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Free-standing smectic films at high temperature

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Optical reflectivity studies on free-standing liquid crystal films above the bulk smectic temperature range have revealed different melting phenomena. Our measurements are performed on tilted smectic phases (smectic C*, smectic C) using optical microscopy in polarized light in order to visualize the changes of the film structure. We observe the formation of two-dimensional defect structures from string-like lines in very thick (about 1000 layers) as well as in thin (about 20 layers) films. In thick films these structures nucleate around the temperature of the bulk smectic–cholesteric phase transition, while in thin films the formation of the defects occurs well above this temperature and just before the thinning transitions. In thick and intermediate thickness films, cholesteric or nematic droplets and a 'quasi-smectic' structure are observed. The films exhibiting the 'quasi-smectic' structure definitely exist at higher temperatures than the smectic films with the same thickness.

1. Introduction

In thin organic films, surface and finite-size effects lead to a number of phenomena unknown in bulk samples. Free-standing smectic films are convenient systems for investigation of these phenomena. These films with two flat surfaces bounded by air can be prepared with various numbers of smectic layers. After preparation, films generally have thickness non-uniformity and dislocations. But owing to the high molecular mobility, the quality of the film can be improved by holding the film near the temperature of the isotropic or nematic phase transition. This method allows one to obtain films of uniform thickness with a rigorously defined number of smectic layers from thousands down to two.

Surface ordering phenomena in films have been observed at the transitions between different liquid crystal phases and are very unusual [1–3]. The first kind of effect was observed at the transitions between smectic phases (i.e. both the low and high temperature phases exhibit a layer structure). It was found that the smectic structure of the low temperature phase remains near the surfaces up to 10–30 K above the bulk transition temperature [4]. On cooling discrete layer-by-layer or continuous changes of structure cause the low temperature smectic phase to fill the whole sample. These phenomena have been studied extensively for the transitions between smectic A, C, C* and B phases [2, 3]. Universal behaviour was found for each transition that does not depend on the molecular structure and the macroscopic characteristics of the individual phases. Other phenomena were reported in films for the transitions between smectic phases and phases without layer ordering (nematic, cholesteric, isotropic liquid). Stoebe *et al.* [5] have observed that smectic films do not melt or rupture above the bulk smectic–isotropic transition temperature on heating, but undergo a series of thinning transitions. At such transitions the thickness of the film decreases in a stepwise manner. As the film thickness decreases, the thinning temperature increases. However, unlike the transitions between smectic phases, the thinning phenomena depend on the compound, film thickness and heating rate [6–10]. In particular, two types of thinning transitions have been observed [7, 9]. The layer-by-layer transitions with reproducible thinning temperatures were designated as 'regular', while the transitions with irreproducible temperatures and multilayer thinning were designated as 'irregular'. 'Regular' transitions can easily be observed in fluorinated compounds. This behaviour may be connected with the lower surface tension γ of those compounds [7, 9]. It should be noted that the essential decrease of γ can also be achieved

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for non-fluorinated compounds upon contact with the vapours of fluoro-organic materials [11]. Up to now, thinning transitions have been extensively studied in fluorinated compounds, but the details and mechanisms of transitions in unfluorinated compounds still remain unknown. Recently 'regular' layer-by-layer transitions were observed over a broad range of thicknesses for non-fluorinated compounds [12, 13]. It was also shown that the thinning transitions may be accompanied by the formation of defect structures [12]. These observations essentially increase the range of materials and of phenomena that can be investigated in smectic films at high temperature.

In this paper we report high temperature investigations on films of unfluorinated material. We used different compounds exhibiting smectic C*–cholesteric (SmC*–N*) or SmC–nematic phase transitions. This allows us to visualize the change in the film structure by polarizing optical microscopy. The phenomena as layer-by-layer and multilayer thinning occur are observed through the formation of a dislocation loop, nucleation of droplets or formation of a 'quasi-smectic' structure, and depend on the film thickness.

2. Experimental

Experiments were carried out on free standing films of chiral *S*-4′-heptyloxybiphenyl-4-yl 4-(1-methylheptyloxy)-benzoate (7BSMHOB), *S*-4′-undecyloxybiphenyl-4-yl 4-(1-methylheptyloxy)benzoate (11BSMHOB) [14], and also on a commercial material ZLI 3488 (Merck). Bulk samples exhibit the following phase transition temperatures: SmC*–92°C–N*–135°C–I (7BSMHOB), SmC*–108°C–N*–123.9°C–I (11BSMHOB), SmC*–61°C–SmA–66°C–N*–85°C–I (ZLI 3488). The tilt angles of 7BSMHOB and 11BSMHOB are large, about 45° below the SmC*–N* transition temperatures [14]. Additionally some measurements were performed on a non-chiral SmC liquid crystal in order to evaluate the influence of chirality on the different phenomena. This material, bis-(4-*n*-decyloxybenzal)-2-chloro-1-4-phenylenediamine (DOBCP), exhibits the following phase transition temperatures: SmC–112°C–nematic–165°C–I.

