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Dielectric properties of a helical smectic liquid crystal

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The temperature and frequency dependences of the permittivity were determined in the vicinity of the phase transition (in either direction) from the smectic C phase to the smectic A phase in a chiral smectic liquid crystal DOBAMBC. The results obtained were compared with the temperature dependences of the tilt angle θ of the molecules in the smectic layers, spontaneous polarization P_s , and helix pitch p_0 . The experimental results were analyzed using a dynamic model based on the phenomenological theory of the chiral smectic C phase put forward by Pikin and Indenbom. The relative role played by piezoelectric effects of various kinds in the spontaneous polarization of DOBAMBC was analyzed and conclusions were drawn.

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INTRODUCTION

The existence of ferroelectric properties in chiral smectic *C* liquid crystals, pointed out by Meyer *et al.*,^[11] has now been confirmed by numerous experimental measurements of the optical and polarization switching properties of this class of compounds.^[2-8] A distinguishing feature of liquid-crystal ferroelectrics is the "orientational" nature of the dipole ordering, associated with the short-range nature of the forces which give rise to a parallel tilt of the molecules in the smectic layers. In this case the polarization is due to piezoelectric effects of various kind allowed by the symmetry of chiral smectic *C* liquid crystals.

A characteristic feature of these crystals is also a helical distribution of the permanent dipole moments of the molecules in the smectic layers (Fig. 1a). In each helix pitch ($\sim 4\mu$) there are $\sim 2 \times 10^3$ discrete directions of the dipole moment in the layers (apart from allowance for thermal fluctuations of the directions of the dipole moment). If a sample is sufficiently thick, this helical structure corresponds to the equilibrium state of the chiral smectic *C* phase and its appearance as a result of a phase transition is due to the limiting nature of the symmetry group of the paraelectric hightemperature phase ($\infty/2$). As in the case of splitting of solid ferroelectrics into domains, the appearance of a helical structure helps to retain this macrosymmetry also in the polar phase.

The interaction of the dipole moments of the molecules with an external electric field results in a homogeneous polarization of a liquid crystal, i.e., it causes "untwisting" of the helix. Therefore, we may assume that the dielectric response of a liquid crystal system to an external alternating field includes important information on the features of the dipole ordering, dynamic processes associated with the deformation of the helix, and relative role of the piezoelectric effects of various kinds in the appearance of the spontaneous polarization. We shall report the temperature and frequency dependences of the permittivity in the vicinity of a phase transition (in either direction) from the smectic C phase





to the smectic A phase) and we shall compare these data with the temperature dependences of the tilt angle θ of the molecules in the smectic layers, spontaneous polarization P_s , and helix pitch p_0 . We shall analyze the experimental results on the basis of the dynamic model based on a phenomenological theory of the C - Asmectic phase transitions.^[9, 10]

MEASUREMENT METHOD

We investigated d-p-decyloxybenzylidene-p'-aminomethylbutyl cinnamate (DOBAMBC), which is a chiral smectic liquid crystal with the $C \leftrightarrow A$ transition at T_c =93°C. This substance was poured into the planar capillary gap 50-150 μ wide between two glass plates coated with conducting transparent films. A homogeneous orientation of the liquid crystal in the smectic Cphase (with the helix axis x in the electrode plane, Fig. 1) was obtained by cooling from the isotropic phase range (T > 116°C) to the smectic C phase in a magnetic field of H=15-18 kOe intensity. Visual examination of the sample in a polarizing microscope demonstrated that the orientation of the liquid crystal was homogeneous throughout the area of the sample.

The field E_p , capable of untwisting the helix completely was deduced from visual observation in the polarizing microscope: it was 400 V/cm at $T \approx T_c$, rising to $E_p = 5000$ V/cm at temperatures differing from the phase transition by $T_c - T = 20$ °C.

