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Band formation in a CPLC subjected to rectilinear oscillatory shear

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The response of a homeotropically aligned columnar phasmidic liquid crystal (CPLC) to rectilinear oscillatory shear was investigated for frequencies between 10^{-2} and 100 Hz and cell thicknesses between 20 and 100 µm. At threshold shear amplitude, we observed a spatial pattern consisting of stationary bands, perpendicular to the direction of shear and localized in the bulk of the columnar sample. For fixed thickness and at threshold shear amplitude, we observed band structure for several frequencies corresponding to integer harmonics v_o, $2v_o$, $3v_o$. We interpret the obtained results in terms of curvature walls that evolve gradually, according to the amplitude, to characteristic developable domain walls of the columnar phase.

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1. Introduction

The response of an aligned liquid crystal to rectilinear oscillatory shear was previously investigated for smectic A [1, 2], nematic [3, 4] and lyotropic [5, 6] phases. The shear induced banding effects in these types of material. This phenomenon has been the subject of much attention in the literature, since the formation mechanism of such bands is of interest in fundamental LC research. Current theory on the formation mechanism is varied and very much an open question.

It is well established that thermotropic hexagonal columnar mesophases can be exhibited by polycatenar compounds, which are molecules with a long rod-like rigid core ending in two half disks and three, four, five or six aliphatic chains in *ortho*-, *para*- and/or *meta*-positions grafted at each end [7, 8]. In previous studies, we examined the structural properties of these mesophases by diffusing biological stains in two geometries, along and perpendicular to the columns [9]. From the generalized hydrodynamic theory [10], a viscous behaviour would be expected. However, using Rayleigh scattering to characterize curvature distortions of the columns, an elastic behaviour has been observed [11]; we pointed out that an optically homogeneous sample is not synonymous to a monocrystal but is formed with a finite boundary.

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In this paper, we are concerned with the viscoelastic properties of hexacatenar columnar mesophases, subjected to rectilinear oscillatory shear with frequency v normal to the columns in homeotropic orientation. In fact, shear instability was observed. At the threshold appears a periodic pattern of bands, which depends on the shear amplitude, the frequency and the sample thickness. We demonstrate the first plastic behaviour of the hexacatenar columnar mesophase. The observed instability is equivalent to a competition between two elastic behaviours with an excited wave vector parallel to the column. In homeotropic orientation (figure 1), the columns perpendicular to the two plates at the

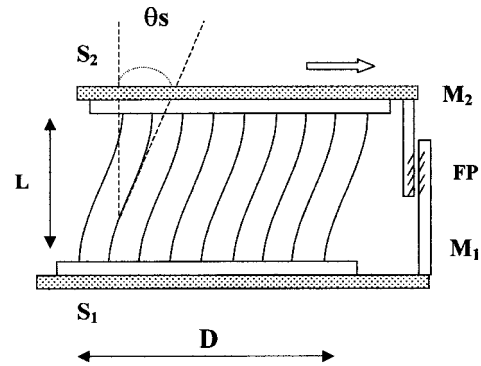

Figure 1. Evolution of the structure when applying alternating shear; the arrow indicates the direction of shear. FP = Fabry–Perot interferometer, M_1, M_2 = Fabry–Perot plates, S_1, S_2 = supports, L = sample thickness.

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sample boundaries have to be anchored. This forces the columns to be normal to these plates. The relative motion of the plates forces the columns to curve. One has two regions of opposite curvature near the upper and the lower plates (boundary layers). The column inclination needs curvature elasticity (which is similar to the splay of the disk planes). Let θ_0 be the inclination of columns out of the boundary layer ($\theta = 0$ on the surfaces). From figure 1 one can write θ_0 versus the displacement u as $\theta_0 = \partial u/\partial z$. If θ varies from 0 to θ_0 ($\theta = \theta_0 \sin 2\pi\nu t$) on a distance l, $\partial^2 u/\partial z^2$ will be of order θ_0/l and $\partial^4 u/\partial z^4$ of order θ_0/l^3. The same relation as in [1] gives

$$l \simeq \frac{m}{\theta_0} \qquad (1)$$

where $m = (K_3/B)^{1/2}$.

