Polymer-Dispersed Liquid Crystal Materials Prepared by Electron-Beam-Initiated Polymerization

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SYNOPSIS

Polymer-dispersed liquid crystal (PDLC) films were prepared using electron beam radiation by a polymerization-induced phase separation process. The composite material was obtained from a blend including a polyester acrylate and the nematic liquid crystal mixture E7. The electro-optic properties of the PDLC films were investigated as a function of film thickness. The obtained transmission vs. voltage curves exhibit a good reproducibility, low threshold voltages, and a high transmission in the on state. © 1995 John Wiley & Sons, Inc.

INTRODUCTION

Polymer-dispersed liquid crystals (PDLCs) are promising new materials for electro-optic applications such as flexible displays and light shutter devices.[1-5] In their most common form, PDLC films consist of low molecular weight nematic liquid crystalline microdroplets dispersed in a solid polymer matrix. These films can be switched electrically from a light scattering off state to a highly transparent on state.

The principle of the electro-optic operation of a normal mode PDLC film (which is opaque in the field off state and transparent in the field on state) is illustrated in Figure 1. The film is sandwiched between transparent conducting electrodes. Because of the strong multiple scattering arising mainly from the refractive index mismatch between the liquid crystal and the polymer matrix, the film appears opaque in the field off state.

When an AC voltage is applied to the electrodes, the resulting electric field aligns the liquid crystal droplets along the field direction. In this case, light incident normal to the film probes, essentially, the ordinary refractive index n_o of the liquid crystal in the droplets. The film appears transparent if the refractive index of the matrix is equal to n_o. Upon removal of the voltage, the droplets return to their original scattering state.

One basic method for the preparation of PDLC films is the formation of microdroplets of liquid crystals in a polymer matrix by a phase separation process.[6] This can be either achieved by thermally activated polycondensation[6] or by radiation-initiated polymerization of a mixture including polymer precursors and liquid crystals (see, for example, Ref. 4).

The use of ultraviolet (UV) light to provoke at will polymerization-induced phase separation (PIPS) was revealed as a most convenient method, offering various advantages of practical concern.[5a,7] In addition, the relative temperature independence of the initiation step allows the thermal control of other important parameters to be freely achieved.[8] The versatility of the UV technique also permits playing with light intensity to control the rate of initiation.[9] It is also possible to cure the matrix with prealignment of the dispersed LC phase upon simultaneous application of mechanical stress, of a magnetic or of an electric field.[10]

A detailed analysis of the chemistry in terms of reactive systems and related treatments reveals that, apart the very conventional thiol-ene system still used by the greatest number of authors, little work is done to develop rationally new systems inducing matrix formation with adequate phase separation. Urethane acrylate prepolymers, nevertheless, appear as an alternative chemistry to the original sys-
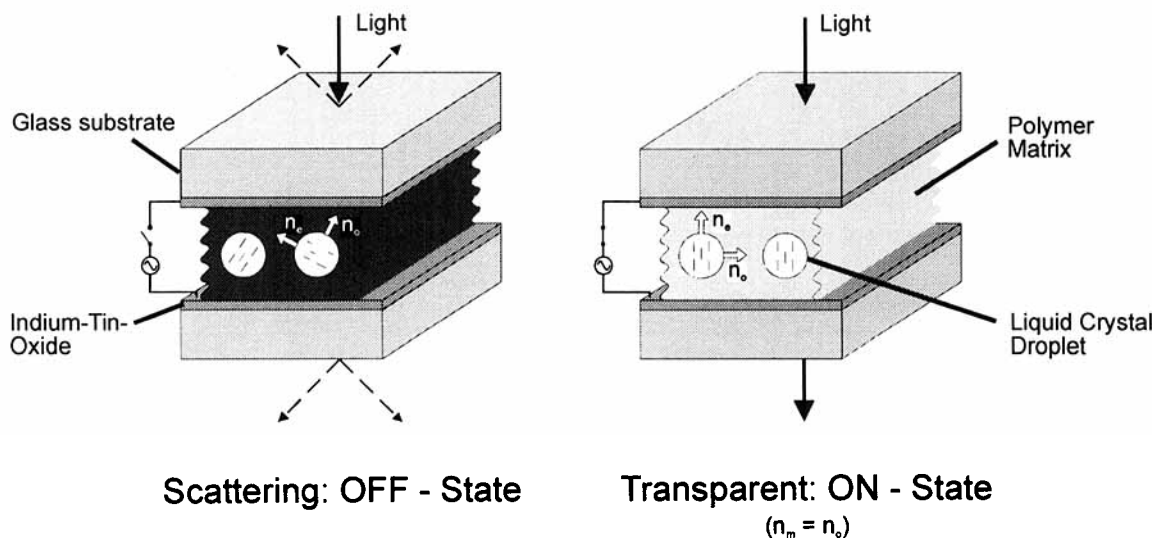
Figure 1 Principle of nematic normal mode PDLC light shutter operation (n_e = extraordinary refractive index, n_o = ordinary refractive index, n_m = refractive index of the polymer matrix).

