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Effect of polymer structure on the morphology and electro-optic properties of UV curable PNLCs

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Abstract

Polymer network liquid crystals (PNLC) were prepared from UV curable polyurethane acrylate (PUA) and a nematic liquid crystal mixture (E7) at a fixed film composition of 1.5/8.5 (polymer/LC) by weight. The polymer networks were obtained upon curing the reactive mixture of hydroxyethyl acrylate (HEA) terminated polyurethane prepolymer, monoacrylate and triacrylate (4/3/3).

The effects of prepolymer molecular structure viz., length of polyurethane (PU) segment, molecular weight (M_n) and functionality (f) of polyol(PPG), and type of hard segment were studied in terms of morphology, voltage–transmittance relationship, off-state transmittance–temperature relationship, and thermal properties of the films. Aromatic diisocyanate (TDI) segment of PU showed greater chemical affinity with aromatic LC molecules and gave smaller domain size resulting in higher threshold (V_{10}) and driving (V_{90}) voltages. The increase in prepolymer molecular weight gave larger polymer–LC phase separation and decreased V_{10} as well as V_{90}, together with smaller nematic–isotropic transition temperature depression. Increase in functionality and decrease in M_n of PPG gave smaller domain size, smaller decay time, greater V_{10} and V_{90}, longer rise time, and greater depression of T_{NI} in film. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: PUA; Polymer/LC composite; PNLC

1. Introduction

Polymer/liquid crystals (LC) composite films have potential for a variety of electro-optic applications ranging from switchable window to information display [1–4]. In one form, they consist of LC droplets dispersed in polymer matrix known as PDLC (polymer dispersed liquid crystal) [2–4]. In the other form LC becomes continuous phase and three-dimensional polymer networks are dispersed in liquid crystals to form PNLC (polymer network liquid crystal) [5,6]. Practically PDLC saves expensive liquid crystals, whereas PNLC reduces driving voltage and response time.

Polymer/LC composite films offer a number of advantages over the conventional nematic devices such as twisted nematic (TN) and supertwisted nematic (STN) as well as ferroelectric or emissive ones [7]: (i) they require no polarizers thus increasing brightness of both direct view and projection displays, minimizing heat control problem and reducing power and weight; (ii) they require no alignment layers or stringent thickness control, thus greatly simplify manufacturing process and enable large area displays; (iii) they have large viewing angle in all directions and a high contrast ratio. Especially for projection system, no use of polarizer is highly appreciated because of the considerable decrease of light losses as compared to more commonly used TN and STN materials.

The polymer/LC composite film operates based on a fairly simple principle. The nematic LC molecule is optically uniaxial, and thus it has an ordinary refractive index (n_o) and extraordinary refractive index (n_e). In the absence of external field (unpowered), the orientation of optic axis and hence the nematic director vary randomly from droplet to droplet, and consequently light propagating normal to the film surface will probe a range of refractive index between n_o and n_e. Since the optical anisotropy ($\Delta n = n_e - n_o$) of LC molecules used in PDLC is sufficiently large for many reasons such as to reduce the driving voltage, rise time and to augment the contrast ratio [8], the effective refractive index is not generally matched with the refractive index of the polymer(n_p), and light will be scattered at the polymer–LC interfaces [9,10].

In the presence of electric field, liquid crystals of positive anisotropy tend to align themselves with the directors parallel to the field direction. In this state, the refractive index for incident light is equal to n_o, and if n_o is matched with n_p, the film becomes transparent.

Morphology and the electro-optic performance of the

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films depend on a number of factors, viz. method of cell preparation, type of polymer, polymer–LC interactions and miscibility, in addition to the film composition [11,12]. Regarding the type of materials, more requirements are imposed for use at high multiplexing level which can be realized in an active matrix substrate, usually a thin film transistor. Coates et al. [8] reviewed that the most fundamental of these films must have a high charge holding ratio so that an addressed pixel must retain its charge and thereby the induced orientation of LC until it is readdressed by the refreshing cycle. A holding ratio of at least 95% of the original charge must remain on the pixel after 200 ms [13]. A second requirement is the low operating voltage. Normal PDLC films operate at ca. 25 V_{rms}. However, the operating voltage can be further lowered by using more LCs. In the extreme case, only a network of polymer exists (PNLC) [14,15]. Finally a hysteresis of less than 0.1 V_{rms} is required so that the same grey levels can be achieved irrespective of whether the final voltage is arrived at from a previously higher or lower voltage state.

High charge holding ratio is obtained with terminal chloro compounds rather than the terminal cyano compounds, together with polymer of high resistivity. In contrast, the driving voltage depends on the droplet size, shape, and interaction between the polymer and LC. Hysteresis presumably occurs due to the slow and fast responses of LC molecules adjacent to the polymer walls and those away from the interfaces [16]. That is, hysteresis occurs when the director orientation is not only determined by the electric force (on switching) or elastic force (off switching), but also by the anchoring force at the interface. So, the energy losses in overcoming the anchoring force can cause the hysteresis, and it is inherently coupled with the material properties of polymer as well as LC.