2. Experimental set-up

The sample used for the experiment is a hexacatenar mesogen with six benzene-ring cores, which exhibits two columnar phases. These columns are arranged in a hexagonal network. Unlike in the discotic columnar mesophases, where the disks are stacked one top of the other to form liquid-like columns [12], in the polycatenar mesophases, the slice of a column comprises a cluster of rigid cores arranged side by side and surrounded by aliphatic chains. Each slice is formed by three molecules of the hexacatenar. To distinguish this phase from the disk-like one, it is called 'phasmidic', undergoing the following temperature transitions:

$$\text{Cr } 70^\circ\text{C } \phi_{ob} \ 81.5^\circ\text{C } \phi_h \ 92^\circ\text{C I.}$$

ϕ_{ob} and ϕ_h denote phasmidic mesophases with respectively an oblique and hexagonal two-dimensional lattice.

The homeotropic orientation of the sample is obtained with the two glass plates treated with polyvinyl alcohol (PVA). In order to obtain a good orientation, the sample must be cooled very slowly ($0.01^\circ\text{C min}^{-1}$) from the isotropic to hexagonal phase [9]. So the sample is placed in an oven with a temperature regulation better than 0.1°C, which allows for a very low rate of cooling.

The dynamic shear cell is essentially formed by two glass plates whose thickness and parallelism are adjustable. The corner air angle α between the two glass plates is controlled by means of interference fringes in a parallel laser beam ($\lambda = 0.6328\ \mu\text{m}$). By observing the type of interference fringes and the value of i ($i = \Lambda/2\alpha$), one can characterize completely the parallelism, with a precision in the order 10^{-3} rd.

The thickness L of the liquid crystal sample is measured by a Michelson interferometer. As shown in [9], it is given by the relationship ($L = P_2 - P_1/n_i - 1$), where n_i is the isotropic index and P_1, P_2 are the respective positions of the adjustment of the optical contact in the absence and in the presence of liquid crystal in the isotropic state, with an uncertainty better than 5%.

The cell is composed of two glass plates (figure 2); the lower plate is cemented to a support S_1 the upper is fixed to a support S_2 which is connected to a loudspeaker. The displacement of the loudspeaker membrane allows for an alternating motion perpendicular to the column orientation. The loudspeaker is fed through a low frequency generator and an amplifier operating from 10^{-2} to 10^3 Hz.

The amplitude of the upper plate displacement can be progressively increased from 0.1 μm to several microns. This displacement is controlled by a Fabry–Perot interferometer formed by a mirror M_2, parallel to a separator M_1, which are respectively fixed to the supports S_2 and S_1. During shear, the system M_1, M_2 is illuminated by He–Ne laser beam ($\lambda = 0.6328\ \mu\text{m}$) which gives, by reflection, a system of rings. The defilement of the system of rings, when projected on a photomultiplier, allows us to measure the difference of the step ($\delta = 2e = k\Lambda$) and

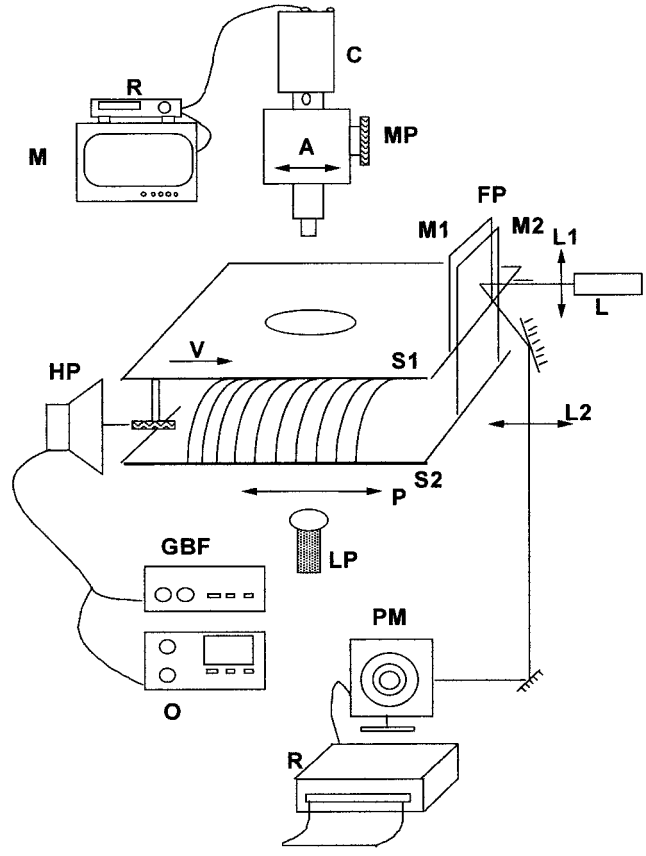

Figure 2. Experimental setup: FP = Fabry–Perot interferometer, M_1, M_2 = Fabry–Perot plates, S_1, S_2 = supports, P = polarizer, A = analyser, L = laser, M = monitor, C = camera, R = recorder, HP = loud speaker, LP = lamp, L_1, L_2 = lenses.

