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PDLC films prepared by electron beam and ultraviolet curing: influence of curing conditions on the electro-optical properties

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Polymer dispersed liquid crystal (PDLC) films were prepared by polymerization-induced phase separation processes using ultraviolet (UV) and electron beam (EB) radiation. A mixture of the nematic LC material E7, an aromatic polyester acrylate, and additional monomeric acrylates was exposed to the EB radiation. A photoinitiator was included in the initial mixture in the case of UV exposure. The electro-optical behaviour of the PDLC films obtained has been investigated as a function of the chosen radiation. The transmission versus voltage curves strongly depend on the curing conditions, and are highly reproducible Threshold and saturation voltages continuously increase with increasing dose values for UV-cured films, whereas plateau values were reached for EB-cured samples. A small memory effect has been observed for UV-cured systems.

1. Introduction

Polymer dispersed liquid crystal (PDLC) materials are of great interest for electro-optical applications such as flexible displays or light shutter devices. They consist generally of low molecular mass nematic liquid crystal (LC) microdroplets dispersed in a polymer matrix [1-3]. In the so-called normal mode, these films scatter light in the off-state and can be electrically switched to a transparent on-state.

The most convenient method used for the preparation of PDLC films is the polymerization-induced phase separation (PIPS) of mixtures composed of reactive polymer precursors and LCs. PIPS initiated by ultraviolet (UV) and electron beam (EB) radiation has been frequently used as a powerful method to obtain well defined PDLC films [1, 2, 4-15]. Compared with the PIPS-UV process, EB-curing does not require the addition of a photoinitiator.

The curing kinetics and the phase separation process control the electro-optical characteristics of PDLC films. The influence of the dose and the dose rate on the sample morphology and on the electro-optical properties has been investigated in recent years [2, 12, 16]. As a result, it was generally found that the LC droplets become smaller with increasing UV intensity. For example, Carter *et al.* [9] observed this effect on the PN393/TL213 system and Lackner *et al.* [8] found this behaviour on cured Thiol-ene/E7 blends. Smaller droplets require higher fields for orientation due to their higher degree of curvature [2]. Other authors like Smith [12] and Nazarenko *et al.* [13] studied the effect of UV dose and curing temperature on the sample morphology and particularly on the droplet size. Only a few results on the effect of the dose on electro-optical properties have been reported for PIPS-EB systems [14, 15, 17]. All these investigations show the need to control the curing parameters to improve the electro-optical performance for a given polymer/LC system.

In this paper, PDLC films were prepared by a PIPS process using UV or EB radiation. The electro-optical properties of the PDLC films obtained, based on a selected representative composition, were studied depending on curing conditions. Two different concentrations of the photoinitiator were used to prepare PIPS-UV samples. The transmission properties have been analysed as a function of voltage at a constant frequency of the applied a.c. voltage. The evaluation of the electro-optical behaviour indicates remarkable differences between UVand EB-cured films. The results are discussed in terms of the transmission in the initial off-state and in the on-state. Threshold voltages (voltage required for 10% of the maximum transmission value) and saturation voltages (voltage required for 90% of the maximum transmission value) have also been considered. Special

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emphasis has been paid to the comparison of the electrooptical curves obtained for both UV- and EB-cured films. To our knowledge such a study has not been reported in the literature so far.

2. Experimental

2.1. Materials

The nematic LC used in this work was a commercial mixture (E7 from Merck Ltd, Poole, UK) containing essentially cyano-*para*-phenylene derivatives. It exhibits a positive dielectric anisotropy at $T = 20^{\circ}$ C and a nematic–isotropic transition temperature $T_{\text{NI}} = 60^{\circ}$ C [18]. The prepolymer chosen consisted of an aromatic polyester acrylate (Rahn AG, Switzerland) diluted in additional monomers including tripropyleneglyc ol diacrylate (UCB, Belgium).

2.2. Preparation of PDLC films

A 30 wt % quantity of the prepolymer and 70 wt % of the LC mixture were mixed at room temperature for several hours and used as the initial reactive mixture for EB-cured samples. Two series of UV-cured samples were prepared from the same mixture containing 1 and 2 wt % (of the acrylate mixture) of a conventional photoinitiator (LucirinTPO, BASF).

