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ORIGINAL PAPER

Study on the effects of isotropic cross-linked pristine morphology and electro-optical properties of PDLC films

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Abstract PDLC films were prepared by polymerization-induced phase separation (PIPS) method along with varying composition ratio between curable epoxy monomers/poly-oxy-propylene-di-amine (POPDA) cross-linking agent/Class α, longitudinal nematic liquid crystal (LC) linear chain polymer mixtures and their optical and pristine morphological properties have been analyzed to investigate their suitability as aligned nematic phase of LC. The refractive index of isotropic cross-linked polymer network could be influenced by the relative content of curable monomers structure PPGDE ~380 and PPGDE ~640 which increased the LC droplet size with POPDA. Meanwhile, it is examined that the decreasing of peel strength and transmittance of all the samples decrease of wavelength in the wavelength range of 300–800 nm due to the lower cross-linking density of the polymer network with effect of strongly influenced by the alkoxy (R-O) and flexible chain length of curable monomers, which also affect the electro-optical properties and droplet size of LC in PDLC films. Our results demonstrate that the LC droplet size of the isotropic cross-linked polymer network could be regulated by adjusting the monomers structure, composition ratio, relative

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wt% of POPDA, PPGDE ∼380 and PPGDE ∼640; then aligned nematic phase of LC have the optical properties of uniaxial crystals and their potential applications for PDLC smart glass market and display fields.

Keywords Polymer-dispersed liquid crystal · Epoxy monomers · Refractive index · Isotropic cross-linked · Morphology

Introduction

Polymer-dispersed liquid crystals (PDLCs) represent an important class of materials with several unique electro-optical (E-O) applications such as intimacy windows, digital signage, different light modulators, complex billboards, fiber optical devices and public display due to high transmittance. Given their advantages, PDLC films have been the subject of much academic and industrial research in the past decades due to good E-O properties, simple manufacturing process and long-term durability. Literature survey on this subject shows that when an electric field is applied to the PDLC film, the nematic LC in the droplets reorient so that the director is parallel to the field, and therefore perpendicular to the plane of the film [1]. If the ordinary refractive index (n_o) of the liquid crystal is matched to the refractive index of the polymer (n_p), then light incident normal to the film does not encounter any variation in refractive index and passes through the film without being scattered. Such PDLC films are therefore opaque in the off state but become clear when a voltage is applied [2, 3]. On the other hand, removal of the electric field, the anchoring forces between the liquid crystal and the polymer droplet walls, acts to restore the LC molecules to their original orientation and the film once again becomes scatter. Usually, the E-O properties of PDLC films can be affected by the LC concentration, film thickness, composition ratio of monomers and morphology of the LC domain size [4]. PDLC films can be prepared by five different techniques: thermally induced phase separation (TIPS), polymerization-induced phase separation (PIPS), solvent-induced phase separation (SIPS), frontal polymerization-induced phase separation (FPIPS) and microencapsulation process (MP). Specifically, the most convenient method to prepare PDLC devices is the PIPS method via heat-curing technique, is more reliable to provide homogeneity, simple processing, less pollution, cheaper, strong bonding power and forms a uniform morphology of polymer networks [5–9]. For instance, the epoxy monomers have own significant features such as high-quality moisture, thermal stability, high cross-linked densities, high mechanical strength and exceptional adhesion to a variety of substrates [10]. To describe the light scattering properties and the transparency of a PDLC film, the concept of an effective refractive index (n_{eff}) was introduced by Wu et al. [11]. According to Wu et al., the refractive index of bipolar droplets in a PDLC films is greater than the refractive index of a uniform director configuration. Kashima et al. examined the influence of cross-linking agents on the morphology and E-O performances of PDLC films [12]. Previously, we have studied two groups with influence of the multi-functional epoxy monomers structure and characterisation on the E-O properties and morphology of PDLC films [13, 14]. Having all these in view, the present work is concerned with research of epoxy monomers and cross-linked agent to investigate briefly the influence of isotropic cross-linked polymer network which effects on the pristine morphology and E-O properties of PDLC films. Meanwhile, this paper will illustrate refractive index of the polymer matrix decreased with increasing the chain length of curable monomers, which effect the reduced mismatching between refractive index of the polymer (n_p) and ordinary refractive index (n_o); thus, on-state transmittance is optimized.

