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Determination of piezoelectric and flexoelectric polarization in ferroelectric liquid crystals

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A method of determining the local polarization in chiral smectic liquid crystals is proposed. The method consists of simultaneous measurement of the electric permittivity and depth of electro-optic modulation in a helical liquid crystal using weak ac electric field. The spontaneous polarization of a single smectic layer determined in this way contains both the flexoelectric and piezoelectric component. On the other hand, the polarization measured using strong electric fields in a switching experiment contains the piezoelectric component only. The comparison of polarization obtained using strong and weak fields makes it possible to determine the value of flexoelectric polarization. This kind of measurement has been performed for two ferroelectric liquid crystals having quite small absolute values of the spontaneous polarization (few $\mathrm{nC/cm^2}$). It turned out that in both cases, the flexoelectric polarization is of the same order of magnitude as the piezoelectric one.

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I. INTRODUCTION

It has been known since the discovery of ferroelectricity in liquid crystals [1] that spontaneous polarization may be produced in chiral smectic liquid crystals due to the two mechanisms called piezoelectric and flexoelectric [2]. The contribution of these two mechanisms to the spontaneous polarization constitutes a classical problem of the physics of ferroelectric liquid crystals (FLCs). It has been discussed in literature for many years, but has not been solved in a satisfying manner yet. The importance of the polarization origin in chiral smectic liquid crystals has even increased lately. Some recently developed theories assume a large impact of flexoelectric polarization on the creation of antiferroelectric liquid crystals and their subphases [3,4]. The flexoelectric effect seems also to be responsible for switching behavior in strong electric fields in the case of ferroelectric liquid crystals with a short helical pitch [5].

Liquid crystals possess orientational order and, if this order is changed, electric polarization may appear. If the liquid crystal molecules are not bar shaped, but have a more complicated shape, then a deformation of the director field may cause an additional order. For example, the bend deformation will force the molecules to align their transversal dipole moments, on average, perpendicular to the director and along the radius of curvature [6,7]. This ordering of dipole moments is equivalent to the occurrence of electric polarization called flexoelectric polarization.

Besides the orientational order, smectic liquid crystals exhibit at least one-dimensional positional order. In chiral tilted smectic-C^* liquid crystals, an effect similar to the piezoelectric one may appear due to the change in the smectic layer's thickness caused by tilting the molecules from the layer's normal [8]. This is responsible for the ordering of dipoles of chiral molecules in the direction perpendicular to the tilt plane. This effect is called piezoelectric, as it resembles the original piezoelectric effect observed in solid crystals, where the dimensions of the elementary cell change under the influence of external forces.

Both polarization mechanisms were already recognized by R. B. Meyer [2]. The vectors of piezoelectric polarization and of the flexoelectric polarization are parallel and appear simultaneously. Nevertheless, they are mutually independent and are described by independent coefficients in the Landau expansion of the free energy [9]. The question arises, how contributions of different kinds of polarization to the total polarization can be recognized and separated. This is not an easy task, as the properties of both kinds of polarization are similar. Both contributions might, however, be distinguished on the basis of symmetry properties. The flexoelectric effect can appear only within the helical structure, no matter whether the molecules are chiral or not. On the other hand, the piezoelectric effect can appear only in substances composed (at least partially) of chiral molecules, no matter whether the helical structure is present or not. Thus, in principle, the way to separate both effects might be measuring the polarization of a given material in two samples: with and without the helical structure. The easiest methods of measuring polarization are switching experiments (using a bridge [10] or the measurements of the switching current [11]). However, these measurements, performed in the absence of the helix, give the value of piezoelectric polarization only. On the other hand, the measurement of polarization in the presence of the helix is much more difficult. That is why it is hardly possible to perform correct measurements of this kind. For this reason, the dependence of flexoelectric polarization on the wave vector of the helix q has been exploited in a few attempts to determine the flexoelectric polarization made so far.

