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Investigation of the liquid crystal alignment layer: effect on electrical properties

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Abstract
We investigate the electrical behavior of a symmetric liquid crystal (LC) cell: electrode-silane-LC-silane-electrode. The silane (chlorodimethyloctadecyl-silane) layer induces a homeotropic orientation of the nematic liquid crystal (NLC) molecules. The wettability technique is used to detect the change of the surface energy of the electrode upon cleaning and silane layer deposition. We report on the dynamic impedance measurements of the nematic liquid crystal cell. It is found that the silane alignment layer has a blocking effect on the liquid crystal (LC) cell. We also study the relaxation behavior of the cell which is later assimilated as an electrical equivalent circuit.

Keywords: liquid crystal, impedance measurement, relaxation processes, electrical equivalent circuit

(Some figures in this article are in colour only in the electronic version)

1. Introduction

In a liquid crystal device (LCD), polymer layers play an important role in molecular alignment and charge injection [1, 2]. It is known that the dielectric anisotropy of nematic liquid crystals (NLCs) plays an important role in the application of the electrical effect to display devices [3]. However, the consumption of electric power increases and ionic impurities, which might cause the misalignment of LC molecules during the operation, shortens the life of the LCD [4–6]. Consequently, the behaviour of ionic impurities included in the LC materials and the mechanism of charge generation on the electrodes is of great concern [7]. It is thus valuable to investigate the behaviour of charge carriers and the effects of the insulating alignment layer used as blocking electrodes under AC and DC excitation in terms of the LCD reliability [8]. Also, the morphology of the alignment layer is of great consequence to the achievement of the desired orientation of the LC molecules.

In this work, we adopted scanning electronic microscopy (SEM) to examine the morphology of a silane layer that induces a homeotropic orientation of NLC molecules.

Electrical impedance spectroscopy may be used to study the dynamics of bound or mobile charge in the bulk or interfacial regions of any kind of solid or liquid material [9, 10].

In this study, we utilized this technique for analyzing the relaxation mechanism in the LC cell and to demonstrate the electrical effect of the silane layer. We also tried to model the studied cells as an electrical equivalent circuit and to determine its component parameters.
2. Sample preparation

2.1. Surface treatment of ITO

Because of its transparency, high conductivity, good etchability, hardness and good adherence on many types of substrates, indium tin oxide (ITO) is usually used as an electrode material. These characteristics allow its use in a wide range of optoelectronic devices, particularly LCDs [8]. Commercially available ITO films (Baltracon-Balzers) with a thickness of around 140 nm on glass substrates and with sheet resistance <20 Ω/□ were used for the experiment.

The surfaces of ITO substrates were cleaned according to the following procedure: 20 min in an ultrasonic bath of acetone followed by 3 h under methanol reflux in a Soxhlet before being dried under a nitrogen flow. This procedure was chosen after a systematic study of the effects of the ITO surface treatments on the performance of LCDs [11].

2.2. Deposition of alignment liquid crystal layers on ITO

A layer of chlorodimethyloctadecyl silane 95% [CH\textsubscript{3}(CH\textsubscript{2})\textsubscript{17}Si(CH\textsubscript{3})\textsubscript{2}Cl] (Aldrich Chemical Company, Inc) was grafted onto the ITO substrate. Before the deposition of the silane layer, the ITO surface was dried under vacuum for 3 h and at 140°C. The ITO slide was impregnated with silane in isopropanol solution; the grafting was achieved under nitrogen atmosphere at 100°C. The sample was then washed using isopropanol to eliminate adsorbed silane molecules.

2.3. NLC cell construction

The cell is comprises two parallel glass plates. These plates were coated with a thin ITO film that was used as the electrode cell and on which the alignment layer was deposited.

A polar NLC belonging to the cyanobiphenyl family, 4-cyano-4-n-hexylbiphenyl (6CB) (BDH Limited Poole England), was used. It is in the nematic phase between 14.5 and 29°C. Its physical properties have been studied extensively. The dielectric anisotropy is positive because the dipole moment of the cyano group in the molecule is nearly parallel to the long molecular axis [12]. The LC material was syringed into a d = 20 μm thick symmetric cell with Mylar spacers between the two functionalized ITO substrates. The effective electrode area was S = 2.5 cm\textsuperscript{2}.

The deposited monolayer favors a homeotropic orientation of the LC molecules. This was checked through observation with a polarized microscope.

