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An unusual chevron structure in an achiral smectic C liquid crystal device

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The layer structure that occurs in an achiral smectic C liquid crystal device has been investigated as a function of temperature using the small angle X-ray scattering facility at the Synchrotron Radiation Source, Daresbury UK. The material studied shows a direct phase transition from the nematic to the smectic C phase. The layer structure proposed on the basis of the diffraction data is relatively complex, containing regions with chevron, quasi-bookshelf and curved structures. A rationale for the formation of the structure is presented, relying on both the phase transition characteristics of the system and the anisotropic layer elasticity in the smectic C phase. Qualitative analysis indicates that the layer constant A_{12} is greater than A_{21} , i.e. layer flexing is easier perpendicular to the plane of the director than parallel to it. It is also demonstrated that the surface chevron angle is several degrees different from the tilt angle of the smectic C phase at temperatures well below the smectic C to nematic phase transition.

1. Introduction

The existence of chevron structures in smectic liquid crystal devices is well known and has been reported in both smectic A (SmA) and ferroelectric chiral smectic C (SmC*) devices. The chevron structure generally occurs when a device with a bookshelf geometry is cooled, resulting in a reduction in the smectic layer thickness. The lowest energy distortion that the layers can adopt to accommodate the reduced thickness is one in which they buckle, forming a chevron, with the production of a defect 'fold' along the centre of the device parallel to the substrates. Most reports of the chevron structure relate to ferroelectric devices containing SmC* liquid crystals, including the first described in 1988 [1], although it is apparent in a SmA device from X-ray data published as early as 1980 [2]. The mechanism causing layer thinning is different in SmC* and SmA systems. In ferroelectric materials the layer spacing reduces when the molecules tilt [3], resulting in a chevron structure which typically forms at an angle approximately the same as the tilt angle of the system (often around 22°). In smectic A liquid crystals, however, the layer thinning occurs as a result of changes in molecular packing rather

than tilt, and the associated chevron angles observed are small, typically less than 10° [4–6].

More complex layer structures have been observed in ferroelectric liquid crystal devices that have been subjected to large electric fields (so-called electric field treatment). In addition to the striped textures [7], a variety of local layer structures depending on the surface alignment conditions and applied field have been reported [8–13]. As well as the undistorted bookshelf and chevron structures, tilted layers, bent layers and asymmetric chevrons have all been observed. In the experiments reported, application of an electric field was necessary to induce the various local layer structures. The layer structure adopted within the device had an influence on the optical properties, the bistability and the measurement of certain liquid crystal parameters [14]. Although the local layer structures in ferroelectric liquid crystal devices are clearly well studied, primarily because of the implications for device applications, the structures formed in achiral smectic C devices have not been considered in as much detail. Further, the work on SmC* systems has concentrated on materials in which the phase sequence observed on cooling is chiral nematic to smectic A to smectic C*, again because of the technological application of such materials; the layer

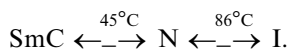
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structures adopted in devices containing materials in which there is a direct transition between the chiral nematic and ferroelectric phases have been largely neglected.

As part of an investigation of the electro-optic properties and elastic constants of the achiral smectic C phase, the layer structure that occurs spontaneously in a flat cell has been studied. The ultimate aim of the work reported in this paper was to determine the elastic constants of smectic C liquid crystals of positive dielectric anisotropy via a study of the Fréedericksz transition in both flat and wedged-shaped devices. Such experiments were first proposed by Leslie *et al.* [15] for very specific device geometries and layer structures within the device. The actual layer structure adopted by the material under investigation was therefore of importance to the electro-optic studies which will be reported in a separate paper. This paper describes the optical and X-ray studies of a device containing the achiral smectic C liquid crystal. The material studied exhibits a nematic to smectic C phase transition. The unusual layer structure adopted by the device, proposed on the basis of optical and X-ray measurements, is described.

2. Experimental

The flat cells of nominal thickness $5\ \mu\text{m}$ were prepared with parallel glass windows and contained homogeneously aligned samples of the smectic C phase of a room temperature smectogen. The glass windows were only $100\ \mu\text{m}$ thick, making them suitable for both optical and X-ray studies, and were coated with a conducting indium/tin oxide layer. The surfaces of the windows were treated for parallel homogeneous alignment by coating them with a layer of rubbed polyvinyl alcohol (PVA). The smectic material used was denoted M3 [16], a mixture of the compounds shown in figure 1. The material has the phase sequence:



The smectic C phase persists to well below room temperature. Above 45°C the material forms a nematic phase and it is convenient to fill the cell with the mesogen in this state and then allow the smectic phase to form on cooling. This process gives rise to the interesting domain pattern which to some extent determines the electro-optic properties of the device and is the subject of this paper.