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to deduce the displacement amplitude ($e = k\Lambda/2$) with a precision better than 0.1 μm, knowing that k is the number of defiling rings for one half period of shear.

3. Experimental results

The experiment was possible because we were able, for the first time, to obtain good specimens with the columns oriented in a direction perpendicular to the plates (homeotropic orientation). The experiment is performed as follows: on a sample of thickness L, we apply the smallest possible shear deformation and measure the deformation amplitude by the Fabry–Perot interferometer.

3.1. *Band formation*

From the homeotropic orientation, the amplitude of shear was increased by an increment of 1 μm. During each increment, the whole of the available frequency range (10^{-2}–10^3 Hz) was swept and the evolution of the texture observed under the polarizing microscope. The experiment showed that for the threshold shear amplitude δ_s (9–11 μm) corresponding to a threshold shear angle $\theta_s = (\theta_o)_{max} \simeq \delta_s/L$, an instability of the structure characterized by the frequency v_0 is generated, over the whole of the sample diameter D (figure 1).

During the instability, we observed the appearance of a regular system of periodic bands perpendicular to the direction of shear, figure 3 (*a*). To detect these periodic bands within the sample, an incident light beam parallel to the columns was propagated through the sample placed between crossed polarizers. The transmitted light showed the periodic bands only for polarizer axes different from the shear axis. These bands were localized in the bulk of the sample, disappearing if the shear amplitude decreased below the threshold amplitude and disappearing also if the frequency was different from the band formation frequency. For threshold shear amplitude, we observed band structure at several harmonic frequencies, figure 3 (*b*). The obtained frequency values v versus the fundamental value v_0 are reported in figure 4. The same figure shows a linear behaviour $v = nv_o$, indicating that the harmonic frequencies are quantified. Note that we never observed the fourth frequency.

It should be noted that for frequencies between 10 and 100 Hz the critical oscillation amplitude for the onset of a spatial pattern, observed in polarized white light, could be reached. At low frequencies (f below about 15 Hz) no periodic band structure was observed. Above 100 Hz the maximum attainable amplitude of the loudspeaker was insufficient for the development of the spatial pattern. The experiment was carried out for cell thicknesses in the range $20\,\mu m < L < 100\,\mu m$. For small thicknesses ($L \approx 20$ μm and below) the contrast to the band

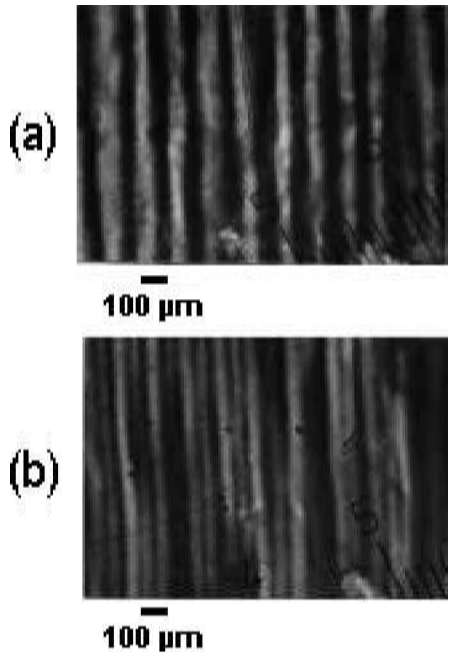

Figure 3. Typical pictures of lines seen under a polarizing microscope, generated when applying alternative shear. For the cell thickness $L = 80$ μm we observe: (*a*) shear lines at $v = 14$ Hz, (*b*) shear lines at $v = 27$ Hz.

