

Surface-induced molecular tilt above the smectic-A –smectic-C phase transition in a nonchiral liquid crystal

Ishtiaque M. Syed and Charles Rosenblatt

Department of Physics, Case Western Reserve University, Cleveland, Ohio 44106-7079, USA

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A polyimide-coated substrate was rubbed in such a way as to possess two competing easy axes for liquid crystal alignment. On cooling a homeotropically aligned liquid crystal through the smectic-A phase toward the smectic-C phase transition, an increasing tilt of the molecules relative to the layer normal was observed. The tilt was localized to within a smectic-C correlation length of the interface, and was found to increase monotonically with the rubbing strength associated with the preparation of the polyimide surface. The results are discussed in light of the dual easy axis model [T. Shioda *et al.*, Phys. Rev. E **67**, 041706 (2003)], and suggest that the two easy axes are not mutually orthogonal.

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The symmetry of a liquid crystal is reduced at an interface. In consequence, the liquid crystal generally adopts some degree of order that is associated with the lower symmetry phase near a phase transition. In the isotropic phase, for example, both dipolar [1–5] and quadrupolar (nematic) [6–13] orientational order may be induced at an interface, where the associated order parameters are related to the particular characteristics of the surface. Specifically, when a polymer alignment layer is deposited on a substrate and rubbed unidirectionally, the principal axis of the induced nematic tensor order parameter lies nearly parallel to the rubbing direction, and its magnitude is related to the “strength” of the rubbing [6–8,14]. Because the anisotropic potential is localized to the polymer–liquid crystal interface, the induced nematic order parameter decreases monotonically with distance z from the interface over a length scale of order the nematic correlation length. An analogous phenomenon is observed at the nematic–smectic-A (Sm-A) phase transition. In this case smectic layering is induced in the nematic phase at a molecularly flat substrate [15–21], and the amplitude $|\psi|$ of the Sm-A order parameter—this corresponds to the amplitude of the periodic density wave—decreases monotonically with increasing distance from the interface over a length scale corresponding to the Sm-A correlation length. For a rough surface it has been shown both theoretically and experimentally that the amplitude of the order parameter $|\psi_0|$ at the interface is reduced from its value at a flat interface [20,21].

The response to *bulk* stimuli of a liquid crystal in the Sm-A phase close to the smectic-C (Sm-C) phase transition is well known [22]. Both magnetic and electric fields have been applied to the Sm-A phase, resulting in a torque that induces a tilt of the molecules by a polar angle θ with respect to the smectic layer normal [23–25]. Here θ corresponds to the amplitude of the Sm-C order parameter. On approaching the Sm-A–Sm-C phase transition temperature T_{AC} from above, the tilt susceptibility $d\theta/d\tau$ diverges, where τ is the torque, which is proportional to H^2 or to E^2 . There are numerous open questions dealing with the critical exponent γ associated with the susceptibility—is the transition mean-field, XY-like, or other? and where is the Ginzburg crossover?

[26]—with ample evidence supporting several different phase transition behaviors [25,27–34].

At an *interface*, the response of the liquid crystal above the Sm-A–Sm-C phase transition may be investigated in two limiting geometries. In the “bookshelf” geometry, a polymer-coated substrate is rubbed unidirectionally, causing the Sm-A layer normal to adopt planar or near-planar alignment parallel to the rubbing direction. No torque is imparted by the treated polymer in the Sm-A phase for the case of a nonchiral liquid crystal, and in consequence $\theta=0$ for $T > T_{AC}$. For the case of a chiral liquid crystal, however, the symmetry is further reduced and a nonzero θ obtains. This is the so-called “surface electroclinic effect,” for which there is a polar interaction between the liquid crystal and substrate that gives rise to an induced surface polarization and concomitant polar tilt relative to the smectic layer normal in the Sm-A phase [35,36]. In the second limiting geometry the surface is treated for homeotropic alignment, wherein the smectic layers lie parallel to the interface. As noted above, this geometry may be achieved with molecularly flat surfaces, or instead by applying an appropriate surfactant or side-chain polymer to a substrate. As no torque is imparted by the substrate, $\theta=0$ throughout the Sm-A phase for a nonchiral material. Moreover, since the dipolar coupling between the liquid crystal and the substrate does not couple to tilt, θ also is equal to zero for chiral molecules in the homeotropic geometry.

