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The smectic A–smectic C* phase transition in submicron confined geometry

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The influence of confined geometry on the smectic A–smectic C* phase transition in the ferroelectric liquid crystal CE8 has been analysed. Using an electro-optic response technique, the temperature dependence of elementary excitations in thin, homogeneously aligned, wedge-type cells of thickness from 0.25 to 4 μm has been measured for different thicknesses of the aligning layer. In the case of a thin aligning layer (15 nm), we observe in the smectic C* phase an increase of the relaxation rate of the phase excitations and a decrease of the phase transition temperature, both of which are proportional to the inverse of the cell thickness. For a thick aligning layer (50 nm), the relaxation frequency and the transition temperature are proportional to the inverse square of the cell thickness. This indicates that the surface anchoring energy depends on the thickness of the aligning layer. For CE8 on nylon, we obtain a surface anchoring energy $W = 10^{-4}$ J m^{-2} for a thin aligning layer, and strong anchoring with $W \geqslant 5 \times 10^{-4}$ J m^{-2} for a thick aligning layer.

1. Introduction

Surface stabilization of ferroelectric liquid crystals has attracted much attention since the discovery of a fast linear electro-optic response in surface stabilized ferroelectric liquid crystal cells (SSFLC) [1]. In SSFLC, a ferroelectric liquid crystal is usually oriented by rubbing a thin aligning layer which is deposited on glass plates. The interaction between the liquid crystal molecules and the aligning layer plays an important role because it determines the equilibrium director structure and the electro-optical response properties of SSFLC [2]. Although many investigations have been performed in order to understand and control this interaction [3], the aligning of liquid crystals on rubbed surfaces is not yet completely understood.

The dynamics of the smectic A–smectic C* phase transition in the bulk has been extensively studied by dielectric measurements [4] and light scattering [5]. This phase transition was also used to study the structures and dynamic properties of ferroelectric liquid crystals in very thin cells [6]. The thickness dependence of the dielectric response of SSFLC has been the subject of several theoretical [7, 8] and experimental studies [9–13]. The dielectric experiments in thin SSFLC have been performed in different cells with thicknesses larger than several micrometers. In this paper we present detailed measurements of the electro-optic response of FLC confined to wedge-type cells with variable thickness from 0.3 to 4 μm and with different thickness of the aligning layer in the temperature range close to the smectic A–smectic C* phase transition. We observe pronounced effects of the confinement on the dynamic and static properties. The results indicate a strong influence of the thickness of the rubbed aligning layer on the surface anchoring. This influence is reflected in the dynamical properties of the submicron, confined ferroelectric smectic C* phase and in the smectic A → smectic C* phase transition temperature. The dependence of surface anchoring on the thickness of the rubbed aligning layer in the case of the nematic liquid crystal phase has also been studied by Alexe-Ionescu *et al.* [14].

2. Theory

In the following we analyse the influence of the cell thickness *d* on the smectic A–smectic C* phase transition temperature and on the dynamics of the smectic C* phase. We consider very thin cells with the material in the bookshelf structure, where the thickness is smaller

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than the bulk period of the helix and the helicoidal smectic C* structure is unwound [15]. We therefore assume that the ferroelectric phase is inhomogeneous only across the cell (x direction) and is homogeneous in the y and z directions (figure 1).

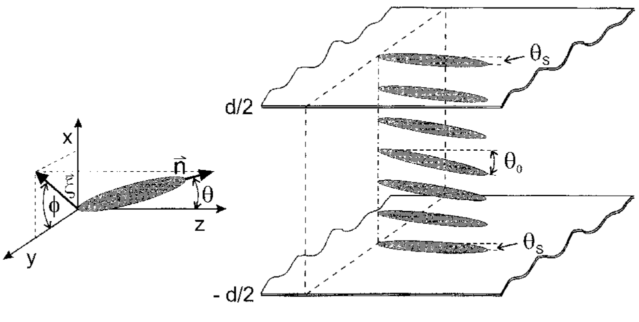
Using the equal elastic constant approximation, we write the free energy density f as a function of the order parameter $\xi=(\theta\sin\phi,\theta\cos\phi)$:

