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# Optical textures in TGBA mesophases

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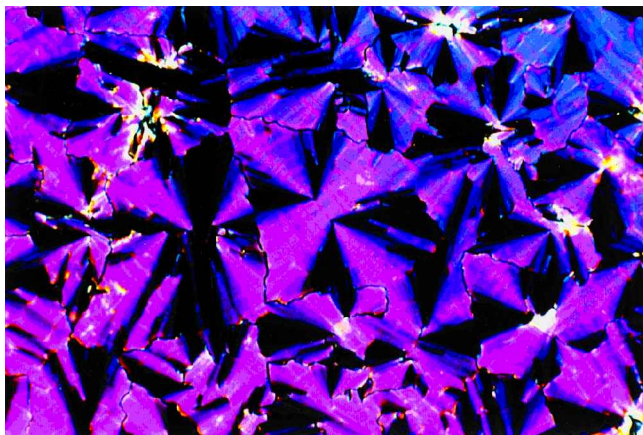
In this paper, we present the results of polarized optical microscopy observations performed on different series of compounds exhibiting twist grain boundary mesophases (TGB). From all the compounds investigated, it appears there are two main typical optical textures which characterize TGBA mesophases in general. One type of texture is characteristic of a planar helical structure, while the other is a cylindrical (or marginally cone-like) domain texture, resembling the developable domains observed in columnar systems. Moreover, both textures can coexist in the same sample over the whole temperature range of the TGB phases. The detection, at the same temperature, of these two types of textures could then be considered as one of the signatures of the TGBA mesophases.

## 1. Introduction

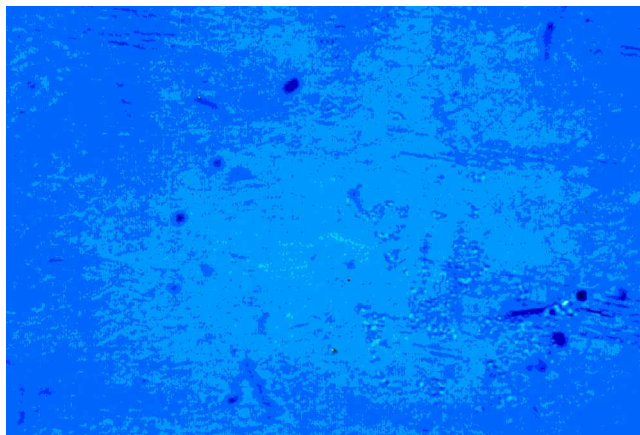
The TGBA and the TGBC mesophases, respectively predicted by Renn and Lubensky in 1988 [1] and by Renn in 1991 [2], were experimentally observed for the first time in 1989 by Goodby *et al.* [3] and in 1992 by Nguyen *et al.* [4]. In 1994, we reported for the first time the characterization of a mesogenic compound with two chiral centres, exhibiting a TGBA mesophase and showing simultaneously different optical textures, usually observed in the separate cases of helical and columnar ordering [5]. Following this work we then reported in 1998 [6] the characterization of two new diastereomers belonging to the same series of compounds studied before. A TGBA mesophase in a large temperature domain was observed with one of the two diastereomers, while both TGBA and TGBC mesophases were observed

in a similar temperature domain with the other. With both diastereomers and in both mesophases, it was possible to observe the coexistence of two different types of optical textures, namely planar helical textures usually encountered in cholesteric samples, and textures apparently similar to the developable domains textures observed in columnar mesophases and first described in this way by Kléman [7]. The theoretical interpretation concerning the observation of developable domain textures in the TGB mesophases was also presented in [6]. These types of textures are more precisely made of cylindrical (or marginally cone-like) domains (CC) which may be seen as particular focal-conics, but also as developable domains [6]. According to our previous interpretation, the observation of both kinds of textures, namely helical textures and CC textures, could be in general a specific feature of all TGB mesophases. However, we must remark that the detection of the CC type textures in TGB mesophases has only been described, till now, for compounds belonging to the series studied in [5, 6].

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(a)



(b)

Figure 1. Optical textures (magnification  $\times 210$ ) observed in the TGBA mesophase of a compound of series **I** ( $n = 10$ ). (a) CC domains observed at  $T = 86.5^\circ\text{C}$  on decreasing the temperature very slowly from the isotropic phase; (b) planar helical texture observed at  $T = 93^\circ\text{C}$  after applying a shear stress to the preparation with a texture similar to that shown in (a).

