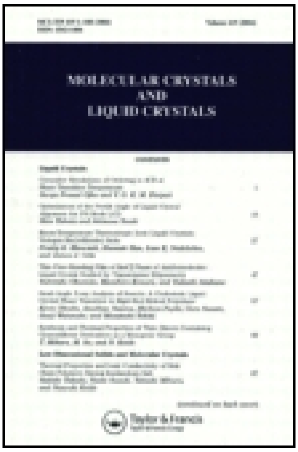

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Ionic Interface-effects in Electro-optical LC-cells

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Ionic Interface-effects in Electro-optical LC-cells

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Abstract The work discusses ionic electrode processes in LC-cells driven by voltage. Ions in LC-material are generated by dissociation of residual impurities or (dye) additives, but by ageing of LC-material as well. Therefore even in carefully purified electro-optical cells temporal decomposition of the base material causes slow, but permanent increase of ion concentration. It is apparently manifesting in non-linear electrical response of LC-cell in the case of quick switching by pulse driving. Every change of voltage on cell electrodes induces re-charging process and causes flow of the space-charge limited current across LC-layer. Existence of the ions influences electro-optical performance of LC-cell. The ionic flow changes field distribution in LC-layer and can temporary distort molecular ordering. Ionic processes may superimpose an undesired intrusion on optical switching response of LC-cell and may result either in faster or in slower switching, depending on driving source parameters. In this paper we propose a simple model of ionic processes to explain the mechanism of non-linear ionic polarisation. Observed voltage - polarisation characteristics, space-charge current flow and an anomalous electro-optical behaviour of LC-cell can be explained by the model assumed. Concentration of the considered ions, their mobility, and activation energy is also evaluated.

Keywords: liquid crystals; ionic transport; ionic polarisation; electrode interface

INTRODUCTION

Ionic processes in liquid crystals (LCs) have been studied very long time[1 -15]. Recently, ferroelectric phases of LCs are investigated more intensively[16 -20], since it became clear that the ions could influence the performance of widely used electro-optical devices: electro-optical characteristics, speed of switching, lifetime. The ions migration in LC-cells is responsible for image sticking phenomenon and faster degradation of LC-material. Special problem appears in ferroelectric SSFLC cells, where ions - from one side - can increase the required product $V \bullet \tau$ for switching and make it harder, but from the other side, ions field distribution can destabilise the state just switched. It is because ferroelecrtric depolarisation field, which in thin ferroelectric layers reaches high values[21] causing the ions to accumulate at the electrodes. The ions form compensating field and additionally stabilise actual state, but, because of slow ions transport, after switching to the opposite state those both fields summarise and destabilise the new state for a time much longer than structure switching time. Therefore good understanding the ionic processes in electro-optical LC-cells seems to be of great importance.

The source of ions in LC devices are not only residual impurities, which in fabrication process are maintained on relatively low level, but probably also decomposition of the basic LC-material during device operation and ageing processes. This statement points out, that in a LC-cell may exist several kinds of ions with different properties.

Although LCs are dielectric, they exhibit some specific ion effects not met in most other ordinary dielectric liquids. One of them is a specific ionic polarisation of LC-cells, which dependence on applied voltage shows pronounced hysteresis. Such a hysteresis was first observed in 1965 by Kapustin and Vistin[22] in nematic paraazoxyphenetole (PAP). They suggested ferroelectric nature of the phenomenon. Of course, they were wrong. First ferroelectric LC was synthesised so many as ten years later. Examination of the phenomenon was continued by Williams and Heilmeier (1966)[23], who observed polarisation hysteresis in three other nematics (PAA, PBBA, PMCA), also in isotropic phases, and even in some non liquid-crystalline liquids. They stated that the polarisation magnitude depends on cell thickness and electrode surfaces. They also supposed it might be ferroelectric although they considered it very generally as the ionic effect as well. The problem left in fact unsolved until 1969 when Kessler et coll. clearly evidenced the ionic mechanism of the polarisation effects observed[24]. Nevertheless, Vistin still in 1972 persisted by ferroelectric explanation[25]. However three years later Khachaturian pointed out also by theoretical analysis[26], that ferroelectricity in pure nematic phase is not possible, because depolarisation field must change nematic ordering and induce a chiral phase. Since than, ionic processes in LCs were studied by various methods and techniques such as current - voltage - time measurements [1-4,6,7,14-17], polarografic examinations[5], dielectric measurements [9,14,27], optical microscopic scanning[28]. With this all work we have not yet full understanding of this processes and further studies are still needed.