tem.[11,12] A limited number of authors have used unsaturated polyesters.[13,14] Structure–properties relations, with special emphasis on the chemical parameters, are lacking in spite of some efforts focussed recently on this problem.[11,13]

A major investigation in this connection is disclosed in a patent.[15] Interestingly, the use of multifunctional thiol associated in high concentration with acrylate prepolymers and diluents in the photosensitized composition permits achieving with UV irradiation a sort of step-wise growth of the network. Substantial transfer during the radical polymerization is obviously expected to play a major role in the construction of the polymer matrix.

As a part of our current projects in the field of radiation curing of polymers, we have undertaken a study aiming at the improvement of PDLCs performances by adjusting the nature of the polymer precursors and by proper control of the treatments. The preparation of PDLC films by electron beam (EB) processing of polymerizable mixtures was described in detail some years ago.[16a,b] Though the materials obtained from the conventional thiol-ene system afforded good results, comparing well to the UV cured analogs, the evaluation of the electro-optical properties of the switchable devices indicate poor to only fair results when acrylic functional compositions were used as polymer precursor.[16c]

The present article deals with the preparation by EB processing and with the characterization of PDLC films, special emphasis being paid to the good control of experimental procedures and to the effect of thickness on the film properties. EB as well as UV radiation, indeed, interacts with substrates by frontal exposure, which can cause gradient effects altering the film performances. The results presented thereafter were obtained from a polyester acrylate chosen because it was shown to present good aptitude to index matching.[17] The transmission properties have been analyzed as a function of voltage and frequency of the applied AC voltage.

EXPERIMENTAL

Materials

The liquid crystal mixture E7 (Merck Ltd, Poole, UK) was used during this work, which shows at $T = 20°C$ a positive dielectric anisotropy $\Delta\varepsilon = 13.8$ ($\varepsilon_{||} = 19.0$) at a frequency of 1 kHz. The refractive indices of E7 at $T = 20°C$ are given as: $n_o = 1.5183$, $n_e = 1.7378$ ($\lambda = 632.8$ nm) and $n_o = 1.5189$, $n_e = 1.7395$ ($\lambda = 625$ nm).[18] This leads to birefringences of $\Delta n = 0.2195$ ($\lambda = 632.8$ nm) and $\Delta n = 0.2206$ ($\lambda = 625$ nm).[19] The "prepolymer" chosen consist of an aromatic polyester acrylate diluted in additional monomers (Rahn AG, Zürich, Switzerland). The refractive index of the prepolymer in the cured state is $n_p = 1.5120$ ($\lambda = 632.8$ nm).[17] The glass transition temperature of the cured prepolymer revealed by DSC studies was evaluated to $T_g = 46°C$.[20]

Preparation of PDLC Films

All steps to obtain our PDLC films were performed at room temperature. The prepolymer and the liquid crystals were mixed together in the ratio 1 : 1 by weight until the mixture became homogeneous. The solutions were cast on a flat glass plate coated with a thin transparent layer of conducting indium-tin-oxide (ITO) (Baltracon 257 645 BO from Balzers Ltd, Balzers, Fürstentum Liechtenstein; $R < 20$ ohms/square). The thickness and the uniform application of the mixture was controlled by using bar coaters (Braive, Liège, Belgium). Three bar coaters were used, which allowed the application of the mixed liquid homogeneous composition with thicknesses of 25 μm, 35 μm, and 50 μm. For each thickness, two samples were prepared by coating the prepolymer/liquid crystal mixture on the ITO-covered glass plates. The so-prepared six samples were exposed to the electron beam irradiation to cure the polymerizable compounds.

