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Temperature dependence of the helical period in the ferrielectric smectic phases of MHPOBC and 10OTBBB1M7

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We have measured the temperature dependence of the helical period in the ferroelectric, ferrielectric and antiferroelectric phases of (*R*)-MHPOBC and (*R*)-10OTBBB1M7. A combination of polarizing microscopy, measurements of optical rotation and the frequency dispersion of the electro-optic linear response has been used to determine both the magnitude and sense of the helical period.

1. Introduction

The ferrielectric phases of chiral smectic liquid crystals have attracted much attention recently in view of the continuing debate concerning the validity of the 1D-Ising [1] or 3D-XY clock model [2]. Evidence from various experimental studies has been presented, ranging from sophisticated resonant X-ray scattering experiments [3], optical ellipsometry [4] and optical rotation [5, 6]. The results of these experiments support the validity of the deformed clock model, first proposed by Akizuki *et al.* [7] and Muševič *et al.* [8] in 1999, based on the unusually large magnitude of the optical rotation in the ferrielectric phases. Recently, the original clock model, first proposed by Čepič and Žekš in 1995 has also been refined [9] and seems capable of generating deformed three- and four-layer unit cell structures, as observed experimentally.

One of the specific questions that seems not yet to have been addressed and thoroughly analysed, is the temperature dependence and sense of the helical period in the ferrielectric phases of a given material. As the helical period is a result of a subtle interplay between different and rather strong interactions that drive the onset of the distorted ferrielectric unit cell, it could be a stringent test for the validity of different theories. In this sense, reliable and accurate data on the temperature dependence of the helical period in the ferrielectric phases are clearly needed, as the available data are rather incomplete and difficult to compare. This has been the motivation for the present work.

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2. Experimental results and discussion

Three different experimental methods have been used to determine either directly or indirectly the magnitude and sense of the helical period in MHPOBC and 10OTBBB1M7. These are described below.

2.1. *Polarizing microscope measurements of the period of the helical structure in planar aligned samples*

Glass cells of thickness aroung 50 μm and with untreated surfaces were filled using the isotropic phases of the liquid crystal materials and set up with a temperature controlled stage in a 6.3 T magnet. The temperature of the sample was slowly decreased into the smectic A phase ($4\,\mathrm{K\,h^{-1}}$), which led to a very well aligned planar texture, as observed using a high resolution polarizing optical microscope. The sample was then cooled into the tilted phases. Whereas no change of the structure could be observed in the smectic C_α^* phase, a periodic parallel set of disclination lines was observed in the ferrielectric phases. The period of this periodic texture is directly related to the helical period of the structure. The average period and the error were determined by capturing the images and subsequently analysing them for the period at several places in the sample. This was repeated at different temperatures.

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2.2. *Measurements of the optical rotation (ORP) of the sample*

It is well known that extremely large ORP in liquid crystals is in most cases a direct consequence of the helical structure of the phase. In simple structures, such as the ferroelectric smectic C* and the antiferroelectric smectic C_A^* phase, the ORP can be calculated exactly using de Vries analytical theory. The ORP for light propagating along the helical axis is

$$\rho = \frac{\Psi}{d} = -\frac{2\pi}{p} \cdot \frac{\alpha^2}{8\lambda'^2(1-\lambda'^2)}. \qquad (1)$$

Here, Ψ is the angle of rotation of the polarization with respect to the input polarization, d is the thickness of the sample and p is the helical period. $\lambda' = \sqrt{2}\lambda_0/p(\varepsilon_\| + \varepsilon_\perp)^{1/2}$ is the reduced wavelength, λ_0 is the wavelength in a vacuum, and $\alpha = (\varepsilon_\| - \varepsilon_\perp)/(\varepsilon_\| + \varepsilon_\perp)$. Here $\varepsilon_\| = \varepsilon_2\varepsilon_3/(\varepsilon_2 \sin^2\theta + \varepsilon_3 \cos^2\theta)$ and $\varepsilon_\perp = \varepsilon_1$ are the dielectric contants in the direction perpendicular to the plane of the tilt (i.e. along the direction of polarization) and in the direction of the projection of the tilt onto the smectic layers, respectively. ε_1, ε_2, and ε_3 are the eigenvalues of the dielectric tensor, defined elsewhere [10] and θ is the tilt angle.

Note that for symmetry reasons, the ORP of ferroelectric and antiferroelectric structures with the same structural parameters (tilt, dielectric tensor and pitch) are the same. This is because the directions of θ and $-\theta$ cannot be distinguished by light, travelling along the helix. The helical period can therefore be extracted from the measurements of ORP, using equation (1), if one knows the value of the tilt angle and the dielectric tensor.