Samples for electro-optical studies were prepared by sandwiching the initial reactive mixture between a glass plate (Balzers, Liechtenstein) and a polyethyleneterephthalate (PET) sheet (Renker, Germany), both coated with a thin transparent layer of conducting indium tin oxide. A 100 μ m thick PET sheet was used for the UV curing process. Our EB generator requires a thinner PET substrate of 50 μ m to allow uniform penetration of the applied dose into the depth of the sample. The film thickness was measured by a micrometer calliper (Mitutoyo; uncertainty: $\pm 1 \mu$ m). No temperature control was used during the irradiation processes.

2.3. Electron beam curing

The generator used in our experiments was an Electrocurtain Model CB 150 (Energy Sciences Inc.) with an operating high voltage of 175 kV. The samples prepared as mentioned above were placed in a tray which was passed under the electron-curtain on a conveyor belt. In our experiments, the applied dose range was 60-120 kGy. This was achieved by using a beam current of 4, 5, 6, 7 and 8 mA and a constant conveyor speed of 0.19 m s^{-1} .

2.4. Ultraviolet curing

The UV light source used was a Minicure Model MC4-300 (Primarc UV technology) equipped with a medium pressure mercury arc lamp rated at 80 W cm⁻¹. The samples prepared as mentioned above were placed

on a conveyor belt. The dose received by the sample was varied between 11 and 52 mJ cm^{-2} . This was achieved by using a constant light intensity and changing the conveyor speed in the range $0.43-1.25 \text{ m s}^{-1}$.

2.5. Electro-optical measurements

The electro-optical experiments were performed at room temperature by measuring the transmission of unpolarized HeNe laser light at a wavelength of $\lambda = 632.8$ nm. The PDLC films were oriented normal to the laser beam. The distance between the sample cell and the detector (silicon photodiode) was approximately 30 cm. The collection angle of the transmitted intensity was about $\pm 2^{\circ}$, so that principally forward scattering was detected. The intensity of transmitted light was recorded on a micro-computer using an interface card (DAS 1600-2). The transmission measurements were corrected using appropriate calibration standards.

For electro-optical measurements, an external electric field was applied across the PDLC film. The output of a frequency generator was amplified and used to drive the shutter device. Starting from the electrical off-state, the applied sinusoidal voltage of frequency 145 Hz was increased continuously up to a desired maximum value $V_{\rm max}$. Subsequently it was decreased in the same way. The whole scan up and down ramp was usually performed during 120 s, and an additional measuring time of 60 s allowed the relaxation behaviour of the transmission in the off-state to be followed. The same procedure was repeated several times using appropriate voltage maximum values.

3. Results and discussion

3.1. Electro-optical behaviour

Figure 1(a) illustrates the electro-optical behaviour of UV-cured PDLC films using 1 and 2 wt % of photoinitiator. The latter film will be called '2 wt % UV' whereas the first will be called '1 wt % UV'. In figure 1(b), the results for EB-cured PDLC films are given. All samples obtained appeared opaque in the initial off-state and became transparent if an appropriate electrical field was applied. The sample thickness was about 30 µm in each case presented in figures 1(a) and 1(b). The scaling of the axes of these figures has been chosen to be identical to make further comparison easier. In figure 1 (a), transmission versus voltage curves of representative PDLC films prepared using UV-dose values of 15 and 45 mJ cm⁻² are represented. For the latter dose, samples obtained from initial mixtures including either 1 or 2 wt % of photoinitiator were exposed to the UV irradiation. A similar presentation is given in figure 1(b), where the electro-optical response of EB-cured films (74 and 104 kGy) is exhibited. Both figures show clearly that for higher dose values, the threshold voltage (V_{10}) and the







Figure 1. (a) Electro-optical response of 30 μm thick UV-cured PDLC films prepared using 15 and 45 mJ cm⁻² (2 wt % photoinitiator) and 45 mJ cm⁻² (1 wt % photoinitiator). (b) Transmission properties of 30 μm thick EB-cured PDLC films for 74 and 104 kGy.

saturation voltage (V_{90}) increase for the UV-cured as well as for the EB-cured samples. Obviously, the difference between V_{90} and V_{10} , $\Delta V = V_{90} - V_{10}$ becomes more important by increasing the dose value for both UVand EB-cured films. Figure 1 (*a*) indicates significantly higher threshold and saturation voltages for the 1 wt % UV sample as compared with the 2 wt % UV film. It was also found that the UV-cured samples did not recover their initial value of transmission in the off-state after application of voltage cycles, as shown in figure 1 (*a*). This memory effect has not been observed for the EB-cured samples.