The real part of the permittivity was measured by the conventional bridge method (using a R551 bridge). A modified attachment to this bridge made it possible to carry out measurements during simultaneous application of a static untwisting field E_{m} . The precision of the absolute measurements of the permittivity ϵ was governed primarily by the accuracy of determination of the capillary gap in the area of the capacitor containing the liquid crystal, and it amounted to 4-5%. The measurements of the relative value of ϵ were accurate to within 0.2% in the range f > 800 Hz and to within 1% at $f \approx 40-60$ Hz. The temperature of the sample was kept constant to within 0.01°C by an automatic control system. The temperature was measured with a platinum resistance thermometer using a compensation circuit with a R306 potentiometer of the 0.05 class and this was done to within 0.01°C.

The appearance of a spontaneous dipole ordering in the chiral smectic C phase was accompanied by anomalies of the temperature and frequency dependences of the permittivity ϵ at right-angles to the helix axis. The value of ϵ depended on the amplitude E_{-} and frequency f of the alternating (measuring) field, static field E_{-} , proximity to the smectic $C \leftarrow A$ transition temperature $(T_c - T)$, and wave number q corresponding to the helix pitch p $(q=2\pi/p)$.

We shall now consider the influence of these factors on the dielectric properties of the smectic C phase.

EXPERIMENTAL RESULTS

1. Dependence $\epsilon = \epsilon(f, E_{\star})$ in the $T < T_c$ range. In zero static field the dependence $\epsilon(f)$ was clearly of



FIG. 2. Dependences of the permittivity ϵ on the frequency in various static electric fields $E_{\pm} \cdot 10^{-3}$ (V/cm): 1) 0; 2) 1; 3) 1.5; 4) 2.5; 5) 3.5; 6) 9.0. $T_c - T = 5^{\circ}$ C. Top right-hand corner shows the dependences $\epsilon = \epsilon(E_{\pm})$ obtained at various frequencies f(Hz): 1) 40; 2) 80; 3) 200; 4) 300; 5) 600; 6) 800; 7) 8000.

relaxational nature and the relaxation frequency f_{rel} depended on temperature, amounting to 150-400 Hz (Fig. 2). The application of the static field E_{\pm} reduced the dispersion which disappeared practically completely for $E_{\pm} \ge E_{\phi}$ indicating a relationship between the dispersion of ϵ in the low-frequency range and the helix untwisting dynamics.

2. Dependence $\epsilon = \epsilon(f, E_{\star})$. An alternating measuring field affected, like E_{\star} , the value of ϵ : it reduced considerably ϵ in the f < 200 Hz range (Fig. 3). The application of a measuring field resulted in partial periodic twisting and untwisting of the helix so that during the measurement period at frequencies $f < f_{rel}$ the average pitch $\langle p \rangle$ became greater than the equilibrium value p_0 .



FIG. 3. Frequency dependences of the permittivity ϵ for different values of an alternating measuring field E (V/cm): 1) 60; 2) 200; 3) 600; 4) 1100; 5) 1500; 6) 2150. $T_c - T = 3.5^{\circ}$ C.

The influence of the amplitude of the measuring field on the permittivity ϵ was particularly important at low frequencies in the vicinity of $T \approx T_c$. Therefore, the temperature dependences of ϵ were obtained in the f<800 Hz range using a weak measuring field $E_{-} \approx 60$ V/cm, which was an order of magnitude smaller than E_{ρ} at $T \approx T_c$; it is clear from Fig. 3 that in such fields the value of ϵ was practically constant and independent of the value of E_{-} .

3. Dependence $\epsilon = \epsilon(f, T - T_c)$. The dependence $\epsilon(f, T - T_c)$ definitely indicated a downward shift of the relaxation frequency $f_{rel} = \frac{1}{2}\pi\tau$ on increase of temperature (Fig. 4). Here, τ is the characteristic relaxation time of an ensemble of static dipoles. Consequently, the temperature dependences of ϵ obtained at various frequencies behaved differently in the vicinity of the transition from smectic C to the smectic A phase (Fig. 5). At the lowest frequencies the temperature dependences of ϵ passed through a maximum near T_c but at higher frequencies the maxima became degenerate and shifted toward lower temperatures. It was characteristic that the position of the transition temperature T_c , corresponding to the maximum of the static (f=0) permittivity ϵ_0 became indeterminate.