Free-standing films were prepared by drawing the liquid crystal in a smectic phase across a 4 mm hole in a glass plate or a $1 \times 10\,\mathrm{mm}^2$ hole between two razor blades. Good quality films less than 100 layers thick could be prepared near the bulk transition temperature (T_C) to the cholesteric or nematic phase. For thicker films such a method is not successful; film and meniscus are very non-uniform. Thick films were prepared in two steps. First we prepared a thin film in the bulk temperature range of the smectic phase. This film was then heated above T_C; the material in the meniscus melted and the meniscus became uniform. Then, we cooled the film below T_C; at low temperature the film thickness increased due to material moving from the meniscus. Depending on time and temperature, it was possible to prepare thick uniform films up to 10 μm with a uniform interference tint of the reflected light spectrum. The layer-by-layer thinning transitions were recorded using heating rates of the order of or lower than $200\ \mathrm{mK\ min}^{-1}$.

The experimental set-up enables simultaneous measurements of light reflectivity from the films and optical observations in polarized light by reflected light microscopy. For the films with a number of layers $N > 20$, the thickness of the films is determined from the spectral dependence of the optical reflection in the 'backward' geometry [15]:

$$I(\lambda) = \frac{(n^2-1)^2 \sin^2(2\pi nNd/\lambda)}{4n^2 + (n^2-1)^2 \sin^2(2\pi nNd/\lambda)} \quad (1)$$

where d is the interplanar distance, n is the index of refraction and λ is the wavelength of the incident radiation. For thin films the reflected intensity is approximately proportional to N^2:

$$I(\lambda) \approx N^2 \pi^2 d^2 (n^2-1)^2/\lambda^2. \quad (2)$$

This allows us to determine the number of layers in the films without knowing d and n, simply by comparing the relative reflected intensities from films of different thickness.

3. Results and discussion

In most publications, thin smectic films have been investigated at high temperature. In our studies we used films with thickness from $N = 3000$ (10 μm) to only two layers. In this section we describe different phenomena that occur in free-standing films above the bulk smectic temperature range: layer-by-layer thinning transitions, formation of modulated structures, nucleation of droplets, and formation of a 'quasi-smectic' phase.

3.1. *Layer-by-layer thinning transitions*

For 11BSMHOB, the regime of layer-by-layer thinning transitions can be observed for films with $24 > N > 7$ layers. Figure 1 shows the change in the reflected intensity for the 12-layer film on heating. Thinning transitions are clearly observed as sharp steps in the reflectivity. Irregular change of the intensity occurs between thinning steps. We assume that this change is connected with the optical anisotropy in the plane of the film and the rotation of the *c*-director during the time of the measurements. The temperatures of thinning transitions for films of different thickness are shown in figure 2.

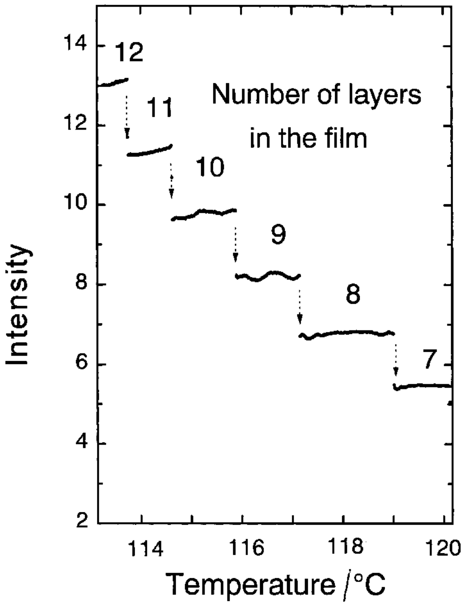

Figure 1. Optical reflectance ($\lambda = 560\,\mathrm{nm}$) above the bulk SmC*–N* transition of 11BSMHOB. At the layer-by-layer transitions the optical reflectance decreases in a stepwise manner. The heating rate is $200\,\mathrm{mK\,min^{-1}}$.

There are several theories describing thinning transitions. In all of them the existence of smectic films above the bulk transition is connected with surface ordering and the finite value of the surface correlation length inducing the smectic order inside the film. The thinning transition is associated with the correlation length decreasing on heating. The relevant questions are: (1) does the material melt in the interior of the film or does the smectic order exist in the whole film before thinning? (2) what is the mechanism of thinning?

The first model [5] was based on the experimental observation that thinning transitions can be described by the power law expression $N(t) = N_0 t^v$, where t is the reduced temperature $t = (T/T_0 - 1)$, and N_0, T_0 and v are fitting parameters. Such behaviour is connected with the temperature dependence of the smectic correlation length. It was shown that thinning transitions in all materials investigated can really be fitted by the power law. However this model does not permit any prediction about the thinning temperatures and does not indicate the mechanism of thinning.

