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Characterization of focal conics in chiral smectic C liquid crystals by X-ray microdiffraction

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Focal conics consisting of an ellipse and a hyperbola in chiral smectic C liquid crystals aligned in surface-stabilized cells of medium thickness (about 25 μ m cell gap) were characterized in relation to the chevron structure by synchrotron X-ray microdiffraction. The focal conic texture is embedded in the chevron structure with a relatively sharp interface. The deformed layer of the focal conics is a kinked bookshelf consisting of a few slightly bent segments for small focal conics, whereas it is a bookshelf layer for large focal conics. Around the focus of the ellipse toward the hyperbola, a complicated layer structure appears, although the core region of small layer curvature has been hardly observed within the present experimental sensitivity. The broad and narrow walls of a zigzag defect in the same cell are analyzed for comparison.

1. Introduction

The study of defects in liquid crystals (LCs) not only has practical importance for the development of various devices but also attracts fundamental interest in the evolution of the LC under various boundary conditions. The focal conic (FC) is the common defect observed in the smectic phase, in which the parallel layer, although it remains equidistant, can be considerably curved according to the shape of Dupin cyclides [1]. In a chevron structure, which characterizes the layer structure of a chiral smectic C (SmC*) phase in a surfacestabilized cell [2], the FC appears as an ellipse and a hyperbola [3], whereas an FC of parabolas intersecting each other perpendicularly is observed in the smectic A (SmA) phase toward the SmA-SmC* transition [4, 5]. In previous studies, it has been discussed how the layer around the FC could be connected to the chevron structure [3-7]. Recent theoretical and experimental studies have revealed new aspects of FCs: the importance of the elastic energy in the FC domain formation [8, 9], the analysis of the defect core structure due to the interface anchoring [10, 11] and the FC nucleation and growth process in the SmA phase [12].

Most studies on defect structure in LCs have been performed using optical techniques. Although X-ray diffraction is more effective in revealing the layer structure directly [4, 6], it has been difficult to investigate an individual FC due to its small defect volume. Recently the layer structure of the defect core in an extremely thin smectic LC deformed through strong antagonistic anchoring has been successfully studied using synchrotron radiation (SR) grazingincident X-ray diffraction [11]. For the study of the FC layer structure of a SmC* phase in a practical surface-stabilized cell, it is attractive to use SR X-ray microbeam small-angle diffraction, which has been applied to soft materials research [13, 14].

This paper reports characterization of the layer structure of an isolated FC in a surface-stabilized SmC* phase by X-ray microbeam small-angle diffraction and its relation to the chevron structure. The modified zigzag defect walls in the same cell are also analyzed for comparison.

2. Experimental

The experimental conditions are briefly described here for convenience (a detailed description is provided elsewhere [14, 15]). The synchrotron X-ray diffraction experiments were carried out on beam-line 4A at the Photon Factory. The incident X-ray energy was 8 keV (1.55 Å) and the X-ray beam size was about $4(h) \times 5(v) \mu m^2$. The sample rubbing direction (X) was

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Figure 1. Schematic representation of a sample cell (a) and coordinate axes (b). An FC consisting of an ellipse (X-Y plane) and a hyperbola (X-Z plane) in a vertical chevron layer is shown schematically.

set horizontally. The layer deflection angles, δ and γ , are defined in figure 1. The χ intensity distribution (χ profile), which reflects the γ deflection, was recorded on a position-sensitive proportional counter simultaneously, whereas the ω -scan intensity profile (ω profile) was obtained by rotating the sample around a vertical axis (Y). In this paper, the ω profile means the integrated intensity with respect to χ as a function of ω and the $\omega - \chi$ profile is obtained by recording χ profiles as a function of ω .

A ferroelectric LC sample, CS-1014 (Chisso), was sandwiched between glass plates (each $150 \,\mu\text{m}$ thick) with polyimide alignment films rubbed unidirectionally. The cell gap was about $25 \,\mu\text{m}$. The experiments were performed in the SmC* phase at room temperature. Since the cell gap was relatively large, the helical stripe line parallel to the smectic layer plane appeared throughout the cell.

3. Results

In a sample cell many isolated FCs were observed of various sizes from a few tens to a few hundred μ m when measured along the major axis of the ellipse. In the micrograph shown in figure 2 a, typical FC texture, which consists of an ellipse (left, major axis is about 60 µm long) and a short segment of a hyperbola (right), are observable due to the distortion of stripes. The stripe bends toward the left inside the ellipse and it shows complicated structure near the ellipse focus and the hyperbola region. The previously proposed layer



Figure 2. (a) An in-situ micrograph of focal conic with helix stripes. A, B C and D indicate positions where X-ray measurements were carried out (see figures 3 A and 4 B–4 D). The scale mark is 40 μ m. (b) A proposed layer structure of focal conics in the chevron layer. The upper figure shows the section at the cell centre in the *X*–*Y* plane, whereas the lower figure corresponds to the section of the *X*–*Z* plane.

structure [1, 3, 5, 6] is shown schematically in figure 2 b for convenience.