In addition, the goal of the present contribution is to report an example of fabrication method using epoxy monomers structure that reinforces the stability and strength of the PDLC films. The epoxy polymer network structure formed which depends on the properties of initial reacting resin-curing agent [15]. Moreover, with Class α, longitudinal nematic liquid crystal (LC) mixture microstructure and the refractive index of isotropically distributed cross-linked polymer networks could be influenced by the relative content of epoxy monomers, owing to their different chemical structures.

Experimental methods

Material

The Class α, longitudinal nematic liquid crystal (LC) mixtures with high birefringence used in this study were SLC-1717 (Shijiazhuang Chengzhi Yang

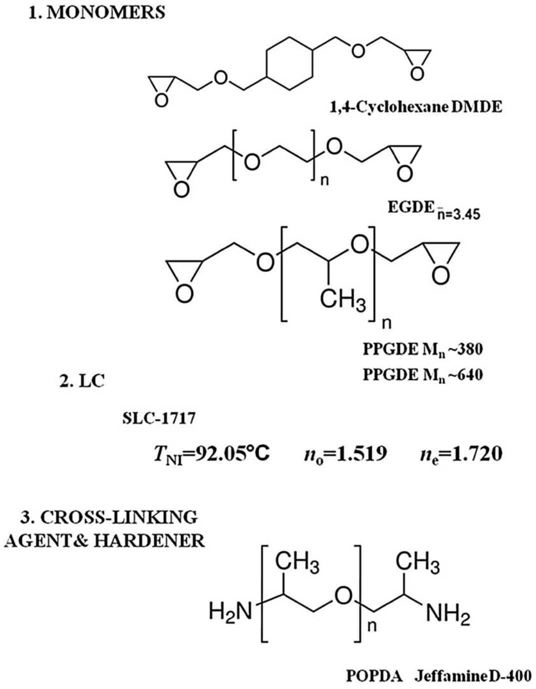


Fig. 1 Chemical structures and physical properties of the materials used without further purifications

Table 1 The compositions of the samples A1–A5 studied

Sample	Monomers mixtures(60 wt%) MM1[a]/MM2[b]/MM3[c]	SLC1717/wt%
A1	05/55/…	40
A2	10/50/…	40
A3	15/45/…	40
A4	20/…/40	40
A5	25/…/35	40

[a] Monomer mixture 1 (MM1[a]) 1,4-CHDMDE /EGDE/POPDA = 2/2/1 (molar ratio)

[b] Monomer mixture 2 (MM2[b]) PPGDE ~380/POPDA = 2/1 (molar ratio)

[c] Monomer mixture 3 (MM3[c]) PPGDE ~640/POPDA = 2/1 (molar ratio)

Huai Display Materials Co., Ltd). The heat-curable epoxy monomers used were a mixture of 1,4-cyclohexanedimethanol diglycidyl ether (1,4-CHDMDE; Meryer (Shanghai) Chemical Technology Co., Ltd), ethylene glycol diglycidyl ether (EGDE) resin (XY 669, Anhui Hengyuan Chemical Co., Ltd.), poly-propylene-glycol-diglycidyl ether (PPGDE ~380), poly-propylene-glycol-diglycidyl ether (PPGDE ~640) (Sigma Aldrich Company) and poly-oxy-propylene-di-amine (POPDA) (Jeffamine D-400, Heowns Biochem Technologies Tianjin). POPDA is also used as a polyamine hardener for epoxy resins in this study. Figure 1 shows the chemical structures of these materials.

All the above materials were used as received without further purification. The compositions of curable epoxy monomers/hardener/LC mixtures are listed in Table 1.

