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Measurement of the viscosity of ferroelectric liquid crystals in free-standing films

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A new implementation of the technique for the measurement of the Goldstone mode rotational viscosity in the S_C^* phase is presented. An electric field is applied parallel to the surface of a free-standing liquid crystal film. The optical transmission change of the film is recorded. The viscosity can be calculated from the reorientation time between optically separated positions of the director. A comparison with the viscosity values measured in thin cells is given. The values determined in cells are always higher than the results obtained from free-standing films. This indicates the great influence of the forces of interaction between the liquid crystal molecules and the surface layer in the cells.

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1. Introduction

Much attention has been devoted to ferroelectric liquid crystals (FLCs) from the standpoint of applications. The Goldstone-mode rotational viscosity η, which is related to rotations along the smectic cone, is one of the most important physical parameters of the smectic C* phase and strongly influences the switching time between the field-induced states of the FLC. Some progress has been made in developing reasonably simple methods for viscosity determinations. Such methods include the electro-optical response, the analysis of the polarization reversal current, and the Sawyer–Tower method [1–4]. A comparison of these methods shows a fairly good agreement of the viscosity values obtained and of the temperature dependence [2]. These measurements are usually performed using thin samples of the SSFLC (surface stabilized FLC) configuration. In the interpretation of the experiments, a perfect bookshelf structure in the cells is assumed: the smectic layers are perpendicular to the cell boundaries, the director is parallel to the solid surfaces, and the helix is unwound by surface action. For the viscosity measurement, an electric field is applied perpendicular to the cell boundaries. If the field reversal occurs abruptly with a sufficient amplitude, domain formation during the switching process is neglected, and it is assumed that the directors move cooperatively on the cone with a constant tilt angle [5]. The equation of motion of the director on the smectic cone is expressed by [1]

$$\eta \frac{d\phi}{dt} = \mathbf{P} \cdot \mathbf{E} \sin\phi + \varepsilon_0 \Delta\varepsilon \mathbf{E}^2 \sin^2\theta \sin\phi \cos\phi, \qquad (1)$$

where ϕ = the azimuthal angle (see figure 1), θ = the cone angle, $\Delta\varepsilon$ = the dielectric anisotropy, $\mathbf{P}$ = the spontaneous polarization of the FLC, and $\mathbf{E}$ = the strength of the applied electric field.

This yields [1]

$$\frac{t}{\tau} = \frac{1}{1-\alpha^2}\left\{\ln\frac{\tan(\phi/2)}{\tan(\phi_0/2)} + \alpha \ln\left[\frac{(1+\alpha\cos\phi)\sin\phi_0}{(1+\alpha\cos\phi_0)\sin\phi}\right]\right\}, \qquad (2)$$

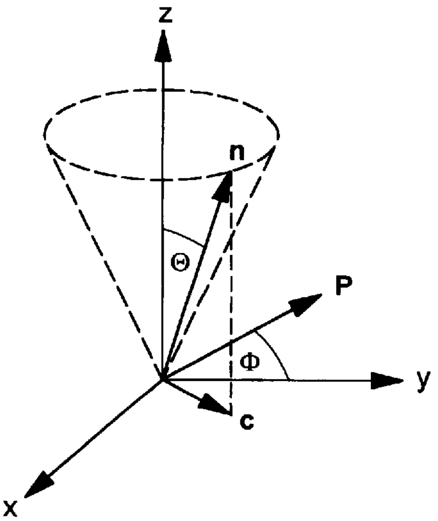

Figure 1. Coordinate system for director motion. The smectic layers define the *xy* plane; the director **n** is tilted from the layer normal (cone angle θ); the vector **c** is the projection of **n** onto the layer plane; the polarization **P** is in the layer (*xy*) plane and normal to **n**; the electric field **E** is applied parallel to the *xy* plane.

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where

$$\tau \equiv \eta/(\mathbf{PE}), \quad \phi_0 \equiv \phi(t=0), \quad \text{and} \quad \alpha \equiv (\varepsilon_0 \Delta\varepsilon \mathbf{E} \sin^2\theta)/\mathbf{P}.$$

The value α characterizes the influence of the dielectric anisotropy of the FLC on the switching process. One can measure the optical response time t after the field reversal and evaluate the viscosity from the formula [5]

$$\eta = \frac{1}{1\cdot75} t_{10-90} \mathbf{P} \cdot \mathbf{E}, \qquad (3)$$

where the time t is measured from 10% to 90% transmission for crossed polarisers.