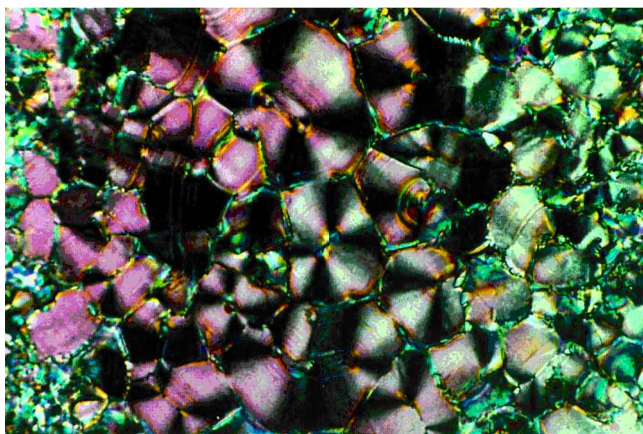


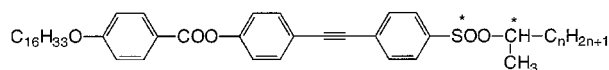
Figure 2. CC type texture detected at  $T = 95.7^\circ\text{C}$  in the TGBA mesophase of compound 18FBT8\*.

In this work, we present the results of careful optical microscopic observations performed on different series of compounds exhibiting TGB mesophases. We provide the experimental evidence that, in these phases, it is possible to observe, in general, the two different types of optical textures described above. We are thus led to propose that the simultaneous observation of these two textures could be considered as a new criterion for the identification of TGBA mesophases.

## 2. Experimental results—optical microscopic observations

The experimental results described in this paper were obtained using a polarizing microscope Orthoplan (Leica) working in the transmission mode and associated with a Mettler hot stage.

Along with an example of our previous observations [5, 6] concerning five compounds of the following series called here **I**



we present and discuss the optical microscopic textures obtained with eight compounds belonging to three other different series (called, **II**, **III**, and **IV**).

### 2.1. Compound of series **I**

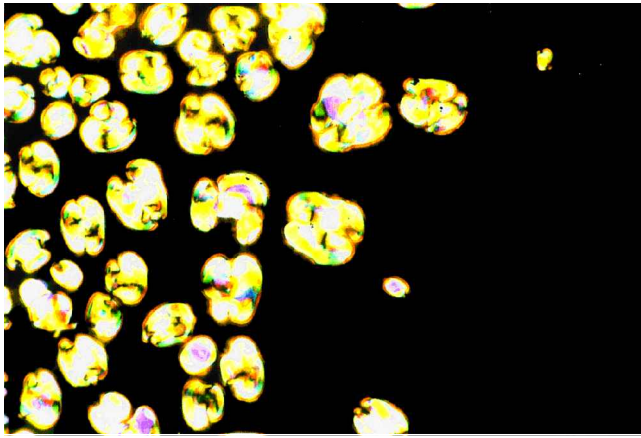
Figure 1(a) presents a texture observed in the TGBA mesophase of a new compound belonging to series **I** ( $n = 10$ , chiral carbon centre in the *S* configuration and chiral sulphur centre in the *R* or *S* configuration) exhibiting the following polymorphism:



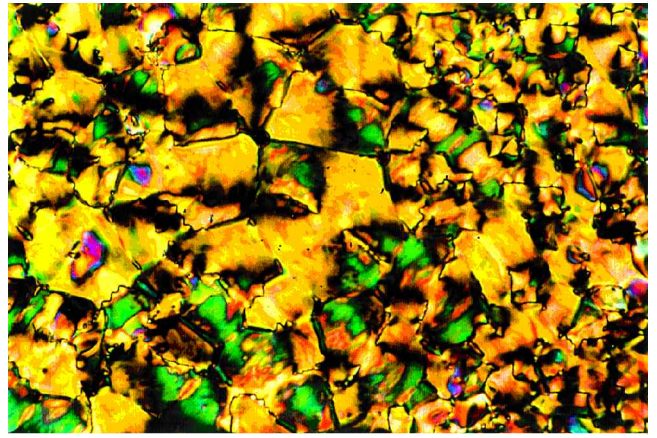
This texture, clearly of the CC type, is made of typical domains with black fringes arising from sharp eyes and rings around them. It was obtained on cooling the sample very slowly from the isotropic phase. For all the compounds of series **I**, by applying a small shear to the preparations exhibiting such textures, the CC domains disappear from a new planar texture characteristic of a cholesteric organisation [5, 6] as can be seen in figure 1(b). Such a texture change is due to the reorientation produced by the shear flow which appears to be strong enough to orient the helical layers† parallel to the cell

† The helical strata or layers in TGB phases give them the mechanical properties of layered systems.

