Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl19

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Version of record first published: 24 Sep 2006

To cite this article: D. N. Stoenescu, H. T. Nguyen, P. Barois, L. Navailles, M. Nobili, Ph. Martinot-Lagarde & I. Dozov (2001): Optical Studies of Chiral Mesophases in Sandwich Cells with Planar Degenerated Anchoring, Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals, 358:1, 275-286

To link to this article: http://dx.doi.org/10.1080/10587250108028287

Mol. Cryst. and Liq. Cryst., 2001, Vol. 358, pp. 275-286
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Optical Studies of Chiral Mesophases in Sandwich Cells with Planar Degenerated Anchoring

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We use a surface treatment allowing to obtain planar anchoring, azimuthally degenerated, by deposition of a silane coupling agent (GLYMO) on an isotropic substrate (bare glass plate). This technique of "passivation" prevents the liquid crystal adsorption on the substrate and avoids memory effects. We present a method of measure of the spontaneous geometric parameters for chiral mesophases by simple optical observations, using a cell with variable thickness and azimuthally degenerated planar anchoring on one of the plates. The cholesteric and the TGB phases were studied for two : $12F_2BTFO_1M_7$(TGB_C) and $10F_2BTFO_1M_7$ (TGB_A). The cell allows to observe the slabs, even for the TGB_A phase, and to measure independently their thickness and twist. Our results are in agreement with previous X-ray measurements.

Keywords: anchoring memory; passivation; ted anchoring; pitch; cholesteric; TGB phase

INTRODUCTION

The interactions between a solid substrate and a liquid crystal result in an "anchoring" of the liquid crystal surface director $\mathbf{n}_s$ along some "easy axis" $\mathbf{n}_e$. The strength of the anchoring is defined by the anchoring energy $W(\mathbf{n}_s, \mathbf{n}_e)$, the anisotropic part of the surface tension. Approximately, W can be decomposed in two parts, zenithal (out-of-plane) and azimuthal (in-plane). The anchoring symmetry and strength are defined by the substrate anisotropy. The liquid crystal (LC) - substrate interface is always anisotropic along its normal N, resulting in a strong zenithal anchoring, usually monostable (homeotropic $\mathbf{n}_s$ ° N, planar $\mathbf{n}_s$ Á N, or tilted). In the plane of the interface, however, the anisotropy is arbitrary and

depends on the substrate nature and treatment. The related azimuthal anchoring can be monostable or multistable (several discrete easy axis directions) and its strength can vary in large limits. The reported azimuthal anchoring extrapolation lengths $L_a = K / W_a$ (here K is a bulk elastic coefficient and W_a is a surface one) vary from less than 50 nm[1] (very strong anchoring) up to a few μm[2] (extremely weak anchoring). For isotropic surfaces, e.g. bare untreated glass, one expects even zero anchoring strength ($L_a \to \infty$) and anchoring degeneration - the easy axis becomes an easy plane ($\mathbf{n}_s \perp \mathbf{N}$) or easy cone (for tilted $\mathbf{n}_s$). In this case the surface does not impose any particular direction to $\mathbf{n}_s$ and the azimuthal anchoring torque vanishes.

The azimuthal anchoring degeneration in liquid crystals is typical for isotropic fluid interfaces or free-standing films[3]. For isotropic solid substrates, however, it is difficult to obtain, because of the anchoring memory. On contact with the mesophase the substrate is modified and becomes anisotropic, mostly by adsorption of an oriented layer of liquid crystal molecules[4]. This adsorbed layer serves as a new anisotropic substrate, imposing relatively strong (L_a<50 nm for 5CB on glass) and monostable anchoring. At short term the local easy axis orientation, often inhomogeneous, keeps the memory of the first LC-substrate contact. At long term, $\mathbf{n}_e$ can drift under external torques, e.g. by slow desorption-readsorption of the anisotropic adsorbed layer[5].