$$f=\sum_{i=x,y}\left[\frac{1}{2}\alpha(T-T_{\mathrm{c}})\xi_i^2+\frac{1}{2}K\left(\frac{\partial\xi_i}{\partial x}\right)^2\right]+f_{\mathrm{s}} \qquad (1)$$

where α is a positive constant and T_{c} is the phase transition temperature in a bulk sample. The second term represents the elastic energy due to deformation of the director profile and K is the corresponding elastic constant. The third term represents the surface energy, which is usually written as a sum of non-polar and polar contributions. Their relative strengths are described by the anchoring constants W_{n} and W_{p}, respectively [2]:

$$f_{\mathrm{s}}=\frac{1}{2}(W_{\mathrm{n}x}\xi_x^2+W_{\mathrm{n}y}\xi_y^2)\left[\delta\left(x+\frac{d}{2}\right)+\delta\left(x-\frac{d}{2}\right)\right]+W_{\mathrm{p}}\xi_y\left[\delta\left(x+\frac{d}{2}\right)-\delta\left(x-\frac{d}{2}\right)\right]. \qquad (2)$$

The molecules of liquid crystals tend to align along the rubbing direction (z axis), and $W_{\mathrm{n}x}$ and $W_{\mathrm{n}y}$ represent the relative strengths of the out-of-plane and in-plane tilt of the molecules from the rubbing direction, respectively. In our experiments we cannot distinguish between the in-plane and out-of-plane anchoring and therefore we put $W_{\mathrm{n}x}=W_{\mathrm{n}y}=W$. On the other hand, polar interactions between the surface and the molecules try to rotate the electric dipole moments of the molecules in a direction normal to the surface, pointing, let us say, into the cell on both surfaces. Such an orientation of molecules at the surface leads to the well known splayed state. The splayed state is stable if the polar surface energy is larger than the corresponding elastic distortion energy [2]. The smectic A–smectic C* phase transition in cells with only polar surface coupling has been analysed by Rovšek and Žekš [16].



(a) (b)

Figure 1. (*a*) The coordinate system for the ferroelectric liquid crystal cell (θ tilt angle, ϕ azimuthal angle, ξ smectic order parameter, **n** molecular director). (*b*) A possible tilt angle distribution across the cell at the smectic A–smectic C* transition. θ_0 and θ_{s} are tilt angles in the middle and at the surface of the cell, respectively.

Let us estimate the influence of the cell thickness d on the phase transition temperature T_{c}. The confining geometry enforces elastic deformation in the cell. As a result, we expect that the smectic A–smectic C* phase transition temperature T_{c}^* in the restricted geometry is lower than the corresponding phase transition temperature in the bulk. Non-polar anchoring can induce a non-uniform distribution of the tilt angle across the cell, and the tilt of the molecules in the middle of the cell can be different from the tilt on the surface. With this in mind, we use a simple approximation for the director field, where the tilt angle increases linearly from the surface to the middle of the cell, figure 1 (*b*):

$$\theta(x)=\frac{2(\theta_{\mathrm{s}}-\theta_0)|x|}{d}+\theta_0. \qquad (3)$$

Here θ_0 is the tilt angle in the middle of the cell and θ_{s} is the tilt angle at the surface of the cell. An additional distortion of the director field can appear in cells with a splayed director structure. If we neglect the electrostatic energy of electric dipoles in the local electric field, we can approximate the phase profile in such cells with $\phi(x)=\pi x/d+\pi/2$ [17].

After inserting expressions for the tilt $\theta(x)$ and azimuthal angle $\phi(x)$ into the free energy density f, equation (1), and integrating f over the cell thickness, the total free energy per unit surface is

$$\frac{F}{S}=\frac{\alpha d}{6}(T-T_{\mathrm{c}})(\theta_0^2+\theta_0\theta_{\mathrm{s}}+\theta_{\mathrm{s}}^2)+\frac{2K}{d}(\theta_0^2-\theta_{\mathrm{s}}^2)+\frac{K\pi^2}{6d}(\theta_0^2+\theta_0\theta_{\mathrm{s}}+\theta_{\mathrm{s}}^2)+W\theta_{\mathrm{s}}^2. \qquad (4)$$

Here, the second and the third terms represent the elastic energy due to the variation of the magnitude and the phase of the tilt angle across the cell thickness. The third term is present only in cells with a splayed director structure. The last term is the surface energy due to the finite value of the tilt angle at the surface.