The q-vector dependence of polarization is reflected in the free energy expansion of the chiral smectic-C^*, which, in the simplest case, has the form [9]

$$F = F_0 + \frac{1}{2}a\theta^2 + \frac{1}{4}b\theta^4 + \cdots + CP\theta + \mu q P\theta + \frac{1}{2\chi_A}P^2 + \cdots, \tag{1}$$

where θ is the order parameter of the smectic-C^* phase (the tilt angle); P—the spontaneous polarization; χ_A—electric

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*Author for correspondence. Email address: wkucz@ifmpan.poznan.pl susceptibility of the smectic-A phase; C and μ—piezoelectric and flexoelectric coefficients, respectively; $q=2\pi/p$—the value of the wave vector of the helix (p is the pitch of the helix); F_0, a and b—constants. Although nine flexoelectric coefficients exist in the smectic-C^* phase [7], only one coefficient μ appears in Eq. (1). All the other coefficients do not contribute to the free energy in the absence of deformations of layers and deformations inside layers. For this reason, only the coefficient μ will be considered below.

The spontaneous polarization, obtained by minimizing the free energy with respect to P, has the form

$$P=\chi_A(\mu q+C)\theta. \tag{2}$$

The q-vector dependence of the spontaneous polarization has been used in various experiments to determine the contribution of piezoelectric and flexoelectric effect to the spontaneous polarization of the ferroelectric smectic. In all the experiments performed to date, the classic ferroelectric liquid crystalline material DOBAMBC (decyloxy-benzylidene-amino-methyl-butyl-cinnamate) was used. The first attempt was made by Ostrovskii *et al.* [12]. They tried to determine the flexoelectric polarization from the measurements of the electric permittivity ε. The measurement made on DOBAMBC revealed that the flexoelectric polarization is dominating [$\mu q \approx (5-10)C$]. In the next paper [13], Ostrovskii, Pikin, and Chigrinov reported the results of measurements of polarization in the samples of the same material with strongly deformed helical structure. From the comparison of the experimental data with the theoretical predictions, the authors drew a contradictory conclusion that the flexoelectric contribution is much smaller than the piezoelectric one. Durand and Martinot-Lagarde [14] investigated the temperature dependence of critical field E_c, which is needed for unwinding of the helix in DOBAMBC. Using the data on the critical field, pitch of the helix and the tilt angle, the authors concluded that the flexoelectric polarization did not exceed 10% of the piezoelectric polarization. In the next experimental paper concerning the discussed subject [15], the authors performed investigations of the pyroelectric effect and electro-optic effects in mixtures of two substances with opposite signs of polarization (one of the substances was DOBAMBC again). They concluded that the flexoelectric polarization was much less significant than the piezoelectric one. In the next paper on the flexoeletric effect [16], the authors stated that the flexoelectric polarization exceeded the piezoelectric polarization about two times.

As it can be seen from this short review, the problem of piezoelectric and flexoelectric contribution to the spontaneous polarization in ferroelectric liquid crystals has not been solved satisfactorily yet. The conclusions drawn from various experiments are not consistent, although the experiments have been performed for the same material. The question arises, what is the reason for such a discrepancy. There are several reasons, but the most important one might be the difficulty in correct determination of the pitch of the helix. This quantity is crucial in all methods used up to now. It is, however, well known, how difficult the correct and accurate measurements of the helical pitch in the smectic-C^* phase are [17,18]. In this situation, it is necessary to develop an experimental method that would deliver the results independent of the pitch measurements (although the measurement must be performed in the presence of the helix, otherwise the flexoelectric polarization does not exist). The best solution seems to be the measurement of polarization in the presence of a nondistorted helix and comparison with the value of polarization, measured in the absence of the helix, e.g., using a switching method. The measurement of spontaneous polarization in the presence of the helix is, however, very difficult and reliable results of such a measurement are not available. Here, we propose the measurement of the local polarization in the presence of the helix and a method of evaluation, which does not demand the helical pitch data.

