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TIME RELAXATION OF THE LIGHT TRANSMISSION OF PDLC FILMS

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The time relaxation of the optical transmission of polymer dispersed liquid crystals after application of an electric rectangular pulse is investigated. The films are made of tripropyleneglycoldiacrylate/E7 mixtures according to the Polymerization Induced Phase Separation (PIPS) process under Ultraviolet (UV) and Electron Beam (EB) curing. The light transmission is measured as a function of time assuming that the origin corresponds to the time where the external field is removed completely. The effects of EB dose and film thickness on the transmission are investigated.

Keywords: electron beam; liquid crystal; polymer; polymer dispersed liquid crystal; ultraviolet

INTRODUCTION

Most applications of Polymer Dispersed Liquid Crystal (PDLCs) are based upon electro-optical response functions [1–4]. Under normal mode conditions, these films are opaque at rest (no electric field applied) and become transparent when they are submitted to an electric field with sufficient amplitude. One of the major criteria for evaluating the performance of these films in terms of electro-optical responses is to achieve a high contrast ratio T_{ON}/T_{OFF} where T_{ON} is the maximum transmission under

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an electric field also called on-state transmission while T_{OFF} is the transmission in the absence of field designated by off-state transmission. This ratio should be as high as possible but commutation from on- to off-state should be instantaneous at least for the sensitivity of the naked eye. This is the reason why a study of the time relaxation of the optical transmission of PDLC films is of primary importance. Multimodal behaviour is found under various conditions in the dynamics of PDLC films. For example, the analysis of the absorbance in the case of PDLCs made of polyvinylacrylate and E7 containing dyes is found to decay according to two processes [1–3] when the exciting signal applied to the film is removed. This response shows a rapid decay followed by a slow process. In a recent investigation, we considered another problem by looking at the time evolution of light transmission across a tripopyleneglycoldiacrylate (TPGDA)/E7 film in the absence of electric field (off state) [5,6]. The purpose was to report results on the relaxation of T_{OFF} for films prepared *in situ* using a combination of Polymerization Induced Phase Separation (PIPS) and Electron-Beam (EB) curing [7–9]. These results show also bimodal decay processes which were analysed in terms of early and late stage phase separation processes. Here, we consider the time relaxation of optical transmission of similar films undergoing a rectangular electric field pulse. We examine the effects of film thickness, radiation dose and make a brief comparison of EB and UV cured systems.

EXPERIMENTAL PART

Materials and Sample Preparation

The eutectic mixture of cyanoparaphenylene derivatives known as E7 (Merck) was used in this work. Tripropyleneglycoldiacrylate (TPGDA) monomer was obtained from Cray Valley (France). A precursor mixture containing 30 weight-% (wt-%) of monomer and 70 wt-% of LC was cured using EB-radiation. Samples for optical studies were prepared by sandwiching the initial reactive mixture between a glass plate (Balzers, Liechtenstein) and a Polyethyleneterephthalate sheet (Renker, Germany), both coated with a thin transparent layer of conducting indium/tin oxide. The EB generator was an Electrocurtain Model CB 150 (Energy Sciences Inc). delivering a high voltage of 175 kV. The samples prepared as mentioned above were placed in a tray which was passed under the radiation source on a conveyor belt. They were exposed to a selected dose expressed in units of kGy (1 kGy = 1J/g) by changing the beam current at constant conveyor speed. This current was changed from 4 to 7 mA by steps of 1 mA. For example 7 mA corresponded in this case to a dose of 105 kGy. Several duplicate samples were prepared to check the reliability of the results.

UV-cured films under similar conditions were also prepared as a brief example of comparison between UV- and EB-cured samples. In UV curing, 2 wt-% (of the acrylate mixture) of Darocur 1173 (Merck, Germany) known as an efficient photoinitiator was added. The UV light source used was a Minicure Model MC4-300 (Primarc UV technology) equipped with a medium pressure mercury arc lamp rated 80 W per cm. The samples were placed on a conveyor belt. Results corresponding to 150 mJ/cm^2 are given.

The film thickness was measured by a micrometer calliper (Mitutoyo; uncertainity: $\pm 1\,\mu m$). No temperature control during the irradiation processes has been performed.