Preparation and conditions

Monomer mixtures of theoretical molar ratio are referred to average functional of multiple curable epoxy resin. It is calculated by Fav = $\Sigma\Phi_i f_i$, where Fav is the average functionality of composite monomer, Φ_i and f_i stand for the relative percentage and functionality, respectively. The curing time is 7 h at 90 °C.

Preparation of PDLC films

In this study, the PDLC films were obtained by the PIPS process. The samples were prepared consisting of heat-curable epoxy monomers, hardener and the same amount of nematic LC. First, the compounds were mixed in different percentages and stirred for 2 h until they had been homogenized. Then, based on capillary action, the mixtures were sandwiched between two pieces of indium tin oxide (ITO)-coated glass substrates, with a thickness of 15.0 ± 1 μm controlled by a polyethylene terephathalate (PET) spacer and optimal preparation condition was 40 % Class α, longitudinal nematic liquid crystal with a curing time 7 h at 90 °C. The composition ratios of the samples are listed in Table 1. Functionality is referred to average functional of multiple curable epoxy resin. It is calculated by

$\mathrm{Fav} = \sum \Phi_i f_i$, where Fav is the average functionality of composite monomer, Φ_i and f_i stand for the relative percentage and functionality, respectively [16]. The theoretical molar ratios of three mixtures 1,4-CHDMDE/EGDE/POPDA,PPGDE ~380/POPDA and PPGDE ~640/POPDA are 2/2/1, 2/1 and 2/1, respectively.

Characterization of PDLC films

The morphology of PDLC films was observed by scanning electron microscopy (SEM) (ZEISS, EVO18, Germany). The samples were first separated and dipped into cyclohexane (C_6H_{12}) for 4 days at room temperature to extract the LC molecules, and then dried for 12 h under vacuum. After the samples were sputtered with carbon, the microstructure of the polymer network was observed under SEM.

The E-O properties of PDLC films were studied using a liquid crystal device (LCD) parameters tester (LCT-5016C, Changchun Liancheng Instrument Co., Ltd.). A halogen tungsten lamp was used as an incident light source and the incident wavelength (λ) through the samples was fixed with the wavelength (λ) filter (632.8 nm). The transmittance of the PDLC films was recorded by a photodiode, and the response of the photodiode was monitored by a digital storage oscilloscope. A square-wave modulated electric field was used and the distance between the sample and photodiode was 300 mm. The transmittance of air was normalized as 100 %.

The refractive index of the polymer matrix was measured using Abbe Refractometer (WYA-2WAJ, Hangzhou Kebo Instrument Co., Ltd.) at room temperature. The polymer matrices were prepared with the molar ratio according to the samples A1–A5, but LC was absent. The curing conditions were the same as that of PDLC films. It is worth mentioning that the influence of LC on the refractive index of polymer network was neglected in this study.

The peel strength of PDLC films were measured by an autograph testing machine (AG-IC 50KN, Shimadzu International Trading (Shang Hai) Co. Ltd.). The PDLC film was cut into pieces with $20 \times 4\ \mathrm{cm}^2$. Measurements were performed at 25 °C at a detachment rate of 20 mm/min. Three independent measurements were made and the averaged value of the results was defined as final peel strength.

The transmittance of the off-state samples was studied using a UV–Vis–NIR spectrophotometer (V-570, Jasco Corp. Tokyo Japan). The wavelengths were measured in the range of 300–800 nm, and the results were recorded with an incident angle, $\delta = 0$.

