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Abstract. — A new phase sequence: twist grain boundary S_A to blue phases without cholesteric phase inbetween has been recently observed in a chiral compound. We have performed X-ray studies on this compound to analyze the smectic ordering through the phase sequence. The main result is that smectic ordering still persists in blue phases with a correlation length lower than the pitch.

Résumé. — Une nouvelle séquence de phase : phase à joints de grains suivie directement par des phases bleues sans phase cholestérique a récemment été découverte dans un composé chiral. Nous avons analysé ce composé en rayons X pour étudier l'ordre smectique à travers la séquence de phases. Le résultat principal que nous avons obtenu est la persistance de l'ordre smectique dans les phases bleues avec une longueur de corrélation inférieure au pas TGB.

Introduction

Twist grain boundary phases, or TGB phases, first predicted by Renn and Lubensky [1], have been extensively studied since they were experimentally discovered in chiral thermal liquid crystals by Goodby et al. in 1989 [2]. Several TGB phases as TGB_A and TGB_C have yet been identified [3]. These phases are usually observed when cooling the isotropic phase and the classical phase diagram is:

Isotropic  -  Blue phases  -  Cholesteric  -  TGB_A  -  TGB_C  -  SmC*.

A twist–grain boundary phase is characterized by both smectic ordering and twist. In the SmC* the twist axis is parallel to the normal to the smectic layers whereas, in the TGB phases, the twist axis is parallel to the smectic layers. A continuous twist of the director in this direction is not compatible with smectic ordering. Thus Renn and Lubensky have proposed a model where twist occurs between blocks or grains, of size l_b in the twist direction, with perfect smectic ordering. This implies the existence of walls between the grains and the presence of an array

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of parallel screw dislocations in each wall. If the screw dislocations within the grain boundary are separated by a distance \( l_d \), then the rotation angle (for small angles) of adjacent blocks is given by \( \Delta \theta = \frac{d}{l_d} \) where \( d \) is the smectic period. The size of the blocks \( l_b \) is also linked to \( \Delta \theta \) through the relation:

\[
\frac{\Delta \theta}{2\pi} = \frac{l_b}{\lambda}
\]

where \( \lambda \) is the twist period. Therefore the two lengths \( l_b \), the size of the blocks, and \( l_d \), the distance between the screw dislocations, are related to \( \lambda \) by:

\[
l_d l_b = \frac{d\lambda}{2\pi}
\]

A reasonable estimation of the size of the blocks can be obtained by taking:

\[
l_d = l_b = l \approx \sqrt{\frac{d\lambda}{2\pi}}.
\]

Thus the size of the blocks is intermediate between the smectic period (typically a few tenths of Å) and the pitch (a few thousands of Å).

Blue phases are observed in compounds with strong chirality and, therefore, they are sometimes observed between the isotropic and the cholesteric phase in the TGB materials [3–5]. Recently [6, 7] a new sequence was discovered in a chiral material where no cholesteric phase has been detected: the announced sequence of phases is, with decreasing temperature:

\[
\text{Isotropic} - \text{Blue phases} - \text{TGB phases} - \text{SmC*}.
\]

Upon heating, only two blue phases have been observed, probably BP2 and BP3 whereas, upon cooling, there appear three blue phases, probably BP1, BP2 and BP3. The phase diagram has been established using calorimetric studies, optical microscopy and the pitch has been measured using the Grandjean–Cano method. Under optical microscope, the blue phases exhibit mosaic textures very similar to that of classical blue phases [6]. The arising questions are then: does the smectic ordering can persist in the blue phases? Can TGB blue phase exist? Such questions are not new since in [8], Önusseit and Stegemeyer have observed a direct smectic to blue phase transition and in [9], Demikhov et al. have reported the observation of a new metastable blue phase called BPS with smectic ordering: this blue phase is observed when the cholesteric range is small enough so that smectic ordering can persist. In our compound there is no cholesteric phase and the blue phases are equilibrium phases. The purpose of this paper is to determine whether or not the smectic ordering persists and with what correlation length. The best appropriate tool to test the local ordering is X–ray scattering and, thus, we present in this paper X–ray diffraction patterns on one of the compound (called FH/FH/HH–18BTMHC) described in [6] and [7]. In the first section, we shall recall the main features of X–ray scattering on TGB phases. Section 2 will be devoted to the description of the experimental set–up. In Section 3, we shall present the main experimental results. The important result of our experimental study is that smectic ordering persists in blue phases with a correlation length which is lower than in the TGB\(_A\) phase. Double twist would occur between finite smectic blocks whose lateral size in all the lateral directions perpendicular to the normal to the smectic planes is the same as the size of the TGB\(_A\) smectic blocks along the pitch direction.