Polymers which can satisfy the above-mentioned various properties can be developed with UV curable acrylate systems [8]. Consequently UV curable materials have most often been encountered as the polymer. Nolan et al. [17] reported that rapid cure (high UV power) gives less LC remained in polymer. The LC which remains in the polymer lowers the glass transition temperature (T_g) of polymer(plasticizing effect) and increase the refractive index of polymer. They showed that T_g of polymer decreased more with slow cure. The increased n_p often causes great index mismatch with n_o, and lowers the on-state clarity of the film.

Fujisawa et al. [18] reported that multifunctional acrylates are essential components for phase separation with LC upon curing and gives excellent properties for multiplexing. The cure temperature, slightly higher than the nematic–isotropic transition temperature (T_{NI}) is also critical to form the desired PNLC morphology. De Baets et al. [16] realized that certain type of monoacrylate drastically reduces the hysteresis width presumably by lowering the anchoring energy of LC molecules to the surface of the cured polymer. Ono and Kawatsuki [19] found that the driving voltage and response time were significantly decreased by adding a small amount of photocurable acrylates to polyvinyl alcohol (PVA)/LC system. The improvement was interpreted in terms of interface modification, i.e. the cured acrylates form an interface layer between PVA and LC.

In UV curable system, liquid crystal and prepolymer (often called polymer precursor) are initially mixed to form a single phase [20]. Prepolymer is oligomer of certain type of acrylate or polyurethane which is encapped with acrylate to allow UV cure in the subsequent step [21,22]. To this mixture, reactive diluent and photoinitiator are added before the mixture is irradiated. Reactive diluents are typically mono and multifunctional acrylates and are added by two reasons, i.e. to reduce the viscosity of prepolymer and to build up molecular weight of the polymer by chain extension (monoacrylate) and/or crosslinkings (multifunctional acrylates). The extent of crosslinking as well as the extent of cure depend on the average functionality of the reactive diluent.

We extensively consider the effects of prepolymer molecular structure on the morphology and electro-optic properties of polymer network liquid crystal. The length of polyurethane (PU) segment in polyurethane acrylate (PUA) (molecular weight of PU prepolymer), molecular weight and average functionality of polyol and diisocyanate structure of PU have been systematically varied and their electro-optic responses have been studied with an emphasis on the role of polymer.

2. Experimental

2.1. Materials and oligomer syntheses

Bifunctional and trifunctional polypropylene glycols with four different molecular weights ($M_n = 1000$, 2000, 3000, and 4000 g/mole) (Korea Polyol) were dried at 80°C, 0.1 mmHg for several hours until no bubbling was observed. A nematic type LC (E7(BDH), $n_o = 1.5216$, $n_e = 1.7462$, $T_{NI} = 61°C$) was employed to prepare the cell. Chemical reagents of isophorone diisocyanate (IPDI), hexamethylene diisocyanate (HDI), toluene diisocyanate (TDI), and hydroxyethyl acrylate (HEA) were used without further purification. Molar excess of diisocyanate was reacted with PPG for 3 h at 80°C to obtain isocyanate terminated prepolymers. The reaction mixtures were then cooled to 45°C before HEA was added and reacted for 3 h at 60°C with agitation to obtain HEA capped urethane acrylate oligomers [23–25].

2.2. Cell preparations

The oligomers are highly viscous and immiscible with LC, which necessitates the use of reactive diluents [26,27]. The reactive diluents used in these experiments are 2-ethyl hexyl acrylate (EHA) and trimethylol propane triacrylate (TMPTA), and composition of oligomer/monofunctional/trifunctional

Table 1
Formulations to prepare poly(hydroxy urethanes)

Structure variables	Poly(hydroxy urethanes)[a]						NCO terminated prepolymer	Vinyl-terminated prepolymer
	Polyol (PPG) (mol)			Isocyanate (mol)			Isocyanates (mol)	HEA (mol)
		Bifunction	Trifunction	HDI	IPDI	TDI	IPDI	
Hard segment structure	A1	2($M_n = 1000$)		1			2	2
	A2	2($M_n = 1000$)			1		2	2
	A3	2($M_n = 1000$)				1	2	2
Length of PU segment	B1	2($M_n = 1000$)			1		2	2
	B2	3($M_n = 1000$)			2		2	2
	B3	4($M_n = 1000$)			3		2	2
Functionality of urethane segment	C1	2($M_n = 1000$)			1		2	2
	C2	1($M_n = 1000$)	1($M_n = 1000$)		1		3	3
	C3		2($M_n = 1000$)		1		4	4
Molecular weight of PPG	D1	2($M_n = 1000$)			1		2	2
	D2	2($M_n = 2000$)			1		2	2
	D3	2($M_n = 3000$)			1		2	2
	E1		2($M_n = 1000$)		1		4	4
	E2		2($M_n = 3000$)		1		4	4
	E3		2($M_n = 4000$)		1		4	4

[a] Oligomer/Monofunction diluent (EHA)/Trifunctional diluent (TMPTA) = 4/3/3 (by weight).

diluents was 4/3/3 by weight (Table 1). LCs were then added to the