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Chirality and polarity transfers between bent-core smectic liquid-crystal substances

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Liquid-crystal phases with both solid and liquid characteristics are unique, since they exhibit chirality on both molecular and mesoscopic levels under special circumstances. We have studied binary composites formed by mixing of a chiral dimer (B-Ch), in which a bent core is covalently linked to a promesogenic cholesterol unit through a flexible spacer, and an achiral bent-core mesogen having two vicinal fluorine atoms (B-2F). The pure materials show smectic phases featuring C_2 symmetry. The dimer B-Ch displays conventional chiral smectic C (SmC^*) phase with a chirality-induced polarization $P_c \sim 30$ nC/cm², whereas B-2F possesses synclinc and anticlinic antiferroelectric “banana” smectic ($SmCP$) phase with a relatively high-polarization (P_b) value of about 900 nC/cm². Mixing these two materials we were able to measure P_c , and we have verified that the molecular chirality contributes to the polarization of banana phases only in synclinc configurations.

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INTRODUCTION

Chirality has been studied for centuries; however, the mechanism of chirality transformations between different length scales is poorly understood. In liquids, macroscopic chirality requires chirality at the molecular level, whereas solid materials can be chiral without chiral molecules. Because chiral materials do not have inversion symmetry, chirality is often related to macroscopic polarization. Liquid-crystal phases, which are a unique state of matter exhibiting the properties of both solid and liquid, display chirality (handedness) on both molecular and mesoscopic levels when they are formed by mesogens possessing certain molecular design features. In layered (smectic) liquid crystal phases, which possess two-dimensional fluid and one-dimensional solid characters, a macroscopic ferroelectric polarization can be induced if the constituent molecules are chiral and tilted with respect to the layer normal, which is generally referred to as the chiral smectic C (SmC^*) phase [1]. On the other hand, bent-shape (banana-shape) molecules can possess layer-scale chirality although they lack molecular chirality [2]. This is due to directed polar packing of the molecules and a tilt of the molecular plane with respect to the smectic layer normal, representing another form of the chirality-polarity-tilt relations. Since these bent-shape molecules (at least those studied first) do not contain any chiral carbons, smectic layers can form two different structures that are non-superposable mirror images of each other. If the smectic phase is ferroelectric [3–5] and synclinc ($SmC_S P_S$), or if it is antiferroelectric [6] and anticlinic ($SmC_A P_A$), then the material has a homogeneously chiral structure, with the same sign of chirality in all layers. By contrast, if the smectic phase is ferroelectric and anticlinic ($SmC_A P_S$), or if it is antiferroelectric and synclinc ($SmC_S P_A$), then the material has alternating right- and left-handed chiral layers, with alternating positive and negative chirality. This latter possibility is generally called a “racemic” structure, although we might use the alternative term “antichiral” [7] to emphasize the rigid alterna-

tion from layer to layer. By applying a sufficiently large external electric field the antiferroelectric (AFE) arrangement can be easily switched to a ferroelectric (FE) which can keep or flip [8] the original layer chirality, or can alter racemic domains to chiral or vice versa [9,10].

Chirality can also be introduced when one or more chiral carbons are incorporated in the bent-shape molecules, for example, in the hydrocarbon terminal chains [11,12], within the bent core [13], or by addition of chiral dopants [14]. It was noted during the early work that the handedness of the homochiral structures is very sensitive to chiral dopants, or even to chiral surfaces [15]. On the other hand, it was observed that banana smectics made of enantiomeric chiral molecules form synclinc-antiferroelectric and anticlinic-ferroelectric [11] domains. This combination of tilt and polar order implies that the phase is racemic, with a rigid alternation of right- and left-handed chiral layers. This shows that the molecular chirality has no or minor effect on deciding about anticlinic or synclinc packing (which is mainly determined by entropic reasons [16]), but it can bias the otherwise degenerate tilt directions.