3. Measurements

3.1. Wettability

The contact angle measurements and the surface energy values of the ITO-treated surfaces were determined by the sessile drop method [13] using a GBX Scientific Instrument (Romans, France). We used the Van Oss theoretical model which gives the surface energy and its dispersive, acid and basic components [14]. Three liquids were used as probes for surface free-energy calculations: diiodomethane (non polar liquid) (Sigma Chemical Co, St Louis, Mo, USA), formamide (first polar liquid) (Sigma Chemical Co, St Louis, Mo, USA) and deionised water (second polar liquid).

3.2. Electrical measurements

The principle of impedance spectroscopy is to measure the electrical characteristics of a material as a function of the frequency of a small-amplitude AC signal. The resulting impedance can be determined using an impedance analyzer. Changes in impedance characteristics with frequency provide information about conduction mechanisms, charge accumulation, dipole behaviour, and the dielectric constant of a material [15]. In our study the dielectric measurements were carried out at ambient temperature (the material being in the liquid crystal phase) and in a frequency range of 10\textsuperscript{3}–12 × 10\textsuperscript{6} Hz. In the lower frequency range (10\textsuperscript{3}–10\textsuperscript{6} Hz), we used the impedance analyzer (Voltalab40 PGZ 301). Impedance measurements in the frequency range from 5 Hz to 13 MHz were taken using the HP 4192A impedance analyzer. For all the measurements, the amplitudes of the AC and DC voltages (bias voltages) were 100 mV and 2 V, respectively. We have chosen to work under polarization to avoid the diffusion effect observed at 0 V DC.

4. Results and discussion

4.1. Wettability

The results of the liquid contact angle and surface energy of the ITO (as received and cleaned) and of the alignment layer of silane are summarised in table 1.

We observe that the cleaning method used gives a lower value of the water contact angle and a higher surface energy compared with the as-received ITO. It makes the surface more hydrophilic and supplies an important basic component of the surface energy, resulting in a favorable surface for the adhesion of the alignment layer. This can explain the ordered structure of the silane layer observed in the SEM
Figure 1. SEM images of silane deposited layer on ITO cleaned with isopropanol (a) and with methanol (b).

images (figure 1) when using this protocol. The interaction between ITO and the alignment film becomes stronger and the distribution of the molecules is more homogenous and well structured in the case of the surface cleaned with methanol compared with that cleaned with isopropanol. We previously reported [11] that these results are obtained thanks to the high polarity and the dielectric constant of methanol.

The deposition of the silane layer makes the surface hydrophobic, considering the large water contact angle, and possesses the lowest surface energy value. This characteristic of the surface is caused by the long alkyl chain in the silane molecule.

It is also very important to link the homeotropic alignment of LC with the chemical structure of the layer. The alignment of LC itself involves complex physicochemical interactions between the LC molecules and the alignment film. It was found that the homeotropic alignment of LC is caused by the weak anchoring [16] and is favoured when the surface is hydrophobic [12].

As described above, the value of surface energy and mainly its acid-base component (polar surface energy) are obviously weak. The polar surface energy affects the pretilt angle dramatically [17]; the pretilt angle increases as the polar surface energy of the alignment film decreases [18]. Therefore, we can assume that the weak polar surface energy plays an important role in the homeotropic alignment of liquid crystal.

4.2. Electrical results

First we examined the effect of the alignment layer on the electric behavior of the cell. Figure 2 shows the frequency dependence of the conductance of two cells with and without the silane layer. We noted stabilization at high frequencies of the conductance for the (ITO-silane-LC-silane-ITO) cell compared with the (ITO-LC-ITO) cell, which indicates that the silane layer has a blocking effect on the electrodes [15]. This last deduction is confirmed from the increase in the real impedance component $Z'$ for all the frequency range (figure 3).

The imaginary impedance part shows two peaks for each cell (figure 4); one in the low-frequency range (around $10^{-2}$ Hz) and the other in the higher range (about $10^{-1}$ Hz). These peaks prove the existence of two kinds of dielectric relaxation in the sample. Thus, we can propose an orientational relaxation (dipolar orientation) of the molecules in the high-frequency range [19, 20] and an ionic relaxation at the LC-Electrode interfaces in the lower one [21, 22]. On the basis of the Murakami study [7], one can consider that the relaxation in the low-frequency domain is due to the formation of the electrical double layer at the electrode/6CB interfaces. The double layer is formed via the adsorption of impurity ions in the liquid crystal on the electrode and therefore the electrode/6CB interface behaves as a parallel-plate capacitor [7, 23]. This leads to the modeling of the cell as an electrical equivalent circuit. This is reported in the following part.

