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A field-reversal method for measuring the parameters of a ferroelectric liquid crystal

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A method for measuring the spontaneous polarization $\mathbf{P}_s$, the switching time, the rotational viscosity γ_φ, and the d.c. conductivity σ is presented. The possibilities of estimating the azimuthal angle φ_0, the dielectric anisotropy $\Delta\varepsilon$ and the dielectric permittivity $\varepsilon_\perp$ in the same experiment are also discussed. It is explicitly shown that the switching delay, though primarily dependent on the material and the applied field, is also dependent on the geometry of the cell.

1. Introduction

A continuing interest in ferroelectric liquid crystals (FLCs), determined by their application to a new generation of electro-optical devices, and the continuing discovery of new, related phenomena, requires a continuing development in measuring techniques and a critical analysis of the existing methods for investigating FLC characteristics. In this paper we present a method which gives most of the important parameters of a FLC in a single experiment.

Among the most important parameters of FLCs are the spontaneous polarization $\mathbf{P}_s$, the rotational viscosity γ_φ, the d.c. conductivity σ, and the dielectric anisotropy $\Delta\varepsilon$. Methods for measurements of $\mathbf{P}_s$ can be divided in two main classes: methods based on the pyroelectric technique [1] and the field-reversal methods [2–4]. Whilst the pyroelectric techniques yield measurements of very low values of the spontaneous polarization with zero external field applied to a LC cell, only the second group of methods allows for the measurement of several parameters of the liquid crystal cell in a single experiment.

2. Experimental

2.1. *The experiment*

Field-reversal techniques can be classified by the shape of the waveform applied to a LC cell. Whilst application of a triangular wave is wide spread due to the simplicity of the method, it has been shown that the square wave technique provides more rigorous measurements [4]. Using developments in testing and measuring equipment, it has also become possible to obtain more information about a liquid crystal using the square wave method.

The set-up given here is a further development of the field-reversal technique [2]. The set-up scheme and the cell geometry are shown in figure 1. A square wave voltage (with magnitude U_{in} up to 200 $\text{V}_{\text{zero-to-peak}}$) is applied to a LC cell. The current i flowing through the cell is integrated by the capacitor C ($RC \sim 10/f_{\text{gen}}$; $f_{\text{gen}} \sim \leqslant 10(t_0 + \tau)$, where t_0 and τ will be defined in § 3); then the voltage across the capacitor is digitized by an oscilloscope (Hewlett Packard 54504A) and the data are stored in the computer for further treatment by a simple program to obtain the various parameters under investigation. The instantaneous value of the voltage over the capacitor C can be presented as [2]:

$$U_{\text{out}} = \frac{1}{\text{C}}\int_{-0}^{t} i\,\mathrm{d}t = \frac{U_{\text{in}}}{RC}t + \frac{2U_{\text{in}}C_{\text{LCC}}}{C} + \frac{\mathbf{P}_{\text{S}}A}{C}\cos(\varphi(t))\big|_{-0}^{t} \equiv U_R + U_C + U_{\mathbf{P}}$$

where $t = 0$ represents the instant of reversing the sign of the generator voltage, R is the equivalent ohmic resistance of the cell (assumed to be a constant in the time scale considered), $C_{\text{LCC}} = (\varepsilon_0\varepsilon_\perp A)/d$ is the capacitance of the cell, $\varepsilon_0 = 8.85 \times 10^{-12}$ F m^{-1}, and $\varepsilon_\perp$ is the dielectric permittivity of the liquid crystal in our geometry. A and d are the area and the thickness of the cell, respectively. Here U_{out} is given as a sum of three components: U_R corresponds to the current through the resistive part of the cell, U_C is due to the recharging of the cell capacitance and U_P is the voltage across C due to the repolarization current in the cell. A typical signal and the definition of the measured parameters are given in figure 2.