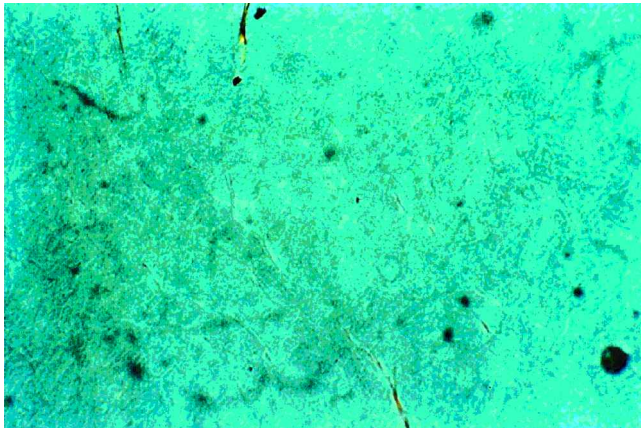




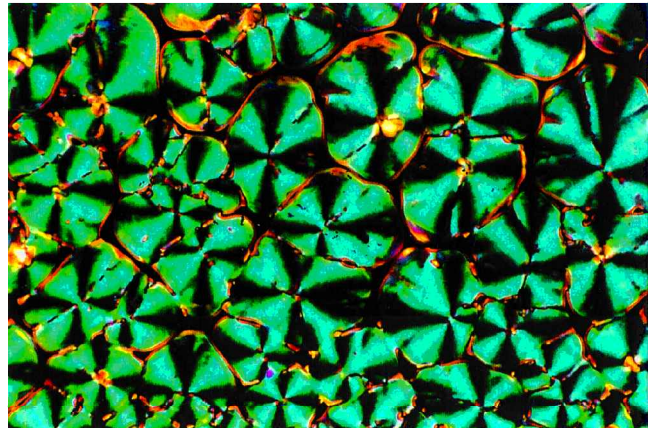
(a)



(b)



(c)



(d)

Figure 3. Textures observed with compound 16BT10\*. (a) Growth of the TGBA mesophase at  $T = 107.5^\circ\text{C}$  on cooling very slowly from the isotropic phase; (b) CC texture observed in the same area as (a) in the TGBA mesophase at  $T = 107.1^\circ\text{C}$ ; (c) planar helical texture detected in the TGBA mesophase at  $T = 106.6^\circ\text{C}$  after applying a shear in a preparation similar to (b); (d) CC type texture detected in the TGBA mesophase at  $T = 107.4^\circ\text{C}$  in another preparation of the same compound.

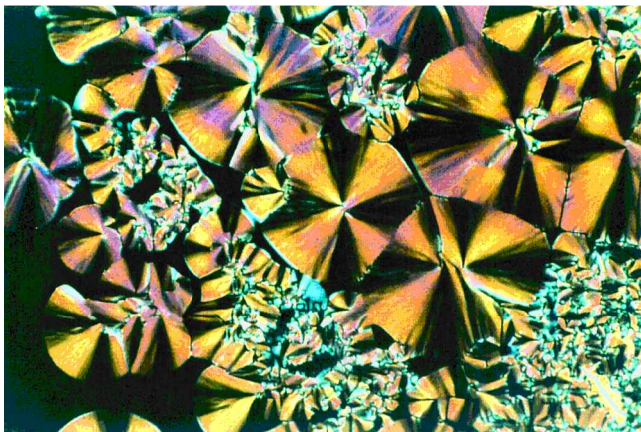


Figure 4. CC texture observed at  $T = 97.6^\circ\text{C}$  in the TGBA mesophase of compound 16BT9\*. In the CC domain at the centre of the photograph, one can observe that the 'two eyes' are separate, though very close to each other.