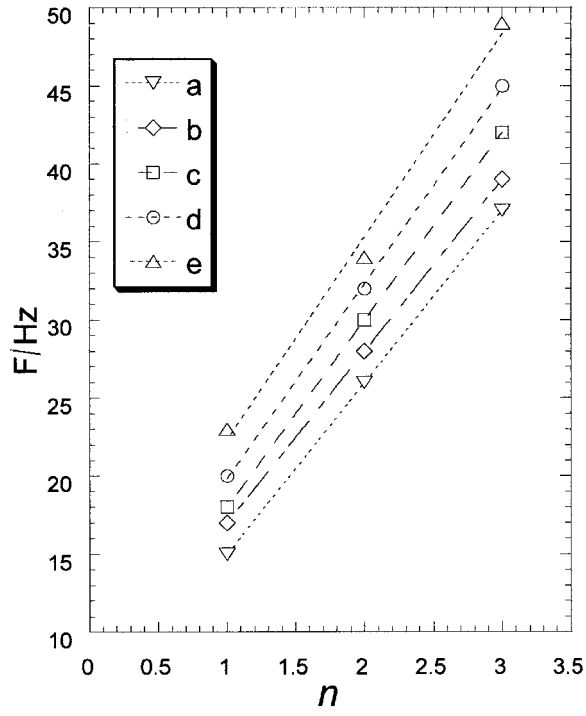

Figure 4. Measured frequency v versus the order n of the instability onset. The data were measured for cell thickness L of (*a*) 92, (*b*) 80, (*c*) 60, (*d*) 42, (*e*) 30 μm. The straight lines are plotted from the relation $v = nv_o$; v_o is the first mode.

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pattern diminished, thus making threshold measurements difficult. For thick samples ($L \geqslant 100\,\mu m$), it was difficult to obtain a good homeotropic orientation.

3.2. *Striations*

The sample was observed between crossed polarizers under a polarizing microscope, the incident polarization being selected with a linear polarizer. First, the angle α between the polarizer and the shear axis was adjusted to 45°, and the thickness fixed at 30 µm. On increasing the shear amplitude slightly above the threshold, the appearance of new group of very fine striations situated in the bulk of the sample, was observed figure 5(*a*). For thickness $L = 30\,\mu m$, the measured striation wavelength was 6 µm. When rotating the crossed polarizers and changing the angle α, the same striation wavelength was observed. However, the maximum optical contrast was observed for $\alpha = 45°$. Note that for $\alpha = 0°$ and 90°, behind the polarizers the extinction of the outgoing light was observed with no striation. It is deduced that the undulated columns remained parallel to the shear axis. The experiment was repeated with increasing thickness L, see figure 5(*b*) as an example; for $L = 60\,\mu m$, the measured striation wavelength was 8 µm. To explain these results, it would be tempting to simply transpose them in the frame of a previously published simple model [13], describing the column undulation instability. If y is the axis normal to the columns and z the axis parallel to them, the free energy density associated with a strain $\partial u/\partial y$ may be written as:

$$f = \frac{1}{2}B(\partial u/\partial y - \theta^2/2)^2 + \frac{1}{2}K(\partial\theta/\partial z)^2. \qquad (2)$$

Here B is a dilation elastic constant which takes into account both the column and the two-dimensional network elasticity; $\theta = \partial u/\partial z$ is the local bend angle of the column. The $\theta^2/2$ term is the additional strain due to the column rotation, taking into account that the number of columns between the two plates remains constant and that the column length is also probably fixed by the edges. The last term is the curvature energy of the columns with a curvature elastic constant K. Minimizing f, one obtains the relationship between the undulation wavelength λ, the sample thickness L and the characteristic length m,

$$\lambda^2 = 4\pi mL \qquad (3)$$

then it is deduced that the two thicknesses have the same approximate characteristic length $m \simeq 0.1\,\mu m$, as measured in previous studies [13, 14]; this result means that for ($\delta > \delta_s$), the structure of a columnar phasmidic liquid crystal (CPLC) undulates. In the next section, we will discuss the nature of the probable evolution of the sample structure.

