ЖЭТФ, 2002, том 122, вып. 4 (10), стр. 840–848

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TEMPERATURE- AND FIELD-INDUCED TRANSITIONS IN FREE-STANDING FILMS OF AN ANTIFERROELECTRIC LIQUID CRYSTAL

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Submitted 17 May 2002

Thin free-standing films of a compound with the smectic-C_A^* and smectic-C_α^* phases were investigated by means of depolarized microscopy and optical reflectivity. In thin films, the smectic phase sequence C_A^*–C_α^*–A is replaced by a series of temperature- and field-induced transitions into states with the coplanar orientation of molecular tilt planes. Transitions are accompanied by a change of the direction of the electric polarization with respect to the tilt plane of molecules. The coplanar structure of these states is consistent with the Ising model.

PACS: 61.30.Eb, 64.70.Md, 68.10.Cr

1. INTRODUCTION

The synclinic smectic-C phase (SmC) and the anticlinic smectic-C_A phase (SmC_A) are the fundamental mesophases with fluid layers that are observed in rod-like molecular systems. When the constituent molecules render the system chiral, the respective phases are denoted as SmC^* and SmC_A^* and become ferroelectric, with the spontaneous polarization perpendicular to the tilt plane, and antiferroelectric [1–5]. The locally averaged molecular tilt direction, called the director, is specified by the polar angle θ and the azimuthal angle ϕ in the liquid crystal frame of reference with the z axis along the smectic layer normal. In the bulk sample, the tilt angle θ is constant at a given temperature. Chirality leads to the formation of a helicoidal structure, $\phi = 2\pi z/p$, where p is the helicoidal pitch. Because p is sufficiently larger than the smectic layer spacing d in general, the azimuthal angle difference between adjacent layers is $\Delta\phi \simeq 0°$ in SmC^* and $\Delta\phi \simeq 180°$ in SmC_A^*; the respective phases therefore remain practically synclinic and anticlinic. In other words, the ordinary chiral intermolecular interaction is weak. The molecular origin of the synclinic and anticlinic ordering in SmC and SmC_A has been studied in detail [6, 7]. It is concluded that the conventional dispersion and steric interactions stabilize SmC and SmC^*, while the orientational correlations of transverse dipoles in adjacent layers actually promote SmC_A and SmC_A^*. We note that these short-range interactions and correlations are not sensitive to the molecular chirality and favor the coplanar synclinic or anticlinic structure.

The transition between synclinic ferroelectric SmC^* and anticlinic antiferroelectric SmC_A^* is of the first order. In many materials, a sequence of polar subphases with periods consisting of more than two layers are formed between SmC^* and SmC_A^*. In this narrow temperature interval, the system is frustrated and both phase structures have nearly the same energy; the subphases must therefore be stabilized by some relatively weak additional factors [7]. Isozaki

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et al. [8, 9] systematically investigated the electric-field–temperature (E–T) phase diagram in several compounds and mixtures [5–8, 10]. They confirmed that at least five subphases can exist between SmC_A^* and SmC^* and the additional one, denoted as SmC_α^*, just below SmA on the high-temperature side of SmC^*, suggesting that the subphases between SmC_A^* and SmC^* consitute a part of a devil's staircase formed by frustration between ferro- and antiferroelectricity. Moreover, it was noticed by Takanishi et al., and Hiraoka et al. [11–15] that SmC_α^* itself is not a simple single phase but can constitute another devil's staircase [16] where the ferroelectric SmC^* and/or antiferroelectric SmC_A^* soft mode fluctuations play an important role because of the extremely small tilt angle. Sophisticated experimental techniques, such as the polarized resonant X-ray scattering [17–19], precision ellipsometry and reflectometry [18, 20–22], and advanced polarizing microscopy [23], have recently been used to determine the detailed subphase structures. It is unambiguously established that the subphases between SmC_A^* and SmC^* with three- and four-layer periodicities are not coplanar and that their azimuthal angle difference between adjacent layers considerably deviates from $\Delta\phi = 0°$ (synclinic) or 180° (anticlinic) [18, 24]. With respect to SmC_α^*, a short pitch helical structure was recently emphasized in contrast with the previous suggestion of its devil's staircase character.