The second model [16, 17] is based on the McMillan mean-field theory of the smectic phase with a short range pair potential for the intermolecular interaction [18]. The behaviour of films upon heating depends on the strength of the surface field [17]. If the field is not 'strong' enough, the whole smectic film should melt above some critical temperature. In this case the theory predicts film rupture. If the surface action is 'strong' enough, heating of the film induces the transition from the smectic phase to a 'quasi-smectic' structure with a melted interior part of the film and smectic structure in the surface layers. The transition temperature to the 'quasi-smectic' structure increases with decreasing film thickness. The boundary layers squeeze out one or a few melted layers and this results in the thinning transition. The thinning transition temperatures T_N are computed as numerical solutions of a set of self-consistent equations. They do not obey any analytical expression. However, the computed dependence of the number of layers N versus the reduced temperature is very similar to the power law that is in qualitative agreement with experimental results.

In the recent theories [13, 19–21], based on the phenomenological pre-smectic liquid model of de Gennes [22], the interior smectic layers do not melt before the thinning transitions. On heating, both the smectic order parameter and the compression modulus decrease essentially for the interior layers. At some critical temperature the balance of the external pressure and the interior

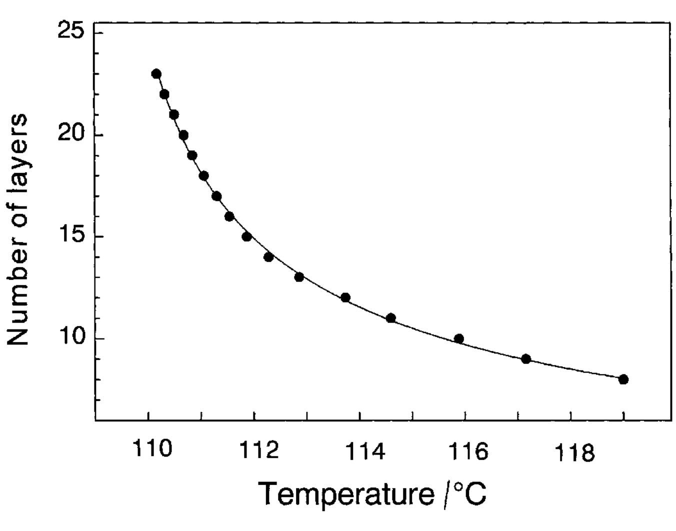

Figure 2. Number of layers N versus temperature for the compound 11BSMHOB. The line is a fit to the equation for the dislocation mechanism of thinning (see text).

elastic forces breaks down giving rise to mechanical instability of the film and thinning transitions. Equations obtained [13, 20, 21] give approximately the same temperature dependence of the film thickness at the thinning transitions. Thickness is proportional to the bulk correlation length within a logarithmic correction. Near the critical temperature of the mechanical instability, the probability of creating a dislocation essentially increases; the thinning transition is then associated with the creation and motion of a dislocation loop. Following this approach, an analytical expression can be obtained for the dislocation mechanism of thinning [20]

$$N = (2\xi/d)\,\mathrm{arcsinh}\,(a_1 t^{-1}) \qquad (3)$$

where $\xi = \xi_0 t^{-1/2}$ is the correlation length for smectic layer ordering. In comparison with experiments, ξ_0, a_1 and T_0 are used as fitting parameters.

The best fit of the experimental data according to equation (3) is plotted as a solid line in figure 2. Although figure 2 demonstrates the success of fitting, the fit leads to a value of $\xi_0/d < 0.1$ that appears to be too low ($\xi_0 = 0.15$ nm; $T_0 = 110.2$°C; $a_1 = 6 \times 10^3$). This would lead to a correlation length ξ lower than the layer thickness in most temperature ranges of thinning transitions. Thus, these theories derived for second order phase transitions in the continuous approximation are not really very appropriate for describing the temperatures of layer-by-layer thinning transitions in our case. We can conclude only that a qualitative correlation exists between experimental data and theoretical predictions.