Recently we demonstrated a surface treatment that results in a robust and reproducible polar tilt angle θ_0 of the nematic director at an interface (corresponding to $z=0$), where θ_0 can be as large as 45° [37]. In that experiment a substrate was spin-coated with the polyimide SE1211 (Nissan Chemicals), baked for a period beyond the manufacturer’s recommendation, and rubbed. We believe that the resulting nematic alignment is a consequence of two competing preferred axes (“easy axes”), one planar and one homeotropic. This explanation was borne out in an experiment [38] in which we showed that an anchoring transition occurs on heating the nematic liquid crystal in a region close to (but above) the nematic–Sm-A phase transition temperature T_{NA} . Just above T_{NA} the surface-induced Sm-A order “stiffens” the director orientation, keeping the director perpendicular to the sub-

strate. However, with increasing temperature the surface-induced smectic order becomes weak, resulting in a reduction in the propensity for homeotropic orientation. At a sufficiently high temperature an anchoring transition occurs in which θ_0 becomes nonzero [38].

This ability to induce a large surface polar tilt of the director is expected to provide a means of inducing a nonzero molecular tilt θ_0 at the interface at temperatures above the Sm-C phase transition. Moreover, because the tilt susceptibility increases on cooling toward T_{AC} , we expect that an appropriately treated substrate, which provides nearly homeotropic orientation ($\theta_0 \sim 0$) well above T_{AC} , can induce an increasing tilt at the interface on approaching T_{AC} . Because $T > T_{AC}$, we also expect that $\theta(z)$ would decrease monotonically to zero into the bulk with the characteristic correlation length ξ associated with Sm-C fluctuations. The purpose of this paper is to report on measurements that examine the surface-induced polar tilt away from the homeotropic orientation above the Sm-A–Sm-C transition temperature. Our central result is that both θ_0 and ξ increase on approaching T_{AC} from above, and that at a given reduced temperature $t = (T - T_{AC})/T_{AC}$, the tilt angle at the interface $\theta_0(t)$ increases with increasing rubbing strength of the polyimide. It should be stressed that, unlike the dipolar-based surface electroclinic effect [35,36] for a chiral Sm-C liquid crystal (also known as the Sm-C* phase) in the bookshelf geometry, this surface-induced tilt is essentially nonchiral in nature.

A microscope slide was cleaned and spin-coated with the polyimide SE1211. The slide was prebaked at 80 °C for 30 min and then baked at 200 °C for 1 h. The slide then was placed in a rubbing machine in which a rotating roller covered by a cotton cloth passed over the slide. The slide was tilted on the bed of the rubbing machine in such a way that it was rubbed harder at one end than at the other, giving a gradient in the rubbing strength across the surface of the slide. The local rubbing strength n_f is defined as the number of fibers passing a position of unit width, and is given by [39]

$$n_f \approx (2r\delta)^{1/2} N\nu L_0 \sigma_f / s, \quad (1)$$

where δ is the depth of the fiber impression, $r = 4$ cm is the radius of the cylindrical roller, $N = 4$ is the number of translations of the substrate under the roller, $\sigma_f = 1040$ cm⁻² is the fiber density, $s = 0.5$ cm s⁻¹ is the translation speed of the substrate beneath the roller, $\nu = 8.33$ rotations per second is the rotation frequency of the roller, and $L_0 \sim 1$ cm is the arc length of the cloth around the roller that makes contact with the substrate. Note that $L_0 = 2\pi r$ if the cloth uniformly covers the roller, although in this experiment only a small part of the total arc length of the roller made contact with the surface during each revolution. A second slide was cleaned, spin-coated with SE1211 polyimide, baked, but *not* rubbed; thus, the alignment at this interface was homeotropic at all temperatures investigated. The two slides were placed together and separated by Mylar spacers of nominal thickness 2 μ m, adjusted for optimal parallelism, and cemented. The cell was housed in an oven that had temperature stability of approximately 10 mK, and was filled in the isotropic phase