Experiments by Binet *et al.* [17], carried out on chiral bent-core materials and on achiral biphenyl core molecules with chiral dopants, reveal that in addition to the polarization P_b , due to the closed packing of the bent-shape molecules, another polarization, P_c is introduced due to the chiral and tilted molecular structure. It was found [17] that in the antiferroelectric racemic domains at low fields, P_b of the synclinc-racemic domains averages out but, due to the synclinc order and of the chiral molecules, a P_c normal to the tilt plane similar to the SmC^* phase is possible. In this case, a relatively low electric field is able to unwind the helical structure, but would not be able to switch the antiferroelectric P_b , which requires a higher threshold. Upon this antiferroelectric to ferroelectric transition, the synclinc structure becomes anticlinic and P_c vanishes, leaving only a P_b . Although the textural observations were consistent with this picture, it was not possible to measure P_c separately, because in case of the enantiomeric molecules isolated metastable

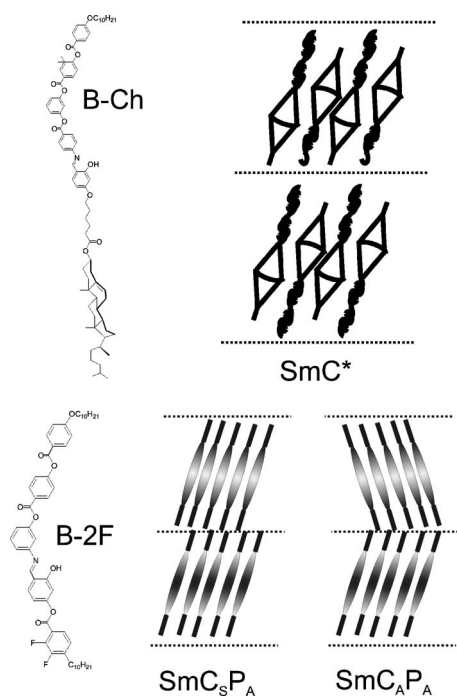


FIG. 1. Molecular structures and the mesophases of the components of the studied mixtures. The bulky cholesterol side chain of the B-Ch molecules prohibits the polar closed packing of the bent cores, i.e., they can be modeled as combination of two cones, resulting in a calamitic SmC^* structure. On the other hand, B-2F molecules may pack closely in antiferroelectric order in the direction perpendicular to the tilt plane corresponding to synclinic $SmC_S P_A$ and anticlinic $SmC_A P_A$ structures at different temperatures.

chiral (anticlinic-antiferroelectric) domains are also present, where both P_c and P_b average out below the transition to the ferroelectric state where the effective polarization becomes $P_b + P_c$.

In this paper we report studies where we were able to separate the chirality-induced polarization P_c from the bent-core packing related polarization P_b by studying dielectric, electro-optical, polarization properties of binary mixtures of low molar mass chiral and achiral bent-core molecules.

MATERIALS

The molecular structures of the bent-core components of the binary mixtures are shown in Fig. 1. The first component is 4-((3-(4-(2-hydroxy-4-(5-(cholesteryloxycarbonyl)pentyl)oxy) benzylideneamino benzoyloxy)phenoxy)carbonyl)phenyl 4-(decyloxy)benzoate, hereafter referred to as B-Ch, in which achiral bent core and the promesogenic cholesterol (as a chiral entity) units are axially tethered through a paraffinic spacer to form a dimer [18]. It exhibits a wide thermal range of amorphous blue phase (BPIII) during cooling from the isotropic phase between 174.5 °C and 153 °C, which is followed by a short pitch cholesteric (N^*) phase down to 138.3 °C, where a transition to a SmC^* phase occurs. The SmC^* is stable down to 52 °C where it becomes glassy, which eventually crystallizes

out [18]. On heating from the crystal state, it directly melts to the N^* phase, which then turns to BPIII state at 7 °C below the clearing point of 175 °C. The SmC^* phase of the bent-core B-Ch means that the bulky cholesterol side chain prohibits the polar closed packing of the bent cores, i.e., the bent cores can basically freely rotate around the long axis (along the line connecting the average two ends of the molecules). On an average, the bent-core units therefore can be modeled as combination of two cones, which is a calamitic structure [18]. The spontaneous polarization therefore is arising only from the chirality-director tilt duality (Meyer’s mechanism [1]), and not due to the closed packing of the bent cores. This picture is consistent with the measured value $P_s \sim 30$ nC/cm², which is an order of magnitude smaller than typical for “banana smectics.” The tilt angle in the whole SmC^* phase is very near to 45°, which is typical for the SmC^* phase with direct transition from the N^* phase. As a consequence of the molecular chirality, the director has a helical structure with submicron size pitch in SmC^* phase range.