After minimization with respect to the surface tilt, $\partial F/\partial\theta_{\mathrm{s}}=0$, we can write the free energy expression as a function of θ_0, which has the role of the order parameter of the smectic A–smectic C* phase transition. At the second order phase transition temperature, the coefficient in front of the θ_0^2 term equals zero. This determines the shift of the phase transition temperature ΔT_c:

$$\Delta T_c = T_c - T_c^* = \frac{4W}{\alpha d} + \frac{24K}{\alpha d^2}\left\{1 - \left[1 + \frac{dW}{6K} + \left(\frac{dW}{6K}\right)^2\right]^{1/2}\right\} + \frac{\pi^2 K}{\alpha d^2}. \tag{5}$$

The last term is present only in cells with the splayed director structure which is caused by strong polar anchoring.

The dependence of the transition temperature on the cell thickness is therefore determined by the competition between the surface and the elastic energy. For strong anchoring, the surface energy is large, and the decrease of the smectic A–smectic C* transition temperature is proportional to $1/d^2$:

$$W \gg \frac{6K}{d} \Rightarrow \Delta T_c = \frac{K(\pi^2 + 12)}{\alpha}\frac{1}{d^2}. \tag{6}$$

On the other hand, for weak anchoring, the decrease of the transition temperature is proportional to $1/d$:

$$W \ll \frac{6K}{d} \Rightarrow \Delta T_c = \frac{2W}{\alpha}\frac{1}{d}. \tag{7}$$

Here we assume that in the case of weak anchoring there is no splayed structure in the cell.

Next, we analyse the influence of the cell thickness on the dynamical properties of the smectic C* phase following the work of Rastegar *et al.* [6]. We assume that the amplitude of the tilt angle θ is constant in the whole cell at a fixed temperature. This CAA approximation is valid far away from the phase transition, where the changes in the magnitude of the tilt angle under the action of external fields or confinement are small. We therefore consider here only the fluctuations of the phase angle. These fluctuations represent the so-called 'thickness mode', which is dominant in this phase. The director fluctuations at the surface are determined by the boundary conditions. These are defined by the minimization of the free energy at the surface and represent the torque balance at each interface

$$K\frac{\partial \Phi}{\partial x}\bigg|_{x=\mp d/2} = \pm W\Phi\bigg|_{x=\mp d/2}. \tag{8}$$

These boundary conditions allow surface fluctuations of the phase angle, but keep the amplitude of the tilt angle constant.

The non-equilibrium phase profile Φ is written as a sum of the equilibrium phase profile Φ_0 and phase excitation Ψ. The linearized Landau–Khalatnikov equation of motion, as deduced from the non-equilibrium free-energy density is

$$\gamma\frac{\partial \Psi}{\partial t} = K\frac{\partial^2 \Psi}{\partial x^2} \tag{9}$$

where γ is the rotational viscosity. The solutions are plane waves with wave vector $\mathbf{q}$

$$\Psi = \Psi_0 \cos(\mathbf{q}x)\exp(-t/\tau) \tag{10}$$

and the relaxation rate τ^{-1} given by

$$\tau^{-1} = \frac{K}{\gamma}\mathbf{q}^2. \tag{11}$$

The wave vector $\mathbf{q}$ is determined by the boundary conditions, equation (8), through the transcendent equation

$$\mathbf{q}\tan\left(\mathbf{q}\frac{d}{2}\right) = \frac{W}{K}. \tag{12}$$

This equation can be analysed in the limiting cases and determines the thickness dependence of the relaxation rate, equation (12). Similarly to the case of the shift of the phase transition temperature, the thickness dependence of the wave vector and relaxation rate is determined by the competition between surface and elastic energies. When the surface energy is large compared with the elastic energy of the distortion, we are in the strong anchoring regime and the relaxation rate is proportional to $1/d^2$:

$$W \gg \frac{2K}{d} \Rightarrow \tau^{-1} = \frac{K\pi^2}{\gamma}\frac{1}{d^2}. \tag{13}$$