EXPERIMENTAL RESULTS

Since we are searching for effects taking place in real devices under operation, all the measurements we have carried out on LCs purified by standard procedures without any external doping with ionic additives. Examined cells were set up mostly without any surfactants, neither, to avoid the risk of shadowing basic material phenomena. Experimental cells were oriented either by rubbing only or not treated at all. The cell thickness was ranged from 5 μm up to 25 μm in plane „sandwich" geometry. Following effects were observed in various LC-materials, including commercial mixtures, without essential differences. Therefore we present here the results derived from examination of only two liquid-crystalline pure substances: positive dielectric anisotropy PCB and negative MBBA. They both are no more used in present devices at least as pure chemicals, however we chose them instead of one of commercial mixtures for two reasons:

- chemical homogeneity of the medium studied,
- well know properties accessible in past reports.

Our measurements were done in a few temperatures covering liquid-crystalline and isotropic phases.

Figure 1. presents polarisation of a nematic cell as a function of applied triangle-shaped periodic voltage. Polarisation was measured in classical Diamant-Drenck-Pepinsky circuit[29] usually used for ferroelectric measurements, allowing compensation of linear capacity and ohmic current of the examining cell. This means, that vertical axis in Figure1A. shows integrated total charge per unit area, flowing through the nematic

cell during one full cycle of linearly increasing and decreasing voltage of the both polarities, which is marked on horizontal axis.

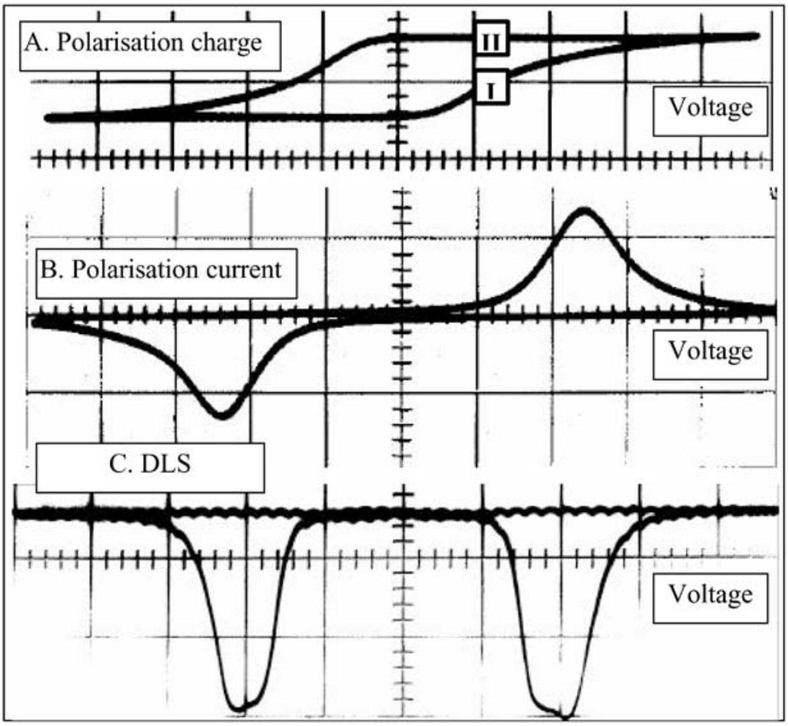

FIGURE 1. Simultaneous records of polarisation charge (1A), current 1(B), and optical transmission without polarisers (1C) of the LC-cell filled with negative anisotropy MBBA. Oscilloscope settings: X=10V/div, Y=0.1(μC/cm^2)/div, sweep rate 0.3 Hz

Such a curve is observed for frequencies up to several tenth Hz, depending on temperature and amplitude of voltage applied. The higher temperature is, the bigger is the polarisation (i.e. cell charge). For higher frequencies the voltage amplitude must also increase to sustain the hysteresis of the curve - otherwise it transforms into ordinary losses ellipse observed in most other dielectric liquids (which in the experimental arrangement is compensated to a straight line along the x-axis). Note important detail of the P(V) dependence: the polarisation charge stays almost constant when the applied voltage on the cell decreases (saturation part (II) of the curve in Figure 1A.) until the voltage changes polarity. Than the sudden re-polarisation of the cell begins causing re-charging current flow.