The other material is 4-[(3-[4-[4-(decyloxy)benzoyloxy]benzoyloxy}phenylimino)methyl]-3-hydroxyphenyl 4-(decyloxy)-2,3-difluorobenzoate [19], we abbreviate as B-2F, expressing the presence of the two fluorine atoms attached to one of the benzene rings of an arm. B-2F has antiferroelectric polar banana $SmCP$ phases with as large as 900 nC/cm² polarization value. The higher temperature phase is synclinic ($SmC_S P_A$) in between 143.3 °C and 114.5 °C. Due to the synclinic structure it shows racemic switching, which eventually transfers to a chiral state when strong rectangular electric field is applied for a few minutes. After field removal an anticlinic chiral state forms and the original racemic synclinic state reforms only gradually within an hour. The lower temperature phase is anticlinic ($SmC_A P_A$) even at zero field and shows linear electro-optical switching almost down to room temperature. Similar to the B-Ch material in the SmC^* phase, B-2F also has a large ($\sim 45^\circ$) tilt angle in the $SmCP$ phases.

RESULTS

Textural observations were made by a combined Kofler’s contact method and by investigating the textures in ten different concentrations. The phase transitions were identified by textural observations, polarization measurements, dielectric studies [Fig. 2(a)] and by their electro-optical properties [Fig. 2(b)].

It was found that basically three distinctly behaving concentration regimes can be distinguished.

Regime I: Mixtures containing less or equal than 40 wt % of B-2F behave similarly to the pure B-Ch. In this range, the blue phase and the cholesteric range continuously shrink and disappear at about 20 wt % and 40 wt % of B-2F concentrations, respectively. The smectic range however only slightly changes. The smectic textures forming under cooling without electric fields are similar to that of the pure B-Ch, i.e., they have low birefringence with chiral patterns indicating tilted layer structure. Under relatively low bipolar electric fields, the optical axis rotates back and forth. In this initial state no

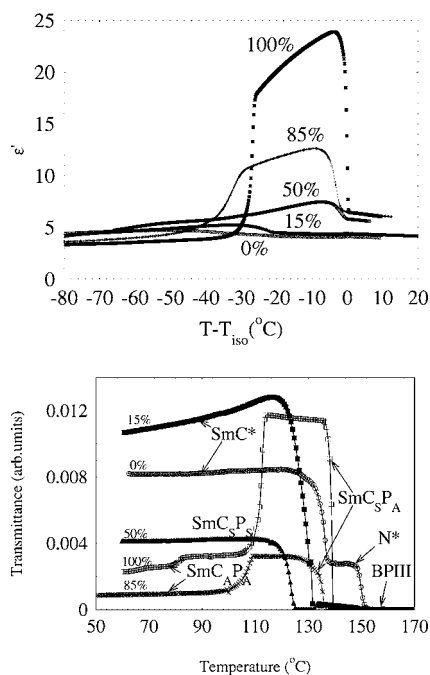


FIG. 2. (a) Real part of dielectric constant measured at 2 kHz as the function of relative temperature and (b) the temperature dependences of the optical transmittances between crossed polarizers of mixtures with different concentrations (in wt % of the B-2F compound).

(or very broad) polarization peak can be resolved in the polarization current under triangular electric field. However, just as for the pure B-Ch, after high-field treatment the texture gradually transfers to a focal conic texture and ferroelectric polarization peak appears (see Fig. 3). The magnitude of the polarization, calculated from the area below the polarization peak, is slightly decreasing from 30 nC/cm² to about 20 nC/cm² at 40 wt %. As the molecular dipoles of the B-2F molecules are much larger than of B-Ch, the fact that B-2F molecules do not lead to an increase of the polarization in the low-concentration range may indicate a locally antiferroelectric packing of the B-2F molecules. Although the B-2F molecules should suppress the free rotation of the B-Ch molecules around the long molecular axis, we assume that at low concentration this distortion is weak (see Fig. 3) and the polar packing of the bent cores may appear only in short (submicron size) range. This means that the phase macroscopically behaves as SmC*. In this state at low-frequency electric field reversals, the texture does not show optical change between crossed polarizers, but the optical axis rotates by ±45°, which can be seen when a birefringent wave plate is inserted at 45° between the crossed polarizers. When the field is removed, fine stripes form, which eventually fill the whole area and the stripes cannot be resolved indicating that the fully formed helical structure has a submicron size pitch.

