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Non-monotonic temperature dependence of the alignment of a nematic mixture on a ferroelectric substrate

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Polarization optic techniques have been applied to study specific features of the anisotropic interaction between a dye-doped eutectic mixture of nematic liquid crystals *p*-methoxybenzylidene-*p*-*n*-butylaniline and *p*-ethoxybenzylidene-*p*-*n*-butylaniline and a polar surface of a ferroelectric triglycine sulphate crystal over the temperature range including the substrate Curie point T_c . It has been found that the temperature-induced structural changes in the nematic layer in the vicinity of T_c are related to the changes in the orientational part of the tensor order parameter Q_{ik} . The temperature dependence of the director angle θ , averaged over the nematic layer, has been obtained from the effective dichroism values of solute absorption. The experimental data were interpreted using the model, in which the anisotropic part of the surface energy has two terms with orthogonal easy axes.

1. Introduction

To date, well established techniques used to control macroscopic (optical, dielectric, etc.) properties of nematic liquid crystals (NLCs) employ external forces acting on a nematic volume and competing with surface anchoring, which stabilizes a particular orientation of the mesogenic molecules [1, 2]. Recently, new methods of controlling liquid crystal orientation have been under intensive development, acting mostly on a LC–substrate interface [3]. Surface director reorientations caused by competition of counteracting alignment mechanisms are of special interest. Such reorientations have much in common with phase transition phenomena and are usually called anchoring transitions [4]. Temperature is often a factor in such transitions, changing the balance of forces on the surface. The transition from planar to homeotropic alignment with varying temperature has been previously observed [5], driven by the competition between dispersive van der Waals and short range anchoring forces on the surface. The competition between polar and dispersive forces of the substrate also favours the orientational transition [6]. At the same time, the surface electric field of the substrate substantially affects the LC alignment because of its dielectric anisotropy, enhancing polar effects at the confining surfaces [7].

The competing effects of dielectric, polar and dispersive forces can be observed on the orienting

surfaces of ferroelectrics [8–11]. The spontaneous polarization field of a ferroelectric single crystal depends on temperature and vanishes at the transition to the non-polar state, which occurs at the Curie point T_c . If the mesophase range includes T_c , the nematic alignment can be observed until the electric field of the substrate completely disappears. It should be noted that the use of birefringence or ellipsometry techniques to study structural changes in LC cells with crystalline substrates is a challenge, since the optical anisotropy of the substrates modifies the LC response to polarized radiation in an unknown way. In addition, the optical indicatrix of the crystalline substrates may also change with temperature. This problem can be partially overcome by means of polarization spectroscopy of solute absorption, which allows one to observe the changes in director orientation, and to control the alignment of mesogenic molecules during field-induced or spontaneous structural transformations in nematics [12, 13].

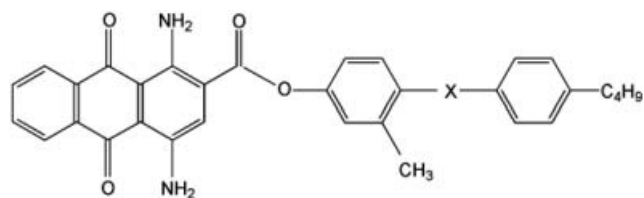
Recently, unusual intensity changes of solute absorption have been observed near T_c in a nematic mixture placed between two ferroelectric substrates [14]. These changes were presumably caused by the surface orientational transition. However, due to the ambiguous temperature behaviour of the absorption caused by the proximity ($\sim 2^\circ\text{C}$) of the nematic-to-isotropic and ferroelectric-to-non-polar state phase transitions, questions about the alignment of nematic molecules above T_c , and about the character of the observed transformations, remained unanswered. In the present paper, we

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apply the same polarization optic technique to study the surface properties of a nematic mixture with a wider mesophase range. The temperature dependences of the director angle $\bar{\theta}$, averaged over the nematic layer, and of the module S of the tensor order parameter Q_{ik} of the mixture, were established by using dichroism and refractive index measurements.