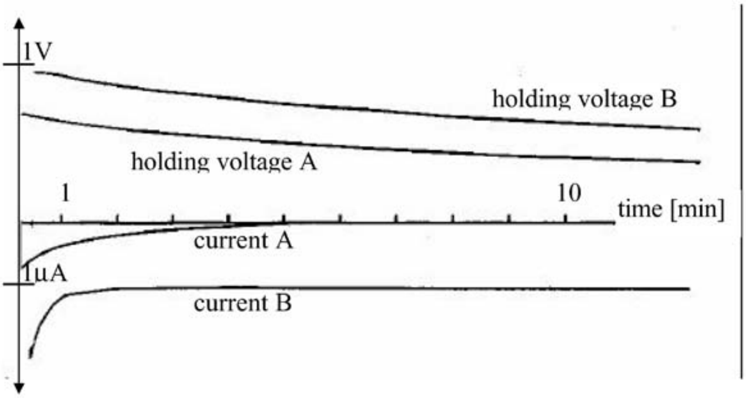

FIGURE 2. The total current and electrode holding-voltage after connection of LC-cell to external DC source of voltages: A=1V, B=9V

Figure 2. presents this current as a function of time in long term range, as well as so called „holding voltage" (V_H), i.e. the voltage remaining on the cell electrodes after removing external driving voltage source. It is observed by simple connecting the LC-cell to an electrometer, while an external voltage is switched on (current measurement) and off - after

a steady state is reached (V_H measurements). After quick initial increase the current decreases and reaches a steady-state value. This value is linear (ohmic) to the applied voltage. If the voltage is removed, the LC-cell holds a part of it like a condenser - curve 2B - for considerably long time (hours), even if it is meanwhile short-circuited. By extending the time scale to magnify the initial picture of the current one can observe (by oscilloscope) a transient current as it is shown in Figure 3.

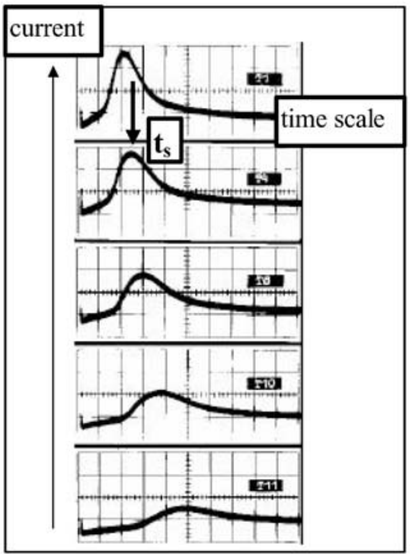

FIGURE 3. Transient ionic current (first maximum) - response of LC-cell on square voltage pulses with amplitudes: from the top respectively 50V, 45V, 40V, 35V, 30V. Oscilloscope settings: X=1ms/div, repetition frequency 3Hz

The current has characteristic local maxima at a time t_s after the DC voltage is switched on. This voltage creates an electric field inside the LC-cell which forces ions to move, but, the ions and the field influence mutually. Some insight on field distribution in the cell can be done by observing reaction of LC-molecular orientation on applied voltage. Oscillogram in Figure 4. reveals time evolution of optical response of LC-cell in the case if applied DC voltage slightly exceeds the threshold of Frederics transition (~1V). One can conclude from the Figure 4. that the LC-layer re-orientates at the first moment giving rise of optical transmission, but with time the layer comes back to initial orientation and the transmission falls down again.

