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Influence of the surface topography on the surface transitions in nematics

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In quasi-homeotropic oriented liquid crystal cells, obtained by oblique SiO deposition on glass and surfactant added to the nematic, a continuous tilt angle *versus* temperature change followed by an abrupt surface transition was observed. At room temperature, the sign of the tilt angle measured from the normal to the surface depends on the SiO deposition angle. A model for the temperature dependence of the tilt angle is proposed.

1. Introduction

Nematic materials are liquids characterized by anisotropic properties typical of uniaxial media. Their anisotropic properties are connected with the tendency of strongly asymmetrical molecules to be parallel to each other giving rise to the mesophase. The average molecular direction, in the meaning of statistical mechanics, is called the nematic director. It is usually denoted by **n**. The degree of order of the molecular direction, along **n**, is called the scalar order parameter and is indicated by S. In the absence of external constraints, the **n** orientation is undetermined. The orientation of **n** can be modified by applying an external field, for example, an electric or magnetic field.

Near to a solid substrate or a free surface, the orientation of **n** follows from nematic–nematic and nematic–substrate interactions. From the experimental point of view, it is evident that in the absence of external interactions, for a given nematic liquid crystal and solid substrate, the orientation of **n** is always the same. Of course this surface orientation minimizes the interfacial energy. It is called the easy direction. The angle made by **n** with the geometrical normal, **k** to the surface, which is assumed to be flat, is known as the easy tilt angle. A number of papers are devoted to the surface properties of nematic liquid crystals. In the following we will be interested in the temperature variation of the easy tilt angle. This temperature induced orientation change is known as the surface transition. It depends, in our opinion, on the interaction of the bulk and the surface, *via* the gradient of the scalar order parameter, as in the ordoelectric model described in § 3.

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Our paper is organized as follows: in § 2 a review of experimental observations is reported. In § 3 the models proposed to interpret the surface transitions are critically reviewed. In § 4 our experimental data regarding the tilt angle dependence on temperature and the effect of the surface topography on this are presented and in § 5 model to interpret these results is proposed. In § 6 the main conclusions of our paper are presented.

2. Previous experimental results

The surface transitions in nematic layers near the clearing temperature at the nematic free surface, the nematic–isotropic interface or at the nematic–solid substrate interface have been experimentally studied by different authors.

Bouchiat and Langevin-Cruchon [1] showed that the molecules of nematic PAA line in the plane of the free surface, while those of nematic MBBA are tilted at an angle $\theta = 15°$ from the normal to the surface. Chiarelli *et al.* [2] discovered a structural transition at the free surface of MBBA when the temperature reaches a critical value T_0. For $T < T_0$, the tilt angle depends on the temperature as $(T_0 - T)^{1/2}$, while for $T > T_0$ a planar alignment is obtained. They measured the anchoring energy of the director at the free surface, which goes to zero as $(T_0 - T)$.

Tilt transitions for nematics on treated substrates were observed by Ryschenkow and Kleman [3] who obtained an approximate value of W for the tilted phase of MBBA at a solid substrate. Känel *et al.* [4] found that in the nematic phase of butoxybenzyl-ideneoctylaniline, on smooth flat glass, the director exhibits a 'transition' from the homeotropic (favoured by the interfacial tension at the surface) to the parallel state, when the temperature is increased. The surface transition could be suppressed by square wave gratings of sufficient depth or short enough period and in such a case the liquid crystal remains in the planar state for all temperatures.

The behaviour of the torsion anchoring strength coefficient near the phase transition at the interface between 5CB and a glass plate treated by oblique evaporation of SiO to produce homogenous parallel alignment of the director with a high anchoring energy coefficient was studied by Faetti *et al.* [5]. They found that the anchoring energy exhibits a roughly linear dependence on temperature, greatly decreasing as the phase transition is approached. DiLisi *et al.* [6] studied the behaviour of the anchoring strength coefficient near the structural transition occurring at a temperature T_s in the nematic phase of a nearly symmetric alkoxyphenyl benzoate monomer from perpendicular, $(T > T_s)$, to tilted $T < T_s$ alignment at a surfactant–treated glass substrate. Using polarizing optical microscopy the sample is found to be uniformly aligned in the homeotropic orientation for $T > T_s$. As soon as T becomes lower than T_s, a schlieren type texture becomes visible, increasing in brightness as the temperature is further reduced. The transition to a tilted state is weakly discontinuous although the resolution in the data is insufficient to be certain. The coefficient of the anchoring energy has been obtained by means of the Fréedericksz transition. For $T > T_s$, on approaching the temperature transition T_s, it rapidly decreases when the temperature decreases and apparently vanishes at T_s.