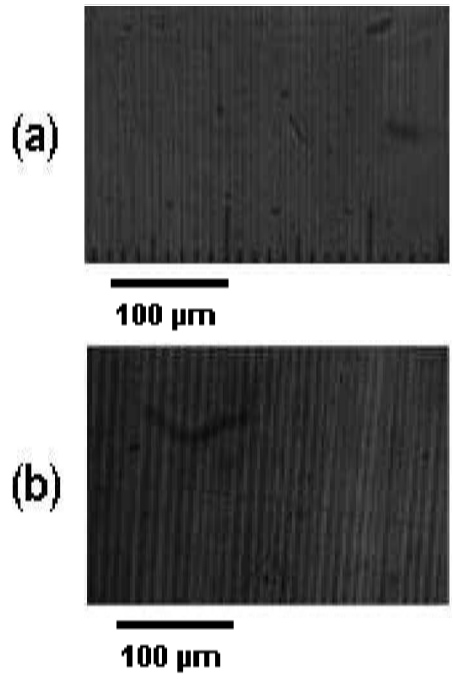

Figure 5. Typical pictures of the undulation mode, observed in polarized white light, above the threshold amplitude ($\delta_s \geqslant 12\,\mu m$), for cell thickness L of (*a*) 30, (*b*) 60 µm.

4. Discussion and models

The main result of the dynamic study shows that when a phasmidic sample is subjected to rectilinear oscillatory shear, an instability appears, manifesting as a set of defects in the form of quasi-periodic bands, perpendicular to the direction of shear and localized in the bulk. In discussing this result, let us first suppose that the number of columns submitted to a flexion deformation are preserved. One has always assumed the first curvature mode of flexion in all deformation to constant thickness [15]. This is probably the case in our study. In these hypotheses, let us now discuss the column configuration during shear and the nature of the sample relaxation.

4.1. *Column configurations*

The obtained instability includes three characteristic configuration types of columnar liquid crystals. First, the existence of bands observed for the first time by Bouligand for a columnar discotic liquid crystal (CDLC) when subjected to rectilinear steady state [16]. These bands are attributed to the existence of developable domain lines or developable domain walls. Second, the observation of fine striations, for ($\delta > \delta_s$), leading to competition between curvature energy and solid-like

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energy (typical of the hexagonal system) [13]. Third, the existence of boundary layers close to the walls limiting the sample studied by Palierne and Durand [17].

4.1.1. *Configuration of the shear instability in the bulk ($\delta = \delta_s$)*

In [16] it is shown that shear generated by the horizontal translation of plates induces many parallel and equidistant bands. These bands correspond to the presence of developable domain lines (DDL), figure 6(*a*). In the present work, using a dynamic shear, the same band type is observed. Qualitatively, the regularity of the bands corresponds probably to an association of many developable domain walls (DDW). Increasing the shear frequency, one creates the dynamic shear mode n with a wavelength $\lambda_n = 2L/n$ and a wave vector $q_n = 2\pi/\lambda_n = n\pi/L$, as represented in figures 6(*a*) ($n = 1$), 6(*b*) ($n = 2$) and 6(*c*) ($n = 3$). However, the experiment showed only the three first modes. The fourth was not observed, probably because of the proximity of walls.

4.1.2. *Column undulations ($\delta > \delta_s$)*

In previous work [14], the response of CPLC in planar geometry to steady dilation was investigated. Undulation of the structure was obtained at threshold dilation. In the same work the square of the wavelength of the stripes versus the thickness was measured. This mechano-optic effect is easily understood if one assumes that, during the time under study, the number of columns present in the sample thickness is constant. Then when the system is expanded, two types of behaviour may occur: either the columns expand uniformly (and this requires a rather high energy, proportional to the elastic modulus B) or they undulate the structure and provoke strong coupling between curvature elasticity and solid-like elasticity. In the present work, the stripes obtained for dynamic shear at amplitudes $\delta > \delta_s$, allow us to measure two wavelengths ($\lambda_1 = 6\,\mu m$ and $\lambda_2 = 8\,\mu m$), comparable to those obtained in previous measurements by dilation deformation [14]. The stripe appearance can be interpreted as follows: by increasing the shear amplitude $\delta > \delta_s$, one probably induces the rupture of the columns near the plates in a width l. When stretching the liquid crystal, there is a flow of matter from the plates to the bulk and the pressure decreases near the plates; this is equivalent to dilation normal to the columns. Then the columns relax by a classic undulation mode. The same found value $m \simeq 0.1\,\mu m$ in both cases seems to confirm our interpretation. Note that this type of relaxation is more general and was observed for the discotic free thin films and thread with thickness L [12]. In all cases the conditions define the same undulated wave vector equal to π/L.