Two conflicting approches have been proposed with the important factor being either the continuous short-pitch evolution of the SmC_α^* helical structure [25–29] or the devil's staircase character not only of the subphase emerging between SmC_A^* and SmC but also of SmC_α^* itself [5, 10, 30]. The first one, called the discrete, clock, or X–Y model, takes competing orientational interactions between nearest- and next-nearest-neighbor smectic layers into account. The minimum of the free energy then corresponds to a uniform rotation of the tilt plane about the layer normal. The formation of subphases with three- and four-layer periodicities and the continuous short-pitch evolution of SmC_α^* can be qualitatively explained by introducing several much more complicated interactions. The second approach is based on the microscopic Ising model with competing repulsive and attractive interactions between nearest and next-nearest neighbors. It is known that a sequence of subphases resembling the devil's staircase is indeed obtained in such a model [31, 32]. But the Ising-like Hamiltonian can hardly be applied to smectic liquid crystals [7]. Neither model can therefore appropriately explain the facts experimentally observed thus far.

Prost and Bruinsma [33, 34] proposed a more consistent model by taking long-range polarization fluctuations into account; they tried to explain the formation of SmC_α^* and its devil's staircase character. This mechanism seems to be very promising and should be taken into consideration in a more general theory [7]. In order to understand the sequence of the subpahses and their nonplanar structures better, further careful experimental investigations are necessary in a variety of compounds and mixtures. The structures of the SmC_α^* phase in bulk samples and in thin films were recently reported to be substantially different [35, 36]. Structures without a short-pitched helix were found above the SmC^* and SmC_A^* phases. Another question is the origin and behavior of the electric polarization in thin films of structures without the net polarization in the bulk sample, in particular, in the SmC_A^* and SmC_α^* phases. The clock and Ising models predict a different behavior of spontaneous polarization as the temperature changes: a smooth variation of the polarization value in the clock model and its steplike changes at certain temperatures in the Ising model. Unusual behavior of antiferroelectric structures in the electric field was found in thin films [37, 38]. These structures possess a ferroelectric polarization: perpendicular (P_Y) to the average direction of the tilt planes in odd-N films (where N is the number of smectic layers) and parallel (P_X) to the average direction of the tilt planes in even-N films.

Because the free energy difference between subphases is intrinsically small, several interface effects in both homogeneous and homeotropic cells disturb the supbphase sequence and structures. Free-standing films [39, 40] are most suitable for making observations that are almost free of these effects. Moreover, applying an electric field can also seriously deform the structure. Consequently, it is essential to study free-standing films by applying a sufficiently weak electric field for controlling the director alignment. In this paper, we present such an example of the direct microscopic observation and optical reflectivity. We believe that the observed results may reflect the bulk property to some extent, although the interface effects must also be considerably large because the number of smectic layers is somewhat small.

2. EXPERIMENT

The material studied was 4-(1-trifluoromethyl-hepthyloxycarbonyl)phenyl 4′-octyl-biphenyl-4-carboxylate (TFMHPBC) [5]. Bulk TFMHPBC exhibits the following transition temperatures between smectic phases:

$$\mathrm{Sm}C_A^* \xrightarrow{74.3\,^\circ\mathrm{C}} \mathrm{Sm}C_\alpha^* \xrightarrow{75.0\,^\circ\mathrm{C}} \mathrm{Sm}A.$$

Two types of cells were used in optical measurements. For the first type, films were prepared in a rectangular frame with two mobile metallic blades. For the second type, free-standing films were drawn over a 4mm-diameter circular hole in a glass plate. Electrodes on four sides of the hole were used to apply an in-plane electric field. The number N of smectic layers was determined by optical reflectivity [39, 41].

Two techniques were used for studies of the temperature- and field-induced phase transitions. The first one was the depolarized reflected light microscopy (DRLM) [42, 43]. For relatively thin films, the reflection intensity is proportional to $(n^2-1)^2$ [44],

$$I \propto (n^2-1)^2\pi^2N^2d^2/\lambda^2,$$

where n is the refraction index and λ is the light wavelength. The refraction index along the tilt plane, $n_{\|}$, is different from the refraction index in the perpendicular direction, n_0. The DRLM allows visualizing domains with different orientations of the tilt plane with respect to the direction of the electric polarization $\mathbf{P}$. The technique uses a polarizer turned by 45° with respect to the electric field direction. The difference between the refraction indices for the two main directions results in an effective rotation of the polarization plane of the reflected light in the direction of the tilt plane. In slightly decrossed polarizers, the domains therefore look dark or bright depending on the orientation of the tilt plane with respect to the direction of the electric polarization. In this method, the low intensity of the image is compensated by a highly contrast pattern.