The anchoring strength coefficient of the same alkoxyphenyl benzoate monomer, aligned parallel to a buffed polymide coated glass substrate, increases with decreasing temperature, while the sample shows no structural transition [7]. A bistable anchoring of nematics on SiO films was discovered [8, 9] by studying the surface orientation of 5CB on thin films (of thickness d) obtained at grazing incidence (α). For a narrow region in the (α, d) plane, a new type of anchoring was found, which is tilted and makes an angle

different from zero or 90° with the plane of evaporation of SiO. In this region, the symmetry allows the anchoring director to have opposite azimuths on both sides of the plane of evaporation. Flatischler *et al.* [10] observed and studied the reversible alignment transition from a low temperature homeotropic alignment to a high temperature planar one in a symmetrical cell in which alignment was obtained by lecithin deposition on SiO and the aligning layer evaporated at an angle of incidence of 60°. The surface alignment transition takes place at a well defined temperature and is abrupt. A surface alignment transition between a quasi-homeotropic and a quasi-planar state was reported by Komitov *et al.* [11] for an antisymmetrical cell made by lecithin deposition on SiO alignment layer evaporated at an incidence angle of 85°. The temperature dependence of the tilt angles for non-twisted and twisted nematic samples was studied by S. Shimoda *et al.* [12]. The nematics used were MBBA, 7CB, E-8, ZLI 1132, ZLI 1083 and the samples were prepared by double evaporation of SiO for tilt angles greater than 80°, by simple oblique evaporation at 85° for medium (70–50°) tilt angles, and by application of a surfactant followed by oblique evaporation at 85° of SiO for small ($\sim$40°) tilt angles. Measurements were made by the magneto-capacitive null method for non-twisted samples and by capacitance voltage for twisted ones. The results are that the tilt angles increase with increasing temperature for both non-twisted and twisted samples, but the twisted cells have a weaker increasing tendency. In the case of a 90° twist a slight decrease in the tilt angle was observed throughout the temperature range.

3. Theoretical models to interpret the surface transitions

Different theoretical models have been proposed to explain the temperature dependence of tilt angles and surface order transitions.

Shimoda *et al.* [12] expressed the surface energy by assuming that there is an easy axis on the surface and a surface tensor order parameter directed along the easy axis. It was, however, shown that the analytical considerations resulting from the model are not correct from a mathematical point of view [13].

Parson [14] proposed a phenomenological model according to which there are two contributions to the surface energy, one due to dipolar and the other to quadrupolar interactions. The two contributions have different temperature dependencies. The surface order transition can be interpreted in terms of Parson's theory. Therefore the model is probably correct, but the dipolar contribution cannot play the role required by the model itself, because in a continuum theory, the polar contribution, which is limited to one or two molecules, cannot be directly taken into account easily.

Känel [4] proposed an explanation for the flip from planar to homeotropic alignment in the nematic phase for the liquid crystal 40·8 which exhibits a nematic to smectic A phase transition. This model takes into account the smectic short-range order imposed by the surface in the free energy of interaction of the liquid crystal with the substrate under weak anchoring situations.

In the phenomenological model of Sluckin–Poniewierski [15] the surface energy is built by means of symmetry arguments in the function of the surface tensor order parameter Q_{ij}. From such arguments, in the surface energy a term of the kind $Q_{ij}Q_{ij}$ is present. The model shows that tilt angle transitions are allowed. An example of the temperature dependence of the tilt angle is deduced by supposing that the scalar order parameter is position independent, which is surely an oversimplifying hypothesis.

In the phenomenological model proposed by Gabbasova *et al.* [16], the surface energy is built by means of symmetry arguments as in the Sluckin–Poniewierski model,

but the term $Q_{ij}Q_{ij}$ is not included because it seems a bulk term. The phase diagrams of tilt angle *versus* temperature for different values of phenomenological coefficients have been obtained in the simplified hypothesis of position independent scalar order parameter.