Results and discussion

Morphology of polymer network of the PDLC films

Figure 2 shows the morphology of SEM micrographs of the isotropic cross-linked polymer network of the samples A1–A5. For the PIPS heat-curing method, the mixtures were homogenized before curing; it took time for the epoxy monomers to react and cross-linked for the LC to coalesce and form droplet size during the curing

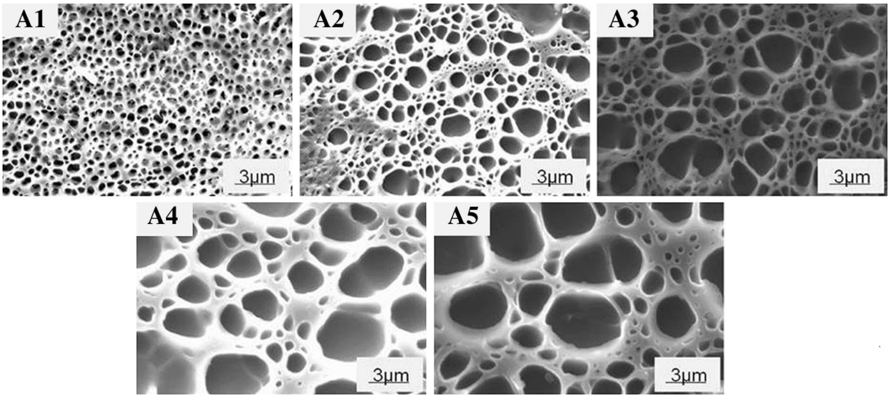


Fig. 2 Scanning electron microscopy (SEM) micrographs of the Isotropic cross-linked pristine polymer network of the samples **A1**–**A5** with a curing time 7 h at 90 °C

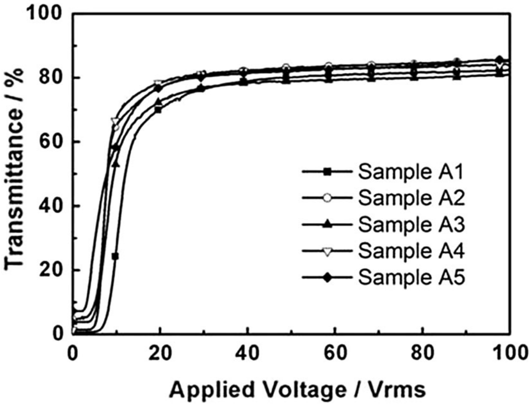
process. As the epoxy monomers polymerized, the LC coalesced forming droplet size and the droplet size got bigger with time. It can be observed that the droplet size of the LC is greatly affected due to the presence of the short flexible chain length EGDE, alkoxy chain length PPGDE ~380, PPGDE ~640 and 1,4-CHDMDE epoxy monomers; in resulting LCs droplet size of the sample A1–A5 increased in sequence. The morphology of PDLC films was gradually formed during the LC droplet nucleation and the polymer gelatins. The LC droplet size was controlled by the relative contents of the polymerizable monomers, the rate of polymerization and some physical parameters such as viscosity, rate of diffusion, experimental temperature, and solubility of the LC molecules in the polymer [17, 18].

In this study, the LC droplet size increased gradually with increasing the alkoxy chain length of the PPGDE monomers. Increasing the alkoxy chain length of the PPGDE ~380 and PPGDE ~640 increases the molecular weight of the epoxy monomers, so the amount of molecules in the same molar ratio of 1,4-CHDMDE monomer decreases, as a result, lowering the cross-linking density and increasing the LC droplet size. In this study, the LC droplet size was also influenced by the relative ratio of the alkoxy chain length of PPGDE monomers, cross-linking agent POPDA and short flexible chain length EGDE. Another essential factor that strongly influences the morphology of PDLC films is the monomers EGDE and PPGDE are more flexible and they show a more rapid rate of polymerisation with POPDA cross-linking agent to form isotropically distributed cross-linked polymer networks. Consequently, we have observed that the curable monomers microstructure showed a vital function during the formation of the isotropic cross-linked polymer network of PDLC films in all samples A1–A5, respectively.