Figure 3 shows a series of ω -dependent γ profiles (ω - γ profiles) as a function of the vertical positions (Y) near the centre of the ellipse along the arrow A in figure 2a. The peaks around $\omega = \pm 18^{\circ}$ and $\chi = 0^{\circ}$ are due to the chevron structure (hereafter, termed the chevron peak) and the broad peak between the chevron peaks is due to the defect. As the measurement position changes along the Y direction, the intensity distribution of the broad peak varies from the low-angle side to the high-angle side in the χ -direction, indicating that the layer bends toward the left in figure 2 a considering the experimental arrangement, which agrees with the deflection direction of stripes. At the FC centre, the ω - χ profile is approximately symmetric with respect to $\gamma = 0^{\circ}$ and $\omega = 0^{\circ}$ and its intensity distribution extends as far as 20– 30° in both χ and ω directions. Since the X-ray microbeam penetrates the cell, the observed broadening of the intensity distribution in the χ -direction is partly due to the average over the depth. A close look reveals that the intensity distribution along the ω direction is not continuous but consists of a few broad peaks (two peaks are observed around $\omega = \pm 10^{\circ}$ from $Y = -5 \,\mu\text{m}$ to $5\,\mu\text{m}$ in figure 3), indicating that the layer consists of a few segments of slightly bent layers, i.e. a kinked bookshelf structure. A slight asymmetric profile in the ω -direction also indicates that the curved layer in the FC is not uniform. The in-plane local layer tilt, which is reflected in the χ profile, is expected from the bent stripe



Figure 3. A series of $\omega - \chi$ intensity profiles along arrow A in the micrograph in figure 2 a. The ω step was 2° with a 30 s accumulation time at each ω angle. The relative position is shown in μ m in each profile (5 μ m apart). The intensity is multiplied by 5 except for chevron peaks (around $\omega = \pm 18^{\circ}$ and $\chi = 0^{\circ}$) to clarify the profile in the low-intensity region. Xray intensities were normalized by the highest (darkest) intensity in each figure. Note the scale difference in the ω and χ angle.

observed in figure 2 a, whereas the in-depth bent layer, the intensity distribution in the ω direction, can be observed only by X-ray scattering.

Figure 4 shows a series of $\omega - \gamma$ profiles as a function of the vertical positions (Y) at B, C and D in figure 2a. At the right side (C) of the FC, the scattered X-rays from the defect center (0 μ m) become broad in the γ direction, indicating that the curvature is large around the ellipse focus. At the hyperbola part (D), the local layer structure still bends both in ω and χ directions similar to the ellipse part of the FC, although it is weak in scattered X-ray intensity. The perturbed region of the hyperbola in the Y direction is much smaller than that of the ellipse part. The complicated layer structure in the region from focus of the ellipse to the hyperbola is also expected from the micrograph (figure 2 a). It is noted that a large deflection angle, both γ and δ , is not observed even near the focus of the ellipse (apex of the hyperbola) within the present experimental sensitivity. Contrary to the deformed FC layer, detailed analysis of the chevron peak showed that the chevron structure that surrounds the FC is not appreciably deformed.

A large FC, with an ellipse major axis of about 200 µm, was also examined (see figure 5 c). A series of ω - χ profiles as a function of the vertical positions (Y) at the centres of the ellipse and the hyperbola are shown in figures 5 a and 5 b, respectively. The scattered intensity distribution and its positional dependence in figure 5 are similar to those for the small FC. Compared to the smaller FC, however, the scattered intensity from the deformed layer concentrates on the narrow ω and χ angular region, indicating that the defect layer is transformed to the bookshelf layer structure and that the transition region from the surrounding chevron layer to the bookshelf layer is negligibly small. This is compared to the layer structure of the small FC where the layer consists of a few bent segments, as already mentioned. Furthermore, the $\omega - \chi$ profiles from various large FCs are more or less similar to that in figure 5, whereas the detail of the kinked bookshelf structure from small ones depends on the FC. It is also noted that the $\omega - \chi$ profile at the hyperbola (figure 5b) is weak and is complicated, similar to the small FC.

The ω profiles with the high angular resolution are shown in figure 6 as a function of X position (at the centre in the Y direction) in the hyperbola part. The broad peak due to the FC appears inside the chevron peak ($\omega = \pm 18^{\circ}$) and it is noted that the ω profile is quite similar to that obtained from the narrow wall of a zigzag defect [15].