DISCUSSION OF RESULTS

1. Characteristics of the permittivity dispersion of the chiral smectic C phase. It is clear from the above data that at low frequencies (f < 400 Hz) there are relaxation processes which clearly correspond to displacements of large ensembles of molecules because of the interaction of their permanent dipoles with an external electric field. This interaction is manifested macroscopically as a periodic variation of the helix pitch in which the molecules perform matched motion on conical



FIG. 4. Frequency dependences of the permittivity at various temperatures near the smectic $C \leftrightarrow$ smectic A phase transition. $T_c - T$ (°C): 1) 12; 2) 10; 3) 8; 4) 6.5; 5) 4.5; 6) 3; 7) 2; 8) 1; 9) 0.5. The asterisk (*) denotes the dependence $\epsilon = \epsilon(f)$ for the smectic A phase.



4) 120; 5) 200; 6) 300; 7) 400; 8) 800; 9) 3000; 10) 8000.

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surfaces keeping the angle θ constant (Fig. 1). The observed low-frequency relaxation differs considerably from the dielectric relaxation effects known to occur in liquid crystals and to be associated with the lag of the rotation of the molecules about their long ($f_{rel} \approx 10^9 - 10^{10}$ Hz) and short ($f_{rel} \approx 10^6$ Hz) axes. ^[11,12]

The observed dependences of ϵ can be explained qualitatively as follows: at higher frequencies of the measuring field the partial untwisting of the helix does not occur because the dynamic viscosity is too strong and, therefore, the contribution of the orientational polarizability decreases at high frequencies. The application of a static field $E_{a} \ge E_{p}$ results in complete untwisting of the helix and then the permanent dipoles of the molecules become oriented along the field E_{a} . This reduces strongly the response of the dipole to the measuring field, the dispersion of the permittivity practically disappears, and there is no further change in ϵ on increase of the field.

We shall now consider in detail the behavior of a helical structure in an external alternating field. The free-energy density of the chiral smectic C phase subjected to an external field is^[10]

$$F = F_{o}(\theta) + \frac{1}{2} K_{s} \theta^{2} \left(\frac{d\varphi}{dz} - q_{o} \right)^{2} + \chi \mu_{i} \theta E \cos \varphi, \qquad (1)$$

where $d\varphi/dz = q$; $E = E_y$; $E_x = 0$; $q_0 = 2\pi/p_0$ (p_0 is the equilibrium helix pitch); K_3 is the elastic constant of the liquid crystal (longitudinal bending constant); φ is the azimuthal angle in the *xy* plane; θ is the tilt angle of the molecule in the smectic layers, which—in weak fields—is independent of $E(\theta \ll 1$, which is true near T_c); χ is the generalized susceptibility; μ_1 is the piezo-electric modulus corresponding to the linear piezo-electric effect of the $P \propto \mu_1 \theta$ type.

The equation describing the distribution of the angle $\varphi(z)$ in an external field is found by minimization of the functional (1) and is of the form

$$K_{3}\theta \frac{d^{2}\varphi}{dz^{2}} + \chi \mu_{i}E\sin\varphi = 0.$$
(2)

In an alternating field $E = E_0 e^{i\omega t}$ the equation of the helix untwisting dynamics derived ignoring the inertial term $(J d^2 \varphi/dz^2)$, where J is the moment of inertia of the molecule) can be written in the form

$$K_{s}\theta \frac{d^{2}\varphi}{dz^{2}} - \gamma_{s}\theta \frac{d\varphi}{dt} + \chi\mu_{s}E\sin\varphi = 0, \qquad (3)$$

where γ_1 is the dynamic viscosity coefficient.

In a weak field $E_0 \ll E_p$, where $E_p = K_3 q_0^2 \theta / 2\chi \mu_1$ is the field needed for complete untwisting of the helix, the solution of Eq. (3) can be found in the form

 $\varphi = q_0 z + A e^{i(\omega t + \alpha)} \sin q_0 z, \tag{4}$

where α is the phase shift between E and φ .