Optical properties of the PDLC films

By utilizing the principle of refractive index matching, the PDLC films exhibit interesting E-O properties. The E-O properties of PDLC films mainly depend on the

Fig. 3 The electro-optical properties of applied voltage dependence of transmittance for samples *A1–A5* with a thickness of 15.0 ± 1 μm



chemical nature of the epoxy monomers and LC. In addition, the influences of epoxy monomer structures on PDLC films can be obtained by analyzing the E-O properties of samples A1–A5. The transmittance applied voltage curves of samples A1–A5 are shown in Fig. 3. With increasing the applied voltage, the transmittance of the samples A1–A5 increases gradually. Comparing Fig. 3 with Fig. 2, the morphology of the polymer network in the PDLC films had a vital effect on the E-O properties of the PDLC films. Good E-O properties of the PDLC films can be obtained when the microstructure of the PDLC film is appropriate. Although for a definite system where the LCs content have fixed, the transmittance increases with increasing LC droplet size and the larger LC droplet size resulting in an insufficient number of scattering centers [19, 20].

As shown in Figs. 2, 3 and 4, the threshold voltage (V_{th}) and saturation voltage (V_{sat}) decreased with increasing LC droplet size. It is shown that the operating voltage of the PDLC film is significantly influenced by the flexible, alkoxy chain length of the epoxy monomers and the LC content. The V_{th} and driving V_{sat} are defined as the electric field required for the transmittance to reach 10 % and 90 % of (Ts) transmittance saturation state, respectively.

It must be mentioned that, the LC droplets size has a strong association with E-O performance in a PDLC system. Generally, the V_{th} is inversely proportional to the radius of LC droplets as shown in following equation [21],

$$V_{th} = \frac{d}{3a} \times \left(\frac{\rho_p + 2}{\rho_{LC}}\right) \times \left(\frac{K(l^2 - 1)}{\Delta\varepsilon\varepsilon_o}\right) \tag{1}$$

where d thickness of the PDLC film, l a/b, the ratio of the length of the semi-major axis, a, to the length of the semi-minor axis, b, ρ_{p} resistivity of the polymer, ρ_{LC} resistivity of the liquid crystal, K elastic constant of liquid crystal, $\Delta\varepsilon$ dielectric anisotropy of the liquid crystal and ε_{o} Vacuum permittivity.

Conventionally, as we examined Fig. 4, the V_{th} and V_{sat} decreased with decreasing of the composition ratio of PPGDE ~380 and PPGDE ~640 curable monomers specially. It must be mentioned that the operating voltage of the PDLC

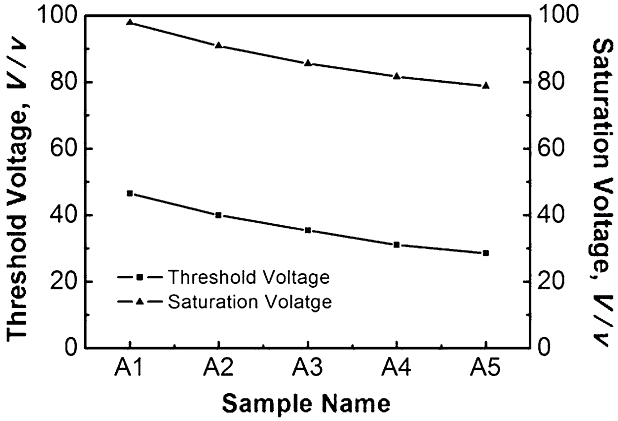


Fig. 4 Threshold voltage (V_{th}) and Saturation voltage (V_{sat}) of samples *A1–A5*

films is significantly influenced by the curable epoxy monomers and Class α, longitudinal nematic liquid crystal (LC) mixtures. It is directly related to the increase of LC droplet size, which indicates that LC droplet size has an important effect on anchoring energy. It is well known that the driving voltages can be thoroughly influenced by the microstructures of the polymer networks. Equation (1) indicates that the E-O properties of PDLC system can be controlled by LC droplet size and other factors such as film thickness, resistivity and dielectric anisotropy of LC. Hence, V_{th} and V_{sat} decreased with increasing of the LC droplet size. As for samples A1–A5, increasing the alkoxy chain length and lowering the cross-linking density alter the values of V_{th} and V_{sat}, which changes from 46.593 to 28.571 V and 97.826 to 78.791 V; respectively. Moreover, with increasing the LC droplet size, the interface between LC molecules and polymer matrix decreased as shown the Fig. 2, which results in decreasing the anchoring effects on LC molecules from polymer matrix. Therefore, the LC molecules are much easier to orient along the direction of the electric field and consequently V_{th} and V_{sat} decreased.