Up to now the anchoring memory has been the main difficulty for the realization of weak or degenerate anchorings on solid substrates. Recently, several approaches to avoid the memory effects have been proposed and demonstrated in nematics. Bryan-Brown et al.[6] showed that a thin layer of oligomeric impurities, spontaneously segregated at the surface, screens completely the memory and gives degenerated anchoring. Recently, Martinot-Lagarde et al.[7,8] proposed that the surface anchoring memory can be "passivated" by adsorbing and fixing on the substrate a thin layer of the isotropic silane coupling agent γ-Glycidoxy propyl trimethoxy silane (GLYMO). All the available adsorption sites being occupied by the passivation agent, there is no more oriented layer adsorption and planar degenerated anchoring is observed. A conic degeneration of nematics has been demonstrated on grafted polymeric brush with highly mobile chains[9]. In this anchoring memory "lubrication" approach, the liquid crystal molecules penetrating the brush or adsorbed on it, can freely reorient together with the "fluid" polymeric chains.

The anchoring constraints, imposed by the boundaries, are transmitted in the bulk by the liquid crystal elasticity. Depending on the anchoring conditions different textures can be induced in the same phase. For example, the nematic (N) phase, spontaneously uniform, can be twisted up to 90° in a sandwich cell by suitable anchorings on the plates. Most chiral phases, e.g. cholesteric (N*) and chiral smectic C (SmC*) are spontaneously twisted. Due to boundary constraints, in thin cells they can be partially or completely untwisted. Even in thick cells,

strong boundary conditions impose "quantification" of the twist angle at integer number of half-turns. In wedge cells this gives the well-known Grandjean-Cano lines[3], on which the total twist angle jumps by π or 2π.

Both strong anchoring and memory impose pitch values different from the spontaneous one P. Even for well studied phases, like N*, this can pose some problems when measuring the pitch variation with the temperature or under external fields. To observe the spontaneous pitch one needs degenerate (or extremely weak) anchorings. This will be advantageous also for more complicated (and less well understood) chiral systems, like the twist grain boundary (TGB) phases. In these phases the nematic and smectic orders are in conflict[10]. Due to the chirality, the nematic director likes to twist, much like in the N*. This twist, however, is forbidden by the smectic layering. In result, the TGB phase is organized in slabs, separated by defect walls, the twisted grain boundary (GB), with smectic order well developed in each slab. At the GB, the smectic wave vector jumps, enabling the director twist within the slab and from one slab to another. In the last ten years the TGB phases have been intensively studied by optical[11,12], electrooptical[11,13] and X-ray techniques[14,15], but up to now their structure is not well established.

The pitch temperature dependence is measured with reasonable precision from the Grandjean-Cano lines in wedge cells[11,12,16], but with an important hysteresis, due to memory effects. The twist angle per slab is accessible from X-ray experiments for commensurate TGB_C[14,15,16] and recently for TGB_A and TGB_C in very thin cells (a few μm)[15]. The very existence of the commensurate TGB_C phase has been questioned[17] and attributed to surface anchoring constraints. The nature of the defects on the GB has also been questioned[18] and it has been proposed that in TGB_C the dislocation line cores melt together to give a planar defect, a melted GB.

The study of the TGB phases under free boundary conditions, without surface torques, could contribute to their further understanding. This kind of study has been impossible up to now, due to the difficulty to realize free-standing films or free drops with planar anchoring in the TGB phase - the smectic layers are anchored strongly parallel to the LC - air interface. However, recent advances in the anchoring memory control enable now the realization of sandwich cells with free azimuthal anchoring.

Here we apply the anchoring memory passivation by GLYMO for the study of the N* and TGB phases. We use sandwich cells with variable thickness and azimuthally degenerated planar alignment on one of the plates. In absence of anchoring constraints on this plate, we observe textures twisted at the spontaneous twist angle, without Grandjean-Cano lines. We measure the pitch by simple optical transmission technique. In the TGB phases we observe optically the slabs (even for the TGB_A phase) and we measure their thickness and twist.