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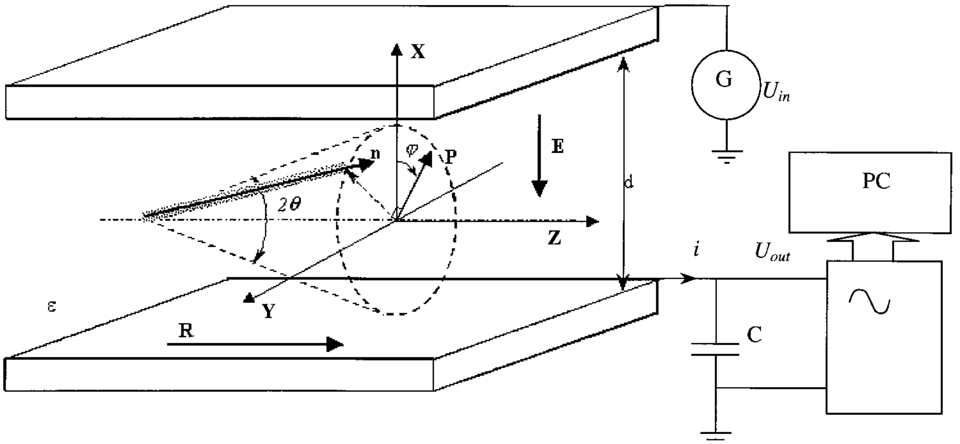


Figure 1. FLC cell in the bookshelf geometry, coordinate system and schematic diagram of the set-up. **R**-rubbing direction, *n*-molecular director, **E**-electric field vector, **P**-polarization vector, φ-azimuthal angle, θ-molecular tilt angle The smectic layers are parallel to the **XY** plane.

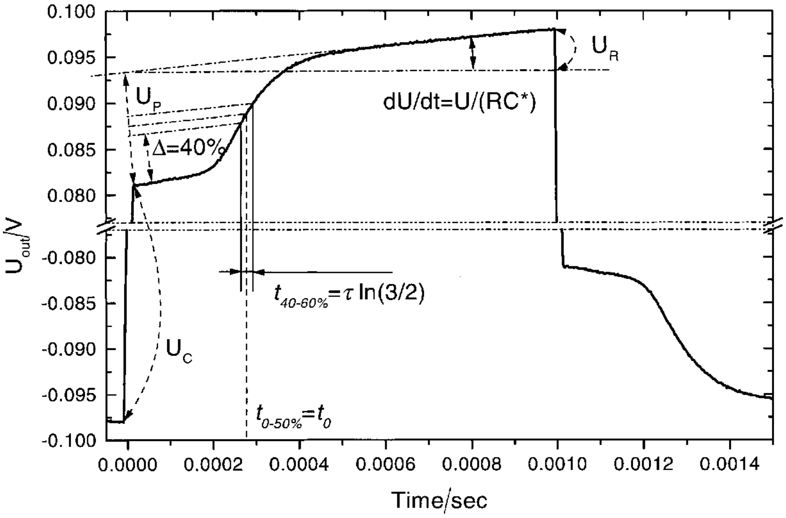
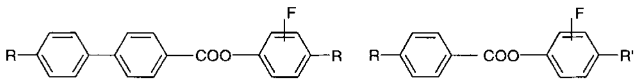


Figure 2. The measured signal and the parameters under investigation. SCE8, $T = 39$°C. U_P, $t_{40\text{-}60\%}$ and $t_{0\text{-}50\%}$ have been determined after subtracting the resistance contribution.

2.2. *The material*

In order to test the technique, a common commercial material was selected. The LC used was a two-component commercial mixture, SCE8, supplied by Merck Ltd. (UK). The chemical formulae of its components are given below.