On the other hand, when the elastic energy is large compared with the surface energy, the anchoring is weak and the relaxation rate is proportional to $1/d$:

$$W \ll \frac{2K}{d} \Rightarrow \tau^{-1} = \frac{2W}{\gamma}\frac{1}{d}. \tag{14}$$

One can see that the surface anchoring is reflected in the thickness dependence of the phase transition temperature and the relaxation rate of the phase mode in the same way. Both are proportional to $1/d^2$ when the anchoring is strong, and are proportional to $1/d$ when the surface anchoring is weak. Measurements of the cell thickness dependence of the phase transition temperature and the relaxation rate can therefore give valuable information on the magnitude of the surface coupling in thin SSFLC.

3. Experimental results and discussion

We have measured the temperature and thickness dependences of the order parameter relaxation rates in the smectic A and smectic C* phases of the ferroelectric liquid crystal material CE8, 4-(2-methylbutyl)phenyl 4′-*n*-octylbiphenyl-4-carboxylate, confined to ultra-thin homogeneously aligned cells.

The experiment was performed in wedge-type cells which were made of commercial glass plates with ITO electrodes and nylon aligning layers. The nylon aligning layer was deposited by dip-coating from a solution of nylon 6/6 in methanol. We used two different concentrations of nylon solution and therefore obtained two different thicknesses of the nylon coating, 15 and 50 nm. The bookshelf structure was obtained by rubbing the nylon surface with a soft velvet cloth.

The thickness of the cell was determined by the 4 μm glass spacers at one edge and a close (proximity) contact at the other edge of the glass. The thinnest part of the cell was approximately 0.2 μm thick in order to avoid direct contact between the two ITO electrodes. The thickness of each individual cell as a function of the position along the cell was determined by measuring the spectral transmittion. A $0.3 \times 2.0\,\mathrm{mm}^2$ slit was used to localize the measuring spot which was placed directly on the glass surface of an empty cell. In this way, the local thickness of the cell was determined with an accuracy better than ±0.05 μm. The resulting wedge angle was of the order of 1.5×10^{-3} and had a negligible effect on the experiment. The cells were filled in the isotropic phase and then slowly cooled to the smectic A phase, where a good homogeneous alignment of the liquid crystal was observed.

For the determination of the relaxation rates we used the linear electro-optic response technique, which is better known as the ‘electroclinic’ experiment when performed in the smectic A phase. Here one measures the real (in-phase) and imaginary (out-of-phase) parts of the linear electro-optic response. It has been shown that this method is an optical analogue of dielectric spectroscopy and can detect polar eigenmodes of the system [17, 18]. The linear electro-optic response of samples was measured at different thicknesses and different temperatures for two different nylon coatings. The thickness dependence was determined very precisely in a single cell, simply by changing the position of the laser beam which was slightly focused onto the surface of the wedge-type cell. In this way, we avoided the experimental errors which would be present if different individual cells of different thickness had been used. In the smectic A as well in the smectic C* phase we observed for a weak measuring electric field ($E < 5\,\mathrm{V\,mm}^{-1}$) a single mode response with a Debye-like spectrum. For stronger electric measuring fields, we observed an additional non-linear mode in the smectic C* phase; this has been analysed elsewhere and is attributed to the switching process in the cell [17]. The relaxation rate of the single linear response mode was determined from the maximum of the imaginary part of the response. The observed mode corresponds in the smectic A phase to the soft mode. In the smectic C* phase it is the transverse phase mode, because it is strongly thickness-dependent. This phase mode has been observed by several authors and is also called the ‘thickness’ mode.

We performed measurements on two different kinds of wedge-cells, which had two different thicknesses of the nylon aligning layer. The results obtained strongly depended on the thickness of the aligning layer.