3.2. Transmission in the initial off-state and in the on-state

Figures 2(a)-(c) illustrate the dependence of both thickness and dose on the transmission in the initial

off-state $T_{off init}$, and on the maximum transmission in the on-state T_{on} . The latter quantity is represented on a logarithmic scale. The relation between $T_{off init}$ and log T_{on} as functions of film thickness and for different UV doses is shown in figure 2(*a*) for the 2 wt % UV system and in figure 2(*b*) for the 1 wt % UV films. The results obtained for EB-cured samples are represented in figure 2(*c*).

For 2 wt % UV films, a small film thickness distribution of $30 \pm 4 \mu m$ was found. Due to the lower rigidity of the $50 \mu m$ PET sheet covering the liquid mixture as compared with the $100 \mu m$ substrate, film thicknesses are less regular and were measured in the range $9-32 \mu m$ for EB-cured samples. The thickness of the 1 wt % UV films also do not remain constant, probably due to the lower mechanical stability of the polymer matrix as compared with the 2 wt % system. These findings explain



Figure 2. (a) Transmission in the initial off-state $(T_{off init})$ and in the on-state (T_{on}) of UV-cured PDLC films (2 wt % photoinitiator) as functions of film thickness and dose. (b) Dependence of transmission on the dose and film thickness in the initial off-state $(T_{off init})$ and in the on-state (T_{on}) for UV-cured PDLC films (1 wt % photoinitiator). (c) Transmission in the initial off-state $(T_{off init})$ and in the on-state (T_{on}) for EBcured PDLC films as functions of dose and film thickness.

the stronger thickness dependence observed in the case of 1 wt % UV and EB systems.

However, the values of $T_{off init}$ for both UV- and EB-cured samples do not show a dependence on the dose and remain roughly below 2%. At a given sample thickness, figure 2(*a*) exhibits slightly reduced values of log T_{on} on increasing the UV-dose. It can be assumed

that this tendency also exists in the case of the 1 wt % UV system as shown in figure 2(*b*). Further experiments will be necessary in order to confirm this behaviour. The log T_{on} values obtained from EB-cured films are in the range roughly from 1.87 (corresponding to 80% transmission) to 2 (corresponding to 100%). An increase of the dose from 59 to 119 kGy does not significantly

change the on-state transmission. At a given dose value, a slight shift to lower transmission values on increasing the sample thickness has been found for all three systems considered.

3.3. Threshold and saturation voltages

Figures 3(*a*) and 3(*b*) illustrate the influence of the dose on V_{10} and V_{90} for selected 30 µm thick 2 wt % UVand EB-cured samples. The 2 wt % UV films exhibited continuously increasing V_{10} and V_{90} values if higher dose values were chosen. This effect has already been observed several times. For example, Lackner *et al.* applied different UV-dose values to the well-known reactive thiol-ene/E7 system, such that the UV intensity was kept constant, but the exposure time was varied [8]. As a result they found that an increase in the dose values leads to increasing threshold and saturation voltages. The authors explained this behaviour on the reduction of the droplet size with increasing UV-dose. Smaller droplets require higher fields for orientation due to their higher degree of curvature. For 30 µm thick EB-cured films, the variation of V_{10} and V_{90} as functions of dose exhibits a completely different behaviour compared with the results obtained from UV-cured films, see figure 3(b). The threshold and saturation voltages of samples cured with 60 and 74 kGy remain nearly constant at approximately 20 and 60 V, respectively. V_{10} and V_{90} change drastically if the dose is changed from 74 to 119 kGy. First V_{10} and V_{90} increase with the dose, passing through voltage maxima around 90 kGy. By further increasing the dose values, V_{10} and V_{90} decrease, reaching constant values around 120 kGy. At high doses, the final plateau values obtained for V_{10} and V_{90} are considerably higher than the constant values observed for low dose values.