3.2. *Formation of modulated structures*

At low temperatures, *thick* films of the *n*BSMHOB ($n = 7$ or 11) compounds give a uniform texture with a reflected colour due to the selective reflection of the light. According to this selective reflection, the pitch of 7BSMHOB varies from 130 nm at 73°C to 300 nm at 89°C. No more transformations are observed by reflected light microscopy up to temperatures near the SmC*–N* transition temperature (T_C). At $T_C - T < 0.2$°C, the continuous field of the *c*-director in the thick films breaks up. Sharp lines appear and grow, figure 3(*a*). The lines may reach several hundred microns in length. Under a slight heating pulse, the line structure becomes unstable: the straight lines begin to undulate and two types of evolution can be observed. The line bends in opposite directions at regular distances like a zigzag. The zigzags of the line are then packed closely together and give rise to a large striated line, figure 3(*b*); the striations are the memories of the zigzag of the original line. The second type of line evolution leads to nearly round structures; such structures appear when the second bend of the line occurs in the same direction as the first, figure 3(*c*). On further heating, two-dimensional structures issue from

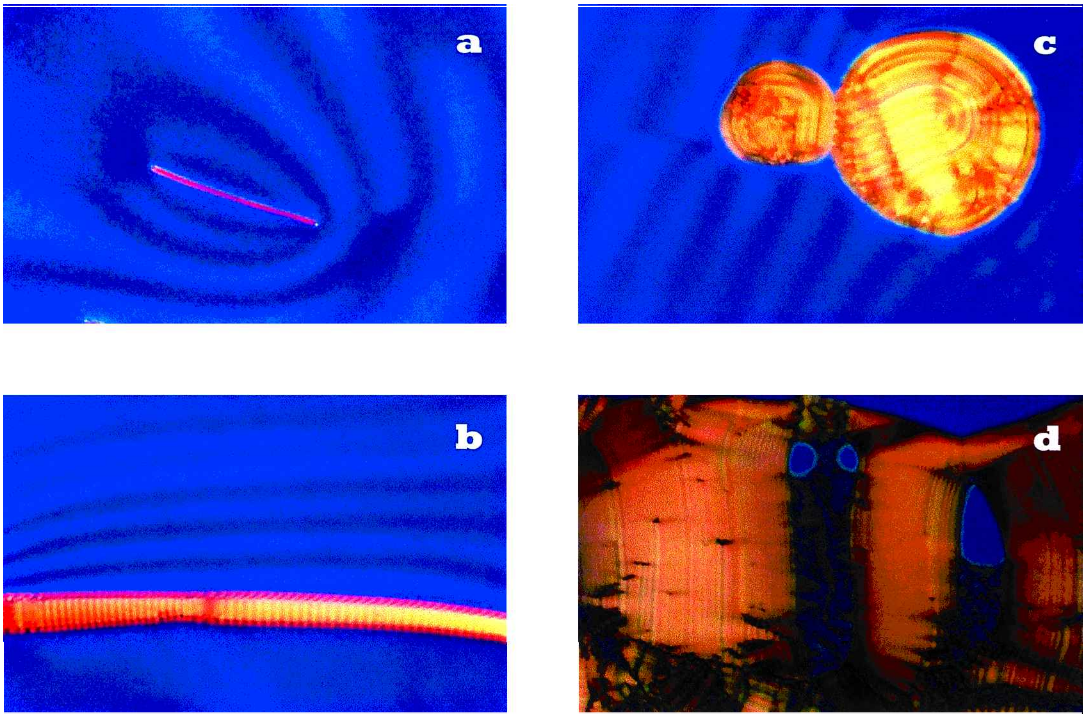

Figure 3. Appearance and evolution of string lines in a thick 7BSMHOB free-standing film near the bulk SmC*–N* transition temperature: (*a*) single line, (*b*) zigzag, (*c*) round defect from lines; (*d*) defect structure fills the whole film. The thickness of the film is 10 µm, reflection between crossed polarizers; the horizontal size of each image is about 270 µm.

these lines and fill the whole film, figure 3 (*d*). Both the formation of lines and the two-dimensional structures are reversible: Cooling leads to their disappearance. Close to T_C the film structure becomes unstable; a part of the material starts to move towards the meniscus and the film thickness decreases. Thick films lose large numbers of layers; for a small increment in temperature, thick films (about 10 µm) may decrease by factors of ten in thickness, but the film does not break, even though most of the material moves to the meniscus.

In *thin* films, formation of a modulated structure from the lines is observed. We found the formation of this structure in 11BSMHOB down to a film thickness $N = 17$, whereas in thick films (more than 1000 layers) a defect structure appears just below T_C; in thin films this forms above the bulk transition temperature. The appearance of the defect structure at high temperature precedes the thinning transitions and its formation temperature increases with decreasing film thickness. With decreasing film thickness, an evolution of defect structures can also be observed. As in thick films, the structure appears as a single line; the widths of the lines decrease on decreasing the film thickness. In thin films, the string-like lines do not pack in zigzag strips; near the thinning transitions they form a modulated structure. These defect structures exist in a sharp temperature range (< 0.05°C); both on cooling and heating (after thinning transition) the modulated structure disappears.

Figure 4 (*a*) shows the usual texture of the SmC* film in polarized light. Dark and bright regions correspond to different orientations of the *c*-director with respect to the direction of the light polarization. Near the thinning transition, the continuous *c*-director field breaks up, figure 4 (*b*), $T_N - T < 0.02$°C. After thinning—$N = 21 \rightarrow N = 20$, figure 4 (*c*)—the continuous *c*-director field is restored. This phenomenon repeats near the next thinning transition: figure 4 (*d*), $N = 20$; figure 4 (*e*, *f*), $N = 19$.