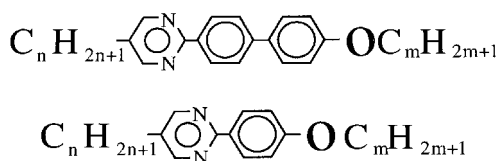


Figure 1. The constituents of the mixture M3.

The optical textures of the sample were observed via polarizing microscopy using an Olympus BH2 polarizing microscope fitted with a Linkam hot stage. The hot stage has a temperature resolution of $\pm 0.1^\circ\text{C}$ and could be used in conjunction with both the microscope and the X-ray apparatus to control the temperature of the sample over its entire phase range. The X-ray diffraction experiments were carried out on station 2.1 at the Synchrotron Radiation Source (SRS), Daresbury Laboratory, Warrington, UK. A collimated beam of X-rays of wavelength $1.54\ \text{\AA}$ and cross section $1\ \text{mm} \times 1\ \text{mm}$ was used. The evacuated camera had a specimen to detector distance of $1\ \text{m}$ and the diffraction pattern was recorded with a two-dimensional gas-filled area detector. The detector was a multiwire chamber with wire spacing of $0.5\ \text{mm}$ constructed at the Daresbury Laboratory [17]. The experimental geometry is shown schematically in figure 2 and has been described in detail elsewhere [6]; it allows the smectic layer spacing to be measured with a resolution of $\pm 0.1\ \text{\AA}$. For convenience, a co-ordinate system is defined in which the direction of the incident X-rays coincides with the z -axis. The orientation of the sample cell with respect to the incident X-ray beam could be changed by rotating (rocking) the cell about a vertical axis in the y -direction. At normal incidence, the plane of the device lies parallel to the xy -plane. The change in the diffraction pattern with rocking angle forms the basis of the model of the domain structure of the sample proposed here.

3. Results and discussion

3.1. Appearance of the cell between crossed polarizers

The optical texture observed in the thin parallel film of M3 in the smectic C phase appears as an array of approximately parallel domains, each about $100\ \mu\text{m}$ long and $40\ \mu\text{m}$ wide. The domains are roughly rectangular, separated by zigzag-like defects and there are suggestions of focal-conic structures in places. The birefringence colours of the sample change only very slightly as the sample is rotated on the stage, implying that there are two predominant orientations of the domains, symmetrically aligned at a small angle on either side of the rubbing direction.

3.2. X-ray diffraction data

The diffraction pattern observed is shown schematically in figure 3. It consists of two pairs of arcs which occur at a Bragg angle of approximately 5° , corresponding to a layer spacing of approximately $18\ \text{\AA}$ within the smectic C phase at 20°C . Such a diffraction pattern can be interpreted in terms of a smectic C structure with the layers tilted in the plane of the device at the angle ψ defined in figure 3. ψ is approximately 20° in the case described here, though neither the angle ψ nor

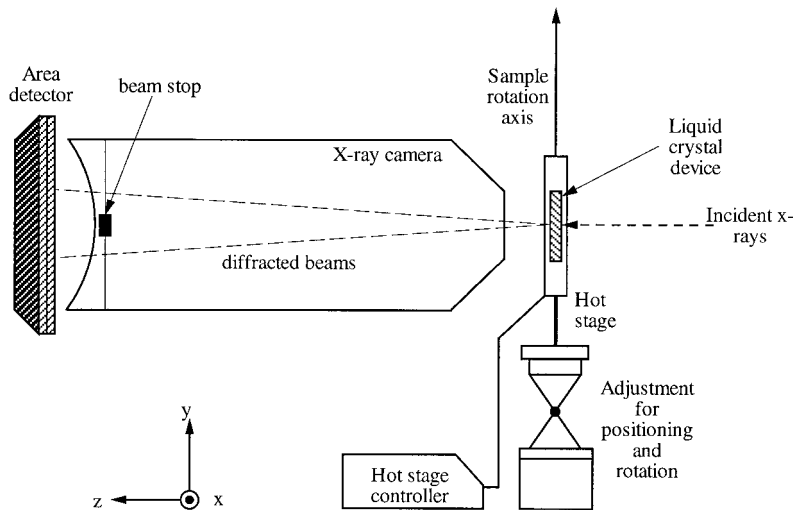


Figure 2. A schematic of the X-ray scattering apparatus used. The x -, y - and z -axes define the co-ordinate system used throughout the paper.