Another technique was used in measurements of the linearly polarized reflectivity from the films in «backward» geometry. In electrooptical measurements, we could change the direction of light polarization and the direction of the electric field in the plane of the film. In tilted smectic phases, the films are optically anisotropic in the plane of the layers. The reflection intensities with the light polarization parallel ($I_{\|}$) and perpendicular ($I_{\perp}$) to the direction of the electeric field were measured. Using this technique, we could determine the orientation of the tilt plane in the films and their optical anisotropy.

3. RESULTS AND DISCUSSION

In the geomety used for imaging, $\mathrm{Sm}C_A^*$ films with an even number of layers look as a dark field with

Fig. 1. Temperature-induced transition in even-N films. (a) The microscope image in reflection of an eight-layer TFMHPBC film in the antiferroelectric phase at $70.7\,^\circ\mathrm{C}$. The $\mathbf{c}$-director is aligned parallel to the electric field. The film appears dark with a bright 2π-wall. (b) Transition on heating ($T = 75\,^\circ\mathrm{C}$): the region of a phase with different orientations of the $\mathbf{c}$-director emerges. At heating, the front moves from bottom to top of the picture. (c) The state at higher temperature with polarization perpendicular to the tilt plane ($T = 75.7\,^\circ\mathrm{C}$). The electric field value is 8.8 V/cm. The orientation of the polarizer ($\mathcal{P}$), the analyzer ($\mathcal{A}$), and the the electric field is shown in Fig. 1c. The horizontal size of each photograph is 370μm

two bright lines in the regions where 2π-walls exist (Fig. 1*a*). Films have the polarization P_X parallel to the tilt plane, and the **c**-director (the projection of **n** on the film plane [3]) is parallel to the vertical axis. Across the 2π-wall, the **c**-director rotates by the angle 2π; as a result, regions with a nearly horizontal orientation of the **c**-director in 2π-walls look as two bright narrow lines. In microscopic investigations, we chose regions of the films with 2π-walls. In these regions, it is easiest to judge on the orientation of the **c**-director with respect to the electric field direction. Heating to 75 °C leads to the appearance of a new phase (Fig. 1*b*). In spite of a small tilt angle, the phase boundary and the phases themselves are easily visualized in films using DRLM above the bulk transition temperature. A transformation of the DRLM picture (dark 2π-walls in the bright field, Fig. 1*b, c*) shows that the direction of the polarization with respect to the **c**-director is drastically different from that in the low-temperature state. The transition may be caused either by a substantial change of the relative orientation of tilt planes in neighboring layers or by the temperature variation of relative values of the polarizations P_X and P_Y and the rotation of the **c**-director of the film in the electric field. The existence of a sharp boundary and the behavior of 2π-walls near it show that the transition is not caused by a smooth temperature decrease of the P_X component of the polarization with respect to the P_Y one. In that case, the transition front would be blurred and the regions of 2π-walls with the horizontal orientation of the **c**-director could smoothly turn into a new oriented state.

In films with an odd number of layers, we also observed the transition to a new structure (Fig. 2). A transformation of the DRLM picture indicates the transition from the structure with the polarization P_Y perpendicular to the tilt plane to a structure with the dominant polarization P_X. Thus, films having polarizations of different types in the SmC_A^* phase (P_Y in odd-N films and P_X in even-N films) after the transition also possess different polarization directions in the plane of the layers (P_Y in even-N films and P_X in odd-N films). We observed such a behavior in all investigated films with the thickness from three to more than twenty layers. But the transition temperatures strongly depend on the film thickness (in a three-layer film, the transition occurs at about 85 °C). Figures 1*b* and 2*b* show two realizing possibilities for the coexistence of 2π-walls and the boundary of two phases. In the antiferroelectric phase, the 2π-walls are either continued into the new phase (Fig. 1*b*) or split into walls that are localized near the boundary as in Fig. 2*b*.