In the current response method, t_w is the half height width of the current peak. The rotational viscosity is usually calculated by means of a relation that is similar to equation (3) [5]

$$\eta = \frac{1}{1\cdot76} t_w \mathbf{P} \cdot \mathbf{E}. \qquad (4)$$

The deduction of viscosity values from response time measurements is influenced by several factors, e.g. by deviations from the perfect bookshelf structure. The smectic layers are often tilted or have a chevron structure with zig-zag defects. Depending on the degree of distortions in the cells very different response times can be measured for the same FLC substance [2]. The relations (3) and (4) are only valid for the bookshelf structure, which is rarely achieved in real FLC cells. Moreover, in thin cells with a structure unwound by surface action, one would expect strong boundary influences on the switching process [6, 7] and, therefore, on the measured values of the rotational viscosity. Because of these uncertainties, dielectric methods for the determination of η in thick samples have been developed [8, 9].

In this paper we present a new implementation of the technique for the measurement of η using free-standing liquid crystal films [10]. After the description of the experimental set-up, we will give an analysis of the optical response curves. The values of η calculated in this way will be compared with the results derived by two methods using thin cells with various orientational layers.

2. Experimental

The experimental set-up for the investigation of the free-standing film is given in figure 2. A thin FLC film is drawn on a conical hole in a non-conducting substrate. The thickness h of the film studied is between 400 and 600 nm (about 150–200 smectic layers), and the diameter of the free-standing part is about 1 mm. Electrodes on the substrate make it possible to apply an electric field parallel to the FLC film: this can be either a square wave field between one pair of electrodes (1/2 or 3/4) or a rotating field by using all four electrodes [11]. For the second case, the impulse diagram for one electrode is drawn in figure 3. The sequence for the opposite electrode starts with the negative value $-V_A$, and at the two other electrodes the cycle begins with the field-free state. The amplitude V_A is adjusted between 20 and 150 V (voltage between the electrodes $V = 2V_A$). The impulse length is selected in the range 50 ms–100 µs. The distance d between a pair of electrodes is 1·2 mm. Therefore, $\mathbf{E} \equiv V/d$ will be smaller than $3\,\mathrm{kV\,cm^{-1}}$. The FLC film takes up the whole area between the electrodes. The optical measurements are performed in a spot (diameter 0·1 mm) which is adjusted to lie at the centre of the film. For this experimental set-up, a homogeneous field distribution is assumed in the film area investigated.

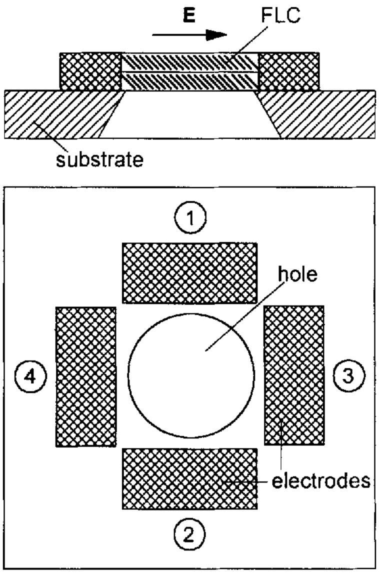

Figure 2. Experimental set-up; the FLC which covers the whole area between the electrodes is omitted in the lower drawing.

The free-standing film is placed on a hot stage between crossed polarisers and illuminated normally with white light. Homogeneous films without layer-steps or disclinations are used in the investigations. A heating/cooling cycle is useful to obtain these homogeneous films. In free-standing films, the smectic layers are arranged parallel to the surfaces of the film [12, 13]. The molecules

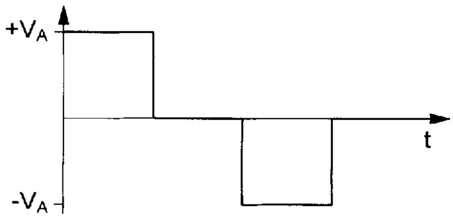

Figure 3. Impulse sequence applied to one of the four electrodes.

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can rotate along the smectic cone whose axis is perpendicular to the film. For the FLCs used in our experiments, the pitch p_0 of the helical structure was in the range between 2·3 µm and about 10 µm ($h < p_0$). In the case ($h \gg p_0$), the helix is unwound above a critical field strength $\mathbf{E}_c$ [14].

$$\mathbf{E}_c = \frac{4\pi^2 \sin^2 \theta K}{\mathbf{P} p_0^2}. \qquad (5)$$

For practical values of the torsional elastic constant K (10^{-12} N [8, 9, 14]), $\mathbf{E}_c$ is of the order 100 V cm^{-1}.