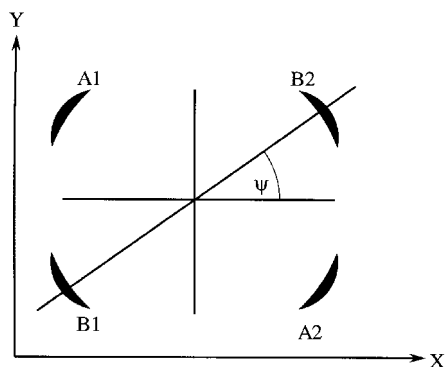


Figure 3. The appearance of the diffraction pattern obtained from the device containing the smectic C liquid crystal. The angle ψ and the pairs of Bragg peaks are defined for later discussion.

the intensities of the Bragg peaks were constant as the sample was rocked about the y -axis. Exact details of the diffraction patterns obtained are described quantitatively below.

As the sample cell is rocked through an angular range of 30° either side of the position where the beam strikes the cell normally, each of the reflections (A1, A2, B1, B2) peaks three times as shown in the rocking curve in figure 4 which was recorded at a temperature of 19.3°C . The intensity data presented in figure 4 have been corrected for the difference in absorption of the glass at different rocking angles and for various apparatus properties that have been described previously [6], but no background has been subtracted. Figure 4 shows three peaks, and such rocking curves can result from either multidomain structures which include regions with layers in chevron, tilted bookshelf and bookshelf orientations, or from structures with more complicated layer geometries through their depth. Rocking curves with three peaks have been obtained for the smectic A

phases of achiral materials [4, 6]. Takinishi *et al.* [4] attribute the three peaks in their rocking curves to kinking of smectic A layers (initially in a bookshelf geometry) as the sample is cooled. A quasi-bookshelf structure coexisting with areas of chevron structure was reported. Morse and Gleeson [6] observed the hybrid behaviour in a material again in a smectic A phase at temperatures well below the N to SmA phase transition. In the latter reference, a small angle chevron structure was reported to form initially at the nematic to smectic A transition, deforming at lower temperatures to a uniform structure with some chevron and some bookshelf characteristics. Observation of the optical textures of the device studied, in addition to the rocking curves recorded, enables distinction to be made between multidomain structures in which several types of layer geometry coexist in different domains within the device and more uniform systems with slightly more complex layer structures.

The rocking curve of figure 4 is interpreted broadly in terms of layers in three orientations as shown in figure 5 since the optical observations for the device showed no evidence of coexisting domains with different layer structures. The X-ray pattern does not enable the relative positions of these three orientations within the depth of the cell (i.e. along the z -axis) to be specified, but there are two obvious possible structures, as shown. The arrangement (a) with two chevron arms and a central bookshelf region is thought to be the more probable structure. The pattern of observed intensities shown in figure 4 implies that the greater part of the sample lies in the chevron arms and only a minor part in the central bookshelf region, although the angular resolution of the rocking curves does not allow differentiation between symmetric and asymmetric chevrons. Further, the peaks are very broad and there is substantial X-ray scattering around 0° and $\pm 22^\circ$, indicating some