4.3. Electrical equivalent circuit

To gain more information on the relaxation mechanism, we assimilate the LC cell into an electrical equivalent circuit to determine its component parameters. The electrical equivalent circuit (figure 5) used was suggested by Schadt [24] and it includes contributions from the electrodes, the LC alignment
Figure 3. Variation of the real impedance part with frequency for ITO-6CB-ITO and ITO-silane-6CB-silane-ITO cells in the frequency ranges of 1 mHz–100 kHz (a) and 5 Hz–5 MHz (b).

Figure 4. Variation of the imaginary impedance part with frequency for ITO-6CB-ITO and ITO-AL-6CB-AL-ITO cells in the frequency ranges of 1 mHz–100 kHz (a) and 5 Hz–5 MHz (b).

Figure 5. Schematic representation of the electrical equivalent circuit associated with the liquid crystal cell. \( R_s \) is the resistance of the electrode (ITO). \( R_0 \) and \( C_0 \) are associated with the cell volume; \( R_1 \) and \( C_1 \) correspond to the resistance and capacitance, respectively, of every LC/silane interface.

Figure 6. Fitting of imaginary impedance part of ITO-6CB-ITO and ITO-silane-6CB-silane-ITO cells in the frequency range of 1 mHz–100 kHz.

layer and the LC in the volume of the cell. On account of the existence of two relaxation times, we assign, in the modeling, a parallel resistor capacitor circuit for every LC/silane interface (\( R_1 \) and \( C_1 \)) and another one for the volume (\( R_0 \) and \( C_0 \)). These circuits are linked serially to a resistance \( R_s \) associated with the ITO electrode. Figure 6 shows that the theoretical fit accurately follows the experimental curve of the imaginary part of the impedance for the previous studied cells (ITO-LC-ITO and ITO-silane-LC-silane-ITO).

On the other hand, the parameters determined using this model are almost as expected (table 2). Indeed, the capacitance values \( C_0 \) and \( C_1 \) are of the order of nF and \( \mu \)F, respectively, which is in agreement with the literature [1–3]. This is also the case for the high resistance values (order of
magnitude of MHz). The relaxation times $\tau_0$ (order of ms) and $\tau_1$ (order of s) are suitable for the contribution to the orientational relaxation and ionic relaxation, respectively.

Moreover, in this table, we note the confirmation of the blocking effect of the LC alignment layer: the increase of the resistance on the one hand and the decrease of capacitance on the other. Also, this result appears in figures 3 and 6 showing the increase in the amplitude of the real and imaginary impedance parts of the ITO-silane-LC-silane-ITO cell compared with those of the ITO-LC-ITO cell.

5. Conclusion

In this paper, we presented an investigation of the characteristics of the liquid crystal alignment layer and its effect on the electrical properties of the LC cell. We found that the weak polar surface energy of the alignment film plays an important role in the homeotropic alignment of liquid crystal.

We used impedance spectroscopy to investigate the alignment layer effect on the conductance and the impedance of the cell. It was found that this layer has a blocking effect on the LC cell since we observed a decrease and an increase in the conductance and the resistance, respectively. We studied, therefore, the relaxation behavior of the entire cell. Two kinds of relaxation were observed; one was attributed to the reoration of the LC molecules in the volume and the second was associated with the ionic relaxation at the LC-silane interfaces. Finally, we attempted to model the LC cell with an electrical equivalent circuit that takes into account the volume and the interfaces of the cell. We obtained good agreement between the experimental and fitted curves. We also determined the different component parameters of the electrical equivalent circuit and we obtained the expected values.

Table 2. Fitting parameters of the equivalent circuit for the LC cell.

<table>
<thead>
<tr>
<th>Fitting parameters</th>
<th>$R_0$(MΩ)</th>
<th>$C_0$(nF)</th>
<th>$R_1$(MΩ)</th>
<th>$C_1$(µF)</th>
<th>$\tau_0$(ms)</th>
<th>$\tau_1$(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ITO-6CB-ITO</td>
<td>5.64 ± 15.6%</td>
<td>7.51 ± 11.4%</td>
<td>2.60 ± 8.5%</td>
<td>10.74 ± 12.5%</td>
<td>42.42 ± 27.9%</td>
<td>27.97 ± 21.0%</td>
</tr>
<tr>
<td>ITO-silane-6CB-silane-ITO</td>
<td>18.52 ± 7.3%</td>
<td>5.72 ± 10.6%</td>
<td>9.59 ± 7.6%</td>
<td>6.56 ± 10.3%</td>
<td>106.1 ± 17.6%</td>
<td>62.94 ± 17.9%</td>
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</table>

References