Optical Measurements

The optical measurements were made at room temperature by recording the transmission of unpolarized HeNe laser light ($\lambda = 632.8\,nm$) at normal incidence to the film. The transmission measurements were corrected using appropriate calibration standards. Measurements of transmission were made as a function of time starting with 30 s in the initial off-state T_{OFF} followed by a period of 60 s in the on-state (transmission at its maximum). The counting relevant for this paper started at the time where the electric field rectangular pulse ceased and have been followed during 60 s. This procedure was repeated for films of different thicknesses and cured under different EB doses. The frequency of the applied signal was maintained at 145 Hz while its amplitude was changed in such a way that it corresponded to the maximum transmission (T_{ON} constant) for each film thickness.

RESULTS AND DISCUSSION

In this work, we pursue our investigation of the TPGDA/E7 system with composition 30 wt-% TPGDA and 70 wt-% E7. The intensity of transmitted light at right angle was recorded as a function of time starting at the time where the applied electric field is removed. Data are taken at room temperature ($\sim 20^{\circ}C$) for different film thicknesses and EB doses. These effects are of primary importance in the evaluation of the time relaxations of LC in electro-optical applications [1,2]. Figure 1 represents data taken on samples prepared under different EB doses. One finds 2 distinct relaxation processes. The curves are plotted in the descending order of the EB dose meaning that the relaxation is faster as the dose is increased. However this phenomenon is somewhat modulated. If the applied dose is increased from 4 to 5 mA, the relaxation speeds up remarkably whereas dose enhancement from 5 to 7 mA yields only a moderate acceleration of relaxation. The relaxation hardly changes above 6 mA. These curves could be

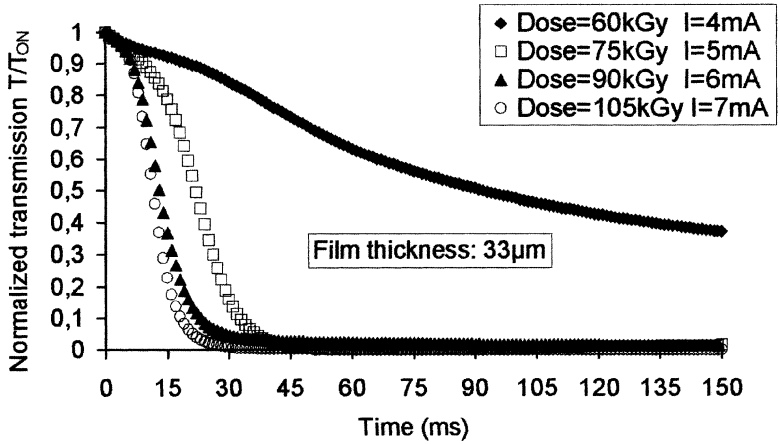

FIGURE 1 T/T_{ON} versus time for TPGDA(30 wt-%)/E7(70 wt-%) EB-cured PDLC films at different doses. In the descending order, doses are 60, 75, 90 and 105 kGy. The electric field applied corresponds to a rectangular pulse lasting 60 s at the voltage applied and the origin corresponds to the time where the field is removed.

fitted with a double exponential function such as $T/T_{ON} = A_f \exp(-w_f t) + A_s \exp(-w_s t)$ where the A's and w's represent the amplitudes and frequencies of the modes, the subscripts s and f refer to slow and fast processes. A more detailed analysis of these parameters is postponed to a future work.

The present results could be useful in exploring the physical properties of the material under investigation and the kinetics of phase separation. For example, by analyzing the results under different films thicknesses, one could extract the cross-section σ_s and the number of scattering centers N_s using a formula of the type $T/T_{ON} = \exp(-N_s \sigma_s d)$ where d is the film thickness. It is clear that the time dependence of T/T_{ON} and concomittent relaxation processes should be quite sensitive to the film thickness. One would expect that the light transmission drops exponentially as the film becomes thicker and the scattering of light should be equally enhanced. In order to confirm those tendencies, we plot in Figure 2 the variation of T/T_{ON} versus time for samples of different thicknesses. At short times, the effects of film thickness are not clear since the curves do not show a clear tendency in terms of the thickness. At longer times, the decay of T/T_{ON} is faster as the thickness increases. The example shown in this figure corresponds to a film cured at the EB-dose of 90 kGy (6 mA) but these observations were found for other doses as well.