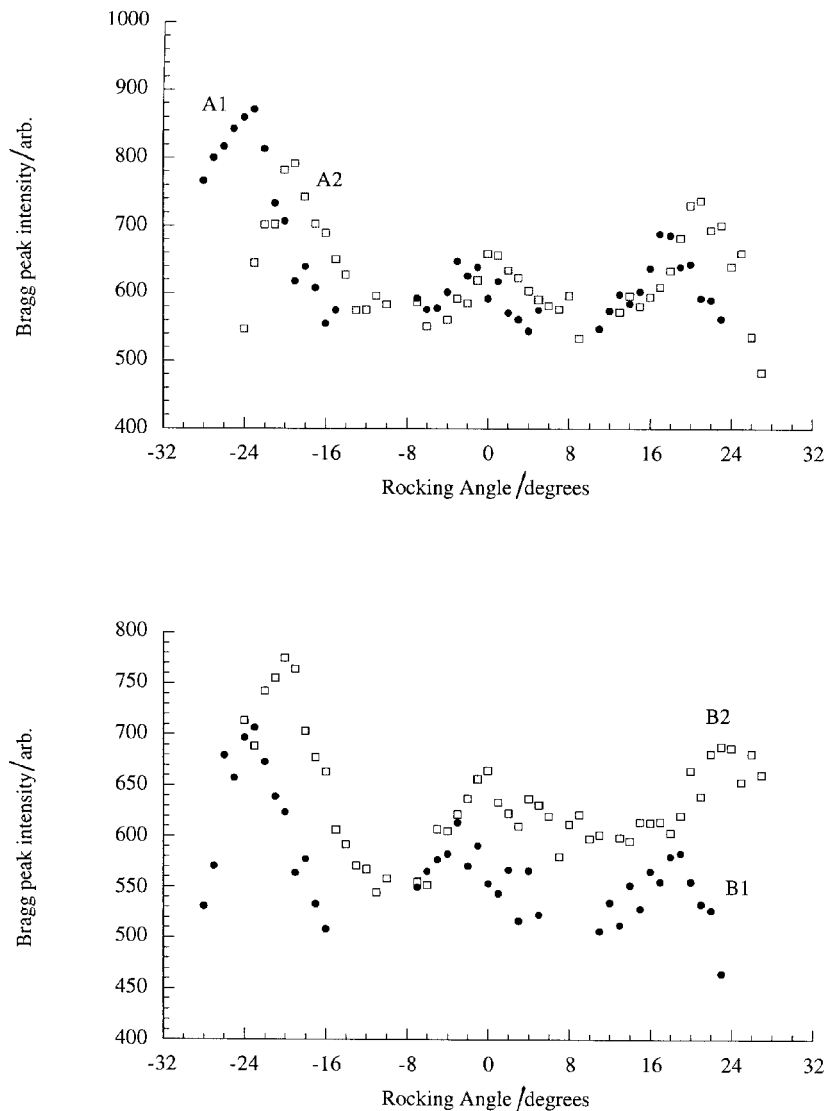


Figure 4. Rocking curves recorded for the device at 19.3°C. The filled symbols correspond to the integrated intensity of the A1 and B1 peaks, while the open symbols refer to the A2 and B2 peaks.

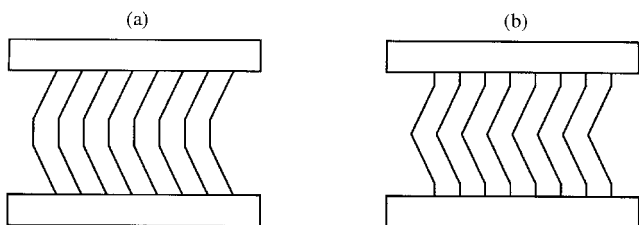


Figure 5. Two possible ways in which the layers can form to produce rocking curves such as those shown in figure 4.

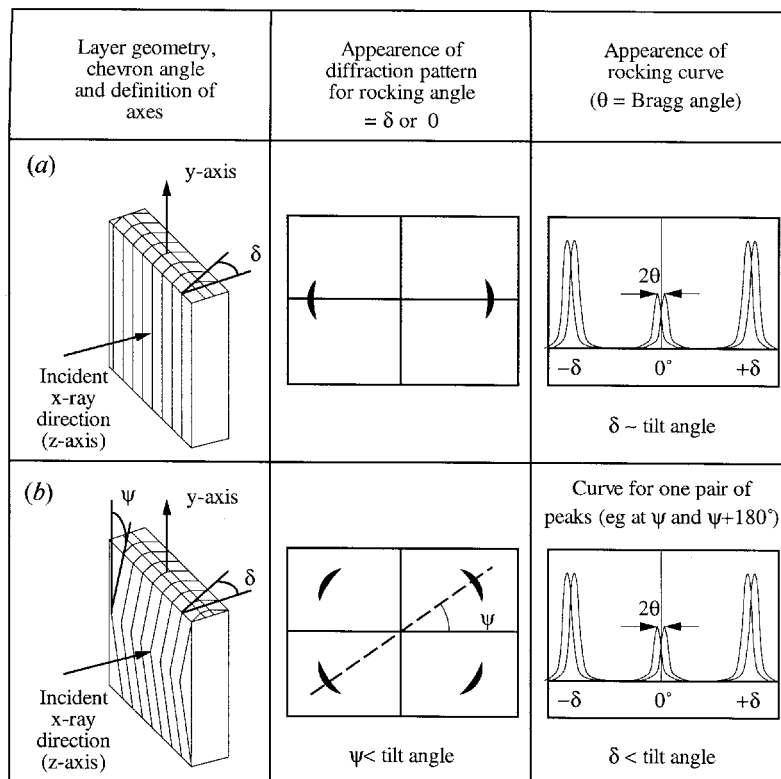
bending of the layers. The chevron angle, δ , deduced from figure 4 is $22 \pm 1^\circ$.