2. Experimental

A mixture of two nematic LCs, 60% of MBBA (*p*-methoxybenzylidene-*p*-*n*-butylaniline) and 40% of EBBA (*p*-ethoxybenzylidene-*p*-*n*-butylaniline), was prepared for this study. The mixture had the following phase sequence Cr–12°C–N–55.3°C–I, showing phase transitions between the crystalline, nematic and isotropic liquid phases. The concentration ratio of the mixture constituents resulted in a substantial increase in the mesophase temperature range. A dichroic dye based on an ester of 1, 4-diaminoanthraquinone-2-carboxylic acid (KD-10) with an absorption band



maximum at wavelength $\lambda=642$ nm [15] was added to the mixture. The non-degenerate long wave $\pi \rightarrow \pi^*$ electron transition of this band, followed by the charge transfer from the amine group to the phenyl ring, is polarized along the longitudinal molecular axis [16]. The small weight concentration ($c_d \sim 0.3\%$) of the dye had no noticeable effect on the clearing point $T_{NI}=55.3^\circ\text{C}$, the sample birefringence or the ordering degree of the nematic mixture.

Cells with polar and non-polar substrates were used in the experiment to orient the nematic mixture. The polar substrates were made by splitting triglycine sulphate (TGS, $(\text{NH}_2\text{CH}_2\text{COOH})_3\text{H}_2\text{SO}_4$) crystal along the cleavage plane normal to the ferroelectric axis b . The plate thickness was in the range 0.5–1.0 mm. A layer of the dye-doped nematic was then placed on a freshly cleaved plate. Photomicrographs of the TGS domain structure made with a polarizing optical microscope are shown in figure 1. The colour contrast is caused by the solute absorption anisotropy of the nematic aligned differently for the ‘+’ and ‘-’ domains. One can see the reversal of the contrast on the conjugated surfaces of the cleavage in figures 1 (a) and (b). In the upper frames, the surface of the ‘-’ domain is an intense light-blue

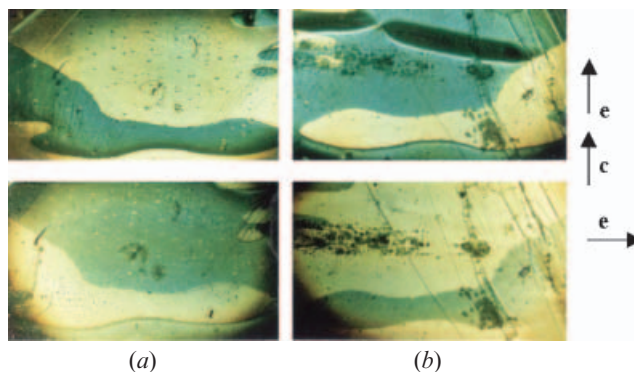


Figure 1. The symmetric domain structure of (a) the conjugated surfaces and (b) the triglycine sulphate crystal cleavage coated by the nematic dye-doped MBBA+EBBA mixture. Arrows indicate the directions of the crystallographic c -axis of TGS and the polarization vector of the light wave e .

colour due to the director orientation along the crystallographic c -axis of TGS, with the polarization vector of the light wave $e \parallel c$. The colour contrast also reverses with the polarizer rotation through 90° (see lower frames). The areas of the TGS cleavages containing the ‘-’ domains served as the polar substrates. The liquid crystal was placed in the space between two coaxially oriented cleaved plates separated by wire spacers of $20 \mu\text{m}$ diameter. The sample orientation quality was inspected by polarizing microscope (POLAM P-113). The cell area chosen for optical measurements extinguished completely via crossed polarizers according to the extinction position of the TGS indicatrix. On the other hand, the area under study appears strongly coloured when using only one polarizer if the light is polarized parallel to the c -axis of the crystalline substrates; the field of view being homogeneous. On rotating the sample through 90° , the field of view becomes colourless. This is the evidence of a uniform planar orientation of the director \mathbf{n} along the c -axis inside the TGS cells.