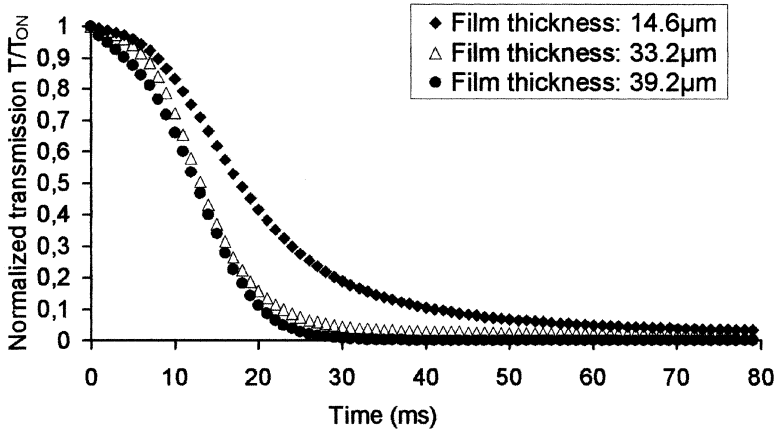

FIGURE 2 T/T_{ON} versus time for EB cured PDLC films with different thicknesses (dose 90 kGy).

In order to get an idea on the relationship between relaxations and the method of film preparation, we give in Figure 3 two typical examples, one dealing with an EB-cured film and the other with UV-cured film under similar conditions. The film thickness and the radiation dose were chosen in such a way that the comparison is meaningful. The frequency of the applied voltage was fixed at 145 Hz and its amplitude chosen to give a constant value of T_{ON}. Doses of 105 kGy for the EB and 150 J/cm^2 for the UV source corresponding to a maximum conversion of double bonds were used with

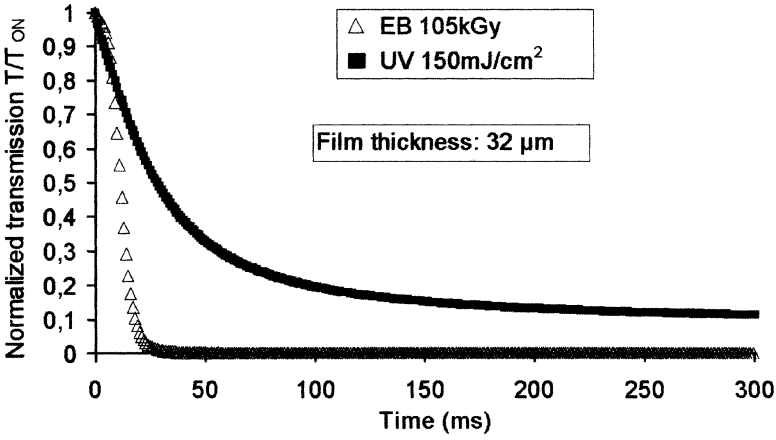

FIGURE 3 T/T_{ON} versus time for UV and EB cured PDLC films.

only a slight difference between the two cases. The results shown here are obtained for films having the same thickness although this is not crucial as long as the thickness does not deviate too much from this value. Clearly, relaxation of the EB-cured film is faster. This is consistent with the general conclusions indicated in the literature [8,9] comparing electro-optical responses of UV- and EB-cured PDLC films. These authors observed that EB-curing gives films with substantial advantages in terms of the characteristic voltages and the contrast ratios. The electron beam is more uniform over the whole sample with a higher penetration depth and a better efficiency in the double bond conversion. Figure 3 shows that the relaxation time after an electric pulse is shorter for EB-cured systems. For example at $t = 50$ ms transmission of the EB-cured system has completely decayed to zero while UV system exhibits a transmission ratio of 30%. Note that the curve of the UV-cured material decays according to a single exponential process whereas the corresponding EB system has at least 2 decay modes.

CONCLUSIONS

The time decay of light transmission of EB-cured PDLC films is analyzed in terms of the film thickness and the dose of curing radiation. Two processes are identified exhibiting a faster decay as the dose of radiation curing increases. At long times, the relaxation speeds up for thicker films. But, at short times, there is no systematic tendency since some of the curves with different thicknesses cross each other in time. In order to elucidate these tendencies additional measurements are needed exploring other parameters such as the temperature of curing and the study of rise times together with decay rates. Comparison of EB- and UV-cured materials indicates that the EB curing option yields materials with uniform droplet sizes characterized by faster relaxation rates. Hence, EB-cured PDLC films are better candidates for electro-optical applications consistent with the conclusions reached earlier [8,9].

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