The surface transitions may also be interpreted in terms of order electric polarization [17]. According to this model, an electric polarization is connected to the spatial variation of the quadrupolar tensor order parameter $Q_{ij}=S[n_jn_j-(1/3)\delta_{ij}]$. When the nematic director is position independent, this polarization is found to be proportional to the gradient of the scalar order parameter S. The connected dielectric energy, proportional to the square of the component of the polarization normal to the interface, gives a contribution to the total free energy depending on the surface tilt angle. The analysis of the total free energy allows one to predict, in some cases, surface order transitions when the temperature changes.

All the previously quoted models refer to a flat and isotropic surface. Consequently, surface energies are built by supposing that the scalar quantities appearing in its expansion are of the kind $k_iQ_{ij}k_j$, $k_iQ_{ij}Q_{je}k_e$ and so on, in which $\mathbf{k}$ is the surface geometrical normal. From this hypothesis, it follows that the angular terms present in the surface energy are powers of $(\mathbf{n}\cdot\mathbf{k})^2=\cos^2\theta$. Consequently θ and $-\theta$ correspond to the same energy, and they are completely equivalent. In particular the predicted surface transitions range in $0<\theta<90°$ or $-90°<\theta<0$. On the contrary, our experimental data, reported in § 4, show in some cases a surface transition in which θ increases from a negative value to 90°. This experimental evidence forces us to assume that the surface is either flat and anisotropic, or characterized by a well defined geometry and locally isotropic. For reasons described in the following, in the case of obliquely evaporated SiO over glass plates, it seems that the surface may be considered to be of the latter type.

We propose in the following, a modified version of the Sluckin–Gabbasova model which takes into account the geometry of the surface by assuming a saw-teeth profile. The equivalent easy axis of the non-uniform surface characterized by a periodic distribution of easy directions is determined by using a simple model recently proposed [18]. This model differs from Yokoyama's model [19] that supposes the existence of two easy directions $\mathbf{k}$ and $\mathbf{e}$ in every point at a geometrical uniform surface, the first being the geometrical normal and the second one due to the epitaxial axis (or to the nematic flow direction during the filling procedure). In that model, the surface energy contains terms of the kind $(e_iQ_{ij}e_j)$, and possibly interference terms of the kind $k_iQ_{ij}e_j$, $(k_iQ_{ij}e_j)^2$ and so on, whose physical meaning is not clear.

4. Experimental results

The samples for experiments were prepared from plane glass surfaces covered by ITO on which a SiO layer was obliquely deposited by vacuum evaporation at incidence angles of $61\pm1°$ or $67\pm1°$. The two glasses forming a cell were mounted so that symmetrical boundary conditions were obtained (see figure 1). In the conditions in which the angles on the surfaces are supposed to be the same, a uniform structure and an undistorted bulk are obtained and, therefore,the results can be straightforwardly interpreted as depending only on the surface properties. Mylar spacers were used and finally the cell gap was measured by an interferometric method. The cells were filled with the nematic ZLI 1623 (Merck) having the nominal nematic range $-25-+80$°C, a negative dielectric anisotropy, and an added dopant for homeotropic alignment provided by the liquid crystal supplier. A direct measurement of T_C for our compound (two years old) gave $77°<T_C<78°$, according to the sample. The liquid crystal was

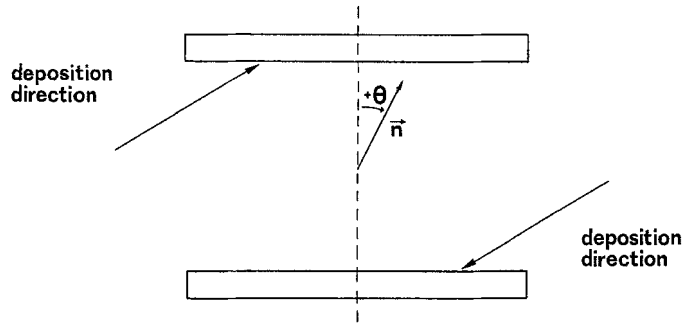

Figure 1. Sample assembly and definition for the sign of tilt angle θ.

introduced into the cell by capillarity in the isotropic phase, in order to avoid easy directions due to the nematic flow. The filling procedure was parallel to the SiO deposition one.

