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Electrically controlled defects at a liquid crystal–polyimide interface

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We report results on the effect of introducing a small quantity of oligomer particles inside a liquid crystal (LC) display. We observe a dramatic change in the properties of the LC–substrate interface. Upon applying an electric field, a π-wall defect network appears that is stable in the absence of further electric perturbation but can be erased by a suitable pulse in a well-defined range of temperature and frequency. The appearance of the defects and their dimensions depend on the cell age, i.e. on the time required by the guest to deposit on the surface. Hypotheses on the phenomenon are reported.

1. Introduction

In the early 1990s, Poulin *et al.* [1] started a new field of research called "liquid crystals colloids" by experimental and theoretical study of suspensions of colloidal particles in a lyotropic liquid crystal (LC). The idea of introducing a large amount of mobile interfaces into the LC, generating non-trivial topological constraints and singularities, has been fruitfully explored in further experimental and theoretical works [2, 3]. Mixtures of spherical particles and thermotropic LC have been studied both experimentally and theoretically [4] along with the interaction forces among elongated particles in a nematic colloid [5]. In all cases it is clear that the presence of a particle in a nematic induces director field distortion in the surroundings that can produce a topological mismatch between the director distribution close to the particle surface and the uniform director field at larger distances.

Subsequently, it has been shown that spherical poly(methyl methacrylate) (PMMA) particles tend to separate from the nematic host giving rise to a so-called soft solid [6].

When oligomers are introduced into a LC cell [2], they separate from the LC, reducing the memory effect and preventing fast surface reorientation. Lower operating ac voltages and steeper electro-optical response are also obtained.

In this paper we report investigations on mixtures of an acrylic monomer with a well-known nematic LC. The typical concentrations of monomer used here are lower than those used in the literature [6]; nevertheless, spinodal decomposition occurs in the mixture and electrically controllable defects are created. After filling a thin cell with the mixture, the spinodal decomposition process causes the monomer molecules to concentrate close to the boundary plates. This fact is supported by several observations that are described in the following. As a consequence of this monomer concentration on the surfaces, a dramatic change in the anchoring properties is observed, making it possible to create a defect network by applying an electric pulse of suitable amplitude and duration.

2. Experimental results

The samples used were standard sandwich nematic LC cells made using two indium–tin oxide (ITO)-coated glass plates. The ITO electrodes were patterned by photolithography and the glass plates assembled to form four pixels. The surface treatment was carried by spin coating a solution of 2 wt% of polyimide (LQ1800 from Hitachi) in 1-methyl-2-pyrrolidinone following a standard procedure [7]. The rubbed polyimide induces a small pretilt angle with respect to the surface. Silica beads were used as spacers and, in order to avoid any influence on the defects formation process, the beads were spread only over the cell perimeter leaving the pixel area free of them. The two plates were assembled in order to obtain a homogeneously tilted director arrangement and the cell thickness was measured by an interferometric method before filling the cell. The thickness was $1.9 \pm 0.1\,\mu$m for all the tested cells.

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The nematic LC mixtures were composed by a well-defined amount of a monomer (Sartomer S349 from Cray-Valley) in LC E7 (1, 2, 3 and 4 wt%) (from Merck) or in 5CB (4%) (from Merck) and, in some cells, 0.5% of a suitable photoinitiator (benzophenone). E7 is a mixture of biphenyl molecules and exhibits a large nematic phase domain around room temperature (from −20 to 62°C), whereas 5CB has a narrower nematic phase range (24–35°C). Both nematics have large dielectric anisotropy. The cell was filled under vacuum at temperatures above the nematic–isotropic transition temperature ($T > T_{NI}$).

An electric pulse of 1 ms width and variable amplitude, generated by a computer-controlled arbitrary waveform generator (HP) and amplified by a broad band power amplifier (Krohn-Hite) was applied to the cell. The temperature was kept constant within 0.2°C by a CALCTEC hot stage.