In films with $N > 10$, we found an increase of the

Fig. 2. Temperature-induced transition in odd-N films. (*a*) The optical microscope picture of a nine-layer TFMHPBC film in reflection in the low-temperature (antiferroelectric) phase ($T = 70.4$ °C). In the odd-N film, the c-director is oriented along the horizontal axis. The film is bright with dark 2π-walls. (*b*) On heating to 75 °C, the film undergoes a transition to the phase with the polarization parallel to the tilt plane. This phase appears in the lower part of the photograph. (*c*) The film after the transition. The entire film appears dark with a bright 2π-wall. The electric field value is 7.5 V/cm. The horizontal size of each photograph is 420 μm

Fig. 3. States with the polarizations parallel and perpendicular to the tilt plane in a twelve-layer film are observed as regions with different brightness levels. A sample with a temperature gradient was used for this observation. The left part of the film corresponds to the temperature about 74 °C and the right part to the temperature about 76 °C. In region *1*, the film is in the SmC_A^* phase. In regions *2–4*, states with different orientations of the tilt plane exist. These states can be aligned by the electric field. The narrow stripe between regions *2* and *3* is a line defect localized at the boundary of the states. The horizontal size of the image is 610 μm, $E = 10$ V/cm

number of structures above the SmC_A^* phase. These structures can be simultaneously observed in a sample with a temperature gradient. An image of such a film is shown in Fig. 3. The temperature increases from left to right. In the left part of the Figure, the film is in the SmC_A^* phase *1*. In regions *2–4*, the sample is in different states with different directions of the polarization. This is clearly seen from measurements of the optical reflectivity from a film for two directions of the light polarization, perpendicular ($I_\perp$) and parallel ($I_\parallel$) to the electric field direction. Figure 4 shows the results of such measurements for an eighteen-layer film. In the SmC_A^* phase (Fig. 4*a*), $I_\perp < I_\parallel$, which implies that the polarization is parallel to the tilt plane. The same polarization direction is observed in state *3* (Fig. 4*c*). In states *2* and *4* (Fig. 4*b*, *d*), $I_\perp > I_\parallel$, which indicates that the tilt plane is perpendicular to the electric field direction and to the film polarization. The sequence of transitions from state *1* to state *4* therefore occurs with the change of the polarization direction. These measurements also provide information about optical anisotropy of the films. This enables us to answer one of the main questions: do short-pitch helical structures emerge above the SmC_A^* phase? Formation of the short-pitched azimuthal helix with the pitch of the order of or less than the film thickness should lead to a substantial decrease of the reflection anisotropy $\left|I_\perp/I_\parallel - 1\right|$. In such a nearly optically uniaxial structure, the anisotropy can occur because the film thickness is not a multiple of the half-pitch. But the magnitude of the anisotropy in short-pitched structures must be several times smaller than before the transition from the SmC_A^* phase. A smooth decrease in $\left|I_\perp/I_\parallel - 1\right|$ in Fig. 4*b*–*d* is due to a well-known decrease in the molecular tilt angle on heating. We found that $I_\perp/I_\parallel - 1$ changes its sign as the result of transitions, but the absolute value changes only insignificantly. Therefore, even in relatively thick films (Fig. 4), transitions occur into structures without a short pitch. At high temperatures, state *4* (Figs. 3 and 4) can be switched to the state with the longitudinal polarization by decreasing the electric field value.

Four pictures in Fig. 5 demonstrate the behavior of an eight-layer film at high temperatures. In the low field, the film is in the state with the polarization parallel to the tilt plane (the bright 2π-wall in a dark field,