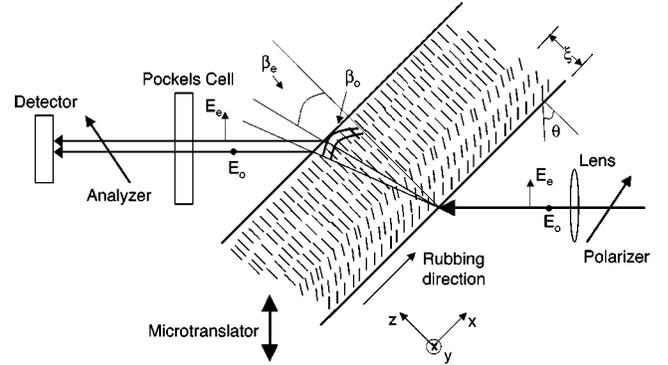


FIG. 1. Schematic representation of experiment. E_e and E_o correspond to extraordinary and ordinary polarizations, and $\beta_e(z)$ and β_o to the refractive angles for the phase fronts inside the cell. $\theta(z)$ corresponds to the tilt angle, and $\xi [= \xi_0 t^{-\nu}]$ to the Sm-A–Sm-C correlation length. The cell is mounted on a vertical translation stage so that different parts of the cell, and therefore different rubbing strengths, may be sampled.

with the racemic liquid crystal mixture SCE12R (Merck). The oven was cooled slowly through the isotropic–nematic transition at $T_{IN} = 118$ °C and through the nematic–Sm-A transition at $T_{NA} = 81$ °C. The oven and cell were tilted by an angle of 45° about an axis perpendicular to the rubbing direction and in the plane of the cell, and placed on a microtranslation stage to facilitate measurements at different values of n_f . Light from a 5 mW He-Ne laser passed through a polarizer (oriented at 45° with respect to the projection of the rubbing direction), a focusing lens, the sample, a Pockels cell, an analyzer, and into a photodiode detector (Fig. 1). The lens focused the light to a spot size of approximately 100 μ m at the sample, minimizing the effects of temperature gradients. The Pockels cell, modulated at a frequency $f = 5800$ Hz, served as an automatic retardation compensator [40], such that the dc voltage V_P applied to the Pockels cell was proportional to the optical retardation $\Delta\alpha$. The temperature of the oven was ramped downward from $T = T_{AC} + 1.3$ °C in the Sm-A phase to $T = T_{AC} - 0.5$ °C in the Sm-C phase at a rate of -0.5 °C min⁻¹, and the temperature and V_P were computer recorded. Retardation data were obtained for seven values of rubbing strength by probing different parts of the sample. Examples of $\Delta\alpha$ vs T are shown in Figs. 2 and 3.

In order to understand the retardation data, we first need to consider the origin of the polar tilt θ , and then the relationship between θ and $\Delta\alpha$. In a previous work, our group examined a rubbing-induced tilt transition in the nematic phase above the Sm-A phase transition [38]. The surface anchoring part F_{surf} of the volumetric free energy density was given as $F_{surf} = F_a \delta(z)$, where

$$F_a = A \sin^2 \theta_0 + B \cos^2 \theta_0 + C \sin^4 \theta_0, \quad (2)$$

$\delta(z)$ is the Dirac delta function, and thus θ_0 corresponds to the polar tilt angle at $z = 0$. The coefficient A corresponds to the usual quadratic anchoring strength coefficient for homeotropic alignment [15,41]. Rubbing of the polyimide induces alignment of the backbone, which we conjectured creates a

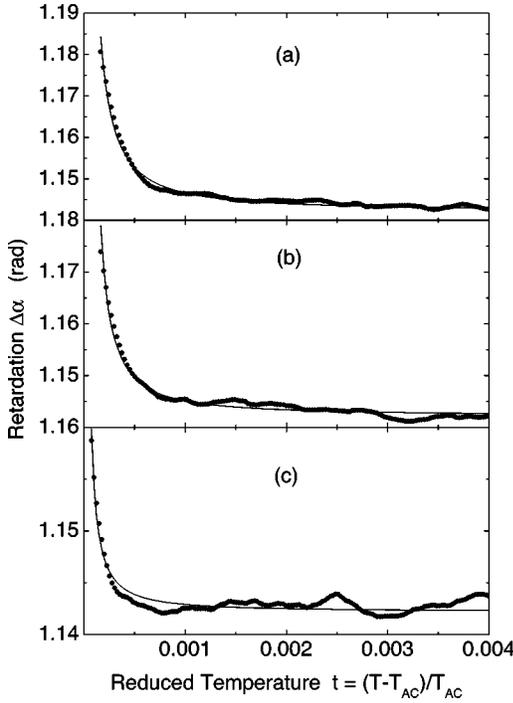


FIG. 2. Retardation vs reduced temperature for three different rubbing strengths. Fits assume that $\xi_0 = 0.5$ nm. Part (a) corresponds to $n_f = 3.55 \times 10^4$ cm $^{-1}$, (b) corresponds to $n_f = 3.32 \times 10^4$ cm $^{-1}$, and (c) corresponds to $n_f = 2.99 \times 10^4$ cm $^{-1}$.