SCE8 has a relatively small spontaneous polarization $\mathbf{P}_s$ and a large birefringence Δn (~0.16). The phase transition temperatures are:

SmC*–59°C–SmA–79°C–N*–100°C–I

All measurements were performed using conventional home-made LC cells. Glass plates with ITO electrodes were spin-coated with polyvinyl alcohol solution (1% by weight concentration), then baked for 1 hour at 140°C and rubbed in one direction on a velvet track in order to achieve a homogeneously planar orientation. Dimensions of the electrodes were 6.5 and 4.5 mm (with an area $A = 29.3\,\text{mm}^2$), and the cell thickness calculated from the measured capacitance of the empty cell was $d \cong 11\,\mu\text{m}$. The LC mixture was introduced into the cell by capillary action in the isotropic phase. A uniform bookshelf structure was obtained using treatment by an a.c. electric field ($20\,\text{V}\,\mu\text{m}^{-1}$, $f \sim 100\,\text{Hz}$) in the SmC* phase and this structure was confirmed using a polarizing microscope.

3. Theory

For planar geometry, the equation for the director motion is:

$$\gamma_\varphi \frac{\mathrm{d}\varphi}{\mathrm{d}t} = \pm \mathbf{P}_S \mathbf{E} \sin\varphi + \varepsilon_0 \Delta\varepsilon \mathbf{E}^2 \sin^2\theta \sin\varphi \cos\varphi \quad (1)$$

where θ is the angle of the molecular tilt in the FLC layer and $\mathbf{E}$ is the amplitude of the electric field in the cell. For the electric field $\mathbf{E}$ directed as shown in figure 1 (*a*), a positive sign of the first term should be selected. Here, by neglecting any contribution from the moment of inertia and the surface interactions and assuming a simultaneous conical rotation of the molecules in the smectic layer with cone apex 2θ, the solution of this equation is [5]:

$$\frac{1}{\tau}=\frac{1}{1-\alpha^2}\left[\ln\frac{\tan(\varphi/2)}{\tan(\varphi_0/2)}+\alpha\ln\frac{(1+\alpha\cos\varphi)\sin\varphi_0}{(1+\alpha\cos\varphi_0)\sin\varphi}\right] \tag{2}$$

where $\tau=\gamma_\varphi/\mathbf{P}_\mathrm{S}\mathbf{E}$ is the switching time, $\varphi_0=\varphi(t=0)$ is the initial azimuthal angle (assumed to be uniform over the sample thickness), and $\alpha=\varepsilon_0\Delta\varepsilon\mathbf{E}\sin^2\theta/\mathbf{P}_\mathrm{S}$.

The polarization part U_P of the voltage across the capacitor C is a function of the azimuthal angle $\varphi(t)$. For the case when the positive slope of the measured signal corresponds by definition to the switching $\varphi(t)$ from φ_0 [$\varphi_0\in(0;90°)$] to $180°-\varphi_0$, the voltage can be presented as:

$$U_p(t)=\frac{A\mathbf{P}_\mathrm{S}}{C}(\cos\varphi_0-\cos\varphi(t)). \tag{3}$$

.

Defining the normalized response $u(t)=-\cos(\varphi(t))$ and substituting $u(t)$, τ and α into equation (1) bring us to the more simple expression:

$$u'_t=\frac{1}{\tau}(1-u^2)(1-\alpha u). \tag{4}$$

By separating variables and integrating on both sides we obtain the solution:

$$\frac{t-t_0}{\tau}=\frac{1}{2(1-\alpha^2)}\ln\left[\frac{(1+u)(1-\alpha u)^{2\alpha}}{(1-u)(1-u^2)^{\alpha}}\right] \tag{5}$$

where the constant of integration $t_0\equiv t(\cos\varphi=0)$ is equal to the switching delay $t_{0\text{-}50\%}$ in our experiment, because the voltage applied is a symmetrical square wave.