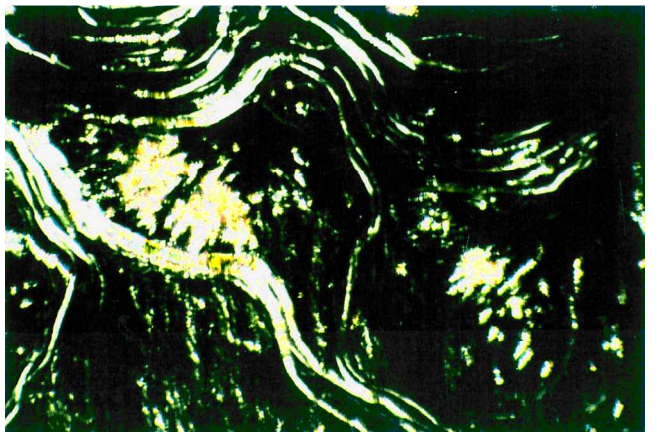
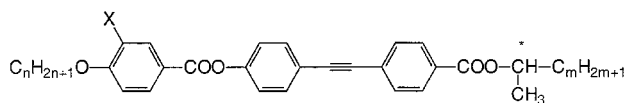


Figure 5. Typical texture with oily streaks, observed at  $T = 107.3^\circ\text{C}$  in the TGBA mesophase of compound 16BT9\*.

plates. As is well known, helical layers yield Bragg colours provided that twice their periodicity is in the wavelength range of visible light. It should be mentioned here that all the compounds of series **I** studied before [5, 6] show TGBA mesophases on decreasing the temperature directly from the isotropic phase. Depending upon the chiral configuration of the sulphanyl group, a TGBC mesophase can also be observed in addition to the TGBA phase [6]. It is also important to remark that CC domain textures and planar helical textures were similarly detected in the TGBC mesophases as in the TGBA phases [6].

We now compare these results with observations performed on eight compounds of three new series (**II**, **III**, and **IV**).

## 2.2. Compounds of series **II**



Six compounds of series **II** have been studied [8, 9]. Depending upon  $n$  and  $m$  and the lateral group  $X$ , they exhibit the following polymorphism:

18FBT8\* ( $n = 18, X = F, m = 6$ )

Cr 67°C SmC\* 93°C TGBA 97°C I

16BT9\* ( $n = 16, X = H, m = 7$ )

Cr 68°C SmC\* 105.8°C TGBA 110°C I

16BT10\* ( $n = 16, X = H, m = 8$ )

Cr 68°C SmC\* 104.6°C TGBA 107.6°C L<sub>1</sub> 110°C I

16FBT9\* ( $n = 16, X = F, m = 7$ )

Cr 76.8°C SmC\* 95.4°C TGBA 97.8°C I

16FBT10\* ( $n = 16, X = F, m = 8$ )

Cr 70°C SmC\* 93.3°C TGBC 93.9°C TGBA 95.2°C L<sub>1</sub> 97.3°C I

18FBT9\* ( $n = 18, X = F, m = 7$ )

Cr 73.2°C SmC\* 94.1°C TGBA 95°C L<sub>1</sub> 96°C I,

where L<sub>1</sub> is most probably the BPIII phase [9].

On cooling the first compound 18FBT8\* very slowly from the isotropic phase, it was possible to observe the growth of textures similar to the one presented in figure 2. This texture is clearly of the CC type similar to the textures observed with the compounds of series **I**. It is, in particular, easily recognised by the presence of domains with two typical 'eyes', corresponding to the axes of the cylinders, and with faint elliptical rings around them. These elliptical lines are locally oriented

perpendicular to the TGB axis. They most probably correspond to fracture lines as their contrast increases on decreasing the temperature within the TGB mesophase, an effect which could be associated with increasing mechanical constraints. The same type of textures is roughly preserved on cooling in the TGBA mesophase. However, on crossing the TGBA–SmC\* phase transition, the CC type texture is completely transformed. As for the compounds of series **I**, on applying a small shear to the preparations in the TGBA mesophase exhibiting a texture similar to that in figure 2, the cylindrical domains disappear and a planar helical texture appears.

With the exception of the second compound, all the four other compounds of this series exhibit CC textures in the TGB mesophases. In fact, for the compounds 16BT10\*, 16FBT9\*, 16FBT10\* and 18FBT9\*, on cooling the samples very slowly from the isotropic phase to the TGB mesophases, it was possible to observe the textures described below.