second easy axis (for planar alignment) having anchoring strength B [38]. Because the two easy axes (homeotropic and planar) compete, we needed to introduce a higher order term in the interfacial free energy, $C \sin^4 \theta_0$, to establish an equilibrium polar angle θ_0 of the director. Based upon results from that experiment, the coefficient C was determined to be positive and may depend upon the rubbing strength. The form F_a in Eq. (2), however, is inconsistent with the results presented herein, whereby θ_0 increases *continuously* with decreasing temperature and no well-defined anchoring transition occurs. In light of Figs. 2 and 3, it is clear that the treated polyimide exerts a torque on the director in the Sm-A phase; nevertheless, we find that the form for F_a in Eq. (2) results in zero torque when $\theta_0 = 0$, and thus needs to be modified. Unlike our previous experiment above the nematic–Sm-A phase transition [38] in which the stylus of atomic force microscope was used to rub the polyimide bidirectionally (back and forth in each successive rub line), the cloth rubbing in this experiment was performed *unidirectionally*. In consequence, the volumetric anchoring free energy density may take the form $F_{surf} = F'_a \delta(z)$, where

$$F'_a = A \sin^2(\theta_0 - \varphi_A) + B \cos^2(\theta_0 + \varphi_B) + C \sin^4(\theta_0 - \varphi_C), \quad (3)$$

and where the positive angles φ_A , φ_B , and φ_C indicate that the easy axes may deviate from the homeotropic and planar directions. The signs of the arguments in Eq. (3) indicate that the two easy axes lie in the range $0 < \theta_0 < \pi/2$. Expanding F'_a for small θ_0 , we find

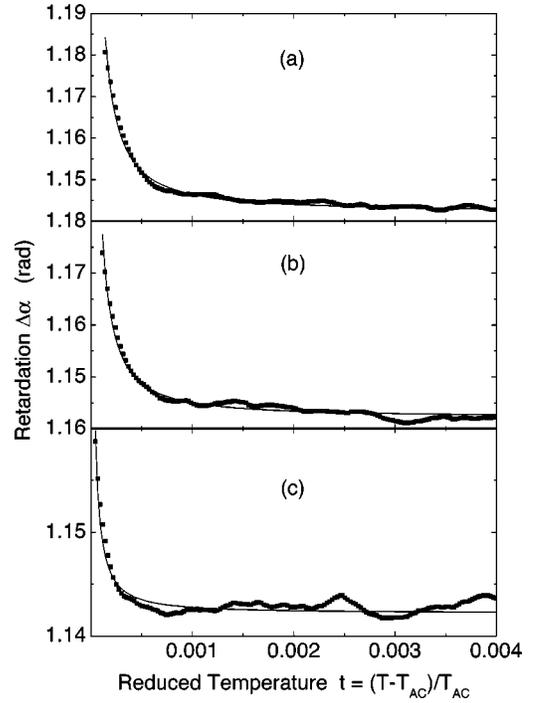


FIG. 3. Retardation vs reduced temperature for three different rubbing strengths. Fits assume that $\xi_0 = 2.0$ nm. Part (a) corresponds to $n_f = 3.55 \times 10^4$ cm $^{-1}$, (b) corresponds to $n_f = 3.32 \times 10^4$ cm $^{-1}$, and (c) corresponds to $n_f = 2.99 \times 10^4$ cm $^{-1}$.

$$F'_a \approx A \sin^2 \varphi_A + B \cos^2 \varphi_B + C \sin^4 \varphi_C - \theta_0 (A \sin 2 \varphi_A + B \sin 2 \varphi_B + 2C \sin 2 \varphi_C \cos^2 \varphi_C) + O(\theta_0^2). \quad (4)$$