The observations and measurements were made with the sample introduced in a Linkham THM 600 hot stage (temperature control precision 0·1°C) and using a polarizing microscope Leitz Laborlux 12 Pol. The orientation of the optical axis of the samples was determined conoscopically and the anchoring tilt angle of the liquid crystal was determined by measuring the optical path difference introduced at normal incidence by the nematic layer, using the tilting compensator of the polarizing microscope. The dependence of optical birefringence of the liquid crystal *versus* temperature was separately measured using a wedge cell made with glass without SiO deposition, giving homeotropic alignment in the temperature range of interest.

At room temperature the samples present a quasi-homeotropic structure with the optical axis lying in the evaporation plane for SiO deposition and tilted in a sense and with an angle which depends on the incidence angle for SiO deposition. For these tilted structures we adopt the following convention: a structure having the optical axis tilted on the same side of the normal to the cell as the direction from which SiO was deposited on one of the surfaces, has a plus sign for the tilt angle (see figure 1). This angle is positive for the cells made with deposition at 61°, but is negative for cells made with deposition at 67°. The values of these angles depend on the thickness of the SiO deposition, but in our experience that parameter was not rigorously controlled.

When the temperature is raised, both type of sample remain uniaxial with the optical axis in the plane of SiO deposition. The tilt angle increases continuously for both samples either from small positive to larger positive angles for the first type of sample, or from negative values, passing through zero, to positive values for the second type of samples, as can be seen from figures 2 and 3. The sign of the tilt angle was established by observing the conoscopic cross position relative to the normal to the cell and the surface deposition direction. θ was determined by the approximate equation

$$\theta = \frac{180^\circ}{\pi} \arcsin\left\{\sqrt{\left(\frac{2\Delta}{h\nu n_0}\right)}\right\},$$

where Δ is the optical path difference introduced by the liquid crystal sample, h the sample thickness, n_0 the ordinary refractive index, $\nu = 1 - (n_0/n_e)^2$ and n_e the extraordinary refractive index.

The main error in the determination of θ is due to the determination of the optical path difference. In the tilting compensator method, this is obtained by estimating the

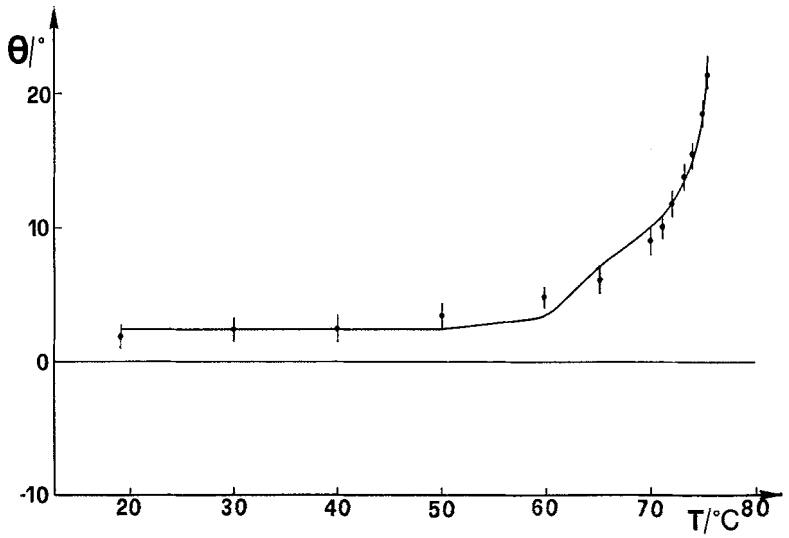

Figure 2. Tilt angle *vs.* temperature for a sample prepared by SiO deposition at 61° incidence angle. The solid line is the best fit with $G_0 = 2{\cdot}5°$, $G_1 = 0{\cdot}22$ and $T_2 = 75{\cdot}3°C$.

