for the layer-normal to change continuously from the chevron at $\gamma \approx 90^\circ$ in the bulk of the cell to layers with their normals twisted away from the bisector at the surfaces. There has to be is some slippage of the surface molecules so that they all become part of a single chevron structure.

The textures observed in Figure 1 support our model. The cell with parallel alignment shows a mosaic like texture, whereas the cell with the skew alignment set at $\pm 18^\circ$ shows a texture characterized by zigzags [15] defects between domains. In the cell with parallel alignment the apex of the chevron-like structures of the domains points at $\pm 20^\circ$ to the rubbing direction so zigzags are not formed. In the cells with the skew alignment there is a chevron like structure that points along the bisector giving rise to the well known zigzag defects.

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