In very thin films we did not observe a modulated structure. However in some cases another modification of the texture before the thinning transition could also be observed. Relatively sharp stripes appear near the thinning transition, figure 5 (*a*), 7BSMHOB, $N = 12$. For comparison figure 5 (*b*) shows the modulated structure in a 19-layer film. Apart from the regions with smooth variation of the line directions, there are regions with sharp change in their orientation, figure 5 (*b*). These stripes can be observed using the microscope when the distance between single string-like lines becomes too small. So, in figure 5 (*a*) we may assume that the lines are not observed because the distances between them are less than the resolution of the microscope. Formation of the string-like lines and the modulated structure are probably connected with the long wavelength instability of the film in relation to the squeezing modes [19]. Near the thinning transitions, the internal part of the film is very soft matter. Recent numerical calculations predict that near the thinning transition temperature, the disjoining pressure in the films reaches several atmospheres and induces a strong compression of the smectic layers [23]. Ferroelectricity and the action of the disjoining pressure in the film may induce a breakdown of the continuous *c*-director field.

3.3. *Nucleation of the droplets*

Together with thinning transitions and the formation of defect structures, the nucleation of cholesteric or nematic (for the SmC–nematic transition) droplets can be observed above the bulk transition temperature. The droplets nucleate in materials exhibiting first order (smectic–cholesteric, smectic–nematic or smectic–isotropic) phase transitions. As a rule the first droplets appear above the bulk transition temperature in parts of the film exhibiting defects. We found that if the film contains a topological point defect, the droplet nucleates in the core of this defect where the *c*-director order is destroyed. Figure 6 (*a*) shows the topological defect between crossed polarizers ($T = 109$°C). The strength of this point defect is equal to one. After heating, the droplet appears in the centre of the defect, figure 6 (*b*), $T = 109.4$°C. Numerical calculation and theory based on a Landau expansion of the free energy in the nematic phase [24, 25] show that the core radius of the topological defect has to increase with temperature and then becomes unstable at some critical temperature; the isotropic phase nucleates. Up to now there has been no comparison between these theories and experiment. Our investigations show that melting phenomena induced by the topological defects are easily observed in the smectic films. Figure 6 (*b*) shows that in the smectic film, the droplet nucleation replaces a single singularity in the *c*-director configuration by two with half the strength. The point defects lie at opposite ends of the bipolar droplet where the *c*-director field escapes from the droplet surface. The distortional free energy of the *c*-director configuration with two defects is less than that of a single defect with double strength.

In regions with a continuous *c*-director field, large thermal fluctuations are needed for nucleation and droplets appear at a slightly higher temperature; this temperature depends on the film thickness and increases with decreasing film thickness. On cooling, small droplets (< 3 µm) disappear, but the largest droplets remain in the smectic film. The number of droplets nucleated near the temperature of thinning transitions ($T_N - T < 0.2$°C) may reach tens or even one hundred, figure 7 (*a*). The