The polarized components of optical density D_j of the dye-doped mixture for the TGS cells were obtained with an automated spectrometer KSVU-23 at fixed temperature $T=23^\circ\text{C}$. The temperature behaviour of the components D_j was then studied on the polarization optic system [13] based on a He–Ne laser ($\lambda=633$ nm). Since the dichroic ratio $N(\lambda)=D_{\parallel}(\lambda)/D_{\perp}(\lambda)$ for the above-mentioned band is independent of λ [16], a one-beam scheme was used. In the following text, the indices \parallel , \perp , i correspond to the direction parallel, or perpendicular to the nematic director \mathbf{n} , and to the isotropic liquid, respectively. To record the components $D_{\parallel,\perp}$, the director of the sample was oriented, respectively, parallel or perpendicular to the polarization direction of light passing through the sample. The

accuracy of adjusting the direction of \mathbf{n} relative to the polarization vector of light wave \mathbf{e} was about $\pm 1^\circ$. Any contribution of background radiation caused by light scattering from the anisotropic substrates was excluded by subtracting the corresponding contribution of the radiation passing through the sample area free from the nematic mixture. This procedure was performed independently for each component of optical density at every temperature point. The temperature uniformity over the whole sample volume and the thermal stability within the mesophase range were within $\pm 0.1^\circ\text{C}$.

When the director field along the z -axis (normal to the substrate) is distorted, some effective optical density D_e is recorded instead of D_{\parallel} [12]. Figure 2 shows the experimental dependences of D_e and D_{\perp} on the reduced temperature $\Delta T = T_{\text{NI}} - T$ for the TGS cell, averaged over a series of samples. Provided for comparison are the temperature dependences $D_{\parallel, \perp}(\Delta T)$ of the dye-doped mixture for a cell of the same thickness with non-polar glass substrates. One can see that within the major portion of the mesophase range the optical density D_e of the TGS cell coincides with D_{\parallel} . However, the two curves diverge sharply in the vicinity of T_c . A significant difference between the values of D_e and D_{\parallel} , exceeding the accuracy of their experimental measurements, was observed at temperature $T^+ \approx 42^\circ\text{C}$. Above T_c , the optical density D_e coincides with D_{\perp} ; finally, above T_{NI} , it coincides with the density of the isotropic phase D_i .

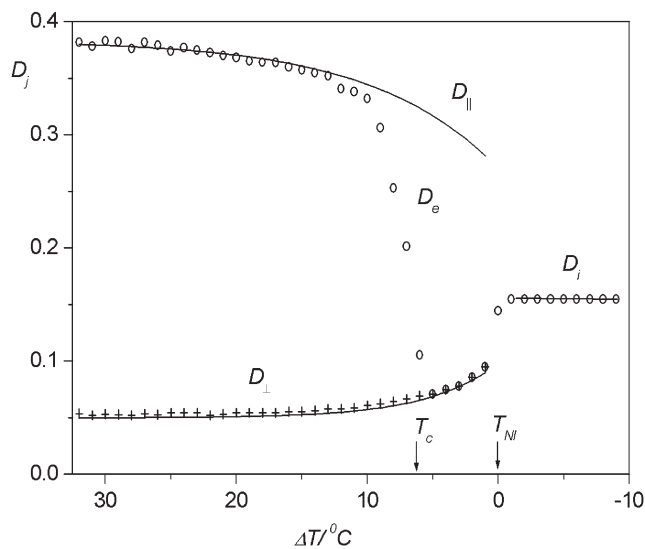


Figure 2. The temperature curves of the polarized components D_j of the KD-10 dye absorption band for the nematic and isotropic phases of the MBBA+EBBA mixture: $D_e(\circ)$ and $D_{\perp}(+)$ in the TGS cell; $D_{\parallel, \perp, i}$ (solid lines) in the glass cell; the cell thickness $d=20\mu\text{m}$, the dye concentration $c_d=0.3\text{ wt}\%$. Arrows indicate the Curie point T_c and the clearing point T_{NI} .

The analysis of the experimental spectra data requires information about the dispersion of the refractive indices of the orienting nematic matrix. The refractive indices $n_{\parallel, \perp, i}$ of the MBBA+EBBA mixture at wavelengths 589 and 633 nm were obtained by the prism technique using a goniometer [17]. Their temperature dependences are shown in figure 3. The refractive indices $n_{\perp, i}$ were also measured with an Abbe refractometer (IRF-454B). A good agreement between the experimental data obtained with the goniometer and the refractometer indicated that the prism technique was applied correctly. The precision for the measured refractive indices was found to be about 10^{-3} .