The dielectric constants of these mixtures are low [see Fig. 2(a)], and are slightly increasing from maximum of four to about six at increasing B-2F concentrations. In SmC* materials, the low-frequency dielectric susceptibility is due to the rotation of the director around the tilt cone and can be

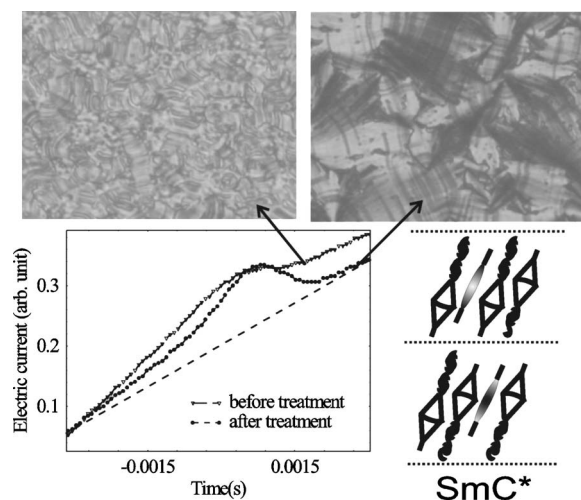


FIG. 3. Typical textures, polarization current curves and proposed mesophases structure of mixtures in Regime I. Upper row: Microphotographs of the textures of 4.5 μm thick samples of a mixture with 6 wt % of B-2F at 130 °C. Left-hand side: virgin texture under $E_{DC}=7 \text{ V}/\mu\text{m}$; right-hand side: at $E_{DC}=7 \text{ V}/\mu\text{m}$ after 10 min. Pictures represent 400 μm × 300 μm areas. Lower row: Time dependences of the polarization currents under triangular voltage excitations before and after high-field treatment; and the proposed structure of the SmC* phase.

expressed [20] with the material parameters as $\chi = (P/q\theta)^2/2\epsilon_o K$, where $P \sim 30 \text{ nC}/\text{cm}^2$ is the spontaneous polarization, q is the wave number of the helical structure, $K \sim 10^{-11} \text{ N}$ is the twist elastic constant, and $\theta \sim 0.8$ is the director tilt angle. The observed $\chi \sim 1-3$ values therefore give that the helical pitch $p=2\pi/q$ increases from 0.25 μm and 0.42 μm as the B-2F concentration increases from zero to 40%. These values are comparable to the pitch in the cholesteric phase, and are in accordance with textural observations. The relaxation frequency $f_r \approx Kq^2/\gamma$ increases with the B-2F concentration from 4.6 kHz to 8 kHz, which with the calculated periodicities give that the rotational viscosity is decreasing from 1.5 to 0.5 Pas in this range. This is consistent with the decreasing average size of the molecules (see Fig. 1) at increasing B-2F concentration.

Regime II: (40 wt % < c_{B-2F} < 70 wt %) In this intermediate concentration range the smectic phase appears directly below the isotropic fluid in form of helical filaments and other exotic chiral superstructures, typical of B₇ textures [21]. The static dielectric constant is still low and its maximum reaches only 7 (Fig. 2). The textures show linear electro-optic response only at very high fields (about 20 V/μm). Similar to that observed previously in some B₇ materials [5], above these fields the texture breaks into small needlelike domains, which rotate with the frequency of the fields (see Fig. 4). Simultaneously with this texture formation, a single polarization peak appears in each half period of the triangular electric wave forms with integrated area corresponding to $P_s \sim 70 \text{ nC}/\text{cm}^2$. Below a transition temperature of about 120 °C this peak disappears, and another polarization peak appears at low fields. The magnitude of this low-field polarization is about 30 nC/cm², slightly decreasing to