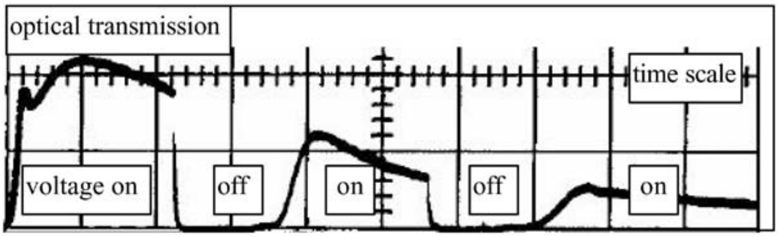

FIGURE 4. Optical response of LC-cell in crossed polarisers (i.e. molecular reorientation) on DC voltage pulse sequentially broken. Time scale X=2s/div

DISCUSSION

The ions existing in a LC-cell originate, as already mentioned, not only from impurities but can be also delivered by dissociation of LC basic material. Mechanism of additional ion generation may be thermodynamic equilibrium process involving decomposition of LC resulted from ageing of the material (hydrolysis, electrolysis e.t.c.). Such processes are widely investigated and clearly documented in MBBA, where they run relatively fast[4-6,8]. They are also present, to more or less extent, in other LC-materials. Thus, one can certainly expect a few different kinds of ions from at least two mentioned sources.

Local generation and transport phenomena of ions in electrolytic medium can be described by following set of formulae[30]:

$$\begin{aligned} dc(t)/dt &= k_R\, n^2(t) - k_D\, c(t) \\ dn(t)/dt &= k_D\, c(t) - k_R\, n^2(t) - (j_i(t) / qd) \\ j(t) = j_i(t) + j_D(t) &= (n^+ \mu^+ + n^- \mu^-)\, q\, E(t) + \varepsilon\varepsilon_o\, dE(t)/dt \end{aligned} \qquad (1)$$

where c(t), $n(t) = n^+ = n^-$, k_R , k_D, q, μ^+ , μ^-, j(t), E(t), are respectively: neutral species concentration, ions concentration, recombination and dissociation constants, ion charge, ion mobilities, current density and electric field in a fixed point of the medium. First two equations describe the local rate of creation of respective products (neutral species and ions) in dissociation/recombination reactions and transport process ($j_i(t)$). The last one expresses current density in the electrolyte as composed with ionic part ($j_i(t)$) and electric displacement part ($j_D(t)$) For the sake of simplicity we assumed macroscopically neutral medium and equivalent ions generated. General solutions of the above equation (1) can not be done analytically. By making, as usual, a reasonable approximation of real situation the solution giving time dependence of sample current can be obtained. First, we can consider the simplest situation if we assume following conditions:

1. constant ion concentration in the medium, n(t) = const,
2. full discharging of ions at the electrodes (no double layers)
3. electrical homogeneity of the LC layer, $n^+ = n^-$, grad n =0 at any point.

Than we have an equilibrium state in the medium; from equation (1) follows $n^2/c = k_D/k_R$ and $n^+ = n^- = [(k_D/k_R)\ c]^{1/2}$, and the steady-state current (by DC voltage and $t \rightarrow \infty$) is :

$$j(t) = (\mu^+ + \mu^-)\ n\ q\ V/d. \qquad (2)$$

This current is ohmic, linearly dependent on applied voltage. Such a picture could not be, of course, a real situation in LC-cell for all the ions present, but can be more or less close to it for some of them, which have diffusion constant sufficiently high to fulfil conditions 1. and 3., at least for small current. The second condition is rather real. W assume equation (2) is the case of residual impurity ions in LC-material, which are usually much smaller than LC-molecules and may have high diffusion constant in the LC-medium. Because of rather small concentration they are almost fully dissociated and they constitute relatively small steady-state part of the total current in a LC-cell.

There are, however, other ions in LCs we have to take into account in our model. Possibly, they come, as mentioned, from dissociation of big LC-molecules and, as the observations indicate, don't obey simplifying conditions given above. Nevertheless, to solve equation (1) for those ions, we can make some simplifications. We expect the LCs to be weak electrolytes with slow dissociation. Thus, after the voltage is applied to a LC-cell, all those ions are quickly removed from the bulk and recombination process is there stopped. By regarding this statement and inserting $n^{+/-} \rightarrow 0$ and $k_R\ n^2 \approx 0$ into equation (1) , the solution for current is obtained[2] in the form:

$$i(t) = (i_o - k_D\ c_o\ q\ d)\ \exp(-2t/\tau_T)\ +\ (k_D\ c_o\ q\ d)\ \exp(-k_D\ t) \qquad (3)$$

where $\tau_T = 2d/\,(\mu^+ + \mu^-)\,E$ is the average transit (drift) time through the cell and ($k_D\, c_o\, q\, d$) is saturation current. For weak electrolyte and low dissociation kinetics ($k_D << 1/\tau_T$) the saturation current is to neglect comparing to i_o , and the exponential current decay with time is so obtained:

$$i(t) = i_o \, \exp(-2t/\tau_T) \qquad (4)$$

Under such circumstances the ions are removed from the bulk where depletion space occurs. The both parts of current, described by equations (2) and (4), form the total current - time characteristics shown in Figure 2. As the discharged ions of the ohmic component diffuse from electrodes back in the bulk, exchange their charge and drift again, being not visible in compensated polarisation - voltage dependence, then the ions of the second part of current must be responsible for hysteresis of P(V) function. To understand mechanism of this effect we have made simultaneous records of polarisation charge, current and optical transmission of the LC-cell filled with negative anisotropy MBBA. The material shows easily dynamic light scattering (DLS) which is directly connected with ionic flow.

The chart in Figure 1. clearly shows, that:

1. current flow (plot 1B) connected with changes of polarisation charge (plot 1A) is caused by massive ions travelling across the LC-cell, what induces synchronic DLS - signal (plot 1C), (then injection effects at the electrodes as the hysteresis origin can be excluded),

2. increasing voltage of each cycle draws the ions from the bulk which accumulate at the electrodes and stay there until the voltage changes sign (refer to saturated part of polarisation hysteresis - plot

1A); than re-charging starts and the polarisation process repeats at the opposite electrode.

The creation of layers of accumulated ions at the electrode-LC interface is also confirmed by other observations:

-decrease of optical signal in Figure 4., indicating decrease of electric field inside the cell during polarisation,

-existence of holding voltage on the cell after removing external voltage.

It is then rather clear that the polarisation charge saturates with increasing voltage as a result of field exclusion of ions (part (I) in Figure 1A). The reasons why accumulated ions don't escape, so that saturation level is maintained until decreasing voltage changes polarity (part (II) in Figure 1A) needs some discussion. The fact of small diffusion constant is far not enough explanation, because in voltage-decrease sequence an inverse ionic field arises in LC-cell forcing drift transport. Formerly it was suggested that the ions can be somehow „firmly trapped" at the electrodes[23]. It seems to us not acceptable, because:

1. such a hysteresis curve was observed up to several tenths of volts by clean (without any cover) electrodes; it is rather impossible that the ions can be firmly trapped and not discharged,

2. a small voltage (about ~ 1V) is sufficient to start recharging process at the electrodes when polarity is changed, though before this moment a comparable reverse electric field also exist in the cell and no re-polarisation takes place,

3. the holding voltage (Figure 2.) on LC-cell: a) does not exceed about 1V independently on how high was previously applied external voltage, b) is maintained a long time (hours), c) remains still if electrodes are short-circuited for some time (is re-built again),

4. optical response of the cell is continued in a sequence of pulses (Figure 4.) indicating that ion charge summarises without essential loss.

This facts incline us to assume following ionic model of an electo-optical LC-cell under operation: the electrode-LC interface of the cell is built prevailingly with discharged, neutral molecules and - in a part only - with those undischarged ions which were unable to reach electrodes being blocked by neutral discharging product. This neutral molecules stick at the electrode surfaces covering them by a more or less insulating sheet. The effect is similar to electrolytic material - deposition and electrodes passivation. Adhered molecules relatively slowly diffuse back with time only by thermal mechanism. If opposite-polarity voltage appears, the stuck molecules are re-charged to ionic form, field-detached from the electrode surfaces and are made free to move. This neutral blocking layer may be not perfectly tight for the rest of accumulated ions because of facts (3) and (4) named above, so that they can penetrate it under some electric field. For the other group of small ions it is probably easily permeable because ohmic current needs the ions having also ohmic contacts. By using simple descriptive symbology, the model of ionic processes we suppose, can be then written as:

$$\underset{\text{bulk dissociation}}{\underleftrightarrow{\mathrm{AB} \Rightarrow \mathrm{A}^{+}}} + \mathrm{B}^{-} - \mathrm{p} - \mathrm{e} \overset{\text{discharging and adhesion}}{\Longleftrightarrow} \mathrm{A}^{o} + \mathrm{B}^{o} + \mathrm{p} + \mathrm{e} \underset{\text{re-charging and re-polarisation}}{\Longleftrightarrow} \mathrm{A}^{+} + \mathrm{B}^{-} \qquad (5)$$

where AB generally symbolises a group of molecules which dissociate onto ionisable species A^{o} + B^{o}, in particular LC molecules. Such a conceptual model, although not precise, can explain observed behaviour of LC electro-optical cells, but not without doubts. One of the still open questions is why that kind of ionic polarisation is observed in most LC cells but not in the prevailing number of other dielectric liquids. From the other hand, one more experimental observation that comply with our model is that the polarisation hysteresis was reduced to usual losses ellipse when the LC-cell electrodes were covered by good insulator sheet, which prevented the accumulated ions to discharge.

The consequence of this model is, in addition to the drawbacks caused by ionic processes in electro-optical LC devices, enumerated in the introduction, that the adhesion of uncontrolled species can deteriorate LC- layer alignment, especially by longer DC operation.

Closer characterisation of ions involved in polarisation effects can be obtained by analysis of LC-cell transient current in a short - time range (Figure 3.). In that case equation (1) have to be solved by different conditions than these for steady - state current (equation (2)). Assume, that square voltage pulses are applied to LC-cell (i.e. pulses of alternating polarity and constant amplitude). Basing on our model we can deduce the situation occurring in the cell: on the beginning of each subsequent voltage pulse there are two thin (~0.1μm[31]) layers of accumulated, re-chargeable species at the both electrodes and the bulk depleted by previous pulse of opposite polarity. Following next pulse induces electric field in the bulk and charges the layers, which during short time play a role of virtual ohmic contacts. Assume, that the layers are thin comparing to the LC-cell thickness d and reduce the problem to one dimension x. So we have:

$$n(x=0,\ t) \rightarrow 0, \quad n(x\neq 0,\ t=0) \rightarrow 0,$$

$$E(x=0,\ t) = 0, \text{ and } \int E(x,t)dx = V = \text{const}, \qquad (6)$$

where x=0, x=d are positions of the parallel cell electrodes. For the sake of simplicity we focus attention on only one electrode and voltages high enough to neglect voltage drop at the interfaces, as well as a small diffusion current, i.e. we assume $n\mu E \ll D\, dn/dx$.

Equation (1) solved with the above conditions (6) leads to the Child - Langmuir law; however it can not be accepted for real LC cells because the electrodes quickly cease to be ohmic. Therefore only the very beginning of the transient solution could be eventually considered. This transit was given by Helfrich and Mark[29] for time period not exceeding a value $\tau_{s,}$,

$$\tau_s = 0.786\, d^2 / \mu\, V, \qquad (7)$$

where the term $d^2 / \mu\, V$ means the ionic flow time by homogeneous field in a perfect isolator. The transient current starts with some initial value i_o and after some increase reaches maximum at the time τ_s , than decreases to steady Child - Langmuir value (assuming constant voltage). The above analysis concerns only one moving charge-carrier. Movement of the opposite ions starting from the other electrode changes this picture. Without complicated calculations one can expect two cases of this situation:

- if mobilities of the both ions are apparently different, the transient current will show two distinct maxima, and the time τ_s for the first one, faster, is not much changed comparing to equation (7) (see Figure (5)),
- if mobilities are close to each other, the transient has only one maximum not expressed by equation (7).

In an intermediate case the transient is broadened and the both maxima - if ever seen - quicker than calculated.