3. Results and discussion

It was assumed earlier [14] that the most probable explanation of the unusual absorption behaviour of the TGS cell is a reorientation of LC molecules from the planar to homeotropic texture, i.e. an orientational transition. Indeed, the change of optical anisotropy of the ferroelectric substrates with temperature is negligibly small over the whole mesophase range of the studied mixture [18] and, therefore, could not lead to the considerable optical density changes seen in figure 2. On the other hand, it is known that the orientational statistics of a uniaxial nematic phase is described in terms of the tensor order parameter [1]

$$Q_{ik} = S \left(n_i n_k - \frac{1}{3} \delta_{ik} \right), \quad i, k = x, y, z. \quad (1)$$

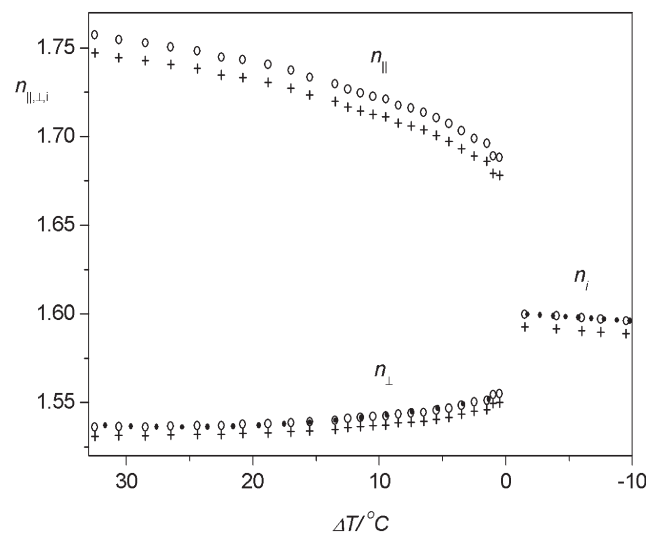


Figure 3. The refractive indices $n_{\parallel, \perp, i}$ of the MBBA+EBBA mixture as a function of reduced temperature at the wavelengths $\lambda=589\text{ nm}$ (\circ) and 633 nm ($+$). The solid points (\bullet) show the values of $n_{\perp, i}$ measured with the refractometer (589 nm).

Here, the module $S = \left(3\overline{\cos^2\theta} - 1\right)/2$ is a scalar uniaxial order parameter, the horizontal bar represents statistical averaging over all molecules, $n_{i,k}$ are the components of the macroscopic director \mathbf{n} , and $\delta_{ik} = 1$ if $i=k$ and 0 if $i \neq k$. Any change in Q_{ik} resulting from an external perturbation can be reduced to the changes of the module S , the direction of \mathbf{n} or both. Hence, it is important to know whether the only change is in the direction of \mathbf{n} or whether the module S also changes. For this purpose, the temperature dependence of the solute order parameter S was found using the results of two independent experiments. In the ferroelectric region of the substrate below T^+ the parameter S was obtained from the relation [19]

$$S = \frac{N_1 g_1 - 1}{N_1 g_1 + 2} \quad (2)$$

where the dichroic ratio $N_1 = D_{\parallel}/D_{\perp} \equiv D_e/D_{\perp}$. In the temperature range $T^+ < T < T_{\text{NI}}$, including T_c and the non-polar phase of the substrate, the parameter S was determined from the relation [19]

$$S = 1 - N_2 g_2 \quad (3)$$

where the dichroic ratio $N_2 = D_{\perp}/D_i \equiv D_e/D_i$. The correction factors $g_{1,2}$ take into account the anisotropy of the local field of the light wave [19]

$$g_1 = \frac{n_{\parallel}}{n_{\perp}} \left(\frac{f_{\perp}}{f_{\parallel}}\right)^2 \quad g_2 = \frac{\rho_i n_{\perp}}{\rho n_i} \left(\frac{f_i}{f_{\perp}}\right)^2. \quad (4)$$