For the case $h < p_0$ the critical field strength is lower than the value given by equation (5) [15]. In our investigations the strength of the applied field was always higher than the critical value to ensure an unwound structure.

A change of field direction switches the director into a new position on the cone. This is visible in the conoscopic picture of a free-standing film by a jump of the optical axis of the liquid crystalline system into a new direction. Conoscopic investigations also provide a means of measuring the cone angle θ. The optical transmission change of the free-standing films induced by the field switching is monitored by a photomultiplier and stored by a digital oscilloscope. The data are transferred to a computer for further analysis.

In principle, only one pair of electrodes is necessary for the switching of the FLC in an alternating field and the study of the viscosity. However, for some FLC substances, the corresponding 180° rotation of the director on the cone is accompanied by an inhomogeneous domain switching of the film, because of the possibility of director rotation in a clockwise *or* in an anti-clockwise direction [16, 17]. Here, the 90° shift in the rotating field is useful to achieve a cooperative switching in the whole area of the film.

On the other hand, the rotating electric field can induce a spontaneous formation of stable ring-shaped domains in FLCs [11, 17–19]. In this case, the 180° rotation of the director in an alternating field can be used to obtain cooperative switching without ring-shaped domains. Dependent on special experimental conditions, one may choose one of these two experimental methods.

We also studied the rotational viscosity in SSFLC cells to compare the results obtained from the free-standing films with those obtained using well-established techniques. The cells were composed of glass plates with a transparent conducting layer and an orientational layer. Different orientational layers (thickness about 20 nm) were used: obliquely deposited SiO$_x$, or spin coated and rubbed layers of polyvinyl alcohol (PVA) or of polyimide (PI). The thickness of the cells (1·5–2 µm, adjusted by spacers) was determined by an optical interference method. After filling the cells with the FLCs in the isotropic phase, they were slowly cooled in a temperature gradient to obtain a homogeneous structure without any visible defects.

Table 1. Ferroelectric mixtures used and their spontaneous polarization P, dielectric anisotropy $\Delta\varepsilon$, and cone angle θ (at 24°C).

FLC mixture	S$_C^*$ phase/°C	P/nC cm^{-2}	$\Delta\varepsilon$	θ°
15/2 (MLU Halle)	16–31	34·7	−1·4	13
FE1 (MLU Halle)	7–43	11·3	5·0	12
SCE12 (Merck Ltd)	< −20–66	16·3	−1·1	20
FELIX-001 (Hoechst)	−7–79	10·5	−0·8	32

In the thin cells, the rotational viscosity was measured using two different techniques: from the half height width of the polarization reversal current peak t_w [2] and from the optical response time $t_{10\text{–}90}$ [1], both using square wave application. The field strength was always sufficient to ensure switching into saturation states ($t \propto 1/\mathbf{E}$). In the optical measurements, the transmission parallel to the smectic layers was studied, contrary to the case of free-standing films, where the optical properties perpendicular to the layers were recorded. The optical response of the cells was registered in the same manner as the transmission change of the free-standing film. All measurements were repeated twice under the same conditions and then repeated with other films/cells of the same material to check the reproducibility and the scatter of the data.

The FLC mixtures investigated are given in table 1. The spontaneous polarization of mixtures 15/2 and FE1 was evaluated by means of the polarization reversal current (triangular wave method, frequency 100 Hz, $\mathbf{E} =$ 100 kV cm^{-1}) [20]. The tilt angle was measured from the conoscopic picture of a free-standing film, and $\Delta\varepsilon$ values are from [21]. The physical parameters of the mixtures SCE12 and FELIX-001 were taken from data sheets.

3. Optical response of the free-standing film

In the investigation of free-standing films, the first polarizer is arranged parallel or perpendicular to the direction of the electric field. If the vector **c**, the projection of the director **n** onto the smectic layer plane (see figure 1), is in the same direction, one observes extinction of transmitted light. The variation of the light intensity I during the motion of the director on the smectic cone is given by

$$I = I_0 \sin^2 2\phi \sin^2\left(\frac{\pi d \Delta n}{\lambda}\right), \qquad (6)$$

where Δn is the effective birefringence and λ is the wavelength. A maximum transmission is obtained after a 45° rotation of **c**. Further director motion decreases I down to extinction at 90°. This transmission change is repeated in the following quadrants. The measured optical response to an alternating field (figure 4) corresponds to this described behaviour. I passes through two maxima followed by extinction. A delay between the field reversal and the beginning of the optical change could not be observed.