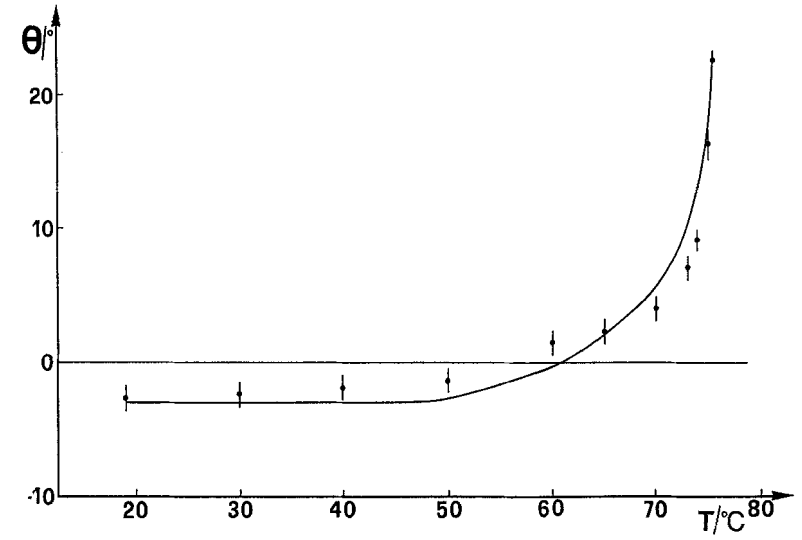

Figure 3. Tilt angle *vs.* temperature for a sample prepared by SiO deposition at 67° incidence angle. The solid line is the best fit with $G_0 = -2{\cdot}8°$, $G_1 = 0{\cdot}243$ and $T_2 = 75{\cdot}22°C$.

middle of the dark band for which the compensation of the optical path difference introduced by the sample takes place. The maximum error in the determination of the middle of that band was taken as equal to 1/4 of the band width.

At a given temperature $T_0 < T_c$ (where $T_c \sim 80°C$ is the clearing point) an abrupt change of superficial orientation takes place. The temperature of this transition ($T_0 = 76 \pm 1°C$) depends on the sample and on the observation point for a given sample. This phenomenon is reversible on condition that the rate of variation of the temperature near T_0 is less than some degree per minute, and the nematic–isotropic clearing point is not reached nor exceeded during the experiment. The transition takes place at both

surfaces separately and this fact can be seen for temperatures near T_0, because a thermal gradient exists across the sample or because there is a small difference in T_0 for the two surfaces (see figure 4). After this surface transition, the director at the surfaces and in the volume remains in the same plane as before the transition, but the tilt angle at this surface changes from a moderate value (approx. 25° for $T < T_0$) to a large value (90° for $T > T_0$) [20]. We were led to these conclusions by:

repeated cycles of observations, for a temperature $T \cong T_0$, do not display the existence of twisted structures both in regions in which the transition domains advanced separately on the two surfaces, and in regions where this transition developed, seemingly, only on one surface;

after the transition has taken place at both surfaces, no change in optical birefringence of the sample may be observed by applying a high frequency, high level voltage; on the other hand, the Williams domains produced by applying a low frequency voltage are oriented in a direction perpendicular to the direction of SiO deposition on the glass surfaces.