Turning now to the bulk Sm-A terms, the appropriate volumetric free energy density in bulk is $F_{bulk} = D \sin^2 \theta + L(d\theta/dz)^2$, which may be approximated by $D\theta^2 + L(d\theta/dz)^2$ for small θ . Here D corresponds to the inverse tilt susceptibility [22] and the coefficient of the gradient term $L = D\xi^2$. Applying the Euler-Lagrange equation to F_{bulk} and solving the resulting differential equation with the boundary condition $\theta \rightarrow 0$ as $z \rightarrow \infty$, we find $\theta(z) = \theta_0 \exp(-z/\xi)$. The second boundary condition involves torque balance at $z = 0$, viz., $Dh\theta_0 + L(d\theta/dz)_{z=0} = (dF'_a/d\theta)_{z=0}$, where h is of the order of the smectic layer spacing. Substituting the form for $\theta(z)$, we obtain $\theta_0 = -(dF'_a/d\theta)_{z=0} / (Dh + \sqrt{LD})$, i.e., $\theta_0 = (A \sin 2 \varphi_A + B \sin 2 \varphi_B + 2C \sin 2 \varphi_C \cos^2 \varphi_C) / (Dh + \sqrt{LD})$. As the correlation length ξ becomes large on approaching T_{AC} , the term $\sqrt{LD} [= D\xi] \gg Dh$. Therefore, in the spirit of the continuum approximation we shall drop the term Dh , and thus

$$\theta(z) = \frac{(A \sin 2 \varphi_A + B \sin 2 \varphi_B + 2C \sin 2 \varphi_C \cos^2 \varphi_C)}{\sqrt{LD}} \times \exp(-z/\xi). \quad (5)$$

In principle, only the A term in F'_a would have been necessary to explain the experimental results; this would correspond to a tilt of the homeotropic easy axis by an angle φ_A .

The A term alone, however, is not sufficient to explain the tilt transition observed in the nematic phase above the Sm-A transition [38]; all three terms are required. Interestingly, because the coefficient of the θ_0 term in the expansion of F'_a is odd in φ_A , φ_B , and φ_C , bidirectional rubbing would result in an anchoring free energy proportional to a constant plus θ_0^2 , consistent with the form F_a used in Ref. [38]. Thus, as required by the combined results presented herein and those in Ref. [38], the appropriate form for the anchoring energy term would correspond to Eq. (3).

Let us now consider how the optical retardation $\Delta\alpha$ is related to the tilt angle θ . The retardation $\Delta\alpha \equiv \alpha_e - \alpha_o$, where α_e and α_o are the extraordinary and ordinary optical phase shifts, respectively. The phase shift of the ordinary phase front is given by $\alpha_o = 2\pi n_o d / \lambda \cos \beta_o$, where d is the thickness of the cell, n_o is the ordinary refractive index, $\beta_o = \sin^{-1}(\sqrt{2}/n_o)$ is the ordinary angle of refraction from Snell's law, and $\lambda = 633$ nm is the wavelength of the light (Fig. 1). Because the temperatures at which the experiment is performed are of order 50°C below the isotropic-nematic transition temperature T_{IN} , the refractive indices may be treated as temperature independent. Using an Abbe refractometer, we found $n_o = 1.4834$ and $n_e = 1.6476$. Turning to the extraordinary phase front and treating the optical dielectric properties as a tilted uniaxial material,

$$\alpha_e = \int_0^d (2\pi n^{eff} / \lambda \cos \beta_e) dz. \quad (6)$$

Here the effective z -dependent extraordinary refractive index $n^{eff}(z) = n_o n_e [n_o^2 \sin^2(\beta_e + \theta) + n_e^2 \cos^2(\beta_e + \theta)]^{-1/2}$, where θ and β_e are functions of z . β_e is the angle of refraction associated with the extraordinary phase front of the light. For calculational purposes the integral will be converted to a sum over thin slices (lamellae), and the retardation will be treated by a simple summation over the slices. Reflections at the interfaces between slices, which can result in interference effects and are accounted for in the Berreman 4×4 matrix approach [42], are not considered due to the small variation of θ through the cell. From Snell's law, for slice i we find that $\beta_{ei} = \sin^{-1}(n_{i-1}^{eff} \sin \beta_{e(i-1)} / n_i^{eff})$, where $n_{i=0}^{eff} = 1$ and $\beta_{e(i=0)} = \pi/4$.