On the other hand azimuthal angles in the unit cells of ferrielectric phases are not exactly known and therefore the helical period in these phases cannot be determined from ORP data directly.

2.3. *Frequency dispersion of the linear electro-optic response*

As an independent test this can be used to determine the helical period. For a given helical and polar structure with a helical period p, the relaxation rate of the lowest polar mode is $\tau^{-1} = (K_3/\gamma)q_c^2$, where $q_c = 2\pi/p$, K_3 is the twist elastic constant and γ is the rotational viscosity.

Figure 1 (*a*) shows an example of disclination lines observed in the planar-aligned smectic C_{FI1}^* phase of MHPOBC, oriented in a strong magnetic field. From a number of measurements of the spacing between disclination lines, the helical period was determined at each temperature. In the smectic C_{FI1}^* phase of MHPOBC it equals approximately 2.5 μm and is practically independent of temperature. Figures 1 (*b*) and 1 (*c*) show similar disclination lines observed for the smectic C_{FI1}^* and smectic C_{FI2}^* phases of planar-aligned samples of 10OTBBB1M7.

Whereas microscope observations can give us only the magnitude, but not the sense of the helical period, this information can be extracted from the sign of the optical rotation of a liquid crystal. We have measured before the ORP in MHPOBC and 10OTBBB1M7 [5], and have also reported on the temperature dependence of the tilt angle and indices of refraction in these compounds [8, 11]. We can therefore reconstruct the sign and the magnitude of the helical period by combining the microscope observations and ORP measurements, as presented in figures 2 and 3.

Figure 2 shows the temperature dependence of the helical period in the ferroelectric smectic C*, ferrielectric smectic C_{FI1}^* and antiferroelectric smectic C_A^* phases of MHPOBC. Similarly, figure 3 shows the temperature dependence of the helical period in the ferroelectric smectic C*, ferrielectric smectic C_{FI1}^* and smectic C_{FI2}^*, as well as the antiferroelectric smectic C_A^* phases of 10OTBBB1M7. In the ferrielectric phases, the data were determined from the microscope observations, as here the helical period is large enough for the distance between disclination lines to be observed and accurately measured in the polarizing microscope. In the ferroelectric and antiferroelectric phases the values of the helical period were determined from the ORP data using

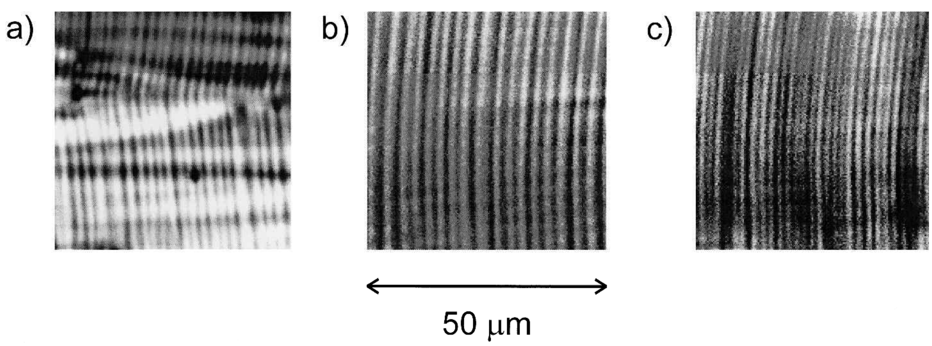

Figure 1. Disclination lines, observed in the polarizing microscope in (*a*) the planar-aligned smectic C_{FI1}^* phase of MHPOBC, (*b*) the smectic C_{FI1}^* phase of 10OTBBB1M7, and (*c*) the smectic C_{FI2}^* phase of 10OTBBB1M7.

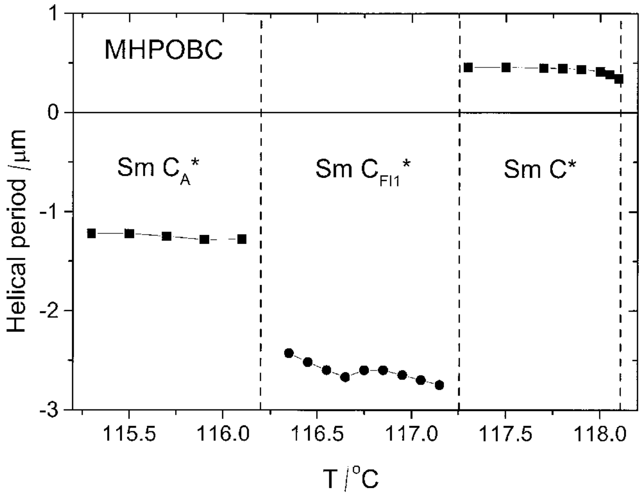

Figure 2. Temperature dependence of the helical period in the smectic C^*_{FI1} phase of MHPOBC measured from microscope observations of the disclination lines (●). The values in the smectic C* and smectic C^*_A phases are calculated from ORP data (■).