X-ray diffraction patterns reported here suggest that the system studied has chevron characteristics both in the depth of the device and in the surface plane. Figure 6 shows the X-ray diffraction patterns and rocking curves that are expected for two specific layer structures in the

plane of the device, both of which assume the structure indicated in figure 5(a) throughout the depth of the device. The first structure, figure 6(a), considers the case in which the layer normal lies in the y -direction in the co-ordinate system used here. Figure 6(b) includes a single chevron in the plane of the device, although a structure with more than one surface chevron would give rise to an identical diffraction pattern. Figure 6(b) has some characteristics of the system reported here, though it does not fully describe the data, as is discussed below.

A close inspection of the way in which the diffraction pattern changes with rocking angle reveals a further feature. The reflections lie in non-equatorial positions indicating that the smectic layers which gave rise to them are not vertical (i.e. the layer normal is not in the x -direction). In the experiment reported here, the reflections at a specific temperature appear at a constant

Figure 6. The small angle X-ray diffraction patterns that would be expected for the idealized geometries shown. (a) No surface chevron, and (b) a single surface chevron. The layer structure in the cross section of the device is in both cases identical to that in figure 5(a). Note, however, that degeneracy is possible; the cross section of figure 5(b) would give rise to identical diffraction patterns and for figure 6(b), there may be many surface chevrons rather than the single one shown. The angle θ is the Bragg angle of the system and all the pairs of peaks on the right hand side of the figure are separated by 2θ . Each peak maps the intensity of one of the Friedel pairs.



Bragg angle within the resolution of the camera used, though the co-ordinates of the diffraction peaks on the recording screen change with the rocking angle as shown in figure 7. The peaks that occur when the rocking angle is close to zero lie furthest removed from the equator, whilst those occurring when the rocking angle is at $\pm 28^\circ$ are significantly closer to the equator. This indicates that the central bookshelf regions are tilted the most in the plane of the device. The X-ray diffraction data recorded during the experiment reported here,

together with the more qualitative optical observations, lead to the proposal of the layer structure within the device shown in figure 8.

X-ray scattering experiments were carried out at several temperatures throughout the SmC phase, though rocking curves were only recorded at 19.3°C and 36.8°C . Figure 9 shows the value of ψ for normally incident X-rays ($\delta = 0$) across the SmC range. It can be seen that there is little temperature variation, the angle falling slowly from approximately 27° to 21° as the temperature

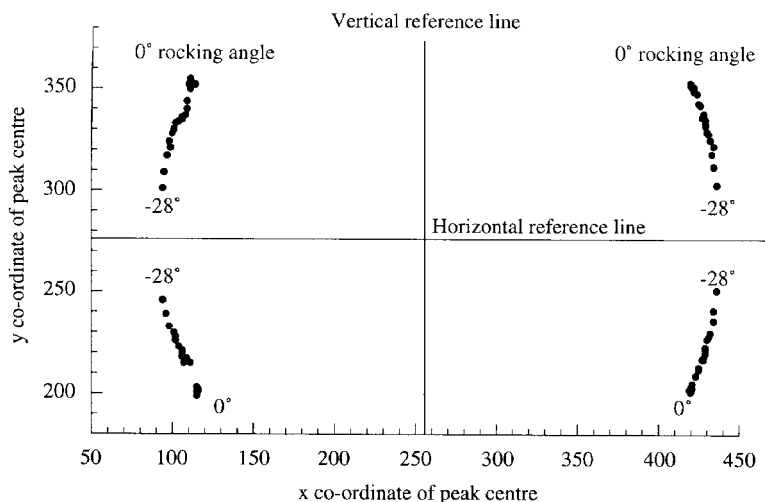


Figure 7. The evolution of the position of the X-ray diffraction peaks obtained from the cell as it is rocked from 0° to -28° .