Substituting Eq. (4) into Eq. (3), we obtain

$$Ae^{i\alpha} = \chi \mu_i E_0 \tau / \theta \gamma_1 (1 + i\omega \tau), \tag{5}$$

where $\tau = \gamma_1 / K_3 q_0^2$. Hence,

$$A = \frac{\chi \mu_i E_0}{\theta \gamma_1 (1/\tau^2 + \omega^2)^{\nu_i}}, \quad \alpha = -\operatorname{arclg} \omega \tau.$$
(6)

The expression for the polarization along the y axis is of the form

$$P_{\nu} = -\chi \theta \left| \mu_1 - \mu_2 q_0 \right| \cos \varphi, \tag{7}$$

where μ_2 is the piezoelectric modulus describing the appearance of the polarization because of the bending and twisting of the director in space (flexoelectric effect). Substituting in Eq. (7) the value of φ from Eq. (4), where A is written in the form of Eq. (6), and bearing in mind that $A \ll 1$, we find that the spatially homogeneous component of the electric polarization along the y axis is

$$\mathbf{P} = \frac{\chi^2 \mu_1 |\mu_1 - \mu_2 q_0|}{2\gamma_1 (1/\tau^2 + \omega^2)^{\frac{1}{\gamma_1}}} E.$$
(8)

In the static case $(\omega \to 0)$ the expression (8) reduces to the expression for $|\mathbf{P}|$ obtained earlier^[10] and it represents the appearance of an additional contribution to the permittivity $\epsilon = \epsilon_{\infty} + 4\pi |\mathbf{P}| / E_0$:

$$\varepsilon = \varepsilon_{\infty} + \frac{2\pi \chi^2 \mu_i |\mu_i - \mu_2 q_s|}{\gamma_i (1/\tau^2 + \omega^2)^{\frac{\gamma_i}{\gamma_i}}},\tag{9}$$

where ϵ_{∞} is the permittivity associated with displacements (in the measuring field) of the induced dipoles and of the components of the permanent dipoles directed parallel to the long axes of the molecules.

The expression (9) describes the Debye relaxation with a characteristic time $\tau = \gamma_1/K_3q_0^2$. Substituting in this expression the typical values^[11] $\gamma_1 = 0.1 - 1P, K_3 = 10^{-6}$, and $p_0 = 4 - 8 \times 10^{-4}$ cm, we find that $\tau = 10^{-3} - 10^{-4}$ sec, which is in agreement with the relaxation frequency of ϵ amounting to $f \approx 400$ Hz. According to Eq. (9), the dispersion of ϵ in the chiral smectic *C* phase is governed by the relationship between the piezoelectric moduli μ_1 and $\mu_2 q_0$ representing piezoelectric effects of different kind, and the relaxation time τ . This relaxation time is governed by the temperature dependence of the helix pitch p_0 .

We shall first estimate the influence of the temperature dependence of the helix pitch on the permittivity and relaxation time. The temperature dependence of this pitch $p_0 = p_0(T)$, measured by us by the diffraction of polarized monochromatic light on the helical structure of the smectic *C* phase, is presented in Fig. 8. The helix pitch rises monotonically up to temperatures $T_c - T \approx 1^{\circ}$ C, where this dependence has a maximum. This behavior of the pitch is confirmed also by visual examination of the sample in a polarizing microscope and is in agreement with the results reported earlier.^[71] The reason for this behavior of p_0 is not yet clear. The increase in the pitch p_0 with rising temperature increases the relaxation time on approach to $T \to T_c$ and reduces the relaxation frequency $f_{rel} = \frac{1}{2}\pi\tau$.

The ratio $\mu_2 q_0 / \mu_1$ can easily be estimated by substituting in Eq. (9) the static permittivity ϵ_0 and the wave number q_0 corresponding to different temperatures. The values of ϵ_0 can be found by plotting the dependence $\epsilon = \epsilon(f)$ using the coordinate axes $(\epsilon - \epsilon_{\infty})^{-2}$ and f^2 , and extrapolating the linear dependence to the frequency range $\omega = 0$ (Fig. 6). It is clear from the data of Figs. 6 and 8 (see below) that the ratio $\mu_2 q_0 / \mu_1$, which governs the relative importance of the piezoelectric effect of the first and second kind is

$$\mu_{z}q_{o}/\mu_{1}=5-10.$$
 (10)

Thus, the polarization and of the dispersion of the permittivity ϵ of DOBAMBC are mainly due to an inhomogeneous spatial distribution of the director. Nevertheless it is clear from Eq. (9) that allowance for the linear piezoelectric effect, governed by the coefficient μ_1 , is necessary for the description of the experimentally observed dispersion of the permittivity at low frequencies.