With compound 16BT10\*, it is possible to see the formation of the TGBA mesophase on cooling very slowly from the isotropic phase, figure 3(a). The texture is very similar then to those observed at the transition from isotropic to the columnar mesophases of disc-like molecules. It is also very interesting to notice that this picture is in a way similar to figure 2(a) presented by Goodby *et al.* [3] in their first publications describing the discovery of the TGBA mesophase. On cooling further, the TGBA mesophase is formed, and it is possible to observe textures similar to that in figure 3(b) with CC domains. However, one may notice that the domains are not so clear as in compounds with a direct isotropic to TGB phase transition, figures 1(a) and (2). The existence of an intermediate phase between the isotropic and TGB phases, here L<sub>1</sub>, most probably prevents the CC domains from forming normally, because it introduces supplementary constraints at their formation. One may also note clear variations of colours in the transmitted light inside the CC domains. This effect could be due to the large thickness of the preparation as we will see hereafter. As for the other TGBA mesophases described above, the texture observed in figure 3(b) is easily destroyed by applying a small shear to the preparation. Then textures characteristic of the helical organization again appear, with the helical layers parallel to the cell plates as shown in figure 3(c). Another clear example of the CC texture observed with the same compound is shown in figure 3(d). In this case, the eyes of the CC domains are practically joined. One also notices that the texture colour is practically uniform, which is consistent with a thinning down of the cell due to the successive pressures exerted on it during the experiment.

Figure 4 presents the texture observed in the TGBA mesophase of compound 16FBT9\*. The texture is roughly similar to those in figures 1(a), 3(b) and 3(d), except for radial streaks inside the domains, instead of faint elliptical rings, giving them a crumpled or broken shape. This effect is not yet understood, but it could arise from stresses of the opposite sign compared with the previous examples where rings are observed. They could themselves result from a different anisotropy of dilation with temperature. Also, the CC domains are generally not complete. However, at the central part of the picture, it is possible to observe an entire CC type domain with the 'two eyes' very close to each other. The effect of a shear into the preparation is similar to that described for the other compounds.

With compound 16FT10\*, the same types of texture are also observed in both its TGBA and TGBC mesophases. In addition, in the TGBC mesophase, some fracture lines decorate the CC domains. Similar results were previously obtained with two compounds of series I [6] exhibiting a similar polymorphism. The effect of a shear on the preparation is similar to that described above for the other compounds.

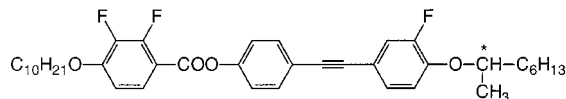
With compound 18FBT9\*, both kinds of texture described above for the other compounds of the series can be obtained in the TGBA mesophase.

With compound 16BT9\*, the experimental results are clearly different from those just described for the other compounds of the series. With this particular material, on cooling the sample very slowly from the isotropic phase, it was always possible to observe the growth of textures similar to that presented in figure 5, and the texture was then preserved until the transition from the TGBA to the SmC\* phase. This texture is characterized by large black areas with birefringent filaments. It clearly looks like a cholesteric texture with the so-called oily streaks [10]. This type of texture is known to arise when the helical pitch is very short, so that the Bragg reflection occurs in the UV range. This indicates that, probably due to a particularly strong planar anchoring of the molecules onto the glass plates, the helical axis of the TGB structure of this compound is spontaneously perpendicular to the preparation (while, with the other compounds presented and studied in the present paper, the helical axis of the TGB structures was approximately in the plane of the preparation, before any shear application). However, it is worth mentioning that textures with very small CC domains can be observed in some parts of the preparation. By applying a slight pressure onto the preparation, the reorientation of the helical layers is not improved at first, and many small domains of high birefringence with many different bright colours (some are visible in the left centre of figure 5) are produced in coexistence with a few oily streaks. Nevertheless,

after some time, most of these domains relax and the original texture reappears, similar to that presented in figure 5.

### 2.3. Compound of series III

The compound of series III that we have studied is the following [4, 11]:



Cr 59.1°C SmC\* 98.4°C TGBA 105.8°C N\* 114.5°C BP 115.7°C I

It is interesting to remark that this compound exhibits both cholesteric and blue phases (BP) at high temperatures, above the TGBA mesophase. On cooling the sample from the isotropic phase until the cholesteric phase, below the BP to the N\* phase transition, it was possible to observe a blue texture containing clearer granulations, indicating a roughly planar cholesteric orientation. On cooling the cholesteric mesophase, an evolution of the colour of the granulated texture towards large wavelengths was detected. Then, crossing over the next transition into the TGBA mesophase, it was possible to observe textures similar to those presented in figure 6(a). These textures seem to be a special kind of CC textures with, however, many defects which, as remarked above, may be due to the existence of intermediate phases between the isotropic and the TGBA phase, in this case, the BP and N\* phases. Three points need to be stressed:

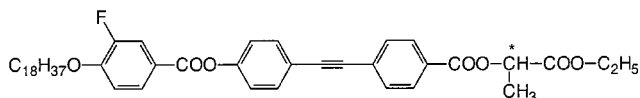
- (i) On heating the sample and on passing the transition temperature from the TGBA to the cholesteric mesophase, the texture is destroyed as shown in figure 6(b).
- (ii) On cooling to the SmC\* phase, the texture changes at the transition between the two mesophases. Several dechiralization lines are detected at the transition and a pseudo-homeotropic texture is observed in the SmC\* mesophase.
- (iii) As observed with the other compounds of series I and II, the textures such as those represented in figure 6(a) are easily destroyed by the application of a small shear to the preparation in the TGBA mesophase which, as shown in figure 6(c), becomes more or less monochromatic, probably due to the imperfect alignment of the helical layers parallel to the plates.

With these remarks, we may conclude that the texture observed in figure 6(a), may be used as a signature of the TGB mesophase, as in the cases of figures 1(a), 2, 3(b), 3(d) and 4.



#### 2.4. Compound of series IV

The compound that we have studied in series IV is [12]:



Cr 64.9°C SmC\* 94.2°C TGBA 105°C N\* 106.6°C BP 107°C I

Like the compound of series III (previous section), this compound exhibits the TGBA mesophase, but also cholesteric and blue phases at higher temperatures. The TGBA mesophase has been identified from measurements of the helical pitch at the transition from the N\* phase (there is a continuous variation of the pitch between N\* and TGBA mesophases, whereas there is a jump of the helical pitch between N\* and TGBC mesophases) [12]. On cooling the sample very slowly from the BP to the N\* phase transition, it was possible to observe the growth of a kind of planar texture characteristic of a cholesteric organization. However, on cooling again into the TGBA mesophase, the texture persists and looks like a cholesteric texture, but with an array of parallel lines, figure 7(a). Moreover, in some regions, two arrays almost perpendicular to each other may be observed, making a roughly square lattice as shown in the right top corner of the photograph. It is interesting to remark that a similar square lattice has already been observed in some TGBC mesophases [13, 14]. However, in the present case, the square lattice seems to be different in nature, since we noticed that its observation depends on its relative orientation with respect to the crossed polarizers. At the present time, we do not propose any explanation for the physical origin of this square lattice observed here in our TGBA mesophase. In the case of a very thin preparation, it was also possible to observe a texture which could be considered as a limiting case of a CC type texture, as shown in figure 7(b).

### 3. Discussion and conclusions

In figure 8, is shown a schematic representation of two cylindrical domain structures in a plane perpendicular to the cylinder axes. Both structures are equivalent. Only the distance between the two 'eyes', associated with the axes of the cylinders, changes. As explained before [6], the solid lines in this figure correspond at the same time to the smectic blocks, the grain boundaries and the layers of the helical structure of the TGB phase coiled around the axis of the cylinders ('eyes'). The molecules inside the TGB slabs are perpendicular to the radial directions coming out from the 'eyes'. More precisely, it is also possible to say that the solid lines correspond to the projection of the molecular direction onto the plane

of the figure. They also correspond to the direction of the molecules when they come into the plane due to the helical rotation. The structure shown in figure 8(a) explains in a clear way the CC textures shown in the different photographs. However, when the distance between the two 'eyes' decreases, it is possible to reach cases corresponding to mechanically stable structures similar to that represented in figure 8(b). In this case, the detected textures must be similar to those represented in figures 3(d) and 4.

From the results presented and discussed above, it is possible to conclude that in general, for compounds showing TGB-isotropic or TGB-L<sub>1</sub>-isotropic phase transitions, it is possible to observe CC textures on cooling very slowly from the isotropic phase. The exception observed with compound 16BT9\* could arise from strong planar anchoring of the molecules onto the glass plates, thereby forcing the helical layers to be parallel to the plates. In general, however, the anchoring should be more or less random on the plates, the more so because their surfaces were not particularly treated. The CC textures could then form preferentially in place of planar helical textures, essentially because of the large versatility of configurations that they offer, with many different shapes, sizes and orientations of the domains; they are thus more easily able to adapt to uncontrolled anchoring conditions than the monocrystalline helical textures.

When a small pressure is exerted onto the cell as described above, the resulting shear, in general, orients the smectic blocks and the helical layers parallel to the plates, subsequently destroying the CC domains. Thus, planar textures characteristic of the helical organization are produced and observed.