Equation (2) provides a relation between the time t and the azimuthal angle ϕ, the angle between the spontaneous polarization and the field direction. This equation shows a divergence at $\phi_0 = 0°$. After a time t_1 the angle between the polarization and the field is ϕ_1, and after t_2 the angle is ϕ_2, or

$$t_{1,2} = \frac{\tau}{1-\alpha^2}\left\{\ln\frac{\tan(\phi_{1,2}/2)}{\tan(\phi_0/2)} + \alpha \ln\left[\frac{(1+\alpha\cos\phi_{1,2})\sin\phi_0}{(1+\alpha\cos\phi_0)\sin\phi_{1,2}}\right]\right\}. \qquad (7)$$

This yields the difference $t_2 - t_1$

$$t_2 - t_1 = \Delta t = \frac{\tau}{1-\alpha^2}\left\{\ln\frac{\tan(\phi_2/2)}{\tan(\phi_1/2)} + \alpha \ln\left[\frac{(1+\alpha\cos\phi_2)\sin\phi_1}{(1+\alpha\cos\phi_1)\sin\phi_2}\right]\right\}. \qquad (8)$$

Thus, the divergence in (2) is removed by the introduction of a time interval. For 180° switching of the field, the angle ϕ increases from 0° to 180°. Strictly speaking, the vector **c** differs by 90° from the spontaneous polarization. However, the characteristic behaviour of the optical response repeats in each quadrant. Therefore, the angles $\phi_1 = 45°$ and $\phi_2 = 135°$ correspond to the maxima in the transmission curve. By measuring the time Δt between these two maxima it is possible to calculate the viscosity by

$$\Delta t = \frac{\tau}{1-\alpha^2}\left[1{\cdot}76 + \alpha \ln\left(\frac{1-0{\cdot}71\alpha}{1+0{\cdot}71\alpha}\right)\right]. \qquad (9)$$

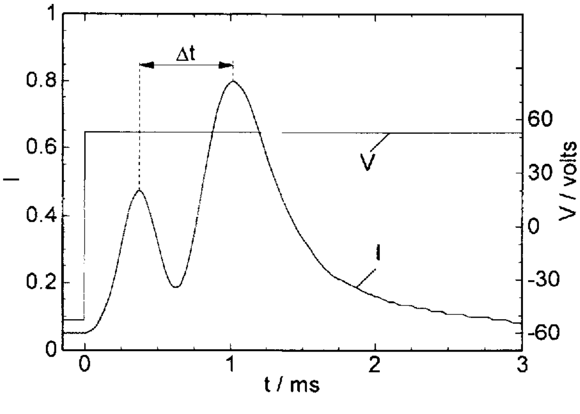

Figure 4. Change of transmitted intensity I at field reversal of the applied alternating field (mixture 15/2).

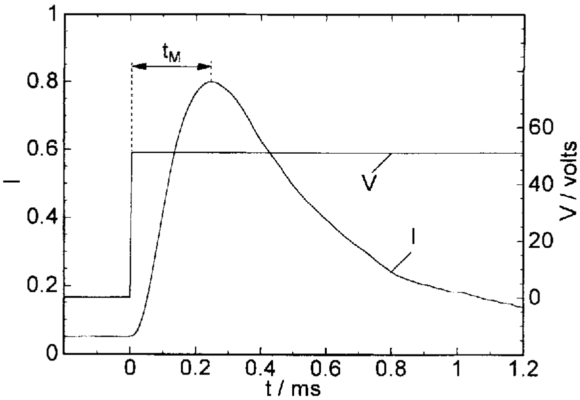

Figure 5. Change of transmitted intensity I when switching of the direction of an applied rotating field occurs (mixture 15/2).

The optical response to the rotating field is given in figure 5. A delay between the field switching and the beginning of the optical change could also not be observed. The vector **c** rotates by 90°, and a single transmission maximum appears after 45°. The initial azimuthal angle is $\phi_0 = 90°$. According to equation (2), the maximum of transmission ($\phi = 135°$) is achieved after

$$t_M = \frac{\tau}{1-\alpha^2}\left[0{\cdot}88 + \alpha \ln\left(\frac{1-0{\cdot}71\alpha}{0{\cdot}71}\right)\right]. \qquad (10)$$

The viscosity can be calculated from t_M, that is the time between the switching of the field into the new direction and the maximum intensity. The time of the whole field-induced reorientation (90° turn of **c**) can be evaluated only experimentally. In our investigations it was about $6t_M$.