II. METHOD OF LOCAL POLARIZATION MEASUREMENT

The proposed method of measurement of the local polarization P_0 of a smectic-C^* phase in the presence of the (quasi)-undistorted helix consists in simultaneous measurement of electric permittivity and electro-optical modulation. Both these physical quantities can be determined from the solution of the equation of motion of the smectic-C^*. In the case of small deformations ($E \ll E_c$), and if the excitation is harmonic, the approximate solution of the equation of motion gives the following for the amplitude of the helix deformation [19,20]:

$$\varphi_0=\frac{P_0E}{Kq^2\sqrt{1+\omega^2\tau^2}}, \tag{3}$$

where K is the twist elastic constant of the $\hat{\mathbf{c}}$ director, ω—angular frequency of the external electric field of amplitude E, and τ—the Goldstone mode relaxation time. The increment of the electric susceptibility χ due to the ferroelectric effect is proportional to the average value of the cosine of the azimuthal angle φ of the $\hat{\mathbf{c}}$ director and was calculated in [21] as

$$\chi=\frac{P_0^2}{2\varepsilon_0Kq^2\sqrt{1+\omega^2\tau^2}}. \tag{4}$$

In a similar way, we can calculate the depth of modulation of the light, passing the sample placed between crossed polarizers. The intensity of light transmitted by the sample and polarizers is

$$I=I_0(\sin^2 2\psi)\sin^2\frac{\rho}{2}, \tag{5}$$

where I_0 is the intensity of the incident light, ρ—the phase difference between extraordinary and ordinary rays on the exit of the analyzer, and ψ—the angle between the optic axis and analyzer direction. After applying the external electric field E the distribution of the azimuthal angle φ changes. The helix deformation, proportional to the quantity φ_0 from Eq. (3), causes changes in optic axis position ψ and, hence, changes in the light intensity I. These changes can be calculated [21] by differentiation of the Eq. (5) as follows:

$$A \equiv \frac{dI}{dE} = 2I_0\left(\sin^2\frac{\rho}{2}\right)(\sin 4\psi)\frac{d\psi}{dE}. \tag{6}$$

We neglected in Eq. (6) the possible changes of birefringence Δn under influence of an electric field. This assumption was supported by measurement of the second harmonic of the electro-optic light modulation, which appeared to be much weaker than the modulation at fundamental frequency [22]. The change in the optic axis position $\Delta\psi$ is related to $\varphi_0 : \Delta\psi = 1/2\varphi_0$ [20]. Thus, the amplitude of the change in the light intensity caused by the external electric field reads

$$A = \frac{BP_0\theta}{2Kq^2\sqrt{1+\omega^2\tau^2}}. \tag{7}$$

In Eq. (7), B represents a coefficient that is dependent on the experimental conditions. It can be either calculated or determined from light intensity measurements by a calibration procedure [22]. As a result, the absolute value (in units of angle) of the electro-optical response can be obtained [23]. It is evident that the form (4) describing electric susceptibility is very similar to Eq. (7) describing the light modulation. In particular, the frequency dependence of both quantities is the same. Hence, dividing Eqs. (4) and (7), we obtain a simple relation between the local polarization P_0 and the dielectric and electro-optic responses

$$\frac{\chi}{A} = \frac{1}{B\varepsilon_0}\frac{P_0}{\theta}. \tag{8}$$

So, the local polarization P_0 can be easily obtained from dielectric and optical measurements. The knowledge of the helical pitch is not necessary to calculate the local polarization. Furthermore, other physical parameters, except the tilt angle, do not appear in Eq. (8) and the local polarization can be very easily calculated as a function of temperature.

Equation (3) was obtained using a very simple model. Nevertheless, this result agrees very well with the results of more exact calculations in the small deformation limit. This simple model has been proved in many experiments [20–23]. Thus, we believe that Eq. (8) is quite accurate and can be used for determination of the local polarization. However, one has to bear in mind that this equation concerns samples with well-developed helical structure. This condition can be better fulfilled in thick samples.

III. EXPERIMENTAL

The experimental setup used for measurement of the local spontaneous polarization P_0 is schematically shown in Fig. 1. The dielectric response was measured with a commercial impedance analyzer HP 4192A (Hewlett-Packard) or with a custom low frequency, six-arm dielectric bridge. The optical response was measured with a photodiode connected to a lock-in amplifier SR850 (Stanford Research). The measuring voltage of either the bridge or the impedance analyzer served also as the source of electro-optic modulation. Both measurements—optical and dielectric—were performed simultaneously—not only in the same sample, but also in the same experiment.