Here $n_{\parallel, \perp, i}$ are the background refractive indices within the dye absorption band coinciding with those of the matrix, and $f_{\parallel, \perp, i}$ are the background components of the local field tensor for solute molecules within their absorption band. The anisotropy of the local field tensor for MBBA was found to be negligibly small [20]. Thus, the approximation of an isotropic local field ($f_{\perp}/f_{\parallel} \approx 1$, $f_i/f_{\perp} \approx 1$) was accepted for the mixture of MBBA with its ethoxy homologue. The values of the refractive indices at wavelength $\lambda = 633$ nm used to calculate the factors $g_{1,2}$ were taken from figure 3. The densities ρ and ρ_i for the nematic and isotropic phases of MBBA were taken from [17].

The temperature dependence of the order parameter S of the KD-10 dye molecules in the nematic phase of the MBBA+EBBA mixture is shown in figure 4. The order parameter S was calculated according to equation (2) in the temperature range $T < T^+$, and according to equation (3) in the temperature range $T^+ < T < T_{\text{NI}}$. As seen in figure 4, the dependence $S(\Delta T)$ is monotonic and is sensitive to the transition of the ferroelectric substrate to the non-polar state within the experimental error. Thus, the specific behaviour of D_e , seen in

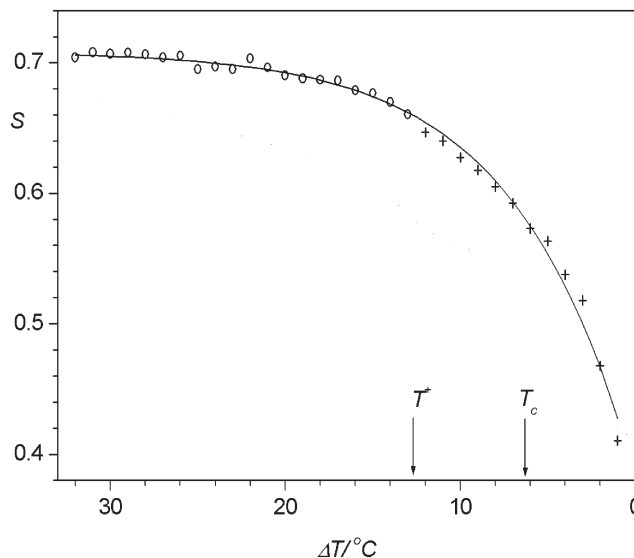


Figure 4. The orientational order parameter S of the KD-10 dye molecules in the nematic phase of the MBBA+EBBA mixture as a function of reduced temperature: open points (\circ) show the values of S calculated from equation (2), crosses ($+$) show those calculated from equation (3); the solid line is the interpolation. The temperature curves $S(T)$ merge at the conjunction point $T^+ \approx 42^\circ\text{C}$.

figure 2, is caused by the changes of the orientational part of the tensor order parameter Q_{ik} , implying purely orientational transition. The continuous form of the dependence $S(\Delta T)$ at the conjunction point T^+ also suggests that the isotropic local field approximation is valid for the mixture studied.

It is well known that the electric field of the substrate is induced by surface charges, which are due to the spontaneous polarization \mathbf{P}_s of the ferroelectric crystal. A nematic energy density in the presence of surface electric field $\mathbf{E} = E(z)\mathbf{l}$, where \mathbf{l} is the unit vector parallel to the z -axis, can be described as [21]

$$-\frac{1}{2}\epsilon_a\epsilon_o(\mathbf{E}\cdot\mathbf{n})^2 - q\frac{\partial E_z}{\partial z}n_z^2. \quad (5)$$