The temperature dependence of the relaxation rate, as measured at three different cell thicknesses for cells with the thicker nylon layer ($d_0 \approx 50\,\mathrm{nm}$) are presented in figure 2(*a*). The relaxation rate of the soft mode decreases linearly in the smectic A phases with decreasing temperature and reaches a finite value at the phase transition. The slope of the soft mode versus temperature plot depends significantly on the thickness of the cell and is smaller when the thickness of the liquid crystal is small, as observed by other authors [19]. After the transition into the ferroelectric phase, we observed the phase mode. Its relaxation rate increases with decreasing temperature and reaches a constant value a few degrees below the transition. This anomalous behaviour just below the transition has been also observed by Pikin *et al.* [7], who explained it in terms of a rapid change

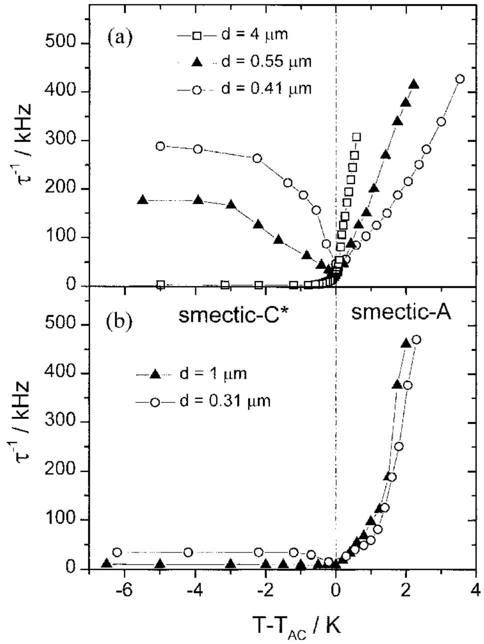


Figure 2. Temperature dependence of the relaxation rate of the soft mode in the smectic A phase and of the phase (thickness) mode in the smectic C* phase of CE8 at different thicknesses *d* of the liquid crystal. The upper graph (*a*) is for the cell with a thicker nylon coating (50 nm); the lower one (*b*) is for the cell with a thinner nylon coating (15 nm).

in the viscosity coefficient. We argue that this anomalous behaviour might appear because of the emergence of the chevron structure, which effectively decreases the thickness of the cell and therefore increases the corresponding relaxation rate of the phase mode.

For a given temperature, the relaxation rate of the phase mode in the smectic C* phase shows a very strong thickness dependence, as shown in figure 3 (*a*). One can see that in the case of the thicker nylon layer, the relaxation rate of the phase mode generally increases as $1/d^2$ on decreasing the cell thickness. The only deviation from this dependence is close to $d \approx 0.7\,\mu m$ ($1/d^2 \approx 2\,\mu m^{-2}$), where the relaxation rate is shifted to higher rates. This shift is caused by the appearance of a polarization-induced electrostatic charge density, and has been explained elsewhere [17]. As the relaxation rate of the phase mode is proportional to $1/d^2$, we conclude that we are dealing with a very strong surface anchoring in the case of the thicker nylon layer. From equation (13) we can determine the lower limit of the surface anchoring energy, $W > 2 \times 10^{-4}\,J\,m^{-2}$ for the thicker nylon coating. Here we used $K = 4 \times 10^{-11}\,N$ [20] and the minimum cell thickness $d = 0.4\,mm$, where this $1/d^2$ dependence is still observed. From equation (13) we can also determine the rotational viscosity $\gamma = 0.011\,kg\,m^{-1}\,s^{-1}$, which is in very good agreement with other experimental data [20, 4].