With the compounds of series III and IV which exhibit more complex polymorphisms, namely cholesteric and blue phases at higher temperatures, the results are very surprising. In fact, for compound III, a special kind of CC texture is observed in the TGBA mesophase. This texture is destroyed on crossing the TGBA-N\* transition with increasing temperature or on crossing the TGBA-SmC\* transition with decreasing temperature, and also as for the other compounds, by applying a shear to the preparation. For compound IV which shows a similar polymorphism to that exhibited by III, we did not observe a similar behaviour, but detected for the first time by optical microscopy an apparent square lattice in a TGBA mesophase. This observation requires further work. However, in special conditions with compound IV, it was possible to observe a kind of limiting case of the CC texture which may also resemble a focal-conic defect as explained previously [6]. The texture presented in figure 7(b) could then be considered as the limiting case of figure 8(b).

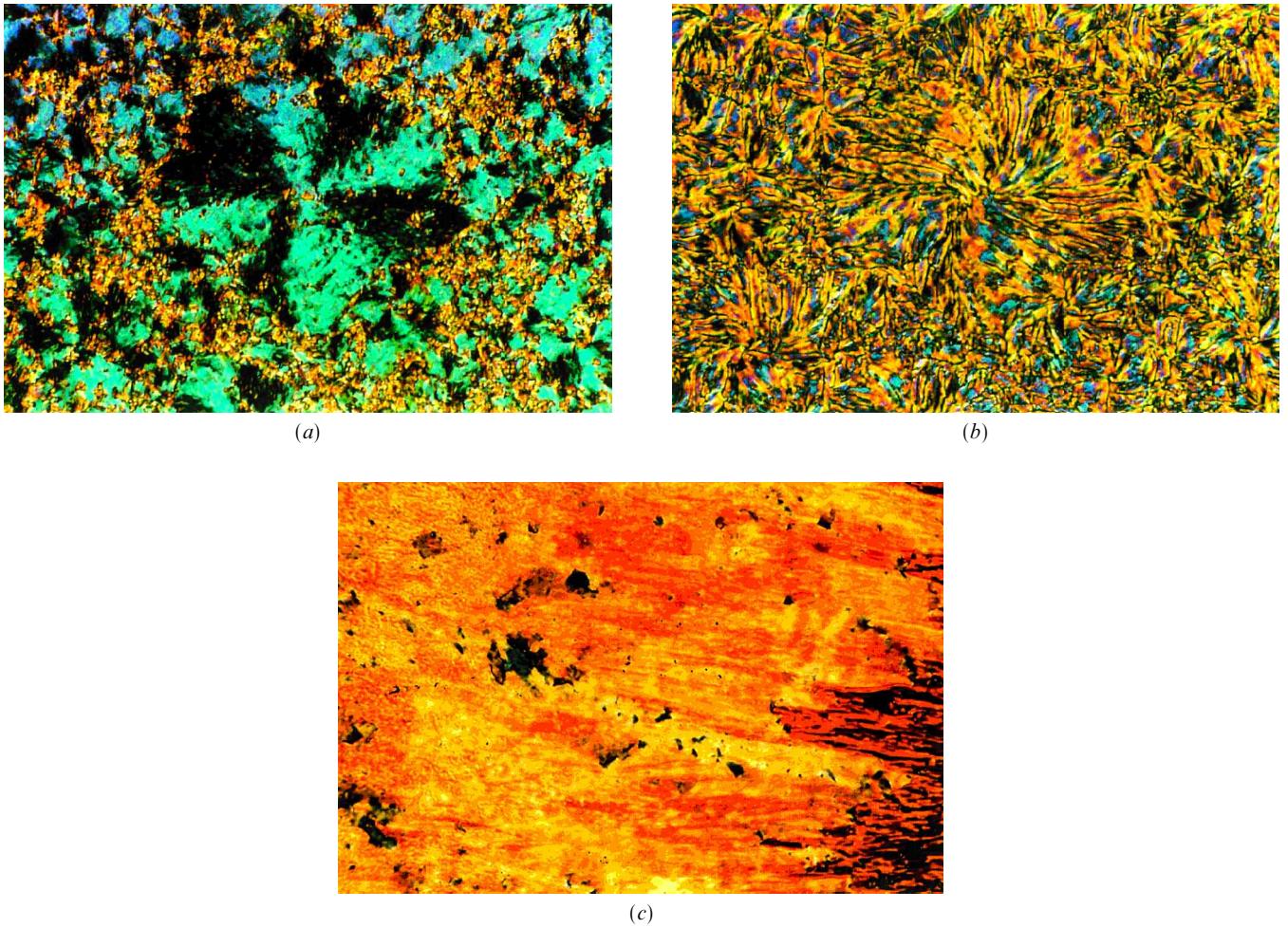


Figure 6. Textures observed with compound **III**. (a) CC texture detected in the TGBA mesophase at  $T = 102^\circ\text{C}$ , spoiled with many defects; (b) texture observed in the same areas as (a) at  $T = 105.8^\circ\text{C}$ , above the TGBA to  $N^*$  phase transition; (c) texture observed in the TGBA mesophase at  $T = 102^\circ\text{C}$ , after applying a shear stress to a preparation similar to that presented in (a).