SAMPLE PREPARATION

Our "wedge" sandwich cell (Fig.1) with variable local thickness d(r), are constructed with one flat plate and one low curvature (R ~ 1 m) spherical glass lens. In this geometry d(r) vary in large limits and can be measured with good precision from the known lens curvature (Fig.1). A special sample holder enables the variation of the cell gap d(0) between 0 and ~100 μm. The cell is placed in an electrical oven with temperature control precision of 25 mK and is observed by transmission polarization microscopy.

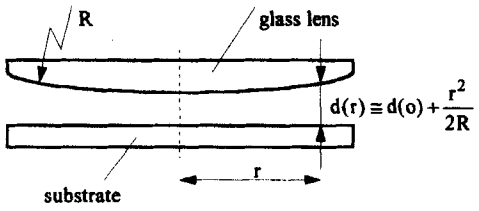

FIGURE 1. Experimental geometry of the cell

The lens surface is covered with vacuum deposited SiO layer (incidence angle 60°, normal thickness 45 nm). This treatment imposes strong planar anchoring for most nematics, and gives us a well defined uniform orientation on the lens. On the opposite flat substrate we realize a memory-free azimuthal degenerated planar anchoring, enabling free in-plane rotation of the surface director, without anchoring torque. We achieve this by anchoring memory "passivation" of the glass substrate[7,8]. The plate is spin coated with a low concentration (0.7 %) GLYMO solution in water and isopropanol. Then it is baked for one hour to cross-link and stabilize the film. The anchoring properties are checked in a separate experiment with the room-temperature nematic 5CB. Although the anchoring can change with the LC compound and phase, our observations confirm that the 5CB based data are relevant also for the present study. With 5CB we obtain a strong planar zenithal anchoring, with extrapolation length of about 37 nm[8]. At short term (<1 day) we observe completely degenerated azimuthal anchoring, with free rotation of $\mathbf{n}_s$ on the passivated surface. At this time scale we do not detect any anchoring memory. In a few days the surface orientation is memorized and a weak anchoring is built up by memory for this direction. The anchoring strength increases slowly and in about one week saturates at a value corresponding to an azimuthal extrapolation length L_a ~ 5 μm. Although measurable, this anchoring can be considered as vanishing for the present experiment (for $P < L_a$ the surface torques can be neglected, compared to the strong bulk ones).

We study in this work two chiral mesogens with rich phase behavior : $12F_2BTFO_1M_7$ (crystal - SmC* - TGB_C - N* - Blue Phase (BP) - Isotropic (I)) and $10F_2BTFO_1M_7$ (crystal - SmC* - TGB_A - N* - BP - I). These mesogens have been choosed because their TGB phases are among the best characterized[11,14], giving us a basis for comparison.

We fill the liquid crystal in the cell in the isotropic phase and to minimize the residual memory effects we keep the cell at this temperature for a few hours before to start the experiment. For the same reason we avoid to remain for a long time with a fixed local surface orientation and between the experiments we heat the cell again to the isotropic phase.

EXPERIMENTAL OBSERVATIONS

a) Cholesteric phase

Decreasing the temperature, we start our observations. We detect the blue phase by a texture variation, but the optical contrast in this phase is too low for unambiguous interpretation of our data. In the N* phase, under crossed polarizers, the sample presents periodic circular fringes on dark field (Fig. 2), each fringe corresponding to a constant thickness of the cell. At high temperature (Fig. 2a) the transmission of the grey - blue "bright" fringes is very low - less than 1%. Decreasing the temperature (Fig. 2b), the fringes transmission increases up to more than 10%, their color changes toward yellow, they move continuously outward from the center of the cell and their period increases. When we vary the cell gap, the fringes move without hysteresis and without sticking on the walls, in order to remain at the same thickness.

The best contrast of the fringes is obtained with a surface director on the lens parallel to one of the polarizers. When the cell turns the contrast decreases and a new fringe appears in the interfringes space. At 45°, the intensity of the new and old fringes equalizes (see Fig.5 for the same effect in the TGB phase).