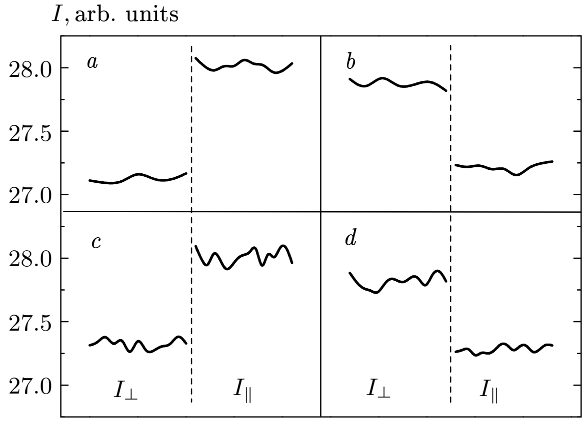

Fig. 4. Optical reflectivity from an eighteen-layer film for two directions of the light polarization: perpendicular ($I_\perp$) and parallel ($I_\parallel$) to the electric field. A sequence of four states was observed: the SmC_A^* phase in state *1* (*a*) and states *2–4* (*b–d*) above the SmC_A^* phase. Transitions between these states occur with the change of the direction of the electric polarization. The tilt plane of molecules is oriented parallel to the electric field and polarization ($I_\perp < I_\parallel$) in states *1* and *3* (*a* and *c*). In states *2* and *4* (*b, d*), the tilt plane is oriented perpendicular to the electric field and polarization ($I_\perp > I_\parallel$). In the SmC_A^* phase, the measurements were made near the temperature T_0 of the transition to state *2*, such that $T_0 - T = 0.3\,^\circ$C; to states *2–4*, correspondingly, at $T_0 + 0.15\,^\circ$C, $T_0 + 0.2\,^\circ$C, and $T_0 + 1.1\,^\circ$C. The data were obtained at the electric field value 35 V/cm

Fig. 5*a*). The increase of the field induces the appearance of the structure with the polarization perpendicular to the tilt plane (Fig. 5*b, c*). The switching electric field E_s is more than three orders of magnitude smaller than the field inducing the anticlinic–synclinic transition in the SmC_A^* phase at low temperatures. Such a small value of E_s is due to the small tilt angle of the molecules. Seemingly, the change of interlayer organization (anticlinic–synclinic or vice versa) occurs in only several layers. This field-induced transition is reversible and the decreasing field returns the film to its initial state (Fig. 5*d*). It is worth mentioning that such a behavior at high temperatures is observed in films of all thicknesses investigated, is independent of the film oddity, and is similar to the behavior of films without the SmC_α^* phase with a large value of the layer polarization [45, 46].

The number of transitions caused by heating depends on the film thickness, its oddity, and the value of the electric field. In thin odd-N films, one transition is observed in the low field and two in the high field. Thin even-N films undergo two transitions in the low field and only one in the high field. The crossover from low to high field is determined by the value of E_s, depends on the film thickness, and typically occurs at about 10 V/cm. In thicker films ($N > 10$), the number of transitions occuring with heating is increased. For odd-N antiferroelectric films, it is odd (even) in the low (high) electric field. For even-N films, the number of transitions is even (odd) in the low (high) electric field. A simpler situation is expected in films with the low-temperature ferroelectric phase: an odd (even) number of transitions in the low (high) electric field in both odd-N and even-N films. As already mentioned, this is the case for compounds with a high layer polarization. For films with a small layer polarization in which the polarization of the high-temperature ground state is perpendicular to the tilt plane [45], the number of transitions is opposite to that described above.

Film surfaces can influence the structure of the film and phase transitions in different ways. The presence of a surface breaks the symmetry existing in bulk samples. For layers near the surface, the nearest and next-nearest interlayer interactions are realized only on one side (only on the side of the film). The missing interlayer interaction near the surface can influence both the modulus θ and the phase ϕ of the order parameter. Symmetry breaking in antiferroelectric films must lead to a smaller magnitude of the polar angle θ and to the low-temperature shift of phase transitions with respect to the bulk sample [47]. The second effect is related to the existence of the surface as a physical boundary between two media. In liquid crystals, this effect mainly manifests itself through the surface tension and the suppression of the layer displacement fluctuations at free surfaces that increase the modulus of the order parameter (surface freezing) and the phase transitions temperatures. To the best of our knowledge, the direct influence of the surface tension on the phase ϕ of the order parameter and suppression of the smectic-layer fluctuations have not been considered theoretically or observed experimentally. Presently, it is still debated which of the above effects prevails for antiferroelectric liquid crystals [47, 48]. The existence of a tilted structure above the bulk transition temperature to the SmA phase and a high-temperature shift of the phase transitions in thin films show that in antiferroelectric films, as in ferroelectric ones, the surface freezing effect dominates. In strong surface freezing conditions, the most distinctive feature of the film is that the profile of θ is essentially inhomogeneous across the film. Above but near the temperature of the bulk transition to the SmC_α^* phase, the modulus of the order parameter on