Electron Beam Curing

The electron beam cure technique was employed in order to prepare the PDLC samples by a PIPS process. The generator used in our experiments was an Electrocurtain Model CB 150 (Energy Sciences Inc.), with an operating high voltage of 175 kV.

The sample placed in a nitrogen atmosphere is submitted to the accelerated electron curtain. The conveyor entrance was equipped with a reverse nitrogen stream to avoid changes of the inertization conditions. It has been shown in our experiments and in several other reports (see, for example, Ref. 21) that the polymerization reactions are not affected if the oxygen content in the processing zone is less than 200 ppm (typically between 130 and 160 ppm oxygen for the present irradiations).

The glass plates, prepared as mentioned above, were placed in a sample tray. This tray was passed under the electron curtain on a conveyor belt. The amount of energy required to cure products is called the dose. In our experiments, the dose applied was 60 kGy (6 MRad). This was achieved by using a beam current of 4 mA and a conveyor speed of 43 feet/min (0.22 m/s). These values have not been changed during our experiments in order to apply the same curing conditions each time. No temperature control during the irradiation process has been performed.

Because our sample thickness did not exceed 50 μm, the dose was essentially delivered in an uniform way. It has been checked by additional FTIR ex-

periments that the amount of remaining polymerisable double bonds after the polymerization process was negligible.[22]

Film thicknesses were measured with a micrometer caliper (Mitutoyo; uncertainty: $\pm$ 1 μm). To minimize errors in the determination of film thickness, 18 different places on each film were taken into account.

Electro-Optic Measurements

The electro-optic experiments were carried out at room temperature using a Cary 219 spectrophotometer (Varian Associates). In order to compare our results to those published in the literature (most experiments were performed with the wavelength of a HeNe Laser at λ = 632.8 nm) and for improving the accuracy of the transmission measurements with our spectrophotometer, the wavelength was set at λ = 625 nm, with a bandwidth of 3 nm. A diaphragm of 3 mm diameter was placed across the beam just in front of the sample to limit the surface of the analyzed area.

The cured PDLC film on the glass plate was sandwiched by another ITO-coated glass substrate, which was separately exposed at the same time to the same dose. Electric contacts were installed previously by applying an electroconductive adhesive (E 206, Epotecny, Vélizy, France). The output of a frequency generator was amplified and used to drive the shutter device.

The transmission properties of the PDLC cells were measured by passing the light through the cells normal to the film surface. The beam intensity with a sample containing of two ITO-coated glass plates that were irradiated at the same time with the same dose as the PDLC samples corresponds to 100% transmission. This procedure is necessary to correct the measurements from the loss of transparency that results from darkening of the glass plates upon EB irradiation.