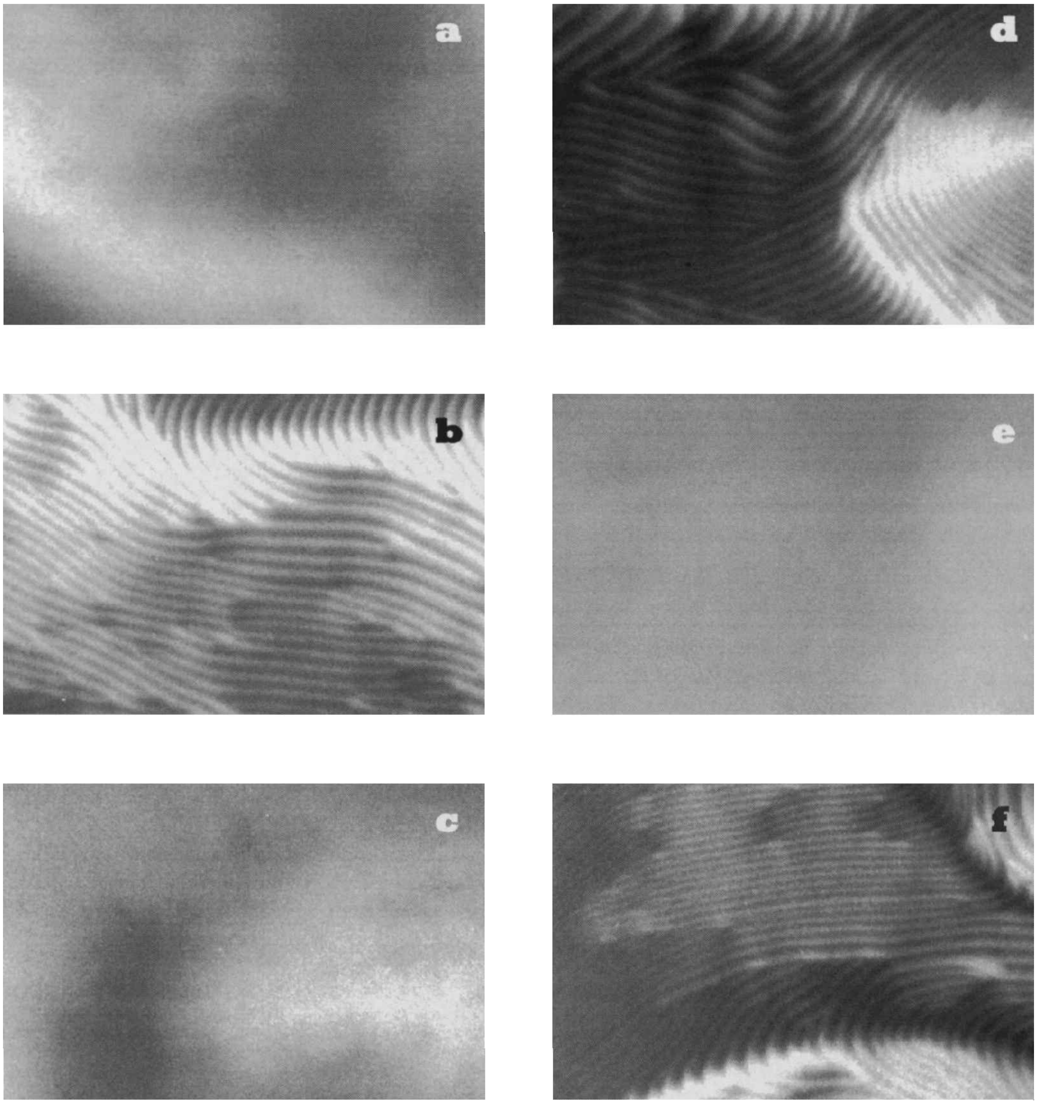

Figure 4. Texture in thin free-standing films, observed far from (*a*), (*c*), (*e*) and near to (*b*), (*d*), (*f*) the temperature of the thinning transitions of 11BSMHOB; reflection between crossed polarizers. The thickness of the film is (*a*, *b*) $N = 21$, (*c*, *d*) $N = 20$, (*e*, *f*) $N = 19$. The horizontal size of each image is about 300 µm.

droplet thickness strongly exceeds the thickness of the film. However, surprisingly in most cases, the droplet nucleation does not induce film thinning. This means that during the nucleation, the film draws off a part of the material from the meniscus. The reason for this phenomenon is not well understood. In DOBCP, large droplets (more than one hundred micrometers) appear on further heating, figure 7 (*b*); these show a continuous variation of interference colours. Estimation of the droplet thickness from the interference colours gives values up to one micron while the smectic film thickness is somewhat less than 0.1 µm. Films with large droplets may exist at a temperature more than ten degrees above the bulk transition temperature. It is interesting to note that on heating, normal thinning transitions are still observed in the smectic part of the film. These transitions only affect the thickness of the smectic regions around the droplets.

3.4. *Formation of the 'quasi-smectic' phase*

Another interesting phenomenon is what we suppose to be the manifestation of a 'quasi-smectic' structure in the ZLI 3488 film and its stability at high temperature. Figure 8 (*a*) shows the film with different numbers of smectic layers ($T = 70$°C). Above the bulk transition temperature, smectic ordering and sharp steps between different thicknesses exist in the film. Steps correspond to a thickness change of kd (k are integers). On further heating the steps become blurred. Figure 8 (*b*) exhibits no more steps, but a continuous change of thickness