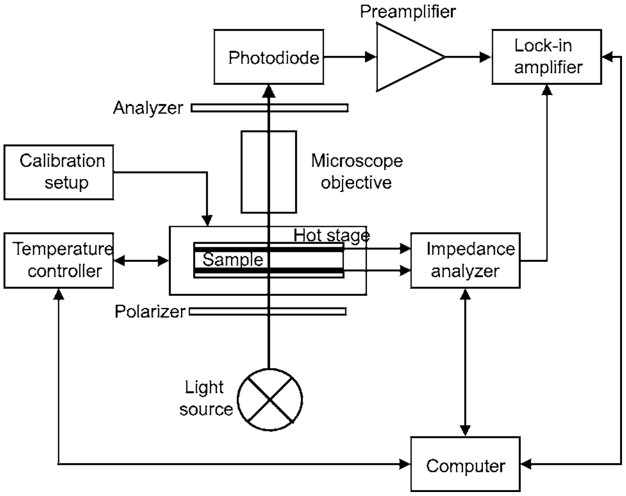
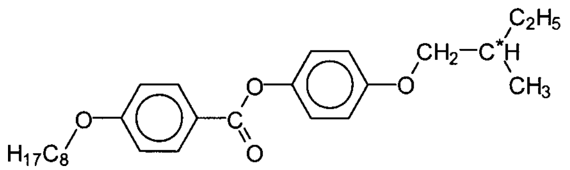


FIG. 1. Scheme of the experimental setup.

This measuring procedure allowed us to diminish substantially the experimental error. Besides, it ensured a univocal interpretation of the experimental results, because systematic errors connected with a possible imperfect orientation of the smectic layers in the sample or deformation of the helix were partially compensated.

For measurement, we used the "classic" FLC material DOBAMBC (4-*n*-decyloxy-benzilidene-amino-metylbuthyl-cinnamate). The temperature of the transition from the smectic-C^* to the smectic-A phase was 95 °C. Another substance used in the experiments, abbreviated here C8 has the following molecular structure:



The substance C8 has the following scheme of phase transitions (temperatures are given in degrees Celsius):

$$\mathrm{Cr}50(\mathrm{S}_3 28)\mathrm{Sm}C^*42\mathrm{Sm}A55\mathrm{Iso}.$$

The liquid crystal sample was introduced into the cell made of two glass plates provided with indium-tin oxide (ITO) electrodes. The electrodes were coated with a thin polyimide layer rubbed in one direction to secure planar alignment. The measuring cell was placed in a modified Mettler hot stage. The temperature of the sample was stabilized using the Unipan Model 650 temperature controller with an accuracy better than 0.05 K. The thickness of the sample was the consequence of a compromise. Thin samples give better alignment of smectic layers, but strongly deform the helix. Thick samples, on the other hand, do not affect significantly the helical structure, but give worse layer alignment. We used samples about 30 μm thick, which revealed rather good layer alignment without seriously disturbing the helical structure. Due to the orienting action of the polymer layers placed on the electrodes and suitable thermal treatment, we succeeded in obtaining a homogeneous planar texture with

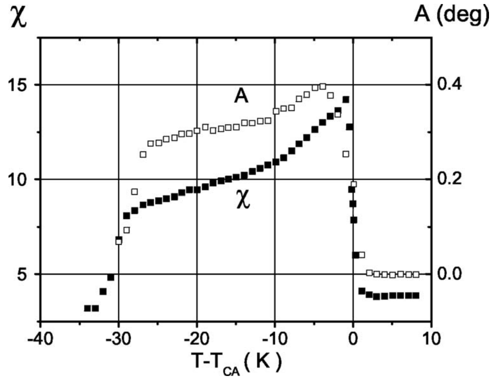


FIG. 2. Electric susceptibility χ and the modulation of the optic axis direction A for the ferroelectric liquid crystal DOBAMBC.

parallel stripes characteristic of a helical structure. In the case of DOBAMBC, the alignment was further improved by applying a strong magnetic field (7 T) during the cooling from the isotropic phase.