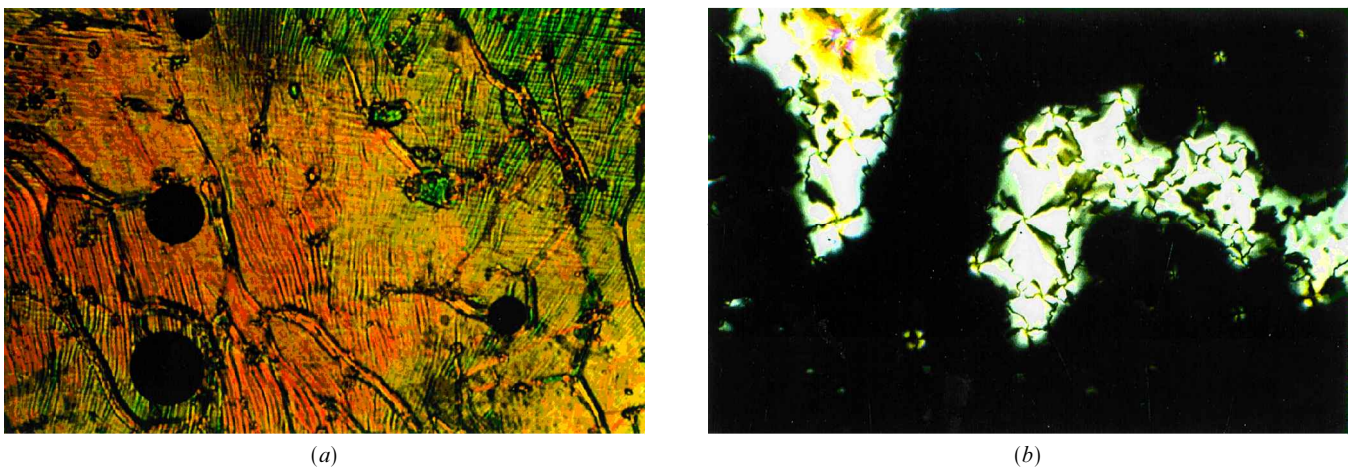


Figure 7. Textures detected in the TGBA mesophase of compound **IV**. (a) Texture with an apparent square grid in the top right corner, observed at  $T = 103.2^\circ\text{C}$ ; (b) CC or focal-conic type texture observed with a thin preparation of the liquid crystal.



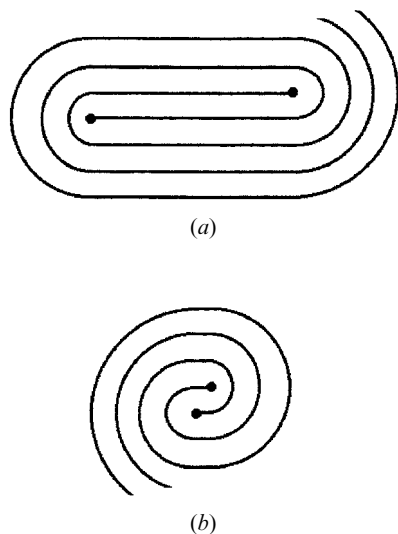


Figure 8. Two cylindrical planar domain structures. Both structures are equivalent, but the distances between the two 'eyes' associated with the axes of the cylinders are different.

In conclusion, we have presented here some experimental evidence that it is possible to observe two different types of optical textures in TGBA mesophases. One (helical texture) is characteristic of a cholesteric organization and the other (CC texture) is apparently similar to textures observed for some columnar mesophases, with however some perturbations when the TGB phase does not form directly from the isotropic phase. Moreover, both textures can coexist in the same preparation. The detection of these two types of texture, in the same compound at a given temperature, could be then considered as one of the signatures of the TGBA mesophase.

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