4. Results and discussion

The optical response parallel to the axis of the smectic cone (figures 4 and 5) supports the picture of the director motion along the cone if an electric field is applied parallel to the smectic layers. In general, this motion is influenced by the ferroelectric, as well as by the dielectric term in equation (1). In our experiments, α is of the order of 10^{-3} or smaller. First of all, this is caused by the low field strength. So, the second terms in (9) and

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(10) can be neglected, and $(1-\alpha^2)\simeq 1$. This yields

$$\eta = \frac{1}{1{\cdot}76}\Delta t\mathbf{P}\cdot\mathbf{E} \qquad (11)$$

or

$$\eta = \frac{1}{0{\cdot}88}t_{\mathrm{M}}\mathbf{P}\cdot\mathbf{E}. \qquad (12)$$

Both relations are independent of the dielectric anisotropy.

The results for the different FLC-mixtures are given in table 2. They are independent of the field strength varied between 0·4 and 3·0 kV cm^{-1}, therefore $t\propto 1/\mathbf{E}$. In the investigation of SSFLC cells, this proportionality between switching time and $1/\mathbf{E}$ indicates that after switching, the director can attain a stable position and the measurement of η is carried out correctly. In the free-standing film, the reorientation of the director into a new, stable state is also directly visible by the constant extinction after the switching. A possible influence of conductivity of the FLC on the switching process has been disregarded here because, for values of conductivity usually measured for LCs ($10^{-9}\,\Omega^{-1}\,\mathrm{cm}^{-1}$), the relaxation time of charges is lower than the impulse length of the applied field only in the low frequency region ($\leqslant$200 Hz) [22]. In our experiments a dependence of the switching time on the impulse length was not observed.

We determined the rotational viscosity of the mixture FE1 by equation (11) as well as by equation (12) and got 33·8 mPa s and 34·6 mPa s, respectively. This difference is within the scatter of the experimental results (up to ±4%). In table 2 the mean value is given. The viscosity of the other mixtures was determined by either (11) or (12). The η values are independent of the film thickness, at least in the range investigated by us. In free-standing films with networks of disclinations or other disturbances of the homogeneous order, we measured essentially longer switching times (up to a factor of 4). The use of these switching times in (11) or (12) would give too high viscosity values.

The method developed here was also used to investigate the temperature dependence of the rotational viscosity. The results for the mixtures 15/2 and FE1 are presented in figure 6. The Arrhenius plot $\ln(\eta)$ versus $1/T$ shows straight lines far away from the $\mathrm{S_C^*}$–$\mathrm{S_A}$ transition. Thus, the temperature dependence of η obeys the Arrhenius law fairly well. The values of the activation energy ($\mu=0{\cdot}95$ eV for 15/2 and $\mu=0{\cdot}61$ eV for FE1) evaluated are of the order of the values obtained for other FLC compounds by other methods [2, 8, 23]. Close to the phase transition we observed the known deviation from the Arrhenius law [2, 8].

Table 2. Rotational viscosity η (in mPa s) at 24°C measured using free-standing films and thin cells with different orientational layers.

FLC mixture	Film	SiO$_x$	PVA	PI
15/2	89	118	107	126
FE1	34	47	42	43
SCE12	166	263	246	232
FELIX-001	49	77	68	69

The switching process in thin cells is connected with a delay between the field reversal and the beginning of the optical change, contrary to the behaviour of free-standing films. However, neither method used by us (polarization reversal current and optical response) requires measurement of this delay time. The applied fields were higher than 20 kV cm^{-1} to ensure that the director can attain a saturation position ($t\propto 1/\mathbf{E}$). The η values derived by these two techniques, and the relations (3) and (4), are in good agreement with each other. The response times were often not symmetrical during the positive and the negative pulses of the applied voltage. This may be attributed to the existence of a pretilt angle favouring one of the bistable states due to its reduced rotation on the helical cone [24]. The mean values obtained from these measurements are shown in table 2. We found a non-systematic experimental error for η of up to ±5%.