The cells were observed using a polarizing optical microscope (Zeiss). First, we checked the texture and then measured indirectly the T_{NI} on heating. Subsequently, we applied an electric field and observed the cell response. The images were acquired using a PC connected to the microscope by a Panasonic camera.

Results obtained using freshly filled and old stored cells are both reported.

E7 freshly filled cells exhibited a typical planar texture and a decrease in T_{NI} with monomer concentration, as shown in table 1. It shows the usual behaviour found in mixtures, i.e. a linear decrease with the monomer weight fraction. The particles are regarded as non-interacting; from a theoretical point of view the factor that controls the distortion is the balance between elastic and anchoring energies at the particle surface; it becomes larger near the nematic–isotropic transition [4].

An electric voltage applied to the cell at this stage does not induce any variation. Dozov *et al.* [8, 9] reported that cell geometry with parallel non-zero pretilt cannot exhibit a planar–twisted transition under the effect of a pulsed electric field since anchoring breakage is forbidden. In fact, we did not observe experimentally in a pure E7 or in a freshly filled cell any transition unless $T \sim T_{NI}$, where small twisted domains appear at voltages higher than 39 V μm^{-1}, probably due to a further decrease of the small tilt angle at the surface. This amplitude is a limit value for our experiments because applying a higher applied voltage implied destroying the cell in most of cases.

Table 1. Variation of T_{NI} as a function of monomer concentration.

Concentration (weight fraction)	T_{NI}/°C
0.01	57
0.02	52
0.03	45
0.04	40

The 5CB mixture appeared isotropic and cells freshly filled were black when observed between crossed polarizers.

After few days of storage at room temperature, the cells start to change. For the E7 cells, the T_{NI} typical of the pure E7 is recovered. Optical observations revealed no changes in the texture. The 5CB cell is no longer in the isotropic phase but also exhibits a uniform planar texture.

The host particles seem to be expelled by the LC bulk and are deposited onto the glass surfaces without changing the optical texture. The mechanism inducing the separation could be the adsorption of oligomers on the glass substrate.

The T_{NI} was measured several times before proceeding with the electrical measurements without any further variation. The interface–monomer interaction appeared to be quite stable and the separation irreversible.

The formation of a defect network texture was observed after pulse application for E7 cells. The defects network appears simultaneously with an increase in T_{NI}; monitoring T_{NI} is a practical method to avoid cell destruction. The defect network was easily erasable by applying electric pulses with lower amplitude or shorter duration than the writing pulse. In order to check the erasure conditions the pulse width is held constant and the amplitude is changed until erasure is obtained; another method is to hold constant the amplitude and vary the pulse width. Figure 1 reports both writing and erasing threshold amplitudes measured as a function of the pulse width at room temperature.

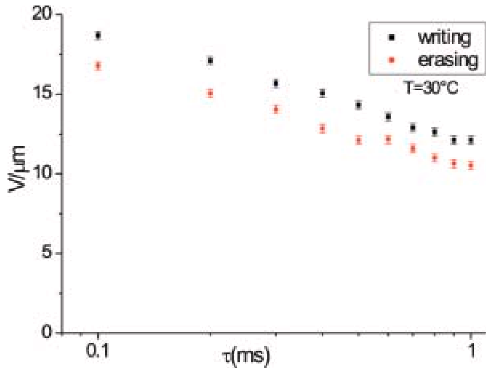

Figure 1. Writing and erasing voltage for the homogeneously tilted cell at $T = 30$°C.

The useful pulse width for this display ranges from 100 μs to 1 ms. Below 100 μs the pulse amplitude needed becomes too large and the cell can be destroyed. Above 1 ms the defect network becomes stable and is difficult to erase using an electric signal. No differences were noticed between positive and negative pulses due to the symmetry of the cell.