For $\alpha=0$ and small φ_0 a simple expression for $U_P(t)$ can be easily obtained substituting $u(t)$ from equation (5) to (3):

$$U_P(t)=\frac{A\mathbf{P}_\mathrm{S}}{C}\left\{\frac{2\exp[2(t-t_0)/\tau]}{\exp[2(t-t_0)/\tau]+1}\right\},\qquad t_0=\tau\ln\left[\frac{1}{\tan(\varphi_0/2)}\right] \tag{6}$$

U_P, t_0 and τ can be obtained with a higher accuracy from the waveform after subtracting the resistance contribution (figure 2). In this case τ is unambiguously connected with the rise time [2] $t_{\Delta,1\text{-}\Delta}$, where Δ and $1\text{-}\Delta$ are the lower and upper thresholds used for the rise time measurements (see figure 2), respectively:

$$t_{\Delta,1\text{-}\Delta}=\tau\ln\left(\frac{1-\Delta}{\Delta}\right). \tag{7}$$

For example, if $\Delta=40\%$, $t_{40\text{-}60\%}=\tau\ln(3/2)$.

For the case of a large voltage, which is essential for obtaining a simultaneous conical molecular rotation, for forming the bookshelf structure and for suppressing surface interactions [6], the parameter α is essentially different from zero. Therefore the exact solution (5) of equation (4) should be used for the calculations.

Figure 3 represents the normalized response $u(t)$ as a function of time (scaled by $\tau=\gamma_\varphi/\mathbf{P}_\mathrm{S}\mathbf{E}$) for different values of α. From these results, we find that the error in the measurement of $\mathbf{P}_\mathrm{S}$ using equation (6) instead of (5) is practically negligible for α up to $+0.9$.

The rise time $t_{\Delta,1\text{-}\Delta}$ significantly depends on α only for $\Delta<30\%$. For $\Delta=40\%$ the switching time $t_{40\text{-}60\%}$ can be used in determining τ using equation (7) for any value of the parameter α [2]. By contrast, the parameter t_0 significantly depends not only on φ_0, but also on α. The dependence is presented in figure 4. For $\Delta\varepsilon>0$, the torque enforces the director to stay parallel to the field, but for $\Delta\varepsilon<0$ it stabilizes in the up and down states [6]. These phenomena speed up or slow down the start of the switching. The most significant contribution to switching delay is caused by small φ_0.

4. Results

The dependence of polarization on temperature for SCE8 is presented in figure 5. It shows a well known dependence of the polarization on temperature near the SmC*–SmA transition for a FLC. The results of measurements are practically the same during heating and cooling cycles. The dependence is also practically indistinguishable for different applied voltages (i.e. for various values of α). The results correspond to those provided by the manufacturer—$\mathbf{P}_\mathrm{S}$ ($T=30°\mathrm{C}$, $d=2$–$7\,\mu\mathrm{m}$, $f=50\,\mathrm{Hz}$, $U=30\,\mathrm{V}$) $=5.1\,\mathrm{nC\,cm^{-2}}$. Some disagreement is explained by inaccuracy in the measurements of the area of the cell and perhaps by small differences in the experimental conditions.

The other parameters, τ and t_0 are presented in figure 6. The relationship t_0/τ significantly decreases with temperature near the phase transition temperature.

Using $\mathbf{P}_\mathrm{S}$ and τ we can obtain the rotational viscosity of the liquid crystal using the formula $\gamma_\varphi=\tau\mathbf{P}_\mathrm{S}U_\mathrm{in}/d$ (figure 7). The rotational viscosity shows practically an exact exponential decrease with temperature far from the phase transition and sharply falls in the vicinity of the transition.