Contrast ratio (CR) is a key measurement of the E-O properties in a PDLC system. CR of PDLC films is used to characterize the difference between a transparent and an opaque state. With the increase of applied voltage, the transmittance of the samples encompasses transmittances of the initial off state (T_0) to the saturation state (Ts).

$$\mathrm{CR} = T\mathrm{s}/T_0 \tag{2}$$

It is commonly known as the switching contrast ratio. The off-state light transmittance (T_{off}) in fixing wavelength (632.8 nm) and the contrast ratio of all samples A1–A5 are shown in Fig. 5. It can be seen that in all samples the CR is continuously decreasing with increasing due to the LC droplet size. Comparing Fig. 2 with Fig. 5, the morphology of the PDLC films has an essential affect on the CR of the PDLC system. Despite the fact that CR is inversely proportional to the alkoxy chain length of PPGDE monomers, it greatly influences the formation of

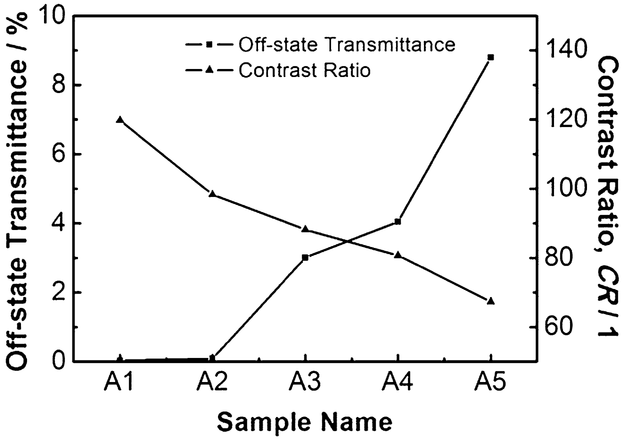


Fig. 5 Off-state light transmittance (T_{off}) and contrast ratio (CR) of samples *A1–A5*

PDLC films. It can be seen in Fig. 5 that T_{off} of all samples A1–A5 increased in sequence. It is well known that T_{off} of PDLC films depends greatly on LC droplet size, with larger LC droplet size resulting insufficient scattering centers. Therefore, T_{off} of the all samples increased with the increasing size of LC droplet; this was also demonstrated by our experiments, and the variation in T_{off} had the same trend as the variation in LC droplet size. The strong scattering in PDLC films can be enhanced by the use of highly birefringent LC, a high droplet density and film thickness. As a result, the PPGDE containing alkoxy chain length can increase the LC droplet size and decrease the light scattering intensity and CR significantly.

Optical contrast in PDLC films comes predominantly from their inherent light scattering property. Table 2 shows the refractive indices (n_p) of the polymer matrices. When comparing the refractive indices of cured epoxy matrix materials with those of the resin hardener mixtures prior to the onset of cure, our finding shows that, in all samples A1–A5, the change of the composition ratio of heat-curable epoxy monomers led to corresponding change of the refractive index of the polymer matrix. As shown in Table 2, the refractive index of polymer matrix decreased in the sequence owing to the composition variation. It is worthy to note that the degree of mismatching of the refractive indices increased at first, and then decreased from sample A1–A5. The larger mismatching degree of the refractive indices of LC droplet size and the polymer matrix shows the smaller is the T_{off} in PDLC films. Meanwhile, the increased T_{off} results in the increased CR. Therefore, the CR of our PDLC films is sensitive to the polarized direction of the incident laser beam [22].