This writing/erasing behaviour was observed at temperatures below 35°C, whereas above this temperature the defects start to relax spontaneously. As expected, both writing and erasing voltages decrease with increasing temperature. The effect of an ac burst signal was also studied: defects are formed and erased with the same mechanism and more or less the same threshold. A small difference of the threshold values might be due to the flexoelectric term in the free energy expression. The defects formed in the cells are π walls and extend across the bulk from one surface to the other. After the voltage is applied, the defect remains stable only when point singularities are formed, otherwise it shrinks and disappears. The cells were found to possess along lifetime of more than one year. The π-wall defects are usually the signature of anchoring breakage.

Defects appear at the surface for all the concentrations used after an electric voltage is applied. For 1% and 2% monomer concentrations erasure is possible, whereas for 3% and 4% the cell remains covered by defects that are no longer erasable unless the temperature rises above the nematic–isotropic transition.

Some observations need to be taken into consideration.

1. If the cell is filled by a mixture without photoinitiator the phase separation does not occur and the T_{NI} increases instead of decreasing. Any time-depending effect is detectable.
2. If the cell is filled by a UV pre-exposed (60 s) mixture the separation speeds up, suggesting a molecular weight influence.
3. Similar behaviour is observed if, following the literature [6], a mixture of PMMA (120 000 molecular weight) and E7 is prepared: the two compounds in the cell segregate quickly, giving rise to defects at the surface without applied voltage and, after the field is applied, the defects remain almost stable. Heating up to T_{NI} and cooling down slowly, the defects disappear and become electrically controllable, as observed for previous mixtures with more or less the same thresholds. The last example belongs to a research field well known in literature, since the dimension of the elongated particles is increased [5]. An investigation of the detailed behaviour of this kind of mixture is still in progress.

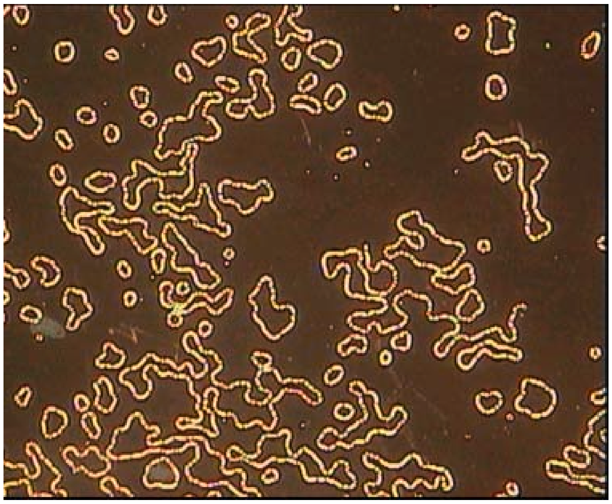

Figure 2. Defects in a cell filled with E7+2% monomer +0.5% photoinitiator under a polarizing optical microscope (20 ×).

As a consequence of the above points, it is probable that monomers aggregate in the LC, separate for energetic reasons and deposit onto the surface, slightly disordering the texture.

3. Discussion and conclusion

In conclusion, the electro-optical behaviour of mixtures of LC with a colloid, acrylate monomer or PMMA has been studied. The nature of the nematic phase is not affected by the presence of particles at low volume fraction; however, due to the elastic interaction induced by the particles, a demixing is expected at the equilibrium with a complete segregation between the nematic phase and the particles [10, 11].

The monomers presumably undergo a slow polymerization process induced either by UV exposure or by the laboratory light. In both cases, linear oligomers may form; hence, the colloidal behaviour should be more similar to that of elongated particles. The same applies for PMMA mixtures.

We have observed electrically controllable defects at the cell surface and, in contrast with previous studies, the defects are not observed by polarized optical microscope before applying an electric voltage. The size of the guest particles, at the beginning, is of the same order as of the LC molecules. A plausible hypothesis is that monomer deposits at the surface giving rise, under suitable electric stimulation, to defects. This feature could play an important role especially for developing bistable displays even if the optical response time is not improved with respect to the usual monostable displays.

The defects in the cells are clearly of the π-wall type. Only the cell with PMMA initially shows a defect-like texture but the homogeneous arrangement is recovered upon heating up to the nematic–isotropic transition and cooling down slowly below T_{NI}.

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