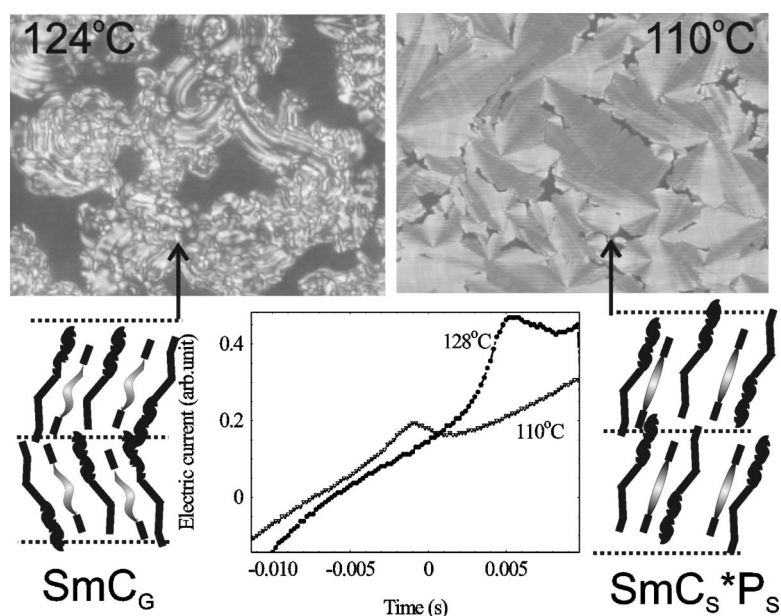


FIG. 4. Textures, polarization currents, and proposed structures in regime II. Upper row: Photomicrographs ($0.4 \text{ mm} \times 0.3 \text{ mm}$ areas) of $4.5 \text{ }\mu\text{m}$ films of 63 wt % of B-2F at zero field in the anticlinic ($124 \text{ }^\circ\text{C}$), and at $22.2 \text{ V}/\mu\text{m}$ $f = 33 \text{ Hz}$ rectangular fields in the synclinic phase ($110 \text{ }^\circ\text{C}$). Lower row: polarization current curves at $128 \text{ }^\circ\text{C}$ and $110 \text{ }^\circ\text{C}$ (at center) and the proposed corresponding structures (at sides).

ward lower temperatures. In this temperature range, steady fan-shaped domains can be seen between crossed polarizers with extinction direction at 45° with the smectic layers. Similar to that observed on some single bent-core compounds with relatively large terminal chains [9], the chiral switching could be transferred to racemic type under triangular fields, which then could be transferred back to chiral when rectangular field was applied for a few seconds. This observation clearly shows that the phase is of SmCP type and not of SmC^* , indicating an isotropic- B_7 - B_2 phase sequence, which has been observed several times [5,22] and discussed in the literature [23].

Regime III: At or more than 70% of B-2F the mixtures resemble to the pure B-2F substance. The optical transmittance shows a transition from a high birefringent texture (indicating synclinic configuration) to a low one (indicating anticlinic structure) at about $20 \text{ }^\circ\text{C}$ below the clearing point.

This transition is reversible and also shows up in the dielectric susceptibility, which decreases from a high value (25 for pure B-2F and 10 for the 75 wt %) to about 3 at the transition to the anticlinic state (Fig. 2). The time dependence of the polarization current shows a ferroelectric-type single peak at relatively low fields, very similar to regime I (Fig. 5, lower row at center). Such a peak is absent during the switching of the pure B-2F, and amounts to a polarization of $75 \text{ nC}/\text{cm}^2$ at 20 wt % of B-Ch content, indicating that it is due to the presence of chiral B-Ch component. However, in contrast to regime I mixtures, above a temperature and concentration dependent threshold the polarization current shows antiferroelectric switching, similar to the pure B-2F material (Fig. 5, lower row at center). It is important to note that once the antiferroelectric peaks appear, the low-field ferroelectric peak disappears and the texture becomes dark between crossed polarizers indicating the formation of anti-