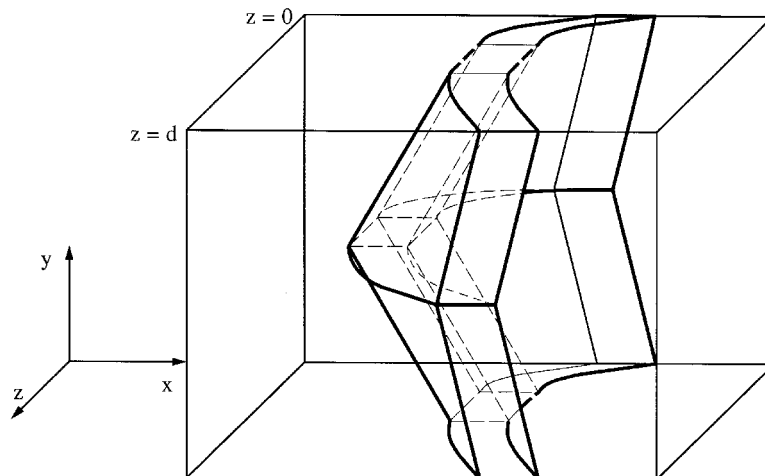


Figure 8. A schematic diagram of the proposed layer structure. The device substrates lie in the xy -plane at positions $z=0$ and $z=d$, respectively. In the experiments described here, X-rays are incident from the z -direction and the axis about which the device is rocked is in the y -direction. The angles are not drawn to scale. At 19.3°C the chevrons lying on the surface of the device ($z=0, d$) make an angle of approximately 21° with the y -axis, while those closer to the centre of the device (the bookshelf region, where the layer normal is in the x -direction) make an angle of $\sim 27^\circ$.

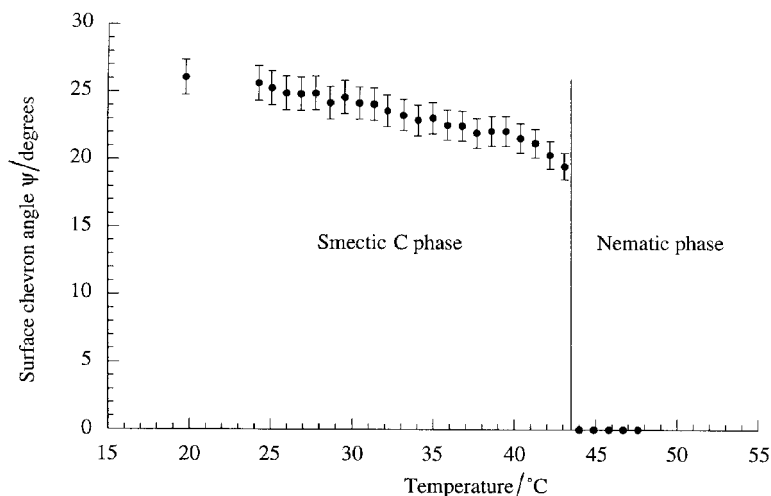


Figure 9. The angle of the chevron at the surface of the device as a function of temperature deduced from the position of the Bragg peaks observed for X-rays at normal incidence to the device. The slight difference in transition temperature from that quoted in the text is due to the different length of cable used to attach the hot stage to the controller at the SRS facility.

is increased and reducing rapidly to zero at the SmC to N phase transition. The transition is clearly first order, as expected. The layer tilt angle of the surface chevron structure, ψ , can be seen to increase as the temperature decreases, and such behaviour is typical of SmC systems. The rocking curve experiments yielded values for the chevron angle δ of 22° and 16° at temperatures of 19.3°C and 36.8°C , respectively. From the temperature variation of the surface chevron angle ψ and the chevron angle δ , it can be deduced that the tilt angle of M3 varies from around 35° at lower temperatures to about 20° at the smectic C to nematic phase transition.