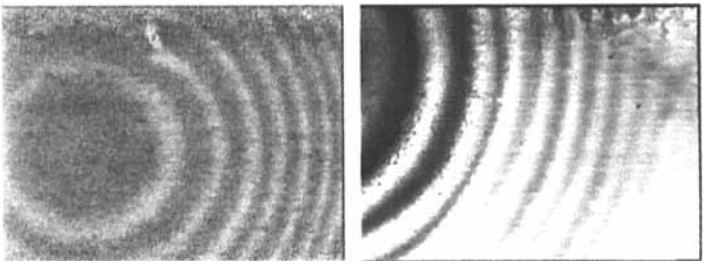

FIGURE 2. Cholesteric phase of $12F_2BTFO_1M_7$ between crossed polarizers, close to the transition N*-BP (left) and the transition N*-TGB_C (right).

The observed textures are quite different from the usual Grandjean textures in wedge-like planar cells. We do not observe the Grandjean-Cano lines, nor any other bulk or surface defects (except for thick samples for which memory effects are not fully suppressed). All the observed features are compatible with azimuthally free anchoring on one of the plates. The helical axis is normal to the plates, with constant azimuth imposed on the lens surface and azimuth $2\pi d/P$ on the GLYMO plate, continously varying with the thickness d and corresponding to the spontaneous pitch P. The fringes observed by us in transmission are quite similar to the Friedel fringes reported for wedge-like samples in reflection[19]. To understand the fringes periodicity, let us first consider the case when $P > \lambda$, but $\Delta n \cdot P < \lambda$, i.e. relatively slow twist, but fast enough to perturb seriously the wave guide regime of light transmission. In this case an analytical geometric optics approximation is valid[20,21], giving for the transmission:

$$T = \left[\cos(\alpha_A + \alpha_P)\cdot\cos(b) + \frac{\cos(\alpha_A + \alpha_P)\cdot\sin(b)}{\sqrt{1+a^2}}\right]^2 + \frac{a^2}{1+a^2}\cos^2(\alpha_A - \alpha_P)\cdot\sin^2(b) \tag{1}$$

where

$\alpha_A = \Phi_{out} - \Phi_A$, $\alpha_P = \Phi_P - \Phi_{in}$, $a = (\Delta n\, P/2\,\lambda)$ and $b = 2\pi d\sqrt{1+a^2}/P$.

Here Φ_P and Φ_A are the orientations of the two polarizers (of input and output respectively), Φ_{in} and Φ_{out} are the liquid crystal orientations on the two cell walls, $\Phi_{out} - \Phi_{in} = \Delta\Phi = (2\pi d/P)$ is the twist angle, Δn is the LC birefringence, P is the pitch and λ is the light wavelength. Choosing $\Phi_P = 0$ and $\Phi_A = 90°$, out of the Mauguin regime ($a \ll 1$) we obtain:

$$T_{(\Phi_{in}=0°)} \cong \left(1 - 3a^2/4\right)\sin^2\left(a^2\Delta\Phi/2\right) + a^2\sin^4(\Delta\Phi) \tag{2}$$

and

$$T_{(\Phi_{in}=45°)} \cong \left(1 - a^2/4\right)\sin^2\left(a^2\Delta\Phi/2\right) + \left(a^2/4\right)\sin^2(2\,\Delta\Phi) \tag{3}$$

From Eqs. (2) and (3) we see that the thickness dependence of the transmission T is a sum of two periodic contributions (Fig. 3). The first one consists of large "chromatic" fringes, i.e. with *λ-dependent* thickness period $\Delta d \cong \left(4\lambda^2/\pi\,\Delta n^2 P\right)$, with strong amplitude (~1). The second contribution is a set of small amplitude ($\ll 1$) "achromatic" fringes, with *λ-independent* period $\Delta d = P/2$ for $\Phi_{in} = 0°$ and $\Delta d = P/4$ for $\Phi_{in} = 45°$. These fringes can be unambiguously identified with the observed modulation of the transmission. Due to the λ-independent periodicity, they are easily detected in white light, their coloration coming only from the λ-dependence of the amplitude.