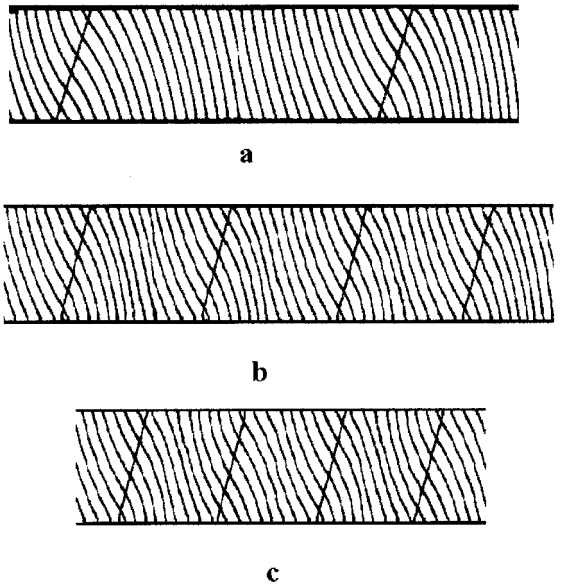

Figure 6. Configuration of the structure in the bulk for the first three modes with a wavelength $\lambda_n = 2L/n$, and a wave vector $q_n = 2\pi/\lambda_n = n\pi/L$. (*a*) $n = 1$, (*b*) $n = 2$, (*c*) $n = 3$.

4.1.3. *Boundary layers*

The boundary layer is the thickness in which some hydrodynamic parameters, in particular the velocity, that have a finite value in the bulk, adjust to a null value near the plates. This notion of boundary layers has been evoked to remove an ambiguity in the problem of layers fluctuations in smectics [10]. For columnar liquid crystals an estimation of this boundary layers thickness l to some micrometers has been made [17]. Using the found value $m \simeq 0.1\,\mu m$, the expression (1) mentioned above can be used to estimate the boundary layers width $l \simeq m/\theta_0$ as a few micrometers. This value is in good agreement with that obtained in [17].

4.2. *The dynamic shear instability frequencies*

We are interested now in the theoretical description of the behaviour of the dynamic shear instability. To calculate the band formation frequencies of the phasmidic sample, we suggest the following model. Let us assume that the sample consists of cylinders forming columns aligned along the z axis (figure 1). We call L the cylinder length and D its diameter. When an alternating shear is applied, the sample is submitted to two restoring forces from volume and surface tensions. Ref. [12] shows that the curvature energy becomes preponderant if $D > \gamma L^2/K_3$, where γ is the surface tension and K_3 the curvature constant. The resulting curvature tension is: $T = K_3 \pi D^2/L^2$ [18].

Assuming a perfect plastic relaxation of the sample, with a linear mass μ, the transverse vibration equation for a displacement y normal to the column axis (figure 1), is written: $\partial^2 y/\partial t^2 = T\mu\, \partial^2 y/\partial z^2$.

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For a cylinder of volume V, using a relationship between the linear mass μ and the volume mass ρ we obtain the relationship

$$\partial^2 y/\partial t^2 = TL/\rho V\, \partial^2 y/\partial^2 z. \quad (4)$$

The solution of equation (4), with a force of frequency $2v$ is

$$y = y_o \sin 2\pi(2vt - z/\lambda). \quad (5)$$

For the wavelength $\lambda_n = 2L/n$, the cylinder dynamic shear frequencies v_n are given by

$$v_n^2 = n^2 T/4\rho\pi D^2 L^2. \quad (6)$$

4.2.1. *Curvature energy*

If only the curvature energy $T = K_3\pi D^2/L^2$ [18] is taken into account, equation (6) gives:

$$v_n^2 = n^2 K_3/4\rho L^4. \quad (7)$$

We note that this expression of v_n is independent of the column diameter. Taking for $K_3 \approx 10^{-1}$ cgs, the effective value found for the phasmidic [14], $\rho \approx 1$ cgs and for the thickness $L = 80\,\mu$m, it is found that the order of the frequency magnitude is of some kHz. This expression allows us to explain neither the experimental frequency values for fixed thickness L, nor the evolution of structure from homeotropic state to the periodical distortions.