The spontaneous polarization and the tilt angle were measured using standard methods [8,10] with an accuracy of about ±5%. The determination of the coefficient B dependent on the experimental conditions [Eqs. (7) and (8)] is of crucial importance in our method. It was performed as a function of temperature by periodic, mechanical rotation of the microscope stage with amplitude 0.2° [22,23].

IV. RESULTS AND DISCUSSION

In the literature, only the data on flexoelectric polarization for DOBAMBC are available. Therefore, we started with measurements of this substance in order to make a comparison of the data obtained by means of different methods. The results of dielectric and electro-optic measurements are presented in Fig. 2.

The amplitude of the measuring voltage applied to 30 μm thick sample was below 0.5 V. As it could be expected, both the dielectric and optical responses were similar. The most important difference is the background in the dielectric response, which is caused by various noncollective mechanisms of polarization. To proceed with the interpretation, this background has to be separated from the susceptibility connected with the collective, ferroelectric polarization. As the dielectric measurement was made simultaneously with the optical measurement, this separation can be performed in an easy and precise way. For further calculations, the quantity $\Delta\chi=\chi\text{-}\chi_A$ was used, where χ_A is the susceptibility in the smectic-A phase.

The piezoelectric component of polarization was measured using the bridge method [10]. In this method, a large field was used for switching the polarization vector by 180°. Then, in the course of measurement, the helical structure does not develop. The flexoelectric component is absent—the measurement gives pure piezoelectric component. During the polarization measurement, the tilt angle was also measured by registration of extinction positions. The results are depicted in Fig. 3. The strong applied electric field might, in principle, influence the dipole ordering and thus increase the apparent polarization. However, the electric field effect on both the measured polarization and the tilt angle was negligible (except the close proximity of the phase transition SmC^*-SmA).

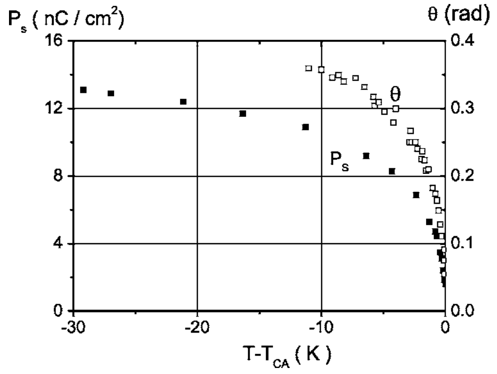


FIG. 3. Spontaneous polarization P_S and tilt angle θ of DOBAMBC measured using strong electric field in the switching experiment.

The final result of the measurement is presented in Fig. 4. In this figure, various kinds of polarization are shown: polarization measured in large fields, containing only piezoelectric contribution [curve (a)], measured in small electric fields containing both the flexoelectric and the piezoelectric components [curve (b)], together with the net flexoelectric polarization [curve (c)]. As it can be seen, at low temperatures the absolute value of the flexoelectric polarization in the investigated material amounts to about 50% of the piezoelectric one. The flexoelectric polarization changes its sign close to the transition smectic-C^*–smectic-A.

The ratio of piezoelectric and flexoelectric polarization obtained in our experiment is similar to that obtained by Chigrinov *et al.* [16], but differs from other results [12–15]. The question remains to be answered, why such a large discrepancy of literature data (shown in Sec. I) on flexoelectric coefficients exists. Because all the former experiments were based on the pitch dependence of the flexoelectric polarization, we suppose that the main reason for this discrepancy was the difficulty in proper measurements of the helical pitch

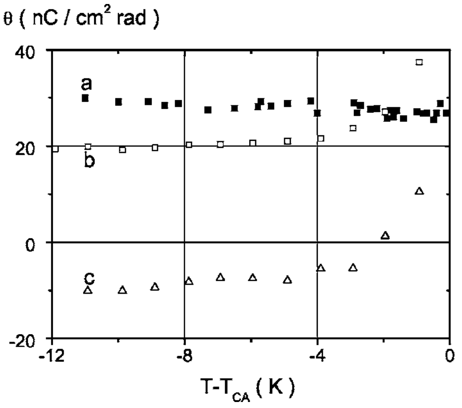


FIG. 4. Ratio of the polarization and the molecular tilt angle θ for DOBAMBC: (a) measured using strong fields in the switching experiment, (b) measured using weak field in the dielectric and electro-optic experiment, and (c) flexoelectric component.