A typical example of the voltage applied to the cells as a function of time is shown in Figure 2. Starting from the electrical off state, the applied voltage is increased in steps of 5 V until the transmission remains nearly constant. During each step, the voltage was kept constant for 1 min. After the first scan up cycle, the voltage is decreased in the same way (first scan down cycle) before the same procedure was repeated (scan 2 up, scan 2 down). In some cases, a third scan cycle was accomplished. Between two complete cycles, the cells were kept for a period of 5 min in the field off state. The transmission values taken for the electro-optic curves are
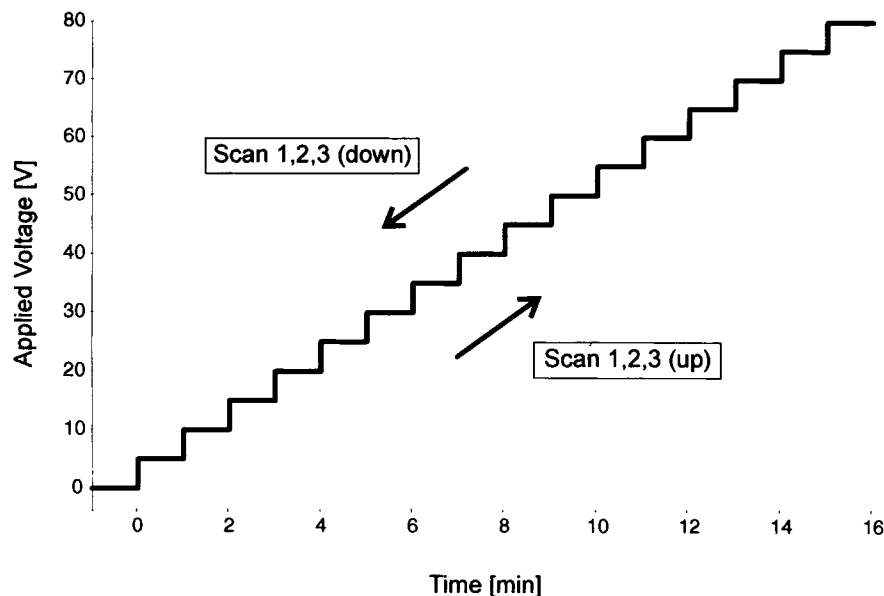
Figure 2   Step-wise variations of the applied voltage scans driving the electro-optic response.

normally those obtained directly before changing the voltage. To obtain reproducible and comparable results, the whole procedure was applied to each of the PDLC cells.

RESULTS AND DISCUSSION

PDLC Film Preparation

Some preliminary curing experiments with several possible compositions of different starting materials have been carried out at first. It was found that a 1 : 1 Polyesteracrylate/Monomer : E7 mixture applied on an ITO-coated glass plate exhibited an immediately and uniform white milky appearance after EB curing.

The conditions of the preparation of the PDLC films are of prime importance in order to get comparable electro-optic responses of the corresponding cells. All parameters concerning the sample preparation were kept constant, i.e., mixing of the components, application of the homogeneous mixture on the glasses, sample curing, electro-optic treatment. Three pairs of individually prepared PDLC samples of the same original composition were obtained by applying the same film preparation conditions (see Experimental part).

The results of the measurements of the film thicknesses are included in Table I. It was confirmed that the thicknesses of the two films of one pair were close together. The measured values were reduced by roughly 30% compared to the liquid state due to the crosslinking processes. Micrometer mea-

Table I   Characteristic Electro-Optic Properties of the Six PDLC Films Investigated

Sample	Film Thickness (μm)	T_0 (%)	T_{10} (%)	T_{90} (%)	T_{100} (%)	$\Delta T = T_{100}-T_0$ (%)	V_{10}	V_{90}	$\Delta V = V_{90}-V_{10}$	V_{H50}[a]
1	13	44.6	49.8	91.4	96.6	52.0	5.3	37.1	31.8	2
2	17	49.3	53.8	90.1	94.6	45.3	5.6	39.7	34.1	3.5
3	23	38.9	44.7	91.5	97.3	58.4	7.5	50.4	42.9	/
4	24	42.3	47.6	90	95.3	53.0	7.7	48.3	40.6	5.2
5	33	25.5	31.1	75.6	81.2	55.7	10.4	53.6	43.2	6.4
6	37	24.2	30.4	80.4	86.6	62.4	12.0	60.3	48.3	/

[a] From the third decreasing scan down half-cycle.

surements indicated variations of the film thickness within the range of ± 2 μm. This is probably due to the mechanical application of the liquid crystal/polymerisable materials.