The permittivity $\epsilon = \epsilon(\omega, \tau)$ of the chiral smectic C



FIG. 6. Dependence of $(\epsilon - \epsilon_{\infty})^{-2} \cdot 10^3$ on the square of the frequency. The notation is the same as in Fig. 4. The top left-hand corner shows the temperature dependence of the relaxation time parameter $(A/\pi) \cdot 10^3$, where $A = \gamma_1 K_3 / 4\pi^2 \chi^4 \mu_1^2 \mu_2^2$.

phase of DOBAMBC is described approximately by

$$\varepsilon = \varepsilon_{\infty} + \frac{2\pi \gamma^2 \mu_1 \mu_2 q_0}{\gamma_1 (1/\tau^2 + \omega^2)^{\gamma_1}}.$$
(11)

Figure 7 shows the theoretical dependences of $\epsilon' = \epsilon - \epsilon_{\infty}$ based on Eq. (11), where they are plotted as a function of frequency ω , relaxation time, and temperature; the dependence $\epsilon(T)$ is given subject to allowance for the temperature dependence of the relaxation time τ $=\gamma_1 p_0^2/K_3(2\pi)^2$ in the region $p_0 \leq p_{max}$. It follows from Fig. 7 that the expression (11) is in good agreement with the experimental data (Figs. 4-6) describing such fine features of the temperature and frequency dependences of ϵ as the shift of the ϵ maxima toward lower temperatures (Fig. 5) and intersections of the dependences of ϵ at different frequencies (Fig. 4). In the limit $\omega \to 0$, Eq. (11) can be written in the form

$$(\varepsilon_{0}-\varepsilon_{\infty})^{-2}=\frac{\gamma_{1}K_{3}}{4\pi^{2}\chi^{4}\mu_{1}^{2}\mu_{2}^{2}}\frac{1}{\tau}.$$
(12)

The temperature dependence of the reciprocal relaxation time obtained from Eq. (12) and Fig. 6 is shown in the inset in Fig. 6. The increase in τ by a factor of 3-4 near the smectic C — smectic A phase transition corresponds to a change in f_{rel} in the frequency range 400-100 Hz.

Estimates $(K_3 \approx 10^{-6}, \chi \approx 1, p_0 = 4 \times 10^{-4} \text{ cm})$ show that $\mu_1 \mu_2 q_0 \approx 200-250$ cgs esu and hence, using Eq. (10), we find that $\mu_1 \approx 4-6$, $\mu_2 q_0 \approx 40-50$, and $\mu_2 \approx 10^{-3}$ cgs esu.

2. Permittivity of the chiral smectic A phase. We shall now consider in detail the effects associated with the change in the permittivity of the smectic A phase and of the chiral smectic C phase with a homogeneous distribution of the director n. In the A phase, when the direction of n coincides with the z axis, the application of a transverse electric field hinders free rotation of the molecules about their long axes and gives rise to the transverse polarization alone. In this case, according to the Neumann principle, the symmetry of the system should decrease to the symmetry group 2 and



FIG. 7. Theoretical dependences of the permittivity ϵ' on the frequency ω , relaxation time τ , and temperature T, plotted using Eq. (11): $\tau_3 > \tau_2 > \tau_1$; $\omega_1 > \omega_2 > \omega_3$.

the director n should be tilted by an angle θ in the plane perpendicular to the axis 2 (this is known as the piezoelectric effect in the A phase^[1,9,12]).

The application of a field $E \ge E_p$ to the *C* phase reduces a single-domain state in which the field *E* gives rise to a homogeneous deflection of the director **n** through an equilibrium tilt angle $\pm \theta$ from the *z* axis in a plane perpendicular to the direction of *E*. The symmetry of the system again decreases from $\infty/2$ (which is the symmetry of the helix) to 2. The spontaneous dipole moment of all the molecules is then directed along the axis 2, which is parallel to **E** (Fig. 1b).