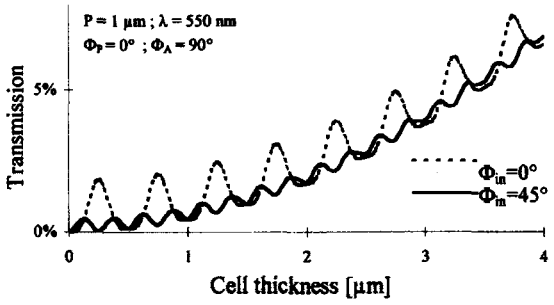

FIGURE 3. Thickness dependence of the optical transmission calculated for uniformly twisted cell between crossed polarizers. The LC orientation on the strong anchoring boundary is parallel (Φ_{in}=0°) or rotated at 45° (Φ_{in}=45°) to one of the polarizers.

For shorter pitches, i.e. $P < n_o\lambda$, the analytical approximation fails, but we obtain by numerical calculation (Berreman's 4x4 matrices method[22]) qualitatively the same behavior of the transmission (Fig. 3). This gives us an easy and reliable technique to measure P from the thickness dependence of the transmission minima. The experimental results for P(T), presented on Fig. 4, are in good agreement with the data obtained previously from the periodicity of the Grandjean-Cano defect lines[11]. We note that our pitch measurement technique is free from the difficulties, commonly arising in the Grandjean texture method - we have no hysteresis and we do not need to know the Burgers vector of the defect lines, often ambiguous.

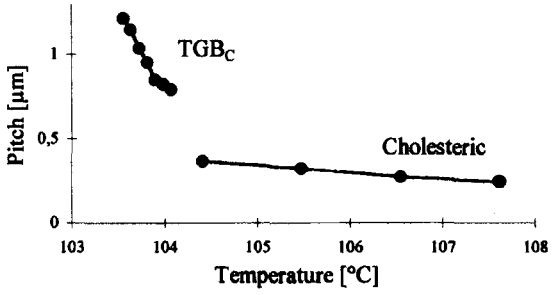

FIGURE 4. Temperature dependence of the pitch for $12F_2BTFO_1M_7$.

b) TGB_C phase

At the N* - TGB_C transition temperature of $12F_2BTFO_1M_7$ we observe a discontinuous change of the texture (Fig. 5). The color of the transmitted light changes from yellow in N* to blue in TGB_C, due to the pitch discontinuity. For the same reason, the periodicity of the fringes changes, enabling us to measure the jump of the pitch (Fig. 4).

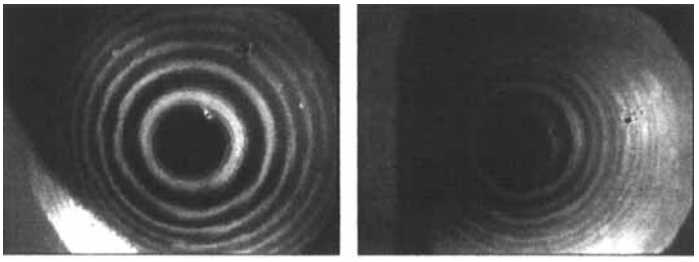

FIGURE 5. Image of the cell between crossed polarizers close to the transition N*-TGB_C with alignment on the lens parallel (left) or at 45° (right) to one of the polarizers.

The important feature is that defect lines appear superposed to the cholesteric-like texture. The periodicity of lines shows that they are not Grandjean-Cano disclinations - there is twenty lines per pitch! Comparing with previous studies, optical[11] and X-ray[16], we identify the lines as edge dislocations of the TGB slabs (Fig. 6). Between two lines the total number of slabs remains the same. At each line an additional TGB slab is created, in order to adjust the total number of slabs to the thickness variation. In this way, the pitch P, the slab thickness l_b and the twist angle per slab $\delta\varphi = 2\pi\, l_b/P$ conserve their spontaneous values.