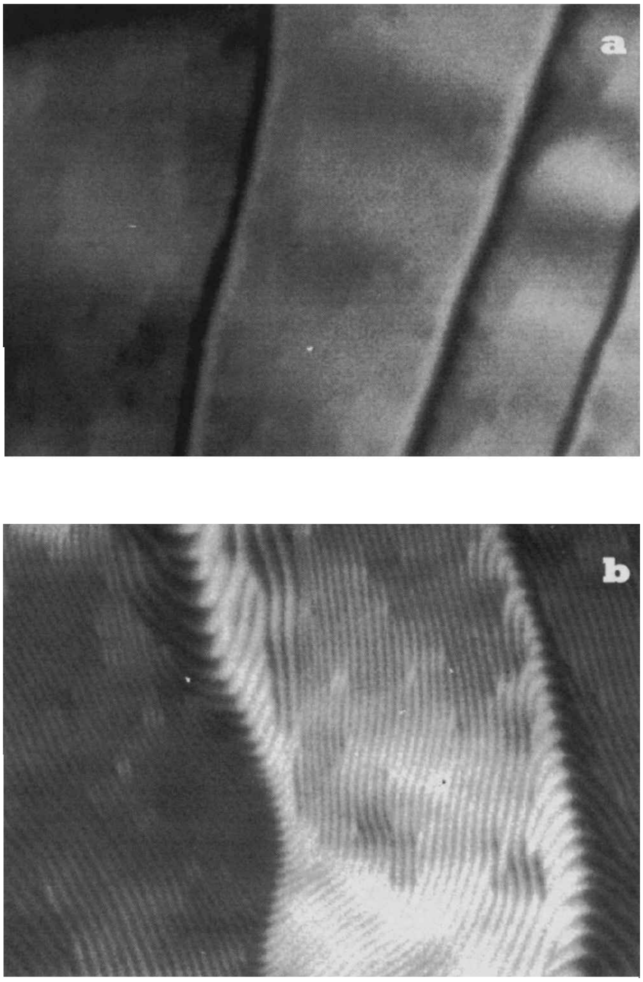

Figure 5. Comparative textures of (*a*) 12-layer and (*b*) 19-layer films of 7BSMHOB near the temperatures of the thinning transitions (reflection between crossed polarizers). The horizontal size of each image is about 300 µm.

($T = 74$°C). Near the film surfaces there is smectic ordering, but now its penetration length is less then $L/2$ (L is the film thickness). In this case interior layers melt completely and the film thickness remains non-uniform, but without steps. Hence, in this state the film thickness can become incommensurate with the layer spacing. Such a structure in free-standing films was predicted and called the 'quasi-smectic' phase [16, 17]. Calculations, based on another density-functional theory [26], also predicted a structure similar to the 'quasi-smectic' phase. According to these theories, such a structure has to be either an intermediate state in the layer thinning transition [16, 17] or a completely unstable state that has to lead to film rupture [26]. In practice the behaviour of the 'quasi-smectic' state differs essentially from predictions. In reality we can observe these films during long periods of time (about one hour) without change of thickness. Such a film can be heated to definitely higher temperatures than smectic films of the same thickness. However, on heating up to 85°C (corresponding to the bulk cholesteric–isotropic transition temperature) the film thickness drastically decreases. The thin films obtained consist of a few smectic layers (about ten or less) and undergo the usual layer thinning transitions on further heating.

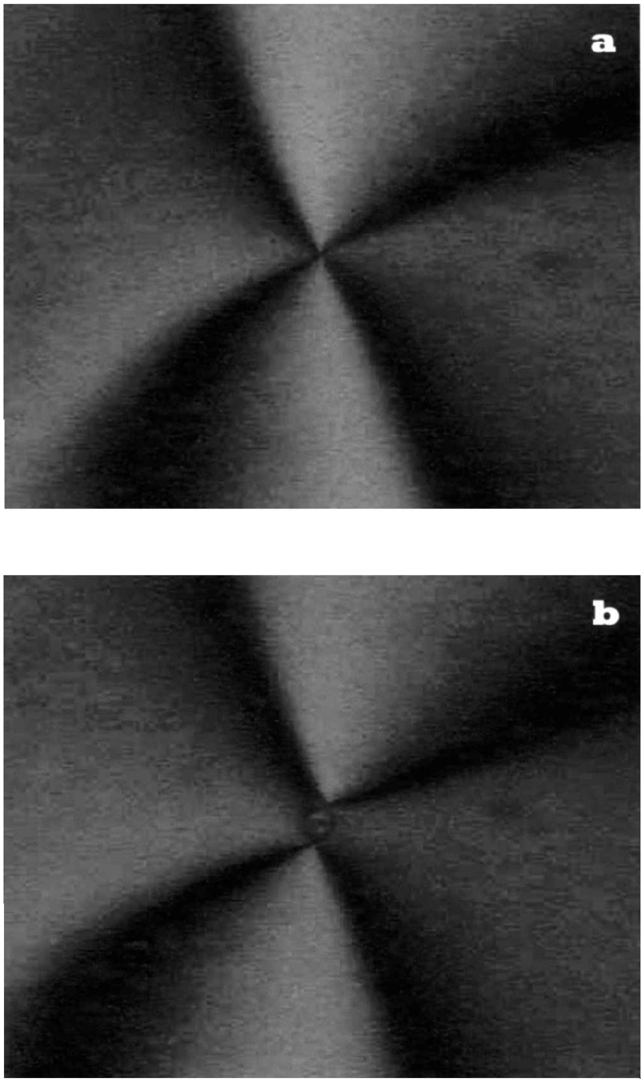

Figure 6. (*a*) Topological point defect in a free-standing film at $T = 109$C° in reflection between crossed polarizers. (*b*) The defect is broken into two parts by the appearance of an N* droplet, $T = 109.4$°C. Film thickness is about 30 smectic layers of 11BSMHOB. The horizontal size of each image is about 150 µm.