Electro-Optic Response

Figure 3 shows representative results of the transmission properties of a 33 μm thick PDLC film as a function of applied voltage. The frequency of the sinusoidal voltage was 100 Hz. The transmission of this example as well as of all other films investigated increases with increasing voltage until a horizontal plateau is reached. A further increase in the voltage does not significantly change the transmission. Two different additional effects could be observed: a small "memory" phenomenon and a hysteresis effect.

The films investigated showed a higher transmission in the field off state after the first voltage scan cycle was completed as compared to the initial off state. After the second and third voltage scan cycles, the same transmission values as after the first cycle were obtained in the field off state. The observation that a transparency is preserved after the voltage is removed is described in literature as memory effect.[11,23-25] Applications of this effect to thermally and/or electrically addressable, and erasable display devices have been proposed.[11,24]

This effect indicates that the liquid crystal molecules did not completely relax back in their original scattering state. They partially tend to maintain their alignment in the direction of the applied field even after the field is removed. In this case, it can be assumed that the effective refractive index of this part of the liquid crystals is still close to the polymer

matrix. The PDLC film conserves, therefore, to some extent its transparent state. The memory effect observed in our samples, however, is small compared to some results shown in the literature.[23]

A hysteresis effect commonly associated with PDLC displays[26-30] was found in all transmission vs. voltage curves of our samples: in an increasing electric field, the transmission at a specified voltage is lower than the transmission at the same voltage in a decreasing electric field. This phenomenon can be explained by a difference of a two-rate process:[29] the liquid crystal directors align during the increasing scan up voltage half-cycle with the electric field, depending on the driving voltage. During the decreasing scan down voltage half-cycle, the rate at which the directors return to their original alignment is a diffusion-controlled process. Surface interactions occurring at the polymer/microdroplet boundary determine this second process: liquid crystal molecules that are next to the polymer interface respond slower to a change in the electric field than molecules that are located further from these interfaces. This difference might be the cause of the hysteresis effect observed in PDLC materials.

The effect of sweeping time on the hysteresis has been studied by several authors.[27-29] It was found that the hysteresis decreases or even disappears with increasing cycle time. In a few cases, however, there is some evidence that the hysteresis effects are observable even in relatively slow up and down voltage scans.[27,29] The largest time scale used in ref. 29 was 4 min and 16 s for one complete continuous voltage cycle, whereas our (discontinuous) voltage sweeps took at least 24 min (highest voltage applied in this case: 60 V). It is evident that our results cannot be
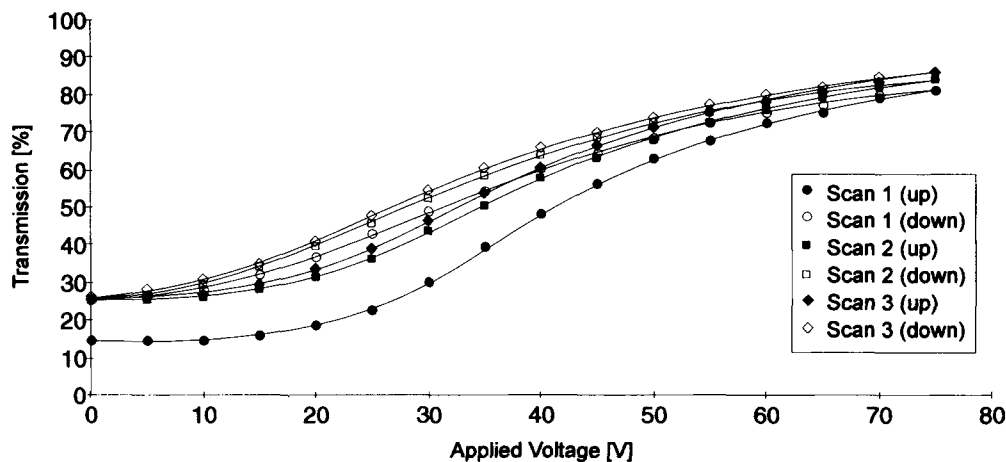

Figure 3 Electro-optic response of a 33 μm thick PDLC film taken in repeated increasing and decreasing voltage scans (λ = 625 nm, ν = 100 Hz).

easily compared with the previously mentioned investigations. Nevertheless, the transmission data presented here show an hysteresis effect even at slow voltage scans.