As is clear from Eq. (5), θ depends upon the critical behavior of the susceptibility at the Sm-A–Sm-C phase transition. Over the years this has been a subject of some controversy, as evidence exists for several types of critical behavior [25,27–34]. Therefore, we performed an electroclinic experiment [43] on the chiral version of this material, SCE12, where we measured the bulk value $d\theta/dE$ vs temperature above the Sm-A–Sm-C* phase transition. From several experimental runs we obtained an average susceptibility exponent $\gamma = 1.2 \pm 0.1$, consistent with $\gamma = 1.20 \pm 0.05$ found in the synclinic/anticlinic material TFMHPOBC [23]. A typical set of data is shown in Fig. 4. Based on this result we performed a three parameter fit of the data using the form

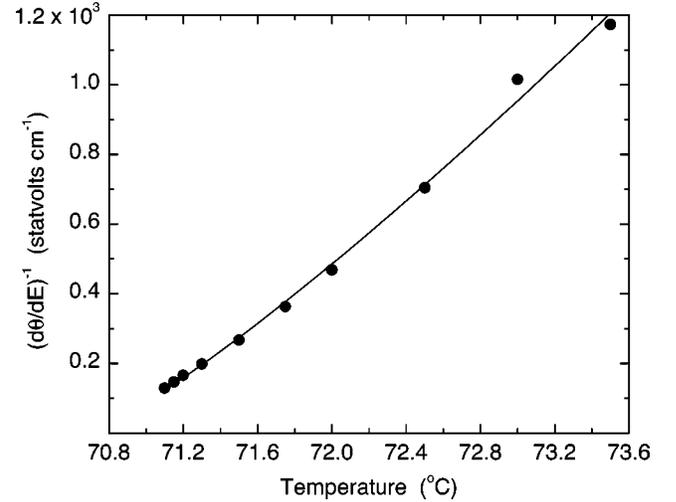


FIG. 4. Inverse tilt susceptibility $(d\theta/dE)^{-1}$ vs temperature obtained from electroclinic experiment for chiral SCE12. Susceptibility exponent $\gamma = 1.2 \pm 0.1$. Note that the transition temperature T_{AC*} for this batch of material is shifted slightly from T_{AC} for the racemate SCE12R.

$$\theta(z) = \theta_1 \left(\frac{T - T_{AC}}{T_{AC}} \right)^{-\gamma/2} \exp \left[- \frac{z}{\xi_0 \left(\frac{T - T_{AC}}{T_{AC}} \right)^{-\nu}} \right]. \quad (7)$$

The factor $\theta_1 t^{-\gamma/2}$ corresponds to the critical behavior of $-(dF'_a/d\theta)_{z=0} / \sqrt{LD}$ in Eq. (5), where the fitting parameter θ_1 is the “bare tilt angle” and depends on the surface treatment, and ξ_0 is the “bare correlation length.” From our electroclinic measurements we used $\gamma = 1.2$ and a correlation length exponent $\nu = 0.6$ in Eq. (7). The three fitting parameters were d , T_{AC} , and θ_1 . To avoid an excessive number of fitting parameters, we chose to fix the bare coherence length using two different values, viz., $\xi_0 = 0.5$ nm [32], which is a value associated with XY-like behavior, and $\xi_0 = 2.0$ nm [25], which is associated with mean-field behavior. Note that our susceptibility exponent $\gamma = 1.2$ falls between these two limiting behaviors: $\gamma = 1$ and $\gamma = 1.32$, respectively. Because our primary goal is to understand the qualitative behavior of θ_1 as a function of rubbing strength, the specific value of ξ_0 turns out not to be of critical importance. To calculate the extraordinary optical phase α_e , initial guesses were made for the parameters d , T_{AC} , and θ_0 . The cell then was divided into $N = 200$ slices each of thickness $w = 5$ nm, plus the remaining thickness $d - Nw$, for which we assumed $\theta = 0$. α_e was calculated as a function of temperature using the discrete summation form of Eq. (6), the discrete form of Eq. (7), as well as the forms for β_{ei} and n_i^{eff} . For the ordinary phase front, α_o was calculated using the initial guess for d . The resulting retardation $\Delta\alpha$ vs T was fitted to our experimental data, such as that shown in Figs. 2 and 3, by adjusting the parameters d , T_{AC} , and θ_1 . In order to assess the quality of the fits, experimental data for $\Delta\alpha$ vs T at each value of n_f were fitted over three ranges of temperature. The closest approach to T_{AC} for the short range fitting was chosen to be about 0.03°C , for the medium range fit it was 0.075°C , and