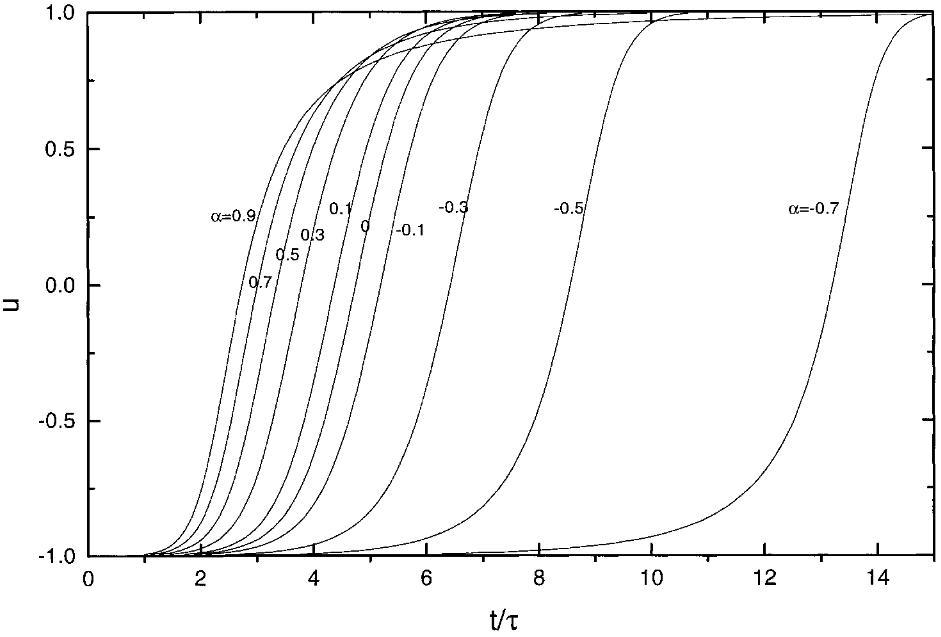


Figure 3. Normalized response u vs. scaled time t/τ for different values of α, $\varphi_0 = 1°$.

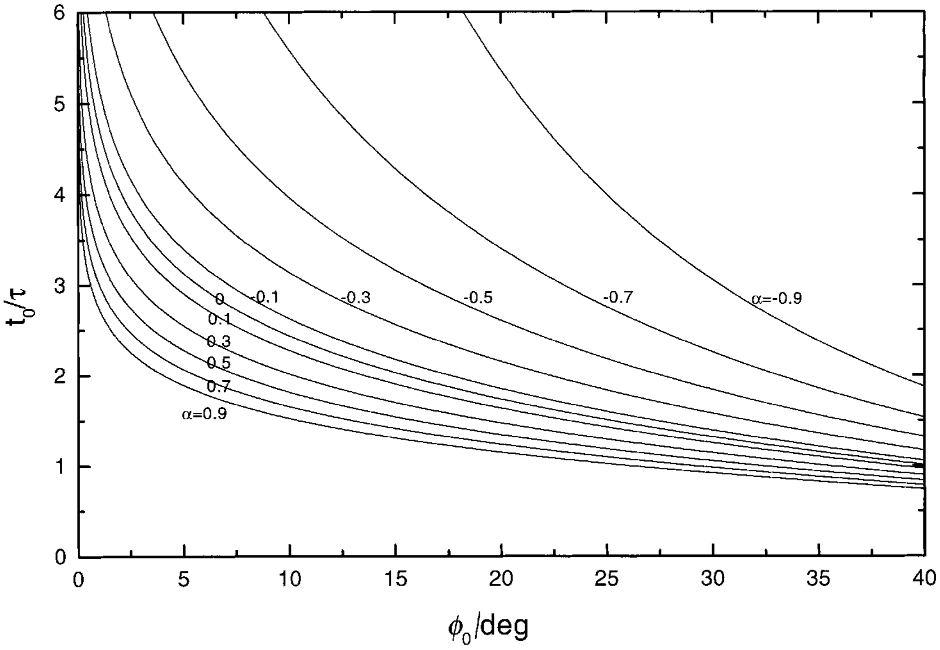


Figure 4. The switching delay as a function of φ_0 (azimuthal angle at $t = 0$) for different α.