3.3. Rationale for the domain pattern

The origin of the domain pattern suggested by the X-ray data and shown schematically in figure 8 can be

explained in terms of the first order phase transition at the N to SmC transition point, together with the influence of the anisotropic elasticity of the system. The layer structures that result in different X-ray diffraction patterns for SmC systems are well documented and the characteristic diffraction pattern shown in figure 3 is usual for systems that have a direct first order transition from the nematic to the smectic C phase. Here, the surface alignment direction imposed on the director in the nematic phase (the x -direction) is maintained through the nematic to smectic C phase transition. The smectic C layers form a surface chevron structure at the transition such that the layer normal is tilted in the xy -plane by an angle equal to the tilt angle of the system. A bookshelf structure is generally thought to occur within the device at the transition, and it would persist if there was no

further change in tilt angle with respect to temperature. However, the data for M3 show that the tilt angle varies by several degrees across the SmC phase. The formation of the unusual chevron pattern observed at lower temperatures can be explained in terms of the following steps:

- (1) The epitaxial alignment of the molecules at the glass surface gives an aligned nematic phase when the cell is first filled.
- (2) As the cell is allowed to cool, a tilted SmC phase forms which, because of surface anchoring of the director, aligns with a bookshelf geometry throughout the depth of the device and a surface chevron structure.
- (3) As the material cools into the smectic C phase the layers decrease in width and expand in area. This produces appreciable compressive strain.
- (4) The strain is relieved by the layers buckling. They billow out in the x -direction as shown in figure 7 and the lateral movement is greatest in the centre of the cell away from the surface anchoring. Because of the asymmetry of the smectic C structure, the layers can bend more easily perpendicular to the plane of the director than parallel to it. The relevant layer elastic constants are defined in figure 10. The structure observed implies that the elastic constant A_{12} (related to the layer bend in the surface xy -plane of the device in this geometry) is greater than A_{21} (the layer flexing in the depth of the device). The layers should therefore appear more curved when viewed in the xz -plane than in the xy -plane. This appears to be compatible with the interpretation of the X-ray diffraction patterns proposed here: the layers as viewed in the xz -plane are curved whereas in the xy -plane they are kinked.

4. Conclusion

This paper reports the way in which the X-ray scattering from a liquid crystal device containing an achiral smectic C phase depends on the relative orientation of the device to the incident X-ray beam. The material

contained in the device exhibits a first order nematic to smectic C phase transition. The domain structure proposed on the basis of the data presented forms spontaneously and consists of slightly curved layers in one plane (through the depth of the device) and kinked layers in an orthogonal plane (the surface plane of the device). The structure suggested here is significantly different from the surface chevron/bookshelf structures assumed for many achiral smectic C systems. It is clear that the angle ψ is not the same as the tilt angle of the material, except at the smectic C to nematic transition, and differs from it by several degrees at low temperatures well within the smectic C phase. The structure suggested is also quite different from the chevron structures reported in smectic A and ferroelectric devices. The striped texture reported in ferroelectric liquid crystal devices includes chevrons in the plane of the substrates and is usually formed in response to a large electric field. More complex layer structures are reported at zigzag defects in ferroelectric devices [3, 18] in which parallelogram-shaped layer elements mediate chevrons of opposite orientation in the device. The structure suggested here includes slightly curved layers, in addition to kinked ones, and is formed spontaneously on cooling the device from the nematic phase into the smectic C state.

The response of the cell to an electric field applied in the z -direction will be described in detail in a subsequent paper, but it is relevant to mention here that the domain structure determines the response which appears to consist of a realignment of the molecules within the existing layers rather than a gross reorganization. This realignment causes the layers to expand in one direction and contract in the other, causing further strain in the sample and culminating in the creation of a new domain structure with units elongated in the y -direction.

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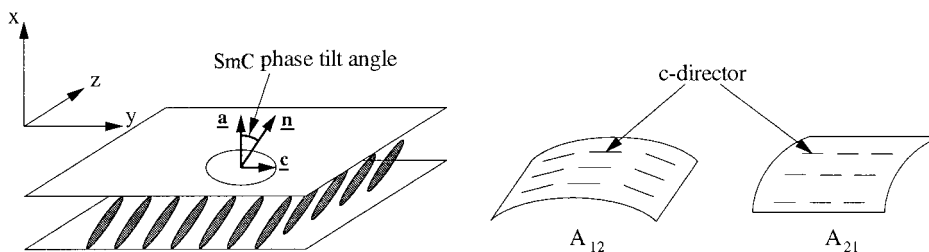


Figure 10. A schematic representation of the elastic constants A_{12} and A_{21} (after Carlsson *et al.* [15]). The layer normal is in the direction of the unit vector, \mathbf{a} , and the vector \mathbf{c} is the projection of the director onto the layer plane.

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