Effect of Thickness on Transmission Curves

The electro-optic transmission curves of the first decreasing scan down half-cycle of six prepared PDLC cells with film thicknesses in the range of 10 to 40 μm are presented in Figure 4. It can be seen that the above-mentioned pairs of films possessing thicknesses that are close together exhibit nearly the same transmission vs. voltage curves. Thus, in spite of the observed memory and hysteresis effects, the electro-optic results can be considered as reproducible.

To compare our results with those reported in the literature, efforts were made to find a general procedure concerning the presentation of electro-optic properties of PDLC films. For typical display applications, the on and off states can be characterized by the points $(V_{OFF}, T_{OFF}) = (V_{10}, T_{10})$ and $(V_{ON}, T_{ON}) = (V_{90}, T_{90})$.[31,32] Hereby, it is assumed that a PDLC film is in its on state if the transmission is at least at 90% of its maximum value. The off state is achieved if the transmission is at or below 10% of its maximum value. The value V_{10} is also known as threshold voltage, whereas V_{90} is called saturation voltage. The hysteresis width is described by the value V_{H50}, which can be defined by the difference of the voltages of the respective increasing and decreasing half-cycles at a transmission of 50%. The results obtained from the first decreasing scan down half cycle (V_{10}, V_{90}, $\Delta V = V_{90} - V_{10}$, V_{H50},

T_0, T_{10}, T_{90}, T_{100}, $\Delta T = T_{100} - T_0$) are gathered in Table I.

An increase in the film thickness results in an increase in the applied voltage necessary for its electro-optic activation.[33-35] This effect is shown in Figure 5, where the threshold voltage V_{10} and the saturation voltage V_{90} are plotted against the thickness of the films. As expected, the dependence of the applied voltage on the film thickness leads to a linear relationship. This result is consistent with the various relations that express for V_{10} and V_{90} a direct proportionality with the film thickness whatever the droplet configuration.[1] Additionally, the difference $\Delta V = V_{90} - V_{10}$ increases with increasing film thickness.

Transmission Properties in the On and Off States

For a collection of droplets that scatter independently, the intensity of a collimated light source through a PDLC sample can be expressed by writing[36-39]

$$I_T = I_0 \exp(-\alpha'd) \tag{1}$$

where I_T is the transmitted intensity, I_0 the incident intensity, and d the optical path length through the sample. In the case of normally incident light, d is considered as equal to the PDLC thickness. The scattering coefficient α' can be written as

$$\alpha' = N_v\sigma \tag{2}$$

where σ denotes the film averaged scattering cross section of a single droplet and N_v the number of