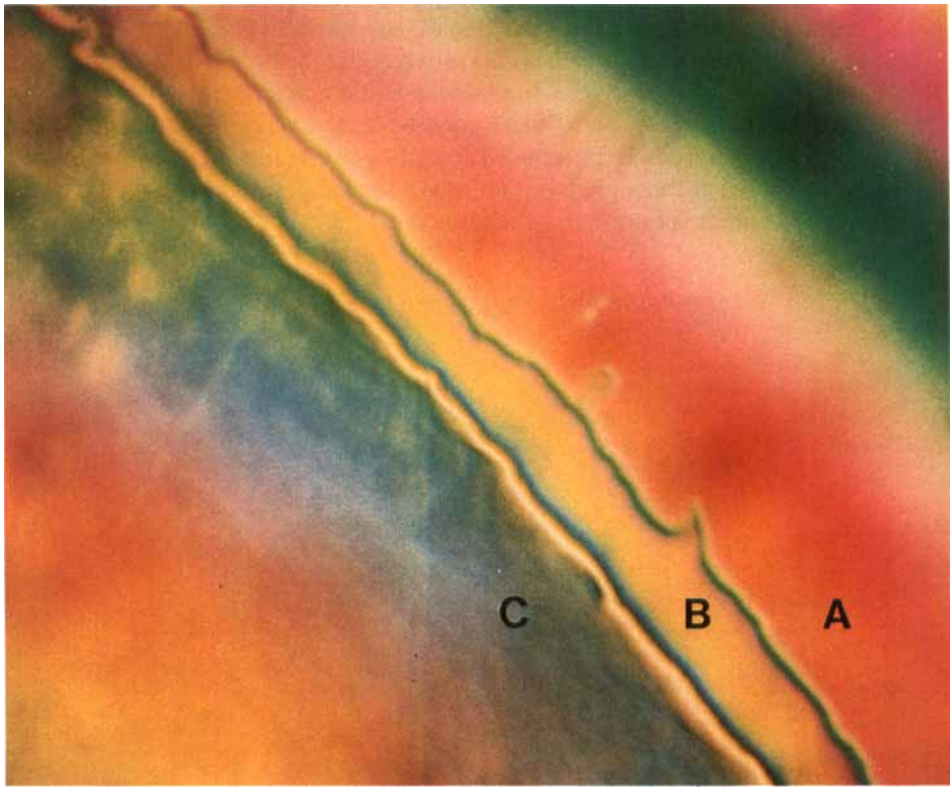

Figure 4. Photomicrograph of liquid crystal sample undergoing a surface transition with crossed polarizers making 45° with the easy surface directions. A tilting compensator is used for enhanced contrast of limits where surface transitions take place. A, region before the surface transition; B, region where the surface transition took place at one surface; and C, region where the surface transition took place at both surfaces.

5. The model

As is shown by electron microscopy [21], the films of SiO obliquely deposited in vacuum, display an inclined columnar growth structure arranged in a row-like order in a perpendicular direction to the incidence plane of SiO. The columnar structure is more or less periodic in a direction parallel to the incidence plane. Let λ be the mean period. The tilted columns have two principal inclined faces whose normals make the angle $\theta_1 < 0$ for the shadowed face and $\theta_2 > 0$ for the second face respectively that is exposed to the vapours during the SiO deposition. The parameters λ, θ_1, θ_2 depend on the incidence angle. For a deposition made at an incidence angle of 60°, [21] gives $\lambda \sim 80$ Å, $\theta_1 \sim -55°$ and $\theta_2 \sim 60°$.

In an attempt to interpret our experimental results we will use a simplified model for the deposited surfaces, keeping some elements resulting from the columnar model (see figure 5). The simplfying hypotheses are the following:

(i) The deposited surface is considered as a plane surface, but with a periodical structure of period λ.

(ii) Over the interval $(0, a)$ a direction denoted by $\mathbf{N}_1$ (having $\theta_1 < 0$ inclination angle) oriented as the normal to the shadowed face of the columns is present, and over the interval (a, λ) a direction denoted by $\mathbf{N}_2$ (having $\theta_2 > 0$ inclination angle) oriented as the normal to the exposed face of columns is present. To put into evidence the directions normal to the column faces $\mathbf{N}_1$ and $\mathbf{N}_2$ as reference directions is a natural way of taking into account the homeotropic orientation tendency of the surfactant added to the liquid crystal.

(iii) On each interval, easy directions $\mathbf{E}_1$ and $\mathbf{E}_2$ respectively (different from $\mathbf{N}_1$ and $\mathbf{N}_2$ respectively) can be introduced according to a model previously proposed by one of the authors [16]. In that model the temperature dependence of the angle γ between the normal to the surface and the easy direction on this surface is given by.

$$\gamma = \arccos\sqrt{\{A(1 - \sqrt{[(T_1 - T_2)/(T_1 - T)]})\}}, \tag{1}$$

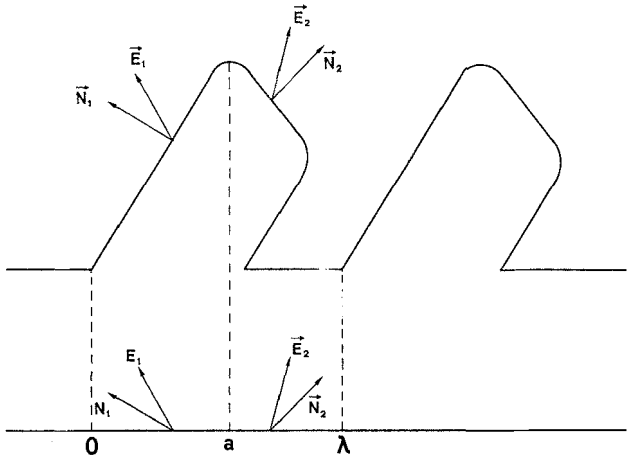

Figure 5. Simplified models of the SiO deposited surface. $\mathbf{N}_1$ and $\mathbf{N}_2$ are the normals to the column faces; $\mathbf{E}_1$ and $\mathbf{E}_2$ are the easy directions at the same surfaces. In the upper part is the columnar model and in the lower part is the corresponding flat model with a periodic distribution of easy directions.