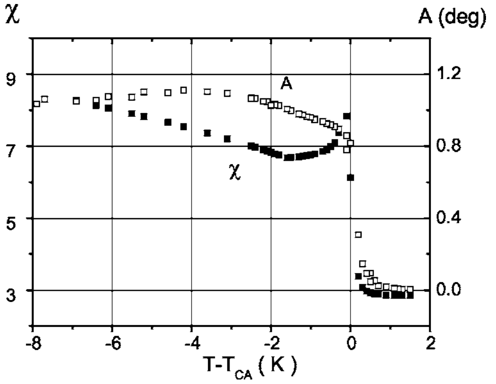


FIG. 5. Electric susceptibility χ and the optic axis deviation A for ferroelectric liquid crystal C8.

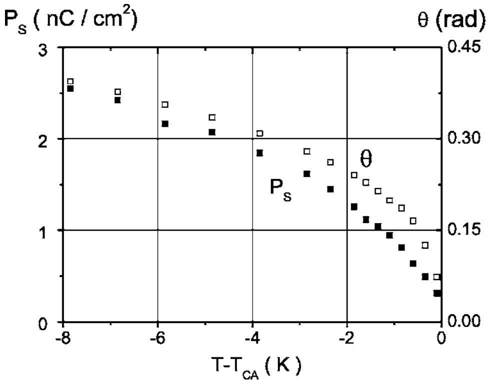


FIG. 6. Spontaneous polarization P_S and the molecular tilt angle θ of C8 measured in the switching experiment.

in the smectic-C^* phase. In samples with planar orientation (smectic layers perpendicular to the glass plates), correct pitch measurement is even impossible. In planar samples, the ideal helical structure described by the form $\varphi = qz$ (φ–tilt direction angle, z–coordinate along the helix axis) does not occur, because the helical structure is not compatible with planar boundary conditions. Glogarova and Pavel [17] demonstrated that in such samples a complicated system of defects exists instead of the helix. These defects are called "dechiralization" or "unwinding" lines. The distance between the lines is close to, but not equal to, the $p/2$ value. This statement was confirmed in many measurements that were performed for planar samples of various thicknesses [18]. Also, the results obtained on heating and on cooling differ strongly, which means that at least one of them must be wrong. It is often assumed that the results obtained using thick samples are correct. Actually, the results obtained even in very thick samples (thickness 100–200 μm) are still not reliable. This opinion is confirmed by the experiment made with thick homeotropic aligned samples [24]. In this geometry, the helical structure is not in conflict with boundary conditions and it is possible to obtain a defect-free sample or a sample with controlled defects (Cano lines) if the thickness varies. Thus, in homeotropic samples, the helix is not deformed and the results of helical pitch measurements are much more reliable than in planar geometry. Unfortunately, dielectric measurements of the Goldstone mode in homeotropic geometry are not possible. The uncertainty of measurements in planar geometry may reach several hundred percent (even for thick samples), especially close to the phase transition SmC^*-SmA, where the slope of $p(T)$ curve reaches high absolute values. This can explain the strong scattering of literature data on the flexoelectric polarization. In our experiments, the value of flexoelectric polarization might be affected by the helix deformation as well, although the sample thickness was quite large (about 30 μm). However, our method is insensitive to the value of the helical pitch [see Eq. (8)] and, therefore, the accuracy of the estimation of the flexoelectric polarization is much better. Of course, the flexoelectric effect depends on the actual helical structure, however, the pitch uncertainty influences the polarization, not the calculation results.