Here $\epsilon_a = \epsilon_{\parallel} - \epsilon_{\perp}$ is the dielectric anisotropy of the NLC, ϵ_o is the dielectric constant of vacuum, q is the difference between the longitudinal and transverse components of the quadrupole electric moment density tensor in the nematic, and \mathbf{n} is the director. The first term in equation (5) arises from the dielectric energy contribution while the second, quadrupolar term, takes into account non-uniformity of the surface electric field. It has been previously established [9] that non-uniformity of the substrate electric field has no significant influence on the nature of the interaction between MBBA and the TGS cleavage surface. To explain this phenomenon, the authors of [9] suggest that the field arising from

polarization charges on the TGS cleavage is screened by the ions present in any nematic material. Therefore, we can assume, as a first approximation, that the corresponding energy per unit area is

$$f_e = -\frac{1}{2} \epsilon_a \epsilon_o \int_0^{\infty} E^2(z) \cos^2 \theta dz \quad (6)$$

where $\theta = \cos^{-1}(\mathbf{n} \cdot \mathbf{l})$. For $\epsilon_a < 0$, which is the case for the MBBA+EBBA mixture, the dielectric energy f_e stabilizes nematic planar anchoring on the TGS cleavage surface. As the temperature increases, the dielectric contribution decreases, vanishing at T_c . The van der Waals dispersion forces of the substrate favour the planar texture, since they are responsible for the different nematic alignment on the '+' and '-' domains of TGS [21]. Thus, the contributions from the electric field and the dispersion forces give rise to torques within the surface plane. At the same time, the asymmetric effect of the solid crystal – LC interface and the nature of the interaction between the ends of molecules and the TGS cleavage surface [22] may give rise to a torque that favours the homeotropic alignment. Under the vanishing field condition, this factor makes an important contribution to the surface free energy of nematic, presumably dominating the dispersive van der Waals forces at the boundaries. According to [23], this contribution takes the form

$$f_o = -\frac{1}{2} w_o \cos^2 \theta \quad (7)$$

where the coefficient w_o serves as the anchoring energy. Taking this into consideration, the total energy per unit surface, which plays the role of the effective anchoring energy for the studied system, can be presented as

$$f_{\text{eff}} = f_o + f_e. \quad (8)$$

At some temperature, the competition between the two terms in equation (8) can cause a deformation of the planar nematic orientation in the TGS cell. For example, some deformed configurations of the director field with different boundary conditions have been considered in [24]. To find out whether the director pattern is homogeneous under conditions of orientational transition, we used the magnetic null method [25] to determine the tilt angle across the sample at certain temperature points within the $T^+ < T < T_c$ range. However, we could not detect a position where the optical transmission of the TGS cell did not change upon applying the external magnetic field. This result led us to conclude that the deformation across the nematic layer corresponds to the non-uniform distribution of the director. This non-uniform deformation of

the nematic layer is probably caused by different active ranges of the surface electric field and the anchoring forces. The question of the real distribution of the director field in the TGS cell remains unclear and needs further investigation.

On the other hand, any deformation of the nematic layer can be characterized by the tilt angle averaged over the thickness of the whole sample, i.e. effective angle $\bar{\theta}$. This angle can be measured using the spectroscopic method described in [12]. Let the director \mathbf{n} of the TGS cell be initially orthogonal to the z -axis, with the orientation angle $\theta = \pi/2$. The nematic deformation caused by the anchoring transition results in the z -dependent orientation angle $\theta(z)$ between the wave vector of the extraordinary beam $\mathbf{k}_{\parallel z}$ and the local direction of \mathbf{n} . Assuming that the correlation length of distortions of the local director field $\mathbf{n}(\mathbf{r})$ in the deformed cell is much larger than the molecular size, we can treat the local optical properties of the system as those of a uniaxial crystal, similar to the case of S or B -deformations under the Fréedericksz transition [12]. Due to the anisotropy of the van der Waals interactions of nematic molecules with the TGS crystal surface [21], we assume that the director field $\mathbf{n}(\mathbf{r})$ of the distorted nematic layer changes only in the z -direction, and remains uniform in the xy -plane. In this case, the effective dichroism $N_e = D_{\parallel} / D_{\perp}$ of solute absorption can be used to calculate the value of the director angle $\bar{\theta}$, averaged over the nematic layer [12],

$$\frac{1}{\cos^2 \bar{\theta}} = \frac{N_e g_e - 1}{Ng - 1}. \quad (9)$$

Here, the correction factor $g_e = 1 + (g-1)(N-1)/(N_e-1)$ takes into account the difference between the group and phase refractive indices of polarized light passing through the distorted nematic layer.