Here T_1 is a temperature higher than the clearing point of the nematic and T_2 is the temperature at which the parallel molecular orientation with the respective surface is reached. The T_2 as well as the A values are obtained by combinations of some phenomenological parameters of the model. Applying that model we suppose that the four directions $\mathbf{N}_1$, $\mathbf{N}_2$, $\mathbf{E}_1$ and $\mathbf{E}_2$ are always in the incidence plane of SiO deposition and also that $\mathbf{E}_1$ and $\mathbf{E}_2$ are oriented in such a way that their tilting angles are given by:

$$\alpha_1 = \theta_1 + \gamma \quad \text{for} \quad \mathbf{E}_1, \tag{2}$$

and

$$\alpha_2 = \theta_2 - \gamma \quad \text{for} \quad \mathbf{E}_2. \tag{3}$$

(iv) A surface having two intervals with different easy directions can be characterized [18] by a mean orientation with tilt angle given by

$$\Theta_e = [a\alpha_1 + (\lambda - a)\alpha_2]/\lambda. \tag{4}$$

Taking into account all the previous hypotheses, the tilt angle dependence on temperature is given by

$$\Theta_e = G_0 + G_1 \cdot \gamma, \tag{5}$$

where

$$G_0 = [a\theta_1 + (\lambda - a)\theta_2]/\lambda, \tag{6}$$

and

$$G_1 = (2a - \lambda)/\lambda. \tag{7}$$

G_0 and G_1 are coefficients that depend only on the structure which was obtained by SiO deposition, and γ contains the temperature dependence. The results of measurements made on the samples presented in figures 2 and 3, as well as for other samples, were fitted according to equation (5). For the whole set of measured samples, the parameters T_1 and A were fixed, while G_0, G_1 and T_2 were allowed to vary so that the best fit was obtained for each sample. The parameter values T_1 and A were chosen so as to assure the best fit for the measurements for the whole set of samples. Their values are $T_1 = 77{\cdot}8°C$ and $A = 1{\cdot}57$. The results for the fit parameters for the two samples of figures 2 and 3 are given below.

For the sample of figure 2 (SiO deposition at $61 \pm 1°$, surface temperature transition equal to $(75{\cdot}8 \pm 0{\cdot}1)°C$), the fitting parameters are: $G_0 = 2{\cdot}5°$, $G_1 = 0{\cdot}22$ and $T_2 = 75{\cdot}3°C$. Taking into account that $\theta_2 = 61°$ and using equations (6) and (7) we obtain $\theta_1 = -35°$.

For the sample of figure 3 (SiO deposition at $67 \pm 1°$, surface transition temperature equal to $75{\cdot}8 \pm 0{\cdot}1°C$), the fitting parameters are: $G_0 = -2{\cdot}8°$, $G_1 = 0{\cdot}243$ and $T_2 = 75{\cdot}22°C$. As $\theta_2 = 67°$, in the same manner as above, we obtain $\theta_1 = -46°$.

We remark that the value of T_2 obtained by the fitting is near to the surface transition temperature for the whole set of samples. On the other hand the temperature T_1 is near to the clearing point of the nematic used for the samples.

6. Conclusions

Surface transitions in nematic liquid crystals induced by temperature have been experimentally detected. By using a nematic mixture containing a surfactant giving a homeotropic alignment, we observed that the topography of the surface has an important influence on the temperature trend of the tilt angle. The experimental results have been interpreted by applying a modified version of the model first proposed by Sluckin and Poniewierski, and taking into account the periodic distribution of easy axes. The agreement between experimental and theoretical predictions is fairly good.

We are indebted to S. Faetti for the experimental determination of birefrigence *versus* temperature, to G. Durand for useful discussions on the influence of the topography on the macroscopic alignment, to A. Stepanescu for SiO evaporation, and to E. Miraldi for the realization of the colour plates. This work has partially been supported by MURST (Italy) and by Unione Industriale di Cuneo.

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