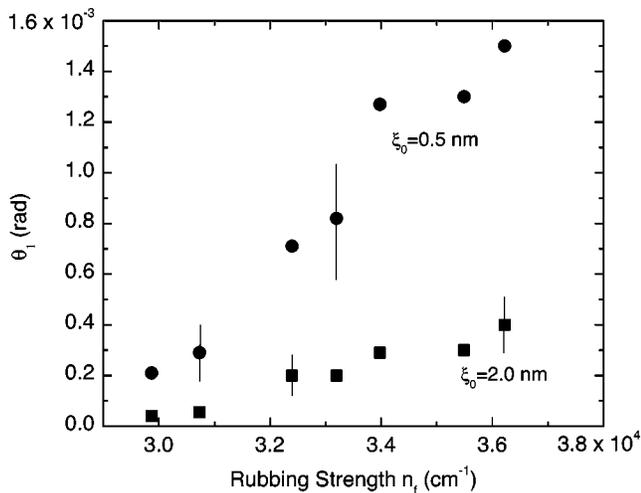


FIG. 5. Fitted coefficient θ_1 vs rubbing strength n_f . Circles correspond to fits using $\xi_0 = 0.5$ nm, and squares correspond to fits using $\xi_0 = 2.0$ nm.

for the long range fit it was 0.12°C . Comparing the three fitting ranges, all three fitting parameters showed consistent behavior for a given rubbing strength. The fitted cell thickness $d \approx 4.3 \pm 0.3 \mu\text{m}$ was found to vary slightly from one rubbing strength to another, as the cell thickness varied slightly from point-to-point in the cell. (Note that d is somewhat larger than the nominal spacer thickness due to curling of the Mylar along its edges.) Similarly, due to temperature gradients in the oven, $T_{AC} \approx 68^\circ\text{C}$ differed slightly from one rubbing strength to another. More importantly, θ_1 was found to exhibit a strong dependence on the rubbing strength, increasing monotonically with increasing n_f . Results for θ_1 are shown in Fig. 5 for both choices of ξ_0 . Due to the weak signal and relatively large noise level, it is not possible to determine a “best value” for the bare correlation length. Nevertheless, both sets of data show similar trends: A minimum rubbing strength is required before the onset of tilt, and the susceptibility increases approximately linearly with rub-

bing strength beyond this minimum n_f . A minimum rubbing strength required for the onset of tilt has been observed previously in the nematic phase [37,44].

Although φ_B , φ_C , and especially φ_A may depend on rubbing strength, we believe that the major effect comes from an increase in the coefficient B with increasing n_f . This would be consistent with our previous experiment involving bidirectional rubbing for which φ_A , φ_B , and φ_C could be taken as zero [38]. There is no reason to expect that B should be linear in n_f , especially given anchoring strength vs rubbing strength results in the literature [13,45,46]. Nevertheless, monotonic behavior certainly would be expected, and is observed in our data (Fig. 5). Finally, one issue that remains perplexing is the apparently small rubbing strength needed to achieve an observable effect. In our original observation of a rubbing-induced nonzero polar tilt angle in the nematic phase [37], a minimum rubbing strength of $n_f \approx 2.5 \times 10^6 \text{ cm}^{-1}$ was required to induce a nonzero polar angle. The interaction of substrate and liquid crystal, of course, depends on the nature of the liquid crystal as well as the substrate. In other experiments we have found qualitatively that SCE12R exhibits a polar tilt in the nematic phase for somewhat smaller anchoring strengths [47]. Thus, the apparently very small anchoring strengths needed in this experiment could reflect the structure of the liquid crystal, or perhaps it could be that the form for n_f given in Eq. (1) may not accurately represent the response of the polyimide to the rubbing. This is a subject for future investigation, and in no way diminishes our principal result that a localized tilt that increases with rubbing strength and that diverges on approaching T_{AC} from above may be induced in a nonchiral Sm-A phase.

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