Qualitatively, the behaviour found in our EB-cured samples can be compared with the results obtained by Nolan *et al.* [10]. These authors investigated the effects of UV-dose and curing temperature on V_{90} for the TL213/ PN393 (80:20) system. At a given temperature, V_{90} first increases slowly and then strongly with increasing UV-dose, before reaching a plateau value where further



Figure 3. (a) Influence of the UV dose on threshold and saturation voltages V_{10} and V_{90} for 30 µm thick PDLC films (2 wt % photoinitiator). The continuous lines are guides for the eye. (b) Effect of EB dose on the threshold and saturation voltages V_{10} and V_{90} for 30 µm thick PDLC films. The continuous lines are guides for the eye. increase of dose does not change the V_{90} value. This behaviour has also been observed for the EB-cured samples which have probably reached chemical stability for dose values exceeding 90 kGy. In order to confirm this hypothesis, additional samples were prepared using dose values of 120 and 132 kGy. The sample thickness was found to be equal, but smaller than 30 µm, so that these data were not included in figure 3 (b). However, the transmission versus voltage curves for these samples do not show a dependence on the dose: the electrooptical curves obtained were identical for 120 and 132 kGy, confirming our argument concerning chemical stability.

Some additional UV-curing experiments were performed at higher doses than those presented in figures 2(a) and 3(a). Our aim was to find out if the entire UV-dose range can be compared to the lower dose range between 60 and 90 kGy used for EB-curing. Several samples have been cured with dose values above 52 mJ cm^{-2} . In each case, increasing the dose value leads to an increase in the electrical field necessary to switch the sample into a transparent state. Constant values of V_{10} and V_{90} were not observed up to an applied voltage maximum of 260 V. These findings indicate that the UV-cured system will still be modified by applying higher dose values, and so a chemically stable state is not attained.

3.4. Comparison of the electro-optical properties

The extent of cure might strongly influence the electro-optical behaviour of the PDLC films prepared using UV- and EB-radiation. The conversion of the carbon–carbon double bond of the acrylate groups was monitored by Fourier transform infrared spectroscopy. For example, the same conversion value of 80% was obtained at approximately 60 kGy (EB) and at 22 mJ cm⁻² (UV 2 wt %) and 32 mJ cm⁻² for the UV 1 wt % system. The corresponding transmission versus voltage curves exhibit striking differences. The threshold voltages increase in the following order: EB, UV 2%, UV 1%; from 20 to 35 to 50 V, respectively. The saturation voltage for the UV 2 wt % system is twice as high as for the EB film, whereas the UV 1 wt % sample exhibits a three times greater value than the EB film.

In all cases, constant conversion values of 90-95% were obtained at dose values exceeding 45 mJ cm^{-2} for the UV systems and 90 kGy for the EB sample. These results show clearly that although approximately the same high conversion values were found for all systems considered here, the electro-optical properties are substantially different.

In order to understand the electro-optical behaviour of UV- and EB-cured samples, thermophysical investigations were carried out. Preliminary DSC measurements allowed us to obtain the glass transition temperature (T_s) of the polymer matrices, the nematic + isotropic/ isotropic (N + I/I) transition temperature, and the corresponding nematic-isotropic transition enthalpy. The results obtained so far indicate similar values for T_{e} around $T = 0^{\circ}$ C in all cases considered here. The fractional amount of LC entrapped in phase separated domains was evaluated at 0.7 for both UV systems. Only slightly increased values of 0.75 have been found for PDLC samples prepared by EB. Significant higher N + I/I transition temperatures were observed in the case of both UV systems compared with EB-cured samples. For EB films, a value of $T_{N+I/I} = 35^{\circ}C$ has been deduced, whereas $T_{N+I/I} = 47^{\circ}C$ was obtained for both UV systems. These results were confirmed by thermooptical studies. Assuming similar network architectures and sample morphologies for the two series of heterogeneous materials, these variations of $T_{N+I/I}$ could be explained by a denser polymer network in the case of UV. The crosslinking density is described by the number of monomer units separating two consecutive crosslinks $N_{\rm c}$. Recently, model calculations have shown that decreasing N_c values will increase the $T_{N+I/I}$ values for mixtures of crosslinked polymer matrices with low molecular mass LC [19]. A more dense network might lead to smaller LC domains which in turn require higher electric fields to align the LC molecules. It is evident that only idealized networks in model systems were considered in the theoretical framework. In particular, experimentally obtained networks might exhibit heterogeneities and therefore, the notion of averaged N_c values can lead to erroneous conclusions. Another interpretation of the results might be found in a change of the polymer volume fraction at crosslinking, in the network elasticity parameters α and β , or in the Flory–Huggins interaction parameter χ . Especially variation of α and β as functions of the irradiation conditions might explain the observed electro-optical behaviour.