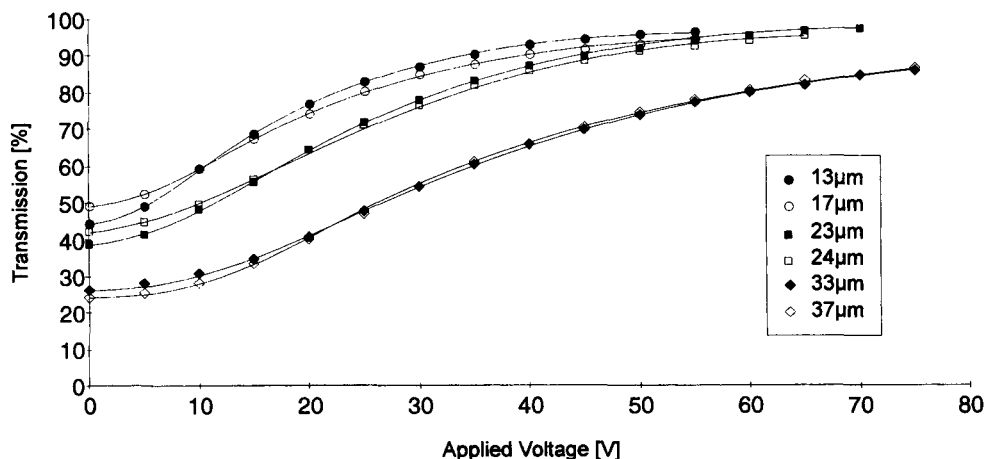**Figure 4** Effect of thickness on transmission curves of PDLC films (voltage scan 1 (down)) (λ = 625 nm, ν = 100 Hz).
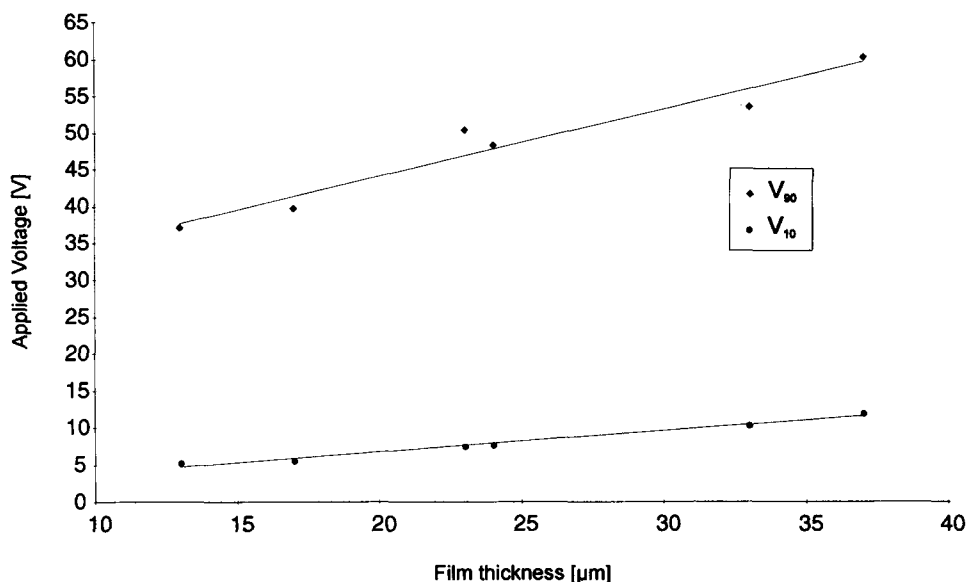
Figure 5 Threshold voltage V_{10} and saturation voltage V_{90} as a function of PDLC film thickness (λ = 625 nm, ν = 100 Hz).

droplets per unit volume.[40] N_v depends on the radius of each droplet and the volume fraction occupied by the droplets. The relationship between I_T and I_0 [eq. (1)] is similar to the Lambert–Beer law used in light absorbing systems.

The transmission T = I_T/I_0 can be expressed by

$$\log T = \log(I_T/I_0) = -\alpha d \qquad (3)$$

as a function of thickness of the films.[41] For a series of PDLC films with identical droplet distributions and droplet densities, but different thicknesses, the logarithm of the transmission would be expected to vary linearly with d.

The scattering cross-section σ is affected by the birefringence of the liquid crystals and the refractive index mismatch between the refractive indices of the liquid crystals and the polymer matrix. It is evident that the birefringence has a much stronger effect on the scattering behavior in the field off state as compared to the field on state. In the field off state, the refractive index mismatch Δn_{mis} can be calculated from the difference of the averaged refractive index $<n>$

$$\left\langle n \right\rangle^2 = \tfrac{1}{3}(n_e^2 + 2n_o^2) \qquad (4)$$

and the refractive index of the polymer matrix n_m:

$$\Delta n_{mis}^{off} = |\left\langle n \right\rangle - n_m| \qquad (5)$$

In the case of the field on state, $<n>$ in eq. (5) can be replaced by the ordinary refractive index n_o:

$$\Delta n_{mis}^{on} = |n_o - n_m| \qquad (6)$$

To illustrate the effect of the refractive index mismatch on the scattering behavior, simple calculations based on eqs. (4) and (5) were carried out. Assuming that the contribution of the remaining dissolved liquid crystal to the refractive index of the matrix is small, thus, $n_m = n_p$, $\Delta n_{mis}^{off} = 0.08$ and $\Delta n_{mis}^{on} = 0.0063$ were obtained. These values can already describe the difference between the slopes of the regression curves shown in Figure 6. This presentation illustrates a linear behavior of log T as a function of d in the case of the electrical on state T(V_{90}) as well as for the different off states [T_{OFF} (initial) and T_{OFF} (scan down cycle 1, 2)]. In the on state, Δn_{mis}^{on} contributes to a small value for the slope in eq. (3). The regression curves shown in Figure 6 corresponding to the off states are characterized by higher values for the slope due to the larger refractive index mismatch. It should be noted that the scattering coefficient α does not change between the different off states during the repeated voltage scan cycles. In particular, the slope in eq. (3) for the initial off state gives the same value compared to the subsequent off states.

Figure 6 also shows that the on state is characterized by transmission values always above 75% so that multiple scattering effects can be neglected.
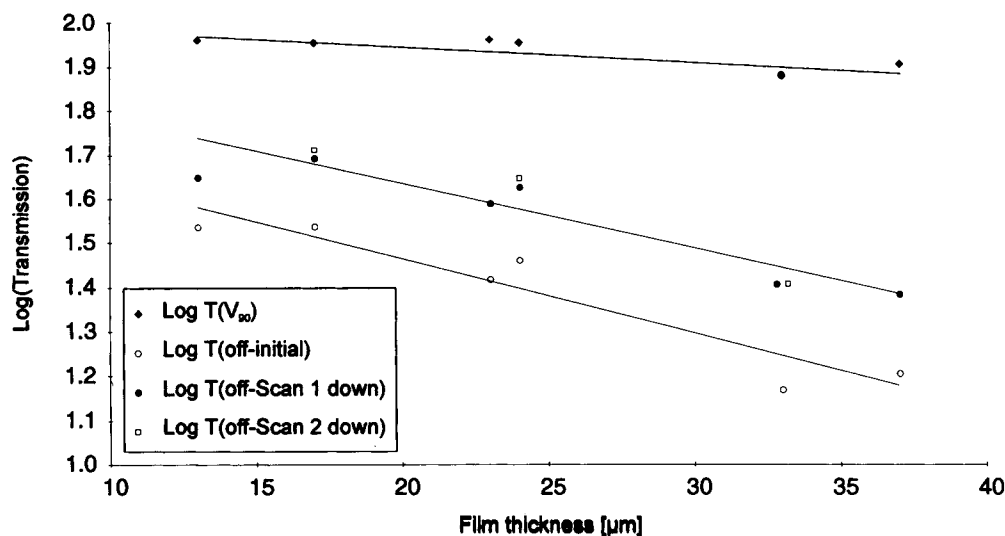
Figure 6 Effect of PDLC film thickness on transmission properties (off and on states)
(λ = 625 nm, ν = 100 Hz).

Because multiple scattering is considered as a principal cause for opacity in the field off state, a non-linear reponse is expected if the other parameters in eq. (3) remain unchanged. Apparently, this is not the case in our experiments. It is assumed that the transmission values in the off states are still too important to influence considerably the presentation of data in Figure 6.

CONCLUSION

PDLC films were successfully prepared by a phase separation process initiated by electron beam radiation. Electron beam processing is confirmed as a promising technique offering various advantages: initiation of the process at desired temperature, high conversion of monomers accessible, controlled degree of crosslinking, adjustable cure rate. Compared with the PIPS process initiated by UV light, electron beam curing has the advantage of not requiring the presence of a photoinitiator, which is detrimental to the PDLC film performances[42] and to long-term aging.

The electro-optic properties of the obtained PDLC films based on a selected representative composition have been investigated as a function of film thickness. In spite of the small memory and hysteresis effects, PDLC samples possessing similar thicknesses exhibit comparable and reproductible electro-optic response. The obtained transmission vs. voltage curves are characterized by low threshold voltages and high transmissions in the field on state. Further characterization of the PDLC materials as

well as the extension of the use of acrylate prepolymers is currently in progress.

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