The normalization values of the parameters $N = D_{\parallel} / D_{\perp}$ and $g = n_{\parallel} / n_{\perp}$ were used for every temperature point independently. The temperature dependences of the dichroic ratios N , N_e and the effective angle $\bar{\theta}$ are shown in figure 5. We see that both a divergence of the dichroic dependences and a variation of $\bar{\theta}$ begin at temperature T^+ . The angle $\bar{\theta}$ decreases smoothly at first and then sharply vanishes near T_c . With further increase in temperature up to T_{NI} , the angle $\bar{\theta}$ remains unchanged. The specific 'delay' above the critical point T^+ makes the form of the $\theta(T)$ curve substantially different from that of the Fréedericksz transition [12]. As we have noted above, it is probably related to specific features of the nematic deformation induced by the surface. The temperature behaviour of $\bar{\theta}$ provides evidence of the director reorientation from the planar to homeotropic texture. We should emphasize that this

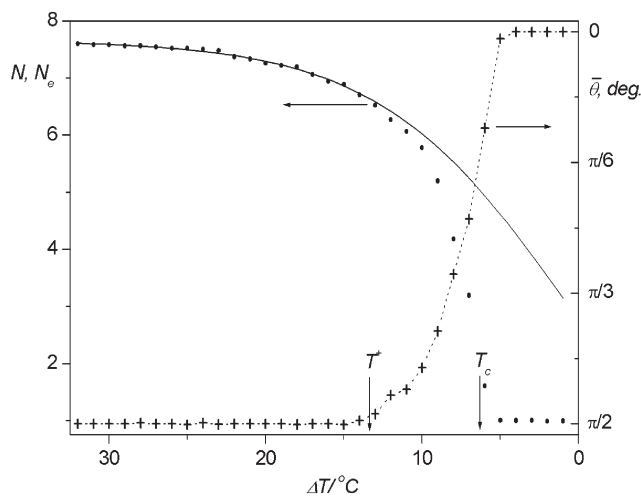


Figure 5. The temperature dependence of the dichroic ratios of the polarized components D_j : $N = D_{||}/D_{\perp}$ (solid line) in the glass cell; $N_e = D_e/D_{\perp}$ (•) in the TGS cell. The temperature curve for the effective angle $\bar{\theta}$ (cross-dotted line) was calculated from equation (9).

reorientation starts far from T_c , where the major orienting factor of the polar substrate, its electrical field, vanishes. This phenomenon can be explained by the substantially different temperature dependences of the competing energy contributions in equation (8), which balance each other at T^+ . As evident from figure 5, the temperature variation of $\bar{\theta}$ exhibits a continuous behaviour at T^+ . Therefore, the change of the director alignment on the polar surface of TGS at this temperature is close to the second order phase transition. Above T_c , the surface electric energy $f_e = 0$, and the interaction between the NLC and the substrate is determined only by the surface anchoring energy f_o , stabilizing the homeotropic orientation.

4. Conclusion

In this study we have clarified the mechanism of the critical behaviour of the nematic MBBA+EBBA mixture on a ferroelectric TGS substrate in the vicinity of the substrate Curie point T_c . The nematic alignment results from the competition of two opposing factors. The electric field of the substrate attempts to 'lay' the nematic molecules onto the substrate plane, while polar effects favour the homeotropic texture. As a result of the different temperature dependences of these competing factors, an anchoring transition occurs at some temperature T^+ . We should note that heating samples above T_c renders the structural changes in the liquid crystal irreversible, since a striped domain structure appears on the TGS substrates after they have been annealed. Nevertheless, complete director reorientation

within a relatively narrow range of temperature close to the Curie point can essentially improve the contrast ratio of the optical parameters of LC cells, and the modulation depth of light transmitted through samples, giving rise to new technological applications of orientational effects. In addition, it would be interesting to study the critical behaviour of NLC – ferroelectric substrate systems which undergo a first order phase transition or a transition to an incommensurate phase.

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