A number of parameters are also used to evaluate E-O properties of PDLC films such as rise time transmittance (τ_{R}) and the initial decay time transmittance (τ_{D}). Figure 6 shows about the response time of the PDLC films with applied voltage dependence of the rise time (τ_{R}) and decay time (τ_D) of samples A1–A5. These are two most important factors when evaluating the performance of PDLC films. The

Table 2 The refractive indices of polymer matrixes

Sample	Refractive index			
	Mixture[a]	n_p^b	n_o^c	$n_p - n_o$
A1	1.5272	1.5520	1.5190	0.0330
A2	1.5119	1.5391	1.5190	0.0201
A3	1.4989	1.5238	1.5190	0.0048
A4	1.4809	1.5021	1.5190	−0.0169
A5	1.4701	1.4981	1.5190	−0.0209

[a] The refractive index of mixture before curing

[b] The refractive index of polymer matrix without dissolved LC material

[c] The ordinary refractive index of SLC-1717 used

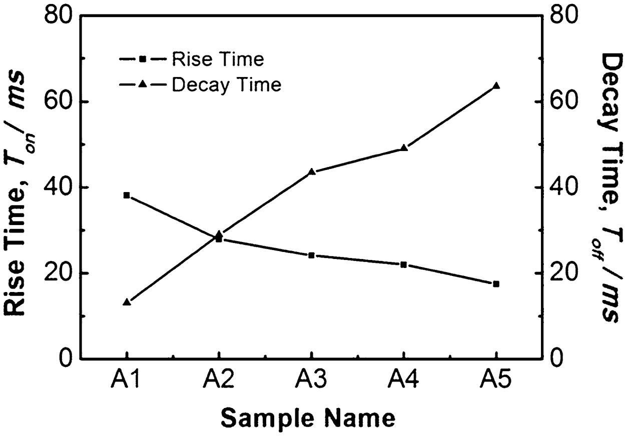
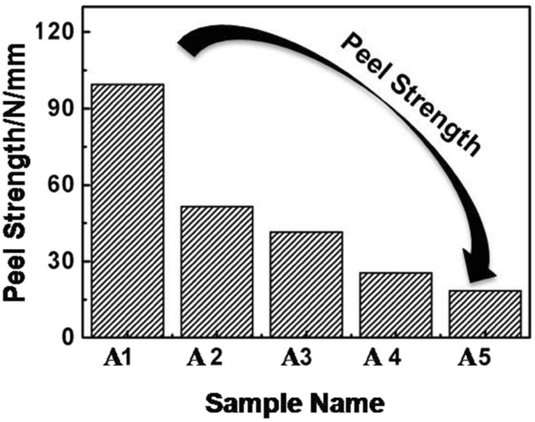


Fig. 6 The rise time (τ_R) and decay time (τ_D) of the samples A1–A5

rise time (τ_R) and decay time (τ_D) are calculated from the time dependence of the transmission coefficient. Rise time is explained as the time, which required to go from 10 to 90 % of the maximum transmittance of the sample upon turning on, and the one which required to go from 90 to 10 % of the maximum transmittance of samples turning off is known as decay time. Usually, a competition between the applied electric field and the interface elastic forces anchoring the LC molecules governs the response time [23]. Smaller the LC droplet size, stronger is the interface elastic forces of the polymer network of the LC molecules. Therefore, smaller LC droplet size needs a longer time to overcome and more interface anchoring energy. Moreover, many studies have indicated that smaller the LC droplet size, the larger is the τ_R and the smaller is the τ_D. As shown in Fig. 6, for each sample, τ_R decreased and τ_D increased along with the increasing LC droplet size. The results have associated to the boundary interaction between the polymer matrix and LC droplet, which manages the re-orientation and orientation of LC droplet under electrical