Fig. 5. An example of the field-induced transition at a high temperature in an eight-layer TFMHPBC film. (*a*) At the low electric field 4 V/cm, the film is in the state with the polarization parallel to the tilt plane. The **c**-director is aligned in the vertical direction (dark background with a bright 2π-wall). (*b*) When the field is increased to 19 V/cm, the region of the high-field state with the polarization perpendicular to the tilt plane emerges as a bright one. This region gradually fills the entire film. The front is moving from the left to the right side of the image. (*c*) The film in the high-field state. (*d*) When the electric field is decreased to 4 V/cm, the film returns to the low-field state. $T = 78.8\,^\circ$C. The horizontal size of each image in about 300 μm

the surface exceeds that in the bulk sample. At the same time, the tilt is smaller in the interior of the film; at some temperature, it corresponds to the value at which transitions with a change of ϕ occur in the bulk sample. In thin films, only few interior layers can undergo the transition. In thick films, the conditions for a change of ϕ arise in a greater number of layers and the number of possible structures with the synclinic and anticlinic tilt is increased. This is related to observed increase in the number of transitions in thicker films.

For the SmC_A^* structure, the appearance of the polarization P_Y perpendicular to the tilt plane P_Y in odd-N films and the polarization P_X parallel to the tilt plane in even-N films is related to different symmetries with respect to the center of the film [37]. Similar symmetry considerations apply to films consisting of synclinic and anticlinic pairs. The change of the number of pairs with synclinic and anticlinic tilts by one can lead to the transition from the P_Y net polarization to the P_X one and vice versa. In even-N films, this reorientation of the tilt can occur with the formation of a symmetric or antisymmetric structure with respect to the center of the film, and accordingly, to the P_X or P_Y net polarization. In odd-N films, formation of a synclinic pair destroys the symmetry with respect to the center of the film. In such films, the P_X or P_Y polarization can dominate. Transition to the P_X polarization with the appearance of a symmetric structure is possible when the molecular tilt becomes zero in the central layer of the odd-N film. We mention that in this case, the decrease of the molecular tilt angle in the center of the film occurs gradually, because we did not observe a substantial decrease of anisotropy in thin films at transitions with the change of the **c**-director orientation.

Two reasons may be responsible for the formation of a coplanar structure in films above the SmC_A^* phase. At low temperatures, the condition for the reorientation of the tilt planes arises only in the central part of the film because of surface freezing, which prevents the formation of a short-pitch structure. The second reason is related to peculiarities of fluctuations of θ in SmA films [47]. In the bulk sample, sinusoidal fluctuations in perpendicular planes can assemble with an arbitrary phase shift, which leads to coplanar, circular or ellipsoidal fluctuations and to possibilities of the formation of tilted pitch structures at a second-order transition. As pointed out in [47], unlike in the bulk sample, two fluctuation modes have the same or the opposite phases in films because of the symmetry with respect to the center of the film, which leads to only coplanar fluctuations and to the transition to coplanar tilted structures. Coplanar structures in thin films may also reflect the interlayer molecular organization in thick films. In optical experiments [20–22] in the range of the SmC_α^* phase, the films possess optical anisotropy and polarization. These may not only be related to surface effects but also be an indication of a distorted staircase character of the SmC_α^* phase in thick free-standing films.

In summary, using depolarized light microscopy and polarized reflectivity, we observed coplanar structures in free-standing films above the SmC_A^* phase. The number of transitions depends on the film thickness, its oddity, and the value of the electric field. Temperature- and field-induced transitions occur as the direction of the electric polarization changes with respect to the tilt plane.

This work was supported in part by the Russian Foundation for Basic Research (project No. 01-02-16507) and a Grant-in-Aid for Scientific Research (№ 12650010) through the Japan Society for the Promotion of Science. We acknowledge very stimulating discussions with D. R. Link and H. Takezoe. One of us (P. V. D.) thanks Shinshu University for his stay as a Foreign Visiting Researcher.

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