4.2.2. *Roles of developable domains*

Ref. [19], suggests the following expression, $E_D = (K_3\theta_s/2)\ln R/r$, for the DDW energy per unit length, where R is the radius of curvature, r the cur radius ($r \simeq 1\,\mu$m). By estimating the radius of curvature according to the thickness ($R \approx L/\cos\theta_s$, θ_s is the angle that the columns make with the normal, $\theta_s \approx 0.15$ rd), one obtains $E_D \approx 10^{-1}$ erg cm^{-1}. The same reference suggests the following expression

$$E_L = \left(\frac{\pi K_3}{2}\right) \ln\left(\frac{R^2 - r_o^2}{r_c^2 - r_o^2}\right),$$

for the DDL energy per unit length, where R is the radius of curvature r_c the cur radius ($r_c \simeq 1\,\mu$m) and r_o the cylinder radius of developable domain, r_o is null [19]. Taking the same experimental values as before, one obtains an estimation of $E_L \approx 1$ erg cm^{-1}.

By substituting the two tension values E_D and E_L into equation (6), one obtains the order of magnitude of v_n, the quantification of the frequency and the evolution of v_n versus the thickness ($v_n \sim n/L$). In particular, for $E_D \approx 10^{-1}$ erg cm^{-1} and sample thickness $L = \lambda_1/2 = 80\,\mu$m, the frequency estimated by relationship (6) is $v_1 \approx 15$ Hz (for $K_3 \approx 10^{-1}$ cgs), which is the order of magnitude of v_n to the first mode. One also obtains the order of magnitude of the second and the third modes ($v_2 \approx 31$ Hz, $v_3 \approx 46$ Hz), respectively for $\lambda_2 = L = 80\,\mu$m and $\lambda_3 = 2L/3 = 53\,\mu$m. These models allow a partial explanation of quantitative experimental observations. The order of the obtained frequency magnitude shows the probable existence of DDL or DDW. Theoretical models and dynamical experiments would be useful for confirming this point.

4.3. *Permeation process*

In the geometry of figure 1, the significant wave vector q is parallel to the columns. The corresponding displacement u must be associated with the characteristic time modulation of the columns. Let us suppose that the fluctuation modes of the columns are dominated by the permeation process. The displacement u versus the pressure p, the absicissa x, the viscosity η and the characteristic length m, obeys the characteristic permeation equation

$$\dot{u} = (m^2/\eta)\frac{\partial \rho}{\partial x} \quad (8)$$

introduced by de Gennes and the Orsay group [10], where $m^2/\eta = \lambda_p$ is the permeation constant. To estimate the permeation characteristic time τ, let us suppose that the sample geometry is a cylinder with diameter D. Writing the equality between the cylinder volume variation and the lateral flux of mass, one obtains the relationship $\dot{u} \sim (\delta/L)D/2\tau$. Then, the characteristic time τ is given by $\tau \sim (\delta/B)(D/2m)^2$, where B is the elastic modulus. Using the experimental values $m \simeq 0.1\,\mu$m, $D = 5$ mm, $B = 5 \times 10^8$ cgs [13] and the usual value $\eta = 0.1$ P, one obtains $\tau \sim 100$ ms. The corresponding order of frequency magnitude $v = 1/\tau \sim 10$ Hz shows that the permeation process can explain the nature of the mass transport from the boundary layers to the bulk.

5. Conclusion

In conclusion, we have presented the first example of phasmidic column instability under rectilinear oscillatory shear. By varying the frequency, we have observed the quantification of the wavelength distortion modes ($\lambda_n = 2L/n$). By increasing the amplitude, we provoked a classic undulation instability. At threshold amplitude $\delta = \delta_s$, we propose that the columns are disposed in developable domain lines (DDL) or developable domain walls (DDW). We interpret the evolution of the columnar structure versus the shear frequency and the shear amplitude, and have demonstrated that the column inclination at the sample boundaries is relaxed in a negligible width l, compared with the sample thickness L. DDW and DDL are not the only defects that can be seen in CPLC. Dislocations and curvature walls may occur, but it seems that plasticity is mainly responsible for the macroscopic fluidity of CPLC.

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This first viscoelastic work on a phasmidic liquid crystal predicts several experimental verifications. Nevertheless, it leaves a first fundamental question concerning the nature of the permeation and the dynamic process of the mass transport from the boundary layer to the bulk. It would be interesting to make the analogous investigation for a discotic liquid crystal, when direct observation of the band structure might be made by the Rayleigh scattering when the distortion mode coherence is sufficient.

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