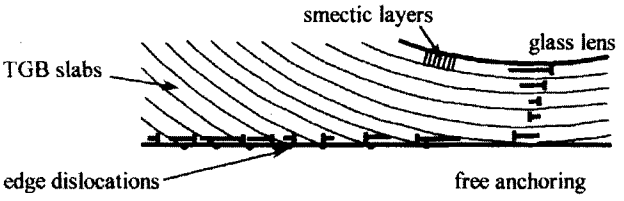

FIGURE 6. Schematic representation of the slab dislocations in the cell. The slab dislocations are on the GLYMO surface, minimizing the total energy.

When we vary the temperature or the cell thickness, both the fringes and the slab dislocations move, in order to adjust to the intrinsic values of P and $\delta\varphi$. There are no visible memory effects, but we observe some sticking of the defect lines on the surface irregularities, indicating that they are on the surface, like sketched on Fig.6, and not in the bulk. At decreasing temperature, l_b increases, the slabs in the center disappear one by one, new defect lines are created and move outward. The growing of the central defect line is strongly anisotropic, with an "easy" direction along the surface director on GLYMO (Fig. 7). This observation confirms that the slab dislocations are on the surface, as shown on Fig. 6, and not in the bulk.

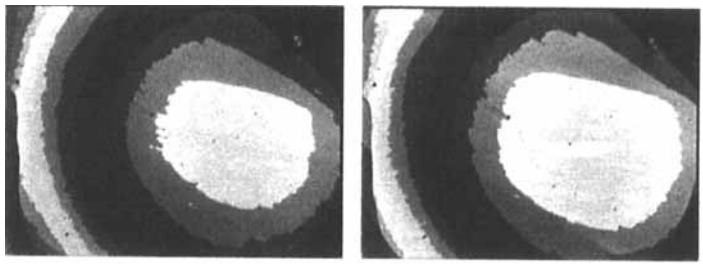

FIGURE 7. TGB_C texture between crossed polarizers. The holes in the slabs present a growing direction parallel to the local director on the GLYMO surface.

The slabs dislocation lines have been already observed optically in TGB_C wedge cells with traditional anchorings[16]. However, due to the strong anchoring and hysteresis it has been impossible before to measure optically $\delta\varphi$. This important structural parameter has been measured only by X-ray experiments in commensurate TGB_C phases[14,15]. Here we easily measure $\delta\varphi$, by counting the number of defect lines between the fringes. For T = 103.8°C we observe $N \cong 16$ slabs per pitch (P = 0.8 μm), giving $\delta\varphi \cong 22°$ and $l_b \cong 50$ nm. These values are in reasonable agreement with the reported X-ray data[14,15,16]. Our present precision, of ±1/2 slab per pitch, is not sufficient to distinguish commensurate from incommensurate phase, i.e. to distinguish integer ratio $2\pi/\delta\varphi$ from an arbitrary one. However, optical techniques[1] enable direct measurement of $\delta\varphi$ with ± 0.2° precision, which should be sufficient for definitive conclusion about the TGB_C commensurability phase. Local measurement of the twist in the domain between two slab dislocations can contribute to the understanding of the TGB_C elasticity and of the structure of the grain boundaries.

c) TGB$_A$ phase

For 10F$_2$BTFO$_1$M$_7$ no discontinuity of the pitch was observed at the N*-TGB$_A$ phase transition (Fig. 8). The pitch increases continuously as the temperature decreases. Simultaneously, the color of the sample changes. Due to the smallest twist angle per pitch ($\delta\varphi \cong 7°\div 9°$) the contrast of the slabs is weak in the TGB$_A$ phase. Therefore, it is difficult to observe the phase transition by the appearance of the edge dislocations. Away from the transition the edge dislocations of the slabs arrangement become perfectly distinguishable (Fig. 9), allowing to measure independently P and l_b. Both of them increase with decreasing temperature. Unlike the TGB$_C$ phase, where the number of slabs per pitch $N=2\pi/\delta\varphi$ is constant, in the TGB$_A$ phase N increases with increasing temperature, showing temperature variation of $\delta\varphi$.