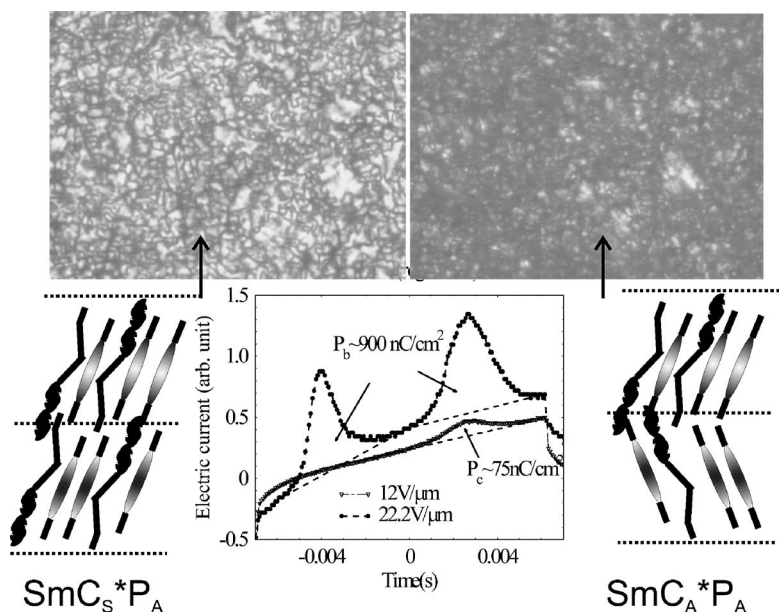


FIG. 5. Textures, polarization currents, and proposed structures in regime III. Upper row: Photomicrographs ($0.4 \text{ mm} \times 0.3 \text{ mm}$ areas) of $4.5 \text{ }\mu\text{m}$ films of 80 wt % of B-2F in the synclinic phase ($117 \text{ }^\circ\text{C}$) and in the anticlinic range ($90 \text{ }^\circ\text{C}$). Lower row: polarization current curves at $130 \text{ }^\circ\text{C}$ at different applied voltages (at center) and the proposed corresponding structures (at sides).

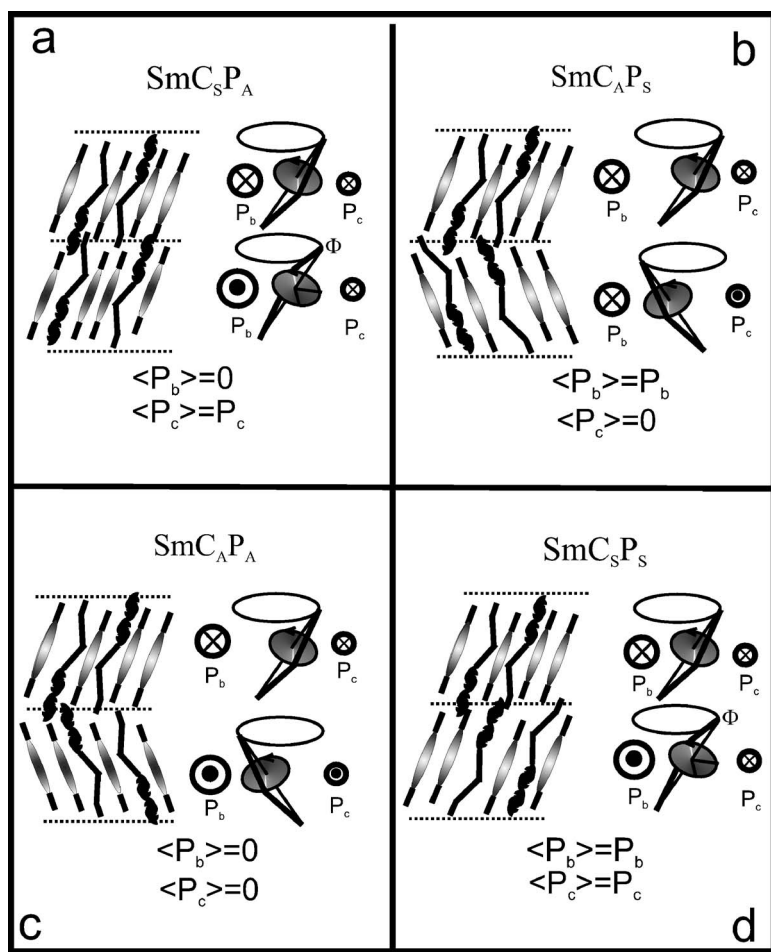


FIG. 6. Illustration of the interplay between P_b and P_c . (a) $\text{SmC}_S P_A$, (b) $\text{SmC}_A P_S$, (c) $\text{SmC}_A P_A$, and (d) $\text{SmC}_S P_S$ structures.

clinic structure. Similar to the pure B-2F, the low birefringent state (with optical axis parallel to the smectic layers) remains metastable after field removal.