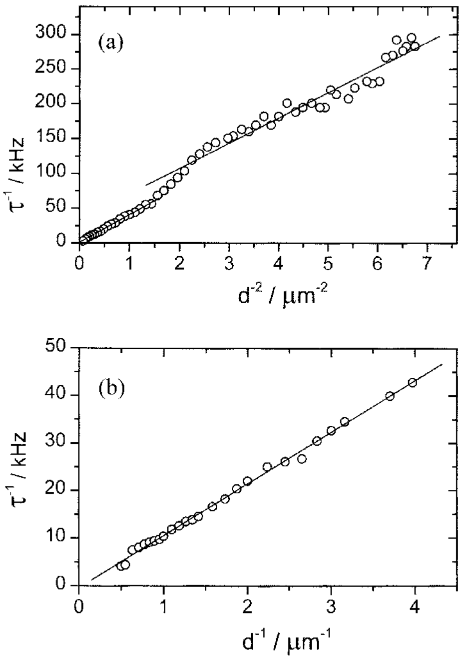


Figure 3. Relaxation rate of the phases (thickness) mode in the smectic C* phase of CE8 at $T = T_c - 8\,K$. This increases with the inverse square of the cell thickness $1/d^2$ in the cell with the thicker nylon coating (*a*) and as $1/d$ in the cell with the thinner nylon coating (*b*); the solid lines are the best fit to a $1/d^2$ and a $1/d$ dependence, respectively.

The temperature dependence of the relaxation rate for cells with the thinner nylon layer ($d_0 \approx 15\,nm$) is presented in figure 2 (*b*). It can be seen that the slope of the soft mode for the thinner coating does not depend on the sample thickness and decreases only in the vicinity of the phase transition. The relaxation rate of the phase mode in the smectic C* phase is for a given cell thickness much smaller than in the case of a thicker coating. For example, considering the thickness of the cell $d = 0.4\,\mu m$, we observe that the relaxation rate of the phase mode is about ten times smaller in cells with a thin coating. This leads immediately to the conjecture of a weak surface coupling in cells with a thinner nylon coating. The relaxation rate of the phase mode in cells with a thin nylon coating is shown in figure 3 (*b*), as a function of inverse thickness. One can clearly see that, in the case of the thinner nylon layer, the relaxation rate of the phase mode increases as $1/d$ on decreasing the cell thickness, as predicted by equation (14). This confirms the conjecture that the molecules are weakly anchored at the surface of this type of cell. Using values for the elastic constant K and viscosity γ from the previous case we can determine the surface energy for the thinner nylon layer; it is in this case $W = 8 \times 10^{-5}\,J\,m^{-2}$.

As an additional effect of the confinement, the phase transition temperature decreases with decreasing sample thickness. For the small cell thickness ($d < 2\,\mu m$), where we have the phase transition into the unwound smectic C* phase [15], the transition temperature was determined from the minimum value of the relaxation rate, which corresponds to the maximum value of the static electro-optic reponse. For the larger cell thicknesses, we have the transition into the helicoidal smectic C* phase ($d > 2\,\mu m$). In this case, the transition temperature was determined from a change of the slope of the temperature dependence of the relaxation rate.

The shift of the phase transition temperature versus cell thickness is shown in figure 4 (*a*) for the case of the thicker nylon coating. One can readily observe that this decrease is proportional to $1/d^2$, which is a clear indication of strong surface anchoring, see equation (6). On the other hand, as shown in figure 4 (*b*), this shift is proportional to $1/d$ in cells with the thinner nylon coating, which is a clear indication of weak surface anchoring, see equation (7). The strength of surface anchoring is directly reflected in the magnitude of the decrease of the phase transition temperature. For example, the decrease of the phase transition temperature relative to the extrapolated bulk value is approximately 4 K at $d = 0.4\,\mu m$ [this corresponds to $1/d^2 = 6.25\,\mu m^{-2}$ in figure 4 (*a*)] in the cell with the thicker nylon layer. On the other hand, the decrease of the phase transition temperature