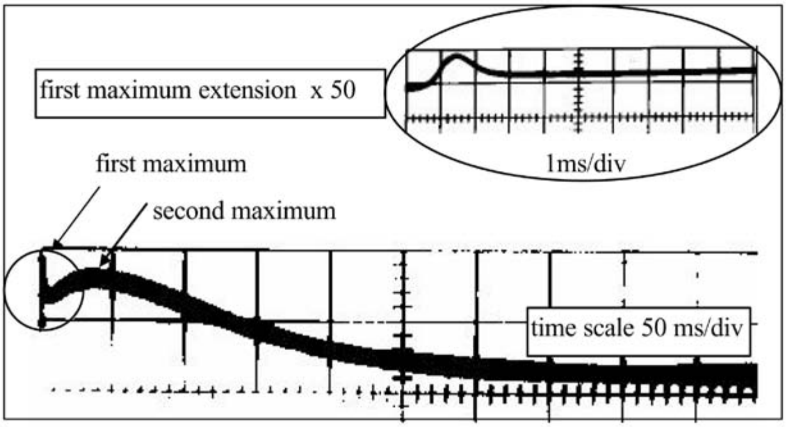

FIGURE 5. Initial transient ionic current in LC-cell exhibiting two distinct maxima; plots by X=1ms/div and X=50ms/div

The above results are more or less approximated and apply to LC-cells with some accuracy by certain restrictions. They are sufficiently high voltage and properly fitted repetition frequency of the pulses allowing the cell to reach assumed initial conditions. But also essential are: cell thickness, kind of electrodes, total concentration of ions and temperature. If the experimental situation meets the analytical considerations, the maximum transient time τ_s according to equation (7) should be linear with the reciprocal voltage value 1/V. The plot of the function $\tau_s= f(1/V)$ for experimental time values is presented in Figure 6. The curve is in fact linear beside low voltage region, not included.

Knowing the LC-cell thickness and using experimental values of polarisation charge, which is equal to charge surface density, we were able to evaluate ionic concentration $n^{+/-}$. Next, from the transient time measurements one can calculate ions mobility μ by use equation (7);

this mobility values taken in various temperatures, by applying Ahrrenius temperature dependence, give mobility activation energy E_{μ} in LC medium. The last calculation was done only for isotropic phase of LCs examined, since mobility in liquid-crystalline phase depends on orientation, which during transient current flow is not defined.

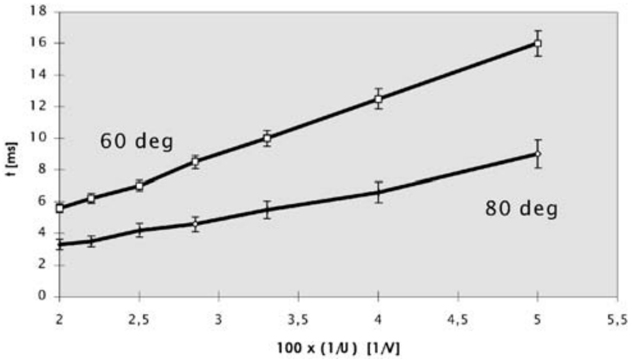

FIGURE 6. The transient time t_s of first maximum of re-polarisation current in LC-cell versus reciprocal of applied voltage

Obtained values for the ions in such a way are presented in Table 1. They are generally in agreement in other examinations[4, 11].

$n^{+/-}$ [1/cm^2],	MBBA 19°C	5• 10^{14}
$n^{+/-}$ [1/cm^2],	PCB 19°C	10^{15}
μ [cm^2/Vs],	MBBA 80/60 °C	7.6/7.6 • 10^{-6}
μ [cm^2/Vs],	PCB 80/60 °C	22/13 • 10^{-6}
μ(80 °C)/ μ (60 °C), PCB+MBBA		1,7
E_{μ} [eV]		0.23

Table 1. Evaluated characteristics of ionic polarisation charge

In the work a model of ionic processes in electro-optical LC-devices was presented. A number of experimental observations was carried out and discussed on the basis of the model. The results of measurements were interpreted in frame of assumed model and gave correct characteristics of the ions present in LCs, i.e. their concentration, mobility, and activation energy, which comply with these obtained by other methods. Although ionic processes in LC-devices were largely investigated earlier, the model given here introduces some new elements. One of the consequences referring to behaviour of LC-electro-optical devices, which result from the presented model, may be essential dependence of their performance on electrochemical properties of electrode surfaces, other than normally was taken into account formerly.

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