Consideration of the contributions from d.c. conductivity is very important for all the previous measurements. In organic liquids like FLCs, the motion of ions determines the conductivity [7]. Fortunately, on the millisecond time scale in the case of polarity reversal of the applied field, change in the ion concentration is negligible and we can consider the cell resistance to be constant. In this case we can easily obtain the conductivity from the same data set by applying a linear regression to the corresponding part of the waveform and then subtracting a straight line from the measured signal (figure 2). While this method appears not to be very accurate for measuring the d.c. conductivity, it is reasonably practicable in order to make corrections to the previously discussed measurements. The d.c. conductivity σ, obtained in our experiment is presented in figure 7; the d.c. conductivity increases with increase in temperature as is found in most organic liquids. For the

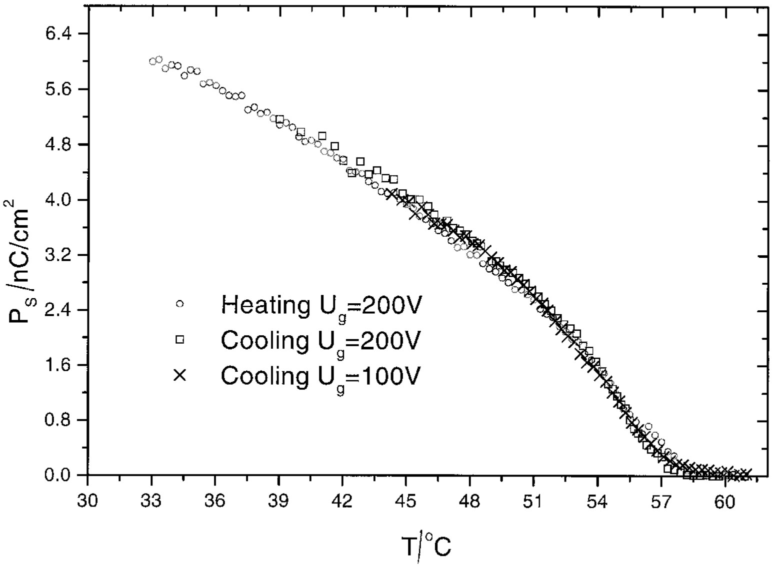


Figure 5. Temperature dependence of the spontaneous polarization.

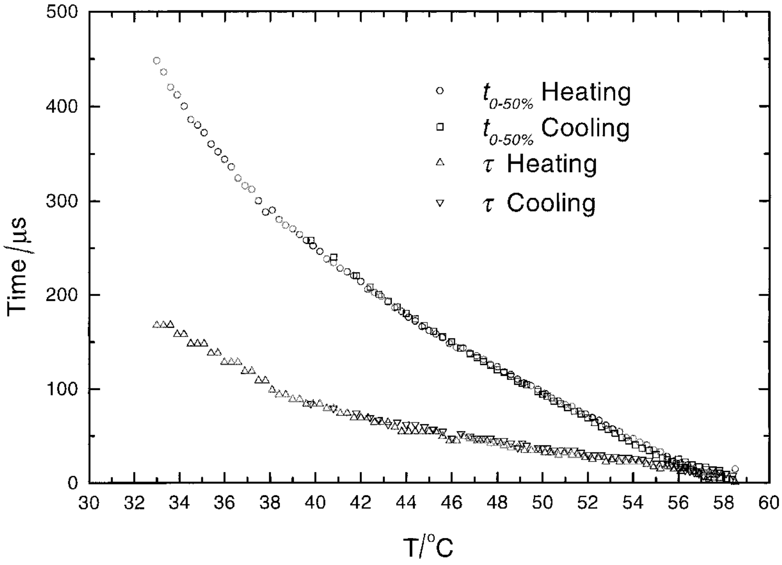


Figure 6. Temperature dependences of $t_{0\text{-}50\%}$ and τ. $U_G = 200$ V.

case of a large field and planar geometry, the d.c. conductivity is found to be almost insensitive to the SmA–SmC* phase transition.

5. Discussion

The parameters α and φ_0 can also possibly be estimated using this technique. The φ_0 ($\varphi(t=0)$) can be easily obtained from equation (2) (see figure 4), if α is known.