3.5. *Memory effects*

For 2 wt % UV samples, figure 4 illustrates the variation of the transmission in the off-state as a function of the dose and voltage maximum applied. In our electro-optical experiments, the maximum of the voltage of the scan up/down cycles was chosen in the following consecutive order: 20, 50, 80, 100, 120, 150, 180, 200, 220 and 260 V, until a plateau value of the transmission had been observed. The values of T_{off} in figure 4 have been taken after the different voltage cycles were completed. This figure shows two different regions. Below a voltage maximum value of approximatel y 150 V, the same transmission values before and after applying the electrical field were obtained; for a given film, these data are identical compared with the $T_{off init}$ values. Above $V_{max} = 150$ V,



Figure 4. Memory effect: dependence of the transmission values in the off states (T_{off}) on the applied voltage maxima for UV-cured PDLC films (2 wt % photoinitiator). The continuous lines are guides for the eye.

the films exhibit continuously increasing $T_{\rm off}$ values in the field off-state by further increasing the voltage maximum. At the highest voltage maximum applied (260 V), the transmission values of the samples were found to be 1-10% higher than the corresponding $T_{\rm off\ init}$ values. In particular, no clear dependence of the curing conditions on this behaviour has been observed. Furthermore, only small variations of the sample thickness ($30 \pm 3 \mu m$) were found and cannot be applied to explain these data.

The 1 wt % UV films exhibit qualitatively the same behaviour. This behaviour is known as the memory effect and has been investigated previously [20-22]. This effect is generally interpreted using the fact that the LC molecules do not completely relax back to their initial scattering off-state, if a sufficiently high electric field has been applied. They remain partially aligned in the direction of the applied field even after it is removed. In this case, it can be assumed that the effective refractive index of this portion of the LC molecules is still close to the refractive index of the polymer matrix. The PDLC film conserves, therefore, to some extent its transparent state. The memory effect in our samples, however, is small compared with some results shown in the literature [20-22].

It was already mentioned that the UV-cured systems apparently do not reach a chemically stable state under the given radiation conditions, and this might be the cause of the memory effect. On the contrary, EB-cured films did not exhibit a dependence of the transmission on the applied voltage in the off-states. These results can also be explained by a change in the network elasticity parameters α and β for UV- and EB-cured films, which influence the relaxation phenomena of LC molecules upon application of an electrical field.

4. Conclusions

A comparative study of three series of PDLC samples prepared by UV- and EB-radiation using a PIPS process has been performed. The initial mixture made of LC E7 and a reactive blend of acrylate derivatives was employed for EB curing whereas different amounts of a photoinitiator were included in the case of UV exposure. PDLC samples prepared by using the same curing conditions and possessing similar thicknesses exhibit comparable and reproducible electro-optical properties. All films show low transmission values in the initial off-state and values higher than 80% in the on-state. The electro-optical behaviour strongly depends on the curing conditions. In particular, the influence of the dose on threshold and saturation voltages exhibits remarkable differences. For UV-cured films the threshold and saturation voltages continuously increase with the dose, whereas plateau values were reached for EB-cured samples. Furthermore, a memory effect has been observed only for the UVcured films. Apparently, chemical stability can be more easily achieved for EB-cured systems. In our experiments, EB-cured PDLC films show a better electro-optical performance compared with the UV-cured samples.

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