The experiments with DOBAMBC are quite difficult because of its bad chemical stability. For this reason, we performed similar experiments with a much more stable substance C8. The experimental results obtained for C8 are presented in the next figures. Figure 5 shows the electro-optic response and the electric permittivity, both measured at 10 Hz. Figure 6 shows the results of measurement of the piezoelectric polarization obtained with the use of strong fields, along with the tilt angle, as a function of temperature.

The comparison of temperature dependencies of the piezoelectric and flexoelectric polarization is presented in Fig. 7. In this case, as well, both kinds of polarization are of the same order of magnitude, similar to the case of DOBAMBC. The figure demonstrates that in a broad temperature range, the total local polarization is less than the piezoelectric one. It means that the sign of the flexoelectric polarization is opposite with respect to the piezoelectric polarization [curve (c) in Fig. 7]. Again, as in the case of DOBAMBC, the flexoelectric polarization changes sign close to the phase transition.

The results obtained for both investigated ferroelectric liquid crystals (DOBAMBC and C8) revealed that the magnitude of the flexoelectric spontaneous polarization is comparable with that of the piezoelectric polarization.

We are aware of the fact that due to the imperfection of the helix, the measured flexoelectric polarization differs from that which would exist in a sample with ideal helix. The

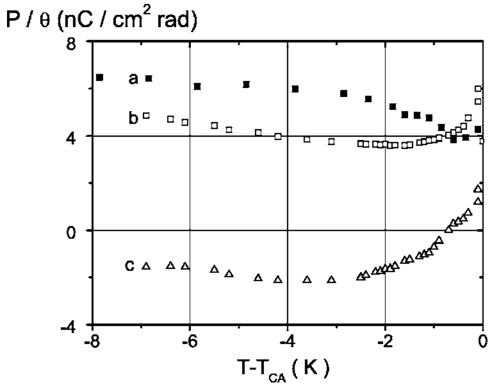


FIG. 7. Ratio of polarization and tilt angle for ferroelectric liquid crystal C8: (a) piezoelectric component measured using strong field, (b) the sum of flexoelectric and piezoelectric polarization measured using weak field, (c) flexoelectric component.

helix deformation could even cause other flexoelectric coefficients (not only μ) to perhaps contribute to the local polarization. To diminish this possibility, we took great care to obtain a good planar alignment of samples, as described in Sec. III. Therefore, we believe that the contribution of the flexoelectric coefficient μ was dominating in our experiments. The method applied in this work allows for the determination of the actual value of the local polarization and does not suffer from the error in pitch measurement. For instance, if there are some areas in the sample without helix, they do not contribute to the flexoelectric polarization and, consequently, are not detected in measurements of the electric permittivity and of the electro-optic response. This situation differs from that taking place in former experiments where a value of helical pitch must be assumed to calculate the flexoelectric polarization. Although our method allows for determination of the correct value of the flexoelectric polarization, we have not determined the absolute value of the flexoelectric coefficient μ, as it is defined only for an ideal helix described by the form $\varphi = qz$.

Both kinds of polarization—piezoelectric and flexoelectric—depend on the molecular structure. So, the investigation of materials consisting of molecules with a different shape may provide information on the conformation of molecules and its influence on the presence of various tilted smectic phases, especially in antiferroelectric liquid crystals.

The presented method of determination of polarization components consists in measuring the local spontaneous polarization. This unique method of measurement can also be applied to other types of investigations: for example, to determine the local polarization in various subphases of antiferroelectric liquid crystals.

V. CONCLUSIONS

We have proposed a method of determining local polarization, consisting in simultaneous measurements of dielectric and electro-optic responses. Using this method, we performed careful measurements of the flexoelectric component of spontaneous polarization in the smectic-C^* phase of two ferroelectric liquid crystals. To make the comparison with literature data possible, we used the classic FLC material DOBAMBC. We found that both components of the spontaneous polarization—flexoelectric and piezoelectric—are of the same order of magnitude. The same conclusion is valid for the other investigated material, C8. We think that the large scattering of the literature data on the flexoelectric polarization is caused by serious uncertainty of the helical pitch determination.

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