There are two main approaches under consideration for determining the dielectric anisotropy contribution α. The first is a direct fitting method using formula (5). Whilst it is the most rigorous method, it has a tendency to give ambiguous results, and the experimental error in this case is difficult to estimate. The second approach is the application of formula (4) directly to the waveform, using the digital differentiation of the signal. In spite of the fact that the differentiation introduces large random errors, caused by the noise in the signal, this method is useful if averaging is carried out over a large number of waveform points. In this case the experimental error is easy to control. For both these approaches, the correct determination of α requires maximal noise reduction during the experiment and perfect alignment of the liquid crystal.

The other capability of the technique is the measurement of the dielectric permittivity $\varepsilon_\perp = U_C C d/(2\varepsilon_0 A U)$. However for small values of the spontaneous polarization $U_P \ll U_C$ and this is a source of error in the measurements of polarization due to a limited dynamic range of the oscilloscope. For this reason simultaneous measurements of the dielectric permittivity and the

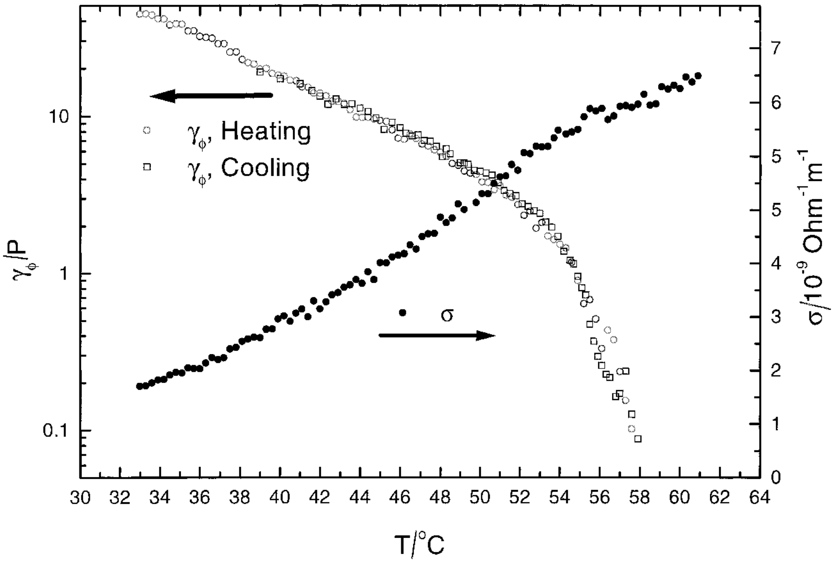


Figure 7. Temperature dependences of γ_φ and σ. Parameters of the experiment are the same as for figure 6.

spontaneous polarization can be used only for the case of large values of the spontaneous polarization, and moderate applied fields.

The measurements of the conductivity can provide ambiguous results because of the instability in the concentration of the ions in the cell, caused, for instance, by the effects caused by d.c. fields described in [8]. Therefore it is recommended that application of a d.c. field to the cell investigation is avoided. These phenomena have little effect on the polarization measurements because the time for ion redistribution sufficiently exceeds the switching time of the cell.

6. Conclusions

The influence of dielectric anisotropy on the measurements of the spontaneous polarization, switching time and switching delay have been investigated. The suggested form of the response equation (3, 4) appears to be useful for practical investigation of the LC parameters using the integral of the repolarization current. The simulation results show a practical independence of the measured value of the polarization and switching time on the parameter α. It has also been shown that if two of the three parameters t_0/τ, $\Delta\varepsilon$ and φ_0 are known, the third can be easily calculated.

It has also been shown that the switching delay time depends on φ_0. If $\varphi_0 = 0$ or 180°, the switching delay is inordinately long. Hence the switching delay, though primarily dependent on the material and the applied field, is also dependent on the geometry of the cell.

The procedure [2] provides correct measurements even for the case of a relatively large d.c. conductivity of the LC cell. In this case some mathematical treatment of the acquired data is required. The experimental results obtained using the developed algorithm are in a good agreement with those reported previously.

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