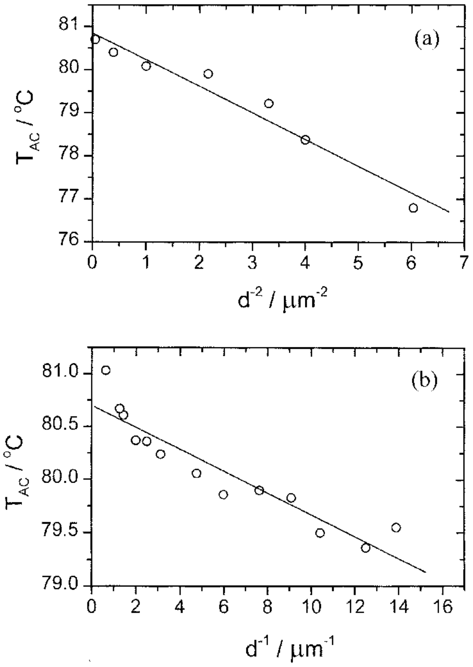


Figure 4. Thickness dependence of the smectic A–smectic C* phase transition temperature T_c in CE8. T_c decreases with decreasing sample thickness as the inverse square of the cell thickness $1/d^2$ in the cell with the thicker nylon coating (*a*) and as $1/d$ in the cell with the thinner nylon coating (*b*); the solid lines are the best fit to a $1/d^2$ and a $1/d$ dependence, respectively.

at $d = 0.4\,\mu\text{m}$ in the cell with the thinner nylon layer [this corresponds to $1/d = 2.5\,\mu\text{m}^{-2}$ in figure 4(*b*)] is of the order of only 0.4 K.

From the thickness dependence of the decrease of the transition temperature, the surface anchoring energy can be estimated. In the case of strong surface anchoring, the lower limit of the surface anchoring energy W can be determined, see equation (6). Following the condition in equation (6) we obtain $W > 5 \times 10^{-4}\,\text{J m}^{-2}$ for the cell with the thicker nylon layer. Here we used $K = 4 \times 10^{-11}$ N [20] and the minimum cell thickness $d = 0.4$ mm, where the $1/d^2$ dependence (strong anchoring) is still observed. This is consistent with the value $W > 2 \times 10^{-4}\,\text{J m}^{-2}$, obtained from the independent measurements of the thickness dependence of the relaxation rates in the smectic C* phase. On the other hand, from equation (7) the surface anchoring energy $W = 1.3 \times 10^{-4}\,\text{J m}^{-2}$ for the thinner nylon layer. This is again consistent with the surface anchoring energy $W = 8 \times 10^{-5}\,\text{J m}^{-2}$, obtained from the measurements of the thickness dependence of the phase mode. Here we have used $\alpha = 2.6 \times 10^3\,\text{N K}^{-1}\,\text{m}^{-2}$, obtained from the temperature slope of the soft mode rate in the smectic A phase and $\gamma = 0.001\,\text{kg m}^{-1}\,\text{s}^{-1}$.

4. Conclusions

In conclusion, we have observed the temperature and thickness dependence of the relaxation rates of the soft mode in the smectic A and the phase mode in the smectic C* phase in ultra-thin ferroelectric liquid crystal cells with two different thicknesses of aligning nylon coatings. Our measurements clearly show that the surface anchoring energy depends on the thickness of the nylon layer. The relaxation rate of the phase mode in the smectic C* phase, as well as the decrease of the smectic A–smectic C* phase transition temperature are proportional to the inverse of the cell thickness for the thinner nylon layer and to the inverse square of the cell thickness for the thicker nylon layer. Following theoretical predictions, this implies weak anchoring on the surface of the thin aligning layer and strong anchoring for the thicker nylon coating. The surface anchoring energy of ferroelectric liquid crystals varies from $W = 1(\pm 0.3) \times 10^{-4}\,\text{J m}^{-2}$ for thinner to $W \geqslant 5 \times 10^{-4}\,\text{J m}^{-2}$ for thicker nylon coatings. It has been reported [14] that surface anchoring energy of nematic liquid crystals decreases with the thickness of the aligning layer. This was explained by considering the screening effect of the polymer film on the anisotropic van der Waals interactions between the liquid crystal and the solid substrate. We think that polar interactions between the rubbed layer and the molecules of the ferroelectric liquid crystal material are important for the anchoring of ferroelectric liquid crystals. However, the physical mechanism, which is responsible for the different surface anchoring of ferroelectric liquid crystals on aligning coatings with different thicknesses is not yet completely understood, and is the subject of further investigations.

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