1. X-Ray Scattering on TGB Phases

Several X–ray scattering experiments on TGB phases have already been performed [10, 11]. They are in good agreement with the theoretical prediction of Renn and Lubensky [1]. In their
model, the rotation of blocks of smectic layers is induced by grain boundaries of screw dislocations. As recalled in the introduction, the size \( l \) of the blocks is typically given by:

\[
l \approx \sqrt{\frac{d\lambda}{2\pi}}. \tag{1}
\]

In our compound, \( \lambda \) is about 0.2 \( \mu \)m and \( d \) is about 46 Å; that gives \( l \approx 120 \) Å. It is important to notice that this value is much smaller than the correlation length of the smectic ordering. The smectic correlation length estimated by Srajer et al. in the TGB\(_A\) phase is indeed larger than 5000 Å [10].

Let us first consider the scattering pattern predicted for an aligned sample. A sample is aligned when there is only one direction of twist. Let us call \( z \) this direction. The normal to the smectic planes is then in the \((x, y)\) plane and, if the angle between the smectic blocks \( \Delta \theta \) is not commensurate with \( 2\pi \), this normal takes all the directions in that plane. The contribution of each block to the scattered intensity is two peaks situated in the \((x, y)\) plane at a distance \( Q_0 = \frac{2\pi}{d} \). Since the blocks have a finite size \( l \) in the \( z \) direction, the peaks are broadened along this direction. The rotation of the blocks around the \( z \) direction generates a scattering cylinder of axis \( z \), of radius \( Q_0 \), whose height is described by a Gaussian of characteristic width \( L \):

\[
L = \frac{2\pi}{\sqrt{\lambda d}} = \frac{\sqrt{2\pi}}{l} \tag{2}
\]

Srajer et al. [10] have performed a detailed comparison between the experimental scattering from the TGB\(_A\) phase and the structure factor predicted for this phase. They showed that the Gaussian line shape of the Bragg peak and its width are in good agreement with the TGB model.

Let us now consider the diffraction pattern of a non-oriented sample of TGB\(_A\). In such a sample there is no preferred orientation of the twist axis. The scattering pattern of a powder can thus be obtained by rotating in all directions the scattering pattern of an oriented sample. One obtains a sphere whose interior radius is \( Q_0 \). The intensity for \( Q > Q_0 \) is given by:

\[
I_P(Q) \propto \frac{1}{Q} \exp\left(\frac{Q^2 - Q_0^2}{L^2}\right). \tag{3}
\]

The real scattered intensity also depends on the resolution of the experimental set-up. Let us call \( I_R(Q) \), the resolution function of the set-up. In the following, \( I_R(Q) \) will be taken as a Gaussian function of width \( R \). The real scattered intensity is then the convolution of \( I_P(Q) \) with \( I_R(Q) \), that is:

\[
I(Q) \propto \int_{Q_0} dQ' \frac{1}{Q'} \exp\left(\frac{-Q'^2 - Q_0^2}{L^2}\right) I_R(Q - Q'). \tag{4}
\]

The resulting profile is asymmetrical: it is broadened for larger \( Q \). Such a profile has already been observed (3) and analysed with use of the function \( I(Q) \) previously introduced. Let us notice that the theoretical profile does not include the fluctuations of the smectic layers, this fact was used by the authors of (3) to explain the slight discrepancy they have found between the theoretical value of \( L \) and the measured one.
Fig. 1. — Calorimetric study of the compound performed on a Perkin–Elmer thermal analysis system (DSC 7; 0.2 °C/min).

2. Experimental Methods

The formula of the compound we have used in our experiments is given below:

\[
\text{CH}_3 \quad \text{COO} \quad \text{C} = \text{C} \quad \text{COO} \quad \text{CH}_2-\text{(CH}_2\text{)}_6\text{H} \\
\text{H(CH}_2\text{)}_{18} \quad \text{F} \quad \text{F}
\]

It has been studied by calorimetry (Fig. 1), optical microscopy and Grandjean–Cano method. We assume that the blue phases which present a platelet texture under microscope are still BP1 and BP2. An optical study using Kossel diagrams would be necessary to confirm the symmetry of these phases.

X–ray experiments were performed in LURE (Orsay, France) using synchrotron radiation. The wavelength (\(\lambda = 1.45 \ \text{Å}\)) was selected using a Ge monochromator. The X–ray beam at the sample position probed a 0.5 mm\(^2\) area and the beam was focused on the detector (imaging plate) situated at about 400 mm from the sample. Powder samples were prepared in a 1 mm–diameter Lindemann capillary. The capillary was placed inside a Mettler hot stage whose glass windows had been replaced by Kapton windows to reduce X–ray absorption. The temperature of the sample could be controlled up to 0.1 °C.

Experiments were performed in two steps. The purpose of the first step was to control the transition temperatures. The set–up we used is schematically described in Figure 2. A temperature ramp (either heating or cooling) was programmed in the Mettler hot stage.
Fig. 2. — X–ray experimental set–up. The wavelength (1.45 Å) of the synchrotron radiation is selected by a curved Ge monochromator. The collimator diameter is 0.5 mm. The compound is contained in a 1 mm diameter Lindemann capillary. This capillary is placed in a Mettler hot stage whose glass windows have been replaced by kapton windows. The detector (Fuji imaging plate) is placed at about 40 cm far from the sample. This distance has been precisely determined using a well–known paraffinic sample. To obtain a continuous recording of the smectic ordering as function of temperature, we keep only the horizontal scattering with the help of an horizontal slit (2 mm width) placed at about 5 cm in front of the detector. The detector is moved vertically (1.5 mm/min) as the temperature of the hot stage is changed at a typical rate of 0.2 °C/min.

The temperature rate was 0.2 °C/min and was the same as that used in the calorimetric studies in order to relate the diffraction patterns to the phase diagram previously established. For a given temperature, the scattering pattern was a regular ring as expected from a powder sample (Fig. 2a). Then horizontal scattering was selected (Fig. 2b) using an horizontal slit (2 mm width). At a given temperature, the selected scattering gave only two horizontal spots on the imaging plate lying on an horizontal line. The detector was moved vertically at a constant speed of 1.5 mm/min while the sample temperature varied at the constant rate of 0.2 °C/min. We thus obtained as scattering pattern two symmetrical lines corresponding to a vertical motion of the selected Bragg spots. This time–resolved scattering pattern was then converted into a temperature–resolved one. In such an experiment, the phase transitions in the sample can be detected by change in the position or in the width of the line, or even emergence of new lines (for instance, when crystallization occurs). When the transition temperatures were well–established, the temperature of the sample was kept constant (second step). The scattering
pattern was detected on the fixed detector without slit (Fig. 2a). In that configuration, the scattering pattern was a ring. Different patterns have been recorded at different temperatures and the profile of the rings have then been analyzed for different phases when the sample was heated or cooled.

3. Experimental Results

3.1. Temperature-Resolved X-Ray Scattering Patterns. — Recordings of the horizontal scattering of a powdered sample as a function of temperature are shown in Figure 3. In Figure 3a, the capillary containing the compound has been slowly heated from 60 °C to 76 °C at the rate of 0.2 °C/min. At low temperature (T < 66 °C), the sample is in a SmC* phase: the horizontal scattering shows two sharp spots. The first change is observed just above 67 °C: the two spots are still observed but they continuously shift towards lower value of Q between 67.3 °C and 68.9 °C. This temperature range corresponds to the TGB_C phase whose smectic period slowly increases with temperature. Then, between 68.9 °C and 71.7 °C, the position of the peaks and its width remains constant. This is the domain of the TGB_A phase whose smectic period is larger than that of the SmC* phase. Above 71.7 °C, we still observe two peaks, but their intensity is lower than in the previously encountered phases. With increasing temperature, these peaks move towards larger Q and get broader. A new change in the diffraction pattern occurs around 73 °C. Above this temperature, only diffuse scattering is observed: the sample is in a liquid state. A slower ramp of temperature (0.1 °C/min) heating from 70 °C to 76 °C (Fig. 3b) enables focusing on blue phases domain.

These transitions have also been observed on cooling. Figure 4 shows a continuous decreasing of temperature from 76 °C to 70 °C at the rate of 0.1 °C/min. At high temperature, we recover the diffuse scattering of the isotropic phase. Different transitions are observed. Around 74 °C, the diffuse scattering gets more localized, that means that the correlation length of the smectic ordering increases. The localization of the scattered intensity still improves around 72.5 °C and a discontinuity in the intensity of the peak is clearly observed at this temperature. The position of the peak moves towards lower Q until the temperature reaches about 71.2 °C. Then the peaks are sharp and do not move significantly. Below 68.5 °C, the peaks move towards
larger $Q$. This behaviour can be related to the phase diagram established by DSC scan and polarizing microscopy: blue phases have been observed between roughly 73 °C and 71 °C, the TGB$_A$ phase between 71 °C and 68.5 °C, and the TGB$_C$ phase below 68.5 °C.

At this point of the discussion, several points are worth emphasizing:

- we recover that the domain of the TGB$_C$ phase is larger when cooling, this is a classical observation for TGB phases. This fact is clear when focusing on the shift towards larger $Q$ which occurs between the TGB$_A$ phase and the SmC* phase through the TGB$_C$ phase. Comparing the continuous recordings, this shift is steeper when heating than when cooling;

- at high temperature, in the liquid state, the smectic peak looks like a classical diffuse peak. At low temperature, in the TGB$_A$ phase, this peak is sharp and intense. It seems that the transition between these two states occurs in several steps at the molecular level. When cooling, we observe first a localization of the diffuse ring around 73.5 °C: this means that the correlation length of the smectic ordering increases. Then, around 72.7 °C, this localization sharply increases but the peak is still broader than in the TGB phase. Then the width of the peak remains constant below about 71.2 °C. Unfortunately, the blue phase domain is so tiny that, with such a method, it is hard to correlate the behaviour of the width of the smectic peak to phase transitions and more precise studies are needed. Nevertheless, we can already conclude that:

  - some phase transitions can really be detected using continuous recording of X-ray scattering as a function of temperature;
  - the transition temperatures we find are in relatively good agreement with those measured by DSC;
  - blue phases which exist between 71.7 °C and 73 °C exhibit local smectic ordering whose correlation length is larger than in the isotropic liquid state.

### 3.2. Rough Analysis of the X-ray Scattering as Function of Temperature

Quantitative variation of the position and the width of the smectic peaks as a function of temperature was performed by recording, at given temperatures, the full diffusion ring of a
powdered sample on a fixed detector. The measurements were done every 0.2 °C in the blue phase region and a slow rate (0.1 °C/min) was used for changing the temperature. The exposure time was 2 min: this implies that the rate of cooling or heating was smaller than in the continuous recording. Some of the patterns obtained by cooling are shown in Figure 5. The position of the maximum intensity and of the width of the diffusion ring have first been determined by a fit with a Gaussian function. Results are presented in Figure 6 for a temperature range close to the blue phase domain.

When heating from the TGBA phase, the position of the maximum of intensity \( Q_{\text{max}} \) increases smoothly when temperature increases. This implies that the length correlated to \( Q_{\text{max}} \left( d = \frac{2\pi}{Q_{\text{max}}} \right) \) slowly decreases with increasing temperature. In the same way, when cooling from the isotropic phase to the TGBA phase, \( Q_{\text{max}} \) decreases when temperature decreases. This observation is in good agreement with the continuous recordings. Looking on the width of the ring provides more information. Above 74.5 °C, one recovers the diffuse ring characteristic of an isotropic liquid phase. Above 73 °C (region II), the width of the ring strongly decreases with decreasing temperature. Between 71.5 °C and 73 °C (region I), the ring width still decreases with decreasing temperature, but smoothly. Below 71.5 °C, the width of the ring remains constant and is probably limited by the experimental resolution. Upon heating, we did not record as many patterns as upon cooling but the behaviour of the ring width seems to be the same.

Unfortunately, the temperatures at which we have observed some change in the scattering pattern upon cooling do not fit precisely with the transition temperatures measured by DSC. In the DSC thermograms, 71.5 °C is the beginning of the domain of the blue phases upon heating, but, upon cooling, 71.5 °C is situated in the domain of the BP2 phase. The temperature-resolved X-ray scattering pattern is not precise enough to calibrate

Fig. 5. — X-ray scattering patterns at given temperatures (upon cooling).
the experiment in temperature. In the future, we plan to use a better controlled hot-stage and to calibrate more precisely the temperature. Nevertheless, from a physical point of view, it seems obvious to associate the correlation length that decreases with the smectic ordering range. This implies the persistence of some smectic ordering in region I which would correspond to the blue phase domain. In this region the correlation length is lower than the resolution of the experimental set-up and does not exceed 1000 Å. Let us recall that blue phases are characterized by a tri-dimensional periodicity of the molecular orientation and usually described by double twist of the director: it is not surprising that, in these complex phases, if a smectic ordering exists, its correlation length is smaller than the size of the elementary cell, that is typically the TGBA pitch (2000 Å). To estimate more quantitatively this correlation length, we have analyzed the profile of the ring for different temperatures between 68 °C and 74 °C.

3.3. INTENSITY PROFILE ANALYSIS BASED ON THE TGB MODEL. — The intensity profile of the scattering pattern of a TGB phase powder sample has been given in Section 1 (Eq. (4)): it is the convolution of the resolution function, a Gaussian of width $R$, and the intensity due to the smectic period, which depends on the length $L$ defined by equation (2). Several fits using the function defined by equation (4) have been made for temperatures below 71 °C (Fig. 7). The best agreement between the experimental data (full line) and the theoretical function (points) has been obtained with: $R = 0.0033$ Å$^{-1}$ and $L = 0.053$ Å$^{-1}$. This value of $R$ is larger than that measured on a lamellar paraffinic sample ($R = 0.002$ Å$^{-1}$) but remains
constant in the TGBₐ phase. The value of $L$, $L₁$, predicted by the theory is $0.02 \, \text{Å}^{-1}$. We find then an experimental value of $L$ which is 2.5 times the predicted one; this is coherent with other X-ray studies since, on oriented samples, Srajer et al. [10] have found an experimental value of $L$ that is twice the predicted one.

Above 71.5 °C (on cooling), that is in region I, the width of the smectic ring increases. Fitting with the function given by equation (4) (which we call the TGBₐ function) can still be done but $R$ and $L$ must both be increased, which is not pertinent. But, if this increase of the ring width corresponds to the appearance of blue phases, that means that simple twist must be replaced by double twist. In that case, the smectic grains are limited (size $L$) not only in one direction, but in all the directions perpendicular to the normal to the layers. Then the function we have to use in the fit is no longer the TGBₐ function which has been described
previously but a TGB$_{BP}$ function defined by:

$$I_{TGB_{BP}}(Q) \propto \int_{Q_0} dQ' \frac{1}{Q'} \exp \left( - \frac{Q'^2 - Q^2}{L^2} \right) I_R(Q - Q').$$  (5)

Fitting with this function in region I ($71.5 \, ^\circ C < T < 73.5 \, ^\circ C$) gives the same value of $L$ as in the TGB$_A$ phase ($T < 71.4 \, ^\circ C$), but the parameter $R$ of the resolution function must be increased. This can be interpreted by a decrease of the correlation length $\xi$ of the smectic ordering while the lateral size of the blocks remains constant. In a first approximation, $\xi$ in region I can be deduced from the value of $R$ given by the fit by taking $\xi = \sqrt{2\pi/R}$. This gives a correlation length $\xi = 280 \, \text{Å}$, that is about 6 layers at $72.2 \, ^\circ C$. In this temperature region, we have noticed that the fit is not sensitive to the form of the resolution function and replacing the Gaussian resolution function by a Lorentzian function of the same width gives the same result for $R$ and $L$.

Above $73.2 \, ^\circ C$, the width of the ring quickly increases: a fit by a TGB$_{BP}$ function gives increasing value of both $R$ and $L$. As we are close to the isotropic phase, the fit with Gaussian functions is certainly not the best and therefore we have tried to fit with convolution of Lorentzian functions. To compare with the previous results, we call $R$ and $L$ the parameters of the Gaussian functions of the same width at medium height as the corresponding Lorentzian functions. Figure 7g shows the fit of the experimental data with the convolution of Lorentzian functions using values of $R$ and $L$ close to those used in the fit of the profile at $73 \, ^\circ C$ done with Gaussian function. The agreement is quite satisfying.
Table I. — Table of the Gaussian parameters $R$ and $L$ given by the different fits for different temperatures.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>fit type</th>
<th>$R$ (best fit) (Å⁻¹)</th>
<th>$L$ (best fit) (Å⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>70.6</td>
<td>TGB$_A$</td>
<td>0.0033</td>
<td>0.055</td>
</tr>
<tr>
<td>71.0</td>
<td>TGB$_A$</td>
<td>0.0033</td>
<td>0.051</td>
</tr>
<tr>
<td>71.2</td>
<td>TGB$_A$</td>
<td>0.0033</td>
<td>0.055</td>
</tr>
<tr>
<td>71.4</td>
<td>TGB$_A$</td>
<td>0.0033</td>
<td>0.055</td>
</tr>
<tr>
<td>71.6</td>
<td>TGB$_A$</td>
<td>0.0055</td>
<td>0.075</td>
</tr>
<tr>
<td></td>
<td>TGB$_{BP}$</td>
<td>0.0040</td>
<td>0.053</td>
</tr>
<tr>
<td>71.8</td>
<td>TGB$_A$</td>
<td>0.0065</td>
<td>0.075</td>
</tr>
<tr>
<td></td>
<td>TGB$_{BP}$</td>
<td>0.0060</td>
<td>0.055</td>
</tr>
<tr>
<td>72.2</td>
<td>TGB$_A$</td>
<td>0.0090</td>
<td>0.075</td>
</tr>
<tr>
<td></td>
<td>TGB$_{BP}$</td>
<td>0.0100</td>
<td>0.050</td>
</tr>
<tr>
<td>73.0</td>
<td>TGB$_{BP}$</td>
<td>0.0120</td>
<td>0.055</td>
</tr>
<tr>
<td>73.5</td>
<td>TGB$_{BP}$Lor</td>
<td>0.0150</td>
<td>0.050</td>
</tr>
</tbody>
</table>

4. Discussion

The main result we have obtained is that smectic ordering which exists in the TGB$_A$ phase disappears in two steps when crossing the blue phase region to the isotropic state. In a first step (region I) the correlation length of the smectic ordering decreases smoothly with increasing temperature and the smectic domains are limited in every direction perpendicular to the normal to the layers. In a second step, the smectic ordering length quickly decreases with increasing temperature, in this region there would be only smectic fluctuations. Although the temperature range corresponding to the region I does not fit perfectly with the blue phases temperature domain as determined by DSC measurements, it seems reasonable to correlate this region with the blue phase domain. This implies that smectic ordering would be compatible with the tricky architecture of blue phases. The simplest and naive way to describe blue phases with smectic ordering is to replace the classical director by blocks of smectic layers with two characteristic length, one parallel to the director ($\xi$) and the other one perpendicular to it ($L$). This would imply the existence of a puzzling network of screw dislocations but probably also of edge dislocations. This model is too naive, but it has guided us to use a rather simple function (TGB$_{BP}$ function) in the fit. The fit with this type of function is not quite satisfying. A more sophisticated theoretical approach of such a phase is proposed by Kamien in [12]. In the future, we have planned new X-ray experiments using a better temperature controlled hot stage in order to properly characterize the molecular ordering of each of the three blue phases. Kossel line diagrams would also be useful to determine the crystallographic symmetry of these phases and the evolution of the cell parameter as a function of temperature. Moreover, although the platelet textures of these phases with smectic ordering are very similar to those of classical blue phases, one can wonder whether this ordering can modify the crystalline habit of blue phases.
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