Obviously, the viscosity values derived in the free-standing films are smaller than those from the thin cells by 20–30%. A faster response of the free-standing film in comparison with cells was also observed in [25]. Additionally, the data in table 2 give rise to the expectation that the values measured for cells depend on the surface orientational layers used. This is in accordance with the findings in [6]. Unfortunately, this effect is of the same order as the experimental error. It was noted that the effective cone angle, as well as the twisted

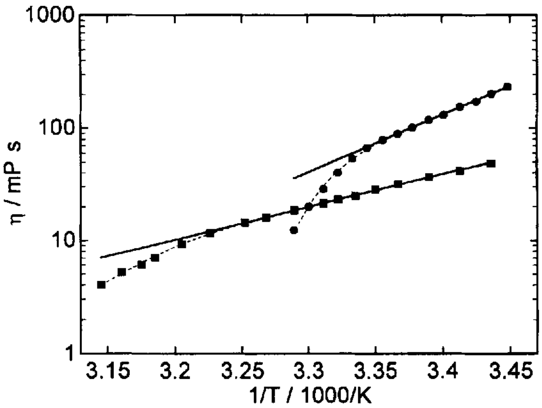

Figure 6. Dependence of the rotational viscosity η of the mixtures 15/2 (●) and FE1 (■) on temperature T.

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structure depend both on the surface orientational layer and on the liquid crystal [26]. Therefore, one may assume that the η values measured for thin cells depend also on the particular combination of FLC mixture and orientational layer. However, if one takes into account that the viscosity values determined for the free-standing films are in principle lower, our results are on the whole in good agreement with the η values given in the data sheets for SCE12 and FELIX-001: 230 mPa s and 64 mPa s, respectively.

Finally, some remarks on the differences between the η values measured for the SSFLC cells and for the film should be given. The equation of motion (1) was derived without consideration of any surface term. This situation is not likely to be found in real cells where the helical structure of the FLC is unwound by strong surface interactions. The molecules near the surface have also to take part in the switching to achieve the situation that most of the LC layer is reoriented in the field as a uniform slab. The switching should be influenced by the nature and strength of interaction between the FLC molecules near the surface and the orientational layer. Because of this interaction, switching near the surface of the sample will probably be slower than that in the bulk, where a relatively free motion on the smectic cone seems possible. For the measurement made using thin cells, an integrated value results.

In the films, the only interaction of the free-standing part with the solid substrate takes place at the edge of the hole. The measurements are however performed in the centre of the free-standing film, far away from the substrate. The only remaining interfacial force in the film centre is the surface tension. This can induce a defined molecular orientation at the free surface with a fixed tilt, but an arbitrary azimuthal angle [27]. Thus, one may suppose that the surface tension does not influence the motion of the director along the cone in any essential way. Therefore, a field-induced reorientation is not hindered at the free surface.

The lower viscosity values always determined with the free-standing films could be a manifestation of the influence of the surface conditions. Our investigations have shown that the influence of the surface may not be neglected in the measurements with the cells. This requires the introduction of an additional term F_{surf} in the equation of motion (1). This surface term is then responsible for the deviations between thin cells with different orientational layers. In homogeneous free-standing films, F_{surf} can probably be ignored. If there are inhomogeneities in the film, these regions probably act similarly to surfaces and hinder the director motion. The increase of response time in free-standing films with a network of disclinations suggests such an interpretation. But, the higher viscosity values obtained from thin cells can also be a result of the inhomogeneous director distribution in real SSFLC cells. It was shown theoretically that an inhomogeneous director field gives higher values of viscosity than a uniform slab [28]. In general, the switching processes in SSFLC cells are not in accordance with the model of a switching, uniform slab. The behaviour of homogeneous free-standing films corresponds better to this model and to its theoretical description.

Preliminary experiments on thin FLC films drawn onto a glass substrate (one free surface) and investigated by the four-electrode equipment have yielded viscosity values that are smaller than those in the case of SSFLC cells and higher than those for free-standing films. This behaviour is in agreement with the influence of solid substrates on the switching process discussed above for thin liquid crystal samples.

In conclusion, we have presented an alternative method for determining the Goldstone mode rotational viscosity in the S_C^* phase based on optical measurements and performed on free-standing films. The derived values agree quite well with the results obtained by other methods. However, they are lower because the measurements were performed on a system that is not influenced by solid surfaces. The dependence on temperature and the activation energy are also similar to the findings of other authors. The drawing of free-standing films is much easier and less time consuming than the preparation of thin cells. The method has been used by us for the measurement of the rotational viscosity of numerous FLCs. However, in this paper the results for only four FLC mixtures are presented, because the corresponding investigations using thin cells were performed only for these mixtures.

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