The application of an alternating measurement field $E = E_0 e^{i\omega t}$ then causes modulation of the angle θ and causes the polarization $P \propto \chi \mu_1 \theta$ to vary in amplitude at a frequency ω . This process is characterized by a definite relaxation frequency ω_{rel} . In the $\omega < \omega_{rel}$ range we may expect dispersion of ϵ . In fact, in the $T > T_c$ range, a weak dispersion of ϵ is observed for the A phase near the transition temperature (Fig. 5) but it becomes degenerate in the $T > T_c$ range. In the single-domain smectic C phase this dependence is manifested very weakly (it is of the same magnitude as the experimental error) for fields $E_{\sim} < E_p$, which is due to the preferential orientation of the permanent dipoles of the molecules along the field E_z .

We shall now consider the characteristics of the dielectric relaxation in the smectic A phase. The additional contribution to the permittivity may be calculated using the expression for the free-energy density of the A phase^[10] in the same way as in the case of the Cphase [see Eq. (9)]:

$$\varepsilon - \varepsilon_{\infty} = \frac{4\pi \chi^2 \mu_1^2}{\gamma_1 (1/\tau_1^2 + \omega^2)^{\frac{1}{2}}},$$
(13)

$$\tau_{1} = \frac{\gamma_{1}}{2a'(T-T_{c}) + K_{s}q_{o}^{2}},$$
(14)

where a' is a constant coefficient. It is important to note that the linear piezoelectric effect plays the dominant role in the A phase.

The expression (13) is identical with (9) and it also



FIG. 8. Temperature dependences of the tilt angle θ of molecules in the smectic layers, ^[5] spontaneous polarization P_s , ^[5] helix pitch p_0 , and ratio θ/p_0 calculated from the other results.



FIG. 9. Temperature dependences of $P_s(\Delta, \bigcirc)$ and $\theta/p_0(\Box)$ plotted on a double logarithmic scale. The points Δ and \bigcirc correspond to different series of measurements.

describes a Debye relaxation process but with a very different relaxation time. In contrast to conventional ferroelectrics, for which the variation of ϵ is described by the term $2a' (T - T_c)$ in the expression for the relaxation time τ (Curie-Weiss law), in the case of the A phase the values of τ_1 and ϵ are finite. At $T \approx T_c$ we have $\tau_1 = \tau = \gamma_1 / K_3 q_0^2$ and the dynamics of reorientation of the A phase is similar to the $T < T_c, E \ll E_p$ case. At $T \gg T_c$ the dominant term contains the transition temperature T_c as a parameter and an increase in temperature reduces ϵ . A comparison of Eqs. (13) and (11) demonstrates that because of Eq. (10) the dispersion of the permittivity ϵ of the A phase is approximately an order of magnitude less than that of the C phase. This is in agreement with the experimental results (Fig. 6).

The small values of the difference $\epsilon - \epsilon_{\infty}$ in the case of the A phase make it difficult to carry out a quantitative comparison of the theory and experiment.

We shall conclude by pointing out that the dynamic processes involving the small changes in the helix pitch in alternating electric fields and the associated dielectric relaxation at low frequencies in the *C* phase can be interpreted quite satisfactorily by the phenomenological theory of Pikin and Indenbom.^[10] In this connection it is interesting to note that the observed^[5] disagreement between the critical indices of the temperature dependences of the spontaneous polarization and the tilt angle of the molecules θ may be associated with the flexoelectric effect in the spirit of the theory of **Pikin** and Indenbom.^[10] In fact, the polarization of the C phase is given by $|\mathbf{P}| \propto \chi | \mu_1$ - $\mu_2 q_0 | \theta$, or if we use Eq. (10), by $|\mathbf{P}| \propto 2\pi\chi^2 \mu_2 \theta/p_0$. Allowance for temperature dependence of the helix pitch p_0 results in complete agreement between the dependence $|\mathbf{P}|(T)$ and the experimentally determined temperature dependence of the spontaneous polarization (Fig. 8). The corresponding dependences $|\mathbf{P}|(T)$ and $P_s(T)$ are plotted on a double logarithmic scale in Fig. 9.

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