Fig. 7 The peel strength of the samples A1–A5

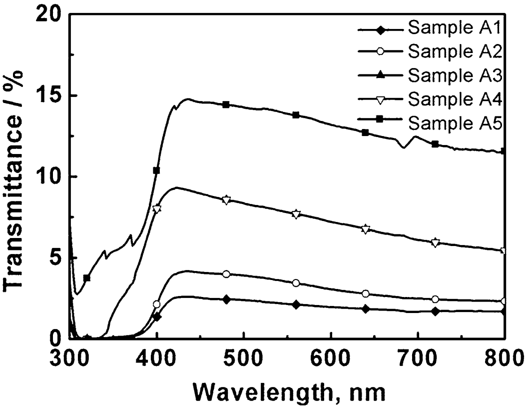


field. It means that if the size of the LC domain is smaller then it contains larger τ_R; as a result, it means that the smaller the LC droplet size, the stronger the interface elastic forces of the polymer network to the LC molecules. Thus, smaller LC droplets need a longer time to overcome more interface anchoring energy. On the other hand, when τ_D is expected to be independent of the voltage because much greater distortion of the director and a greater restoring energy of deformed leading the liquid crystal molecule go back to original position quickly [24, 25]. The PPGDE containing alkoxy chain length curable monomers are inversely proportional to the decay time (τ_D) and directly proportional to the rise time (τ_R) in all samples A1–A5, respectively. It can be seen that in Fig. 6, τ_D increased ceaselessly with decreasing the τ_R. As stated earlier heat content epoxy monomers composition ratio, wt% and microstructure of monomers play an important role in governing the response time of PDLC film.

Figure 7 shows the peel strength of samples A1–A5. The results of samples A1–A5 signify that the peel strength decreases with the enhancing the wt% of PPGDE of epoxy resins. The peel strength is defined as the sum of the energy required to break the bond and deform the backing and polymer [26]. The decreasing of lower cross-linking density and increasing of the LC droplet size increase the cohesive strength of sample A1, which is stronger than other samples A2–A5. Though the peel strength of sample A1 is greater than that of sample A5 due to the wt% of PPGDE of epoxy resins, is also strongly influenced by the alkoxy (R-O) and flexible chain length of curable monomers. Finally, the adhesion force decreases along with the increase of wt% of PPGDE, which ultimately decreases the peel strength.

Figure 8 shows the wavelength (λ) dependence of the off-state transmittance for all samples in the UV–Vis spectra. It can be found that the transmittances of all the samples tended to decrease with the decrease of wavelength in the wavelength range of 300–800 nm. The transmittance of the off-state PDLC films for sample A1 was relatively lower than the other samples, and there was a regular change for the samples A2–A5. This was in good agreement with the results of the LC domain size of the polymer network, as shown in Fig. 2 with a curing time 7 h at 90 °C.

Fig. 8 The wavelength (λ) dependence of the off-state transmittance (T_{off}) for the samples *A1–A5*



Conclusions

In summary, the cross-linked agent, varying composition ratio, wt% and the microstructures of curable epoxy monomers have significant influence on the morphology, E-O properties and the refractive index of the polymer matrix with Class α, longitudinal nematic LC content of PDLC films. On the morphology, microstructures of the PDLC films influenced by increasing the flexible and alkoxy chain length monomers and the existence of POPDA, PPGDE ~380 and PPGDE ~640 forms isotropically cross-linked pristine morphology. As a result, the LC droplet size of PDLC films increased with increasing the τ_D and decreasing CR, τ_R, V_{th} and V_{sat}. Meanwhile, the refractive index of the polymer matrix decreased with increasing chain length of curable monomers, which affect the reduced mismatching between n_p and n_o; thus on-state transmittance is optimized and the peel strength of PDLC films decreased. With the E-O data, it is further believed that it is possible to regulate the LC droplet size and optimize the electro-optic performance by adjusting the composition ratio, relative wt% of POPDA and structure of heat-curable epoxy monomers to obtain PDLC films which possess good E-O properties. Therefore, the results in this research subject show significant advantages for manufacturing PDLC films and developing the PDLC market.

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