DISCUSSION

By mixing strongly chiral B-Ch bent-core molecules with strongly polar achiral ones (B-2F), we identified three distinct regimes.

In regime I, which is dominated by the chiral molecules, the most interesting result is the decrease of P_c with increasing B-2F concentration, which can be explained by the assumption of antipolar packing of the B-2F molecules.

In regime II, where both the molecular chirality and the polar packing have importance, we have observed the formation of a B_7 -type phase. Although we have no direct evidence for the molecular packing in this phase, we wish to put forward the following model that may explain the electro-optical observations. Molecular chirality usually transfers to microscopic ranges and induces a helical structure. In calamitic SmC^* phase helix can form only by varying the azimuth angle of the c director, however in chiral bent-core materials a helix can form also by rotating the angle ϕ between the molecular planes and tilt planes. The rotation of ϕ means that $\phi \neq 90^\circ$ or $\phi \neq 270^\circ$ states, which have C_1 symmetry with out of plane polarization components [24], should ap-

pear periodically. In such structures the layer chirality varies between zero and the maximum of the B_2 phase, representing a direct transformation of the molecular chirality to the layer-scale chirality. Together with twisting in-plane polarization components this structure corresponds to a combined splay and twist polarization modulation. This is different from the splay modulated polarization structure of the B_7 materials without molecular chirality proposed by Coleman *et al.* [25], because there the splay had only in-plane components, whereas now the splay is normal to the layers, and is due to the molecular chirality. Since the out-of-plane components periodically have opposite directions, during switching regions with thickness of half-periods should rotate in opposite sense, thus explaining the observations that switching above this threshold involves breaking of the continuous texture to equally thick needles. This preliminary model therefore is corroborated with earlier models suggesting that some of the B_7 textures have an out-of-plane polarization component (SmC_G) structures [26].

The most important result of our observations is that in regime III below the antiferroelectric-to-ferroelectric bent-core packing transition, the molecular chirality induces a chirality-related polarization P_c , which could be separated from the bent-core packing related polarization P_b . P_c is found to be an order of magnitude smaller than P_b , but larger than that of the pure B-Ch. This is because the molecular dipoles of B-2F with highly polar fluorine atoms are much

larger than of B-Ch. We have also verified the combinations of the P_c and P_b polarizations in the four possible SmCP structures, as illustrated in Fig. 6. In the antiferroelectric racemic domains at low fields, P_b of the synclinc-racemic domains averages out but, due to the synclinc order and of the chiral molecules, a P_c normal to the tilt plane similar to the SmC* phase appears [see Fig. 6(a)]. In this case, a relatively low electric field is able to switch P_c , by rotating the synclinc-antiferroelectric double layers around the tilt cone, but is not able to switch the antiferroelectric P_b , which requires a higher threshold. Upon the antiferroelectric-to-ferroelectric transition, the synclinc structure becomes antclinic and P_c vanishes, leaving only a P_b [Fig. 6(b)]. At lower temperatures when the antclinic-antiferroelectric phase forms, both P_c and P_b average out in the ground state [Fig. 6(c)]. Upon the transition to the ferroelectric state, the effective polarization becomes either P_b+P_c or P_b-P_c [Fig. 6(d)].

Summarizing, we have found three distinctly behaving concentration ranges when mixing chiral and achiral bent-core molecules. Mixtures with low B-Ch content show a ferroelectric polarization P_c at low fields and antiferroelectric banana P_b at high fields. Mixtures of intermediate concentrations are found to have ferroelectric banana polarization P_b at high-temperature ranges and a chiral polarization P_c at lower temperatures. The B-Ch dominated mixtures have lower P_c than of the pure B-Ch, whereas in the B-2F range P_c is larger than of the pure B-Ch. This measurement directly verifies the existence and magnitude of chirality-induced polarization P_c in bent-core liquid crystals.

ACKNOWLEDGMENT

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