Finally, the broad and narrow walls observed in the same cell (see figure 7 c) were studied for comparison. For thick cells, the narrow and broad walls have been



Figure 4. A series of $\omega - \chi$ intensity profiles along the Y and X directions. The rows correspond to positions B, C and D in the micrograph in figure 2 a. In the Y direction, each figure is 10 µm and 5 µm apart for lines B and C and line D, respectively, as shown in the figures.



Figure 5. A series of $\omega - \chi$ intensity profiles along the Y direction for a large FC. (a) and (b) were obtained by scanning along the left (A) and right (B) arrows in micrograph (c) (80 µm apart). In the Y direction, each figure is separated by 15 µm. The scale mark in (c) is 40 µm.



Figure 6. A series of detailed ω profiles as a function of the X-position along the centre line of the FC in the hyperbola part toward the right. The right end column is obtained from the region without FCs. A 0.1° ω step and 1s accumulation time were used for each step.

reported to consist of many FCs [3]. In the present case of a medium thickness cell, as shown in figure 7 c, FClike texture seems to exist in the broad wall, although the ellipse or hyperbola figures cannot be recognized clearly. Figure 7 a shows the $\omega - \chi$ profiles obtained at the inclined narrow wall along the arrow A in figure 7 c. The peak position in the χ direction depends on the ω angle and the maximum χ angle is more than 30°. These characteristics are the same as those for narrow walls reported in the previous study on a thin cell [15] and are in agreement with the uniform structure observed in figure 7 c. At the broad wall (figure 7 b), the $\omega - \chi$ profiles indicate the bookshelf structure, as expected, although the broadening in the χ direction is relatively large compared to the thinner cell, and that the weak deflection in the χ direction is observed, indicating that the broad wall is not pure bookshelf but deformed, similar to the FC.

4. Discussion

The FC is embedded in the chevron structure and no significant deformation of the chevron layer surrounding the FC is observed. It is shown that the layer for the large FC is a bookshelf structure with a negligibly small transition region from the FC to the surrounding chevron structure, whereas that for the small FC is a kinked bookshelf structure consisting of two or three bent segments. The volume ratios of the deformed FC layer to the chevron layer at the ellipse centre can be estimated using the X-ray integrated intensity of the broad scattering peak due to the deformed layer to that of the chevron peak without FC defect, and they are about 9+1 and 3+1 for large (figure 5) and small (figure 2) FC, respectively. This implies that most of the layer structure along the depth at the ellipse changes to the FC deformed layer, although the chevron layer still remains, as seen from sharp chevron peaks. Since the large FC occupies most of the layer volume of the present relatively thick cell, the layer structure at the ellipse is mostly the bookshelf structure reflecting



Figure 7. $\omega - \chi$ intensity profiles (a) and (b) obtained across the narrow and broad wall (arrows A and B, respectively) of the zigzag defect (c). The scale mark in the micrograph is 100 µm. Each profile in the same row is separated by 5 µm and 50 µm for (a) and (b), respectively.

the bulk FC. In contrast, in the small FC, the chevron layer due to the anchoring at the cell interface occupy to a certain extent, relatively thin FC layer produce the kinked layer rather than the uniform bookshelf structure, similar to the layer structure observed at a broad wall in a thin cell [14].

Around the focus of the ellipse toward the hyperbola, the layer structure is complicated and is not clarified with the present experiment. It is noted from the micrographs that the layer distortion around the hyperbola part extends more than expected from the confocal FC equation; for instance, the distorted region corresponding to the hyperbola is about $30 \,\mu\text{m}$ in figure 2 a, whereas the projection length of the hyperbola segment is expected as $15 \,\mu\text{m}$ from the calculation. It has been reported that FCs obey the geometric condition for the thick cell [6]. Since the layer is subjected to the conflicting boundary conditions, i.e. those at the cell interface and along the singular line (hyperbola), for thin cells and/or the large FC, the layer structure becomes complicated at the hyperbola part.

In both FCs (small and large), the large deflection angle has not been observed even near the focus of the ellipse (apex of the hyperbola), i.e. the deflection angles are less than the chevron angle in most FCs of various sizes. This implies that the volume of the highly deformed region (near the core of the FC) is limited in space or the long range layer order is destroyed. Furthermore, the layer curvature seems to obey the $\cos \delta \cos \gamma < \cos \sigma$ condition, where σ is twice the angle of the tilt angle; this also implies that the director continuity at the interface holds also for the FC layer and its surrounding region in the SmC* phase.

In summary, focal conics structure in chiral smectic C liquid crystals aligned in the medium thick surfacestabilized cells have been characterized by synchrotron X-ray microbeam diffraction. The layer structure around the focus of the ellipse and also around the hyperbola seems to be greatly disturbed from that of smooth ideal focal conics.

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