4. Summary

Free-standing films above the temperature range of the bulk smectic phase are unusual liquid crystalline state. The transitions between a smectic phase and less organized phases are accompanied by changes of the

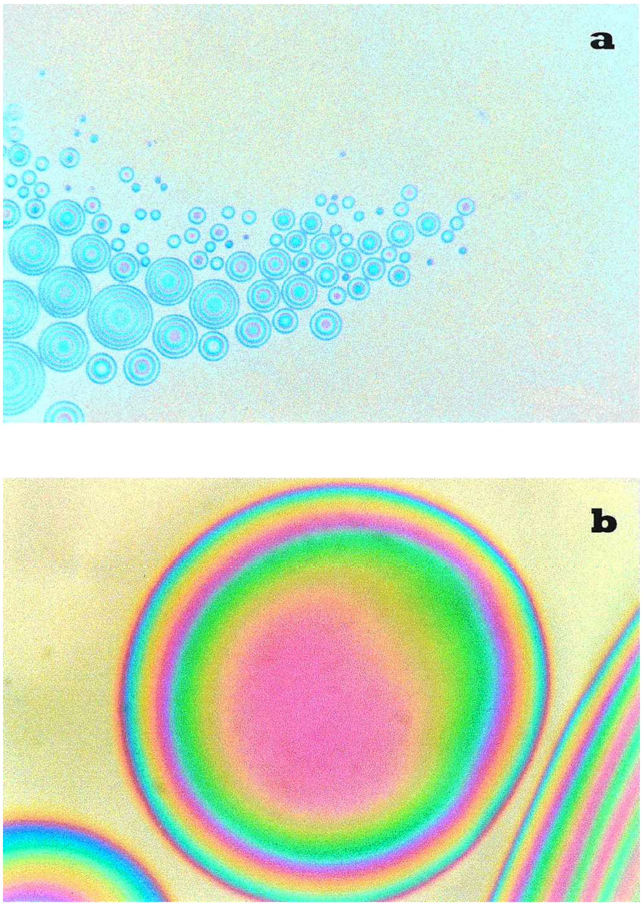

Figure 7. Microscope picture, in reflection, of (*a*) small at 113°C, and (*b*) large at 117°C nematic droplets in SmC films of DOBCP above the bulk transition temperature. The horizontal size of each image is about 580 µm.

smectic correlation length with temperature. Therefore, heating far above the bulk smectic temperature range forbids that the film exists in the former smectic state. Until recent time, studies of free-standing films at high temperature have been mainly devoted to investigations of only one type of response to the change of correlation length on heating, namely, layer-by-layer thinning transitions. Recent papers [12, 27] and our present investigations on unfluorinated compounds reveal a broad range of phenomena in films formed from different compounds: successive formation of modulated structures, nucleation of droplets, layer-by-layer thinning phenomena for some compounds and 'quasi-smectic' structure formation for others. Two types of topological defect existing in smectic films (dislocations and point disclinations) play an important role in the observed phenomena. The phases without smectic ordering (nematic, cholesteric) first nucleate in the core of the point disclination. The nucleation of the dislocation induces the thinning transition that takes the place of the film melting. In contrast, the 'quasi-smectic' structure formation enables the films to exist without thinning at higher temperature than a smectic film of the same thickness. Such behaviour has not yet received theoretical explanation. Further studies, both experimental and theoretical, are necessary for an understanding of the phenomena observed in superheated smectic (pre-smectic) films.

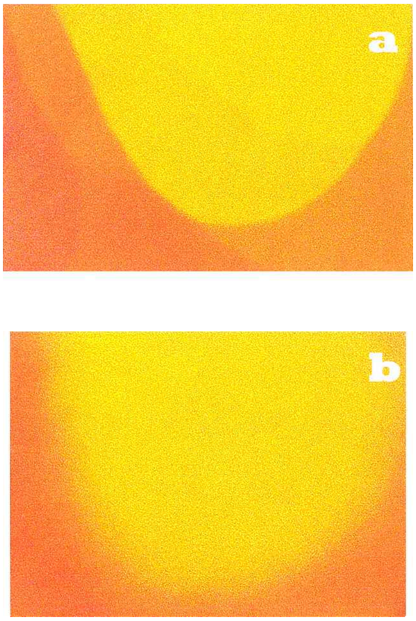

Figure 8. Evidence of the 'quasi-smectic' structure in a ZLI 3488 film observed in reflection: (*a*) at $T = 70°\mathrm{C}$, regions of different thickness are separated by steps; (*b*) film without smectic layer ordering in the interior no longer exhibits steps ($T = 74°\mathrm{C}$). The film thickness is about 100 nm, the horizontal size of each image is about 240 µm.

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