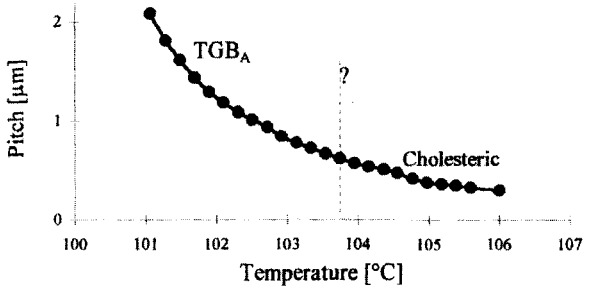

FIGURE 8. Temperature dependence of the pitch for 10F$_2$BTFO$_1$M$_7$. The variation is continuous. The threshold of the N*-TGB transition is not evident.

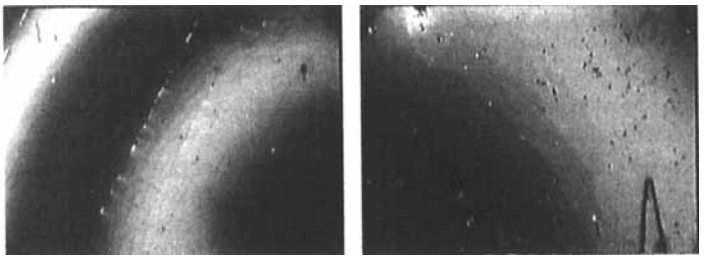

FIGURE 9. TGB$_A$ sample between crossed polarizers. The contrast of the defects is weak, due to the small value of $\delta\varphi$ (7°÷9°).

The Renn-Lubensky model[10] suppose that the GB consists of equidistant dislocation lines. The distance l_d between them is geometrically related to $\delta\varphi$ and to the thickness of the smectic layers m. For small values of $\delta\varphi$:

$$l_d = \frac{m}{2\sin(\delta\varphi/2)} \cong \frac{m}{\delta\varphi} \tag{4}$$

The model predicts a temperature independent ratio $l_b / l_d < 1$. Experimentally, taking $m = 38$ Å [16] we obtain a constant ratio $l_b / l_d \cong 0.85$ in good agreement with the Renn-Lubensky TGB_A model.

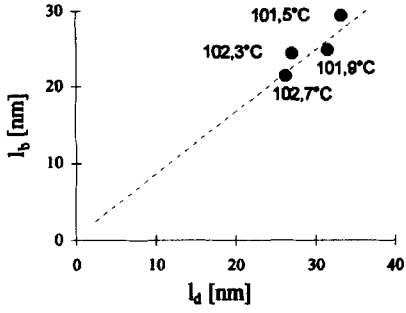

FIGURE 10. Dependence of the slab thickness l_b on the calculated distance between dislocations l_d in the TGB_A phase of $10F_2BTFO_1M_7$.

CONCLUSION

We propose here an original experimental approach to the study of the spontaneous structure of chiral mesophases, without surface imposed constraints. We realize an azimuthally degenerated and memory-free anchoring on one of the plates of a sandwich cell, "passivating" the anchoring memory with a thin GLYMO film, inhibiting the mesogen adsorption.

We study experimentally the cholesteric and the TGB phases of two mesogens in variable thickness cells. On the GLYMO-treated plate, with almost free azimuthal anchoring, we observe a continuous rotation of the surface director following the natural twist of the texture. We propose a simple optical technique for precise measurement of the spontaneous pitch.

In the TGB_C and TGB_A phases we observe also the slab edge dislocations. By simple optical observation we measure the structural characteristics of the TGB phases, like thickness of the slabs and value of the twist angle per slab. Our

results are in good agreement with previous X-ray measurements in the same mesogens.

In principle, our technique enables the structural measurements in thick cells, independently of the phase commensurability. It is less direct, but much simpler than the X-ray studies. Further development of the precision of the optical measurements should allow the study of the TGB elasticity, nature of the grain boundaries and intrinsic (or surface-imposed) commensurability.

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