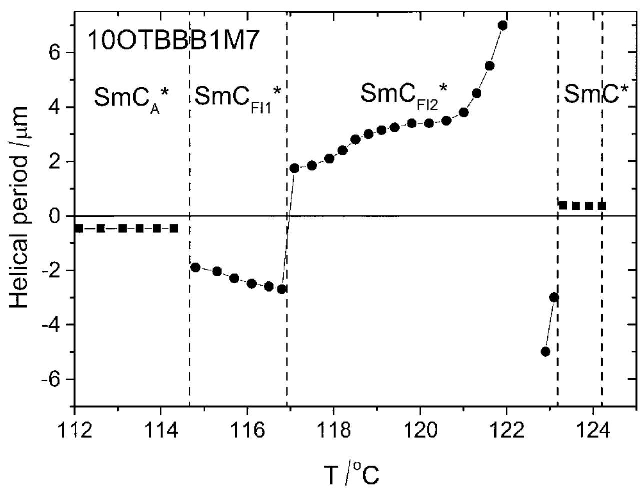

Figure 3. Temperature dependence of the helical period in the smectic C^*_{FI1} and smectic C^*_{FI2} phases of 10OTBBB1M7 measured directly by the observation of disclination lines (●). The values in the smectic C* and smectic C^*_A phases are calculated from ORP data (■).

equation (1). These data have also been used for the determination of the sense of the helical period in all phases for both mesogens. The helical period has the opposite sign to the sense of ORP, if the helical period is larger than the wavelength of the light in the liquid crystal. On the contrary, if the helical period is smaller than the wavelength of light in the liquid crystal, the sense of the helix is the same as the sense of optical rotation. This is, for example, the case for the smectic C* phase of 10OTBBB1M7.

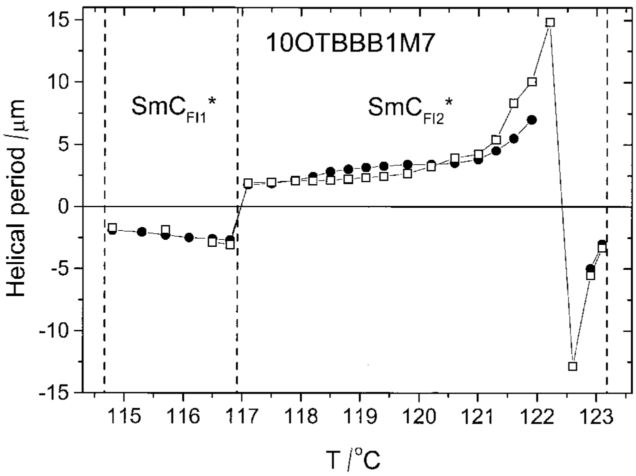

Figure 4. Temperature dependence of the helical period in the smectic C^*_{FI1} and smectic C^*_{FI2} phases of 10OTBBB1M7 measured directly by the observation of disclination lines (●) and determined indirectly from relaxation rates of the phase mode (□).

As an additional check, we present in figure 4 a comparison of the helical period in the ferrielectric phases of 10OTBBB1M7, as obtained from indirect electro-optic response measurements, and direct microscope observations. The helical period in the ferrielectric phases has been calculated from the temperature dependence of the relaxation rates in the ferrielectric phases. In the calculation we have used the ratio $K_3/\gamma = 2 \times 10^{-10}\ \mathrm{m^2\,s^{-1}}$, which was determined for the antiferroelectric phase. It is interesting that the helix in the smectic C^*_{FI2} is unwound and rewound in the opposite sense 0.8 K below the transition from the smectic C* phase. At this temperature the relaxation rate indeed goes to zero and the sign of ORP is changed.

3. Conclusions

We have measured the temperature dependence of the helical period in MHPOBC and 10OTBBB1M7 using three different methods. A consistent set of data was obtained that can be used for further fine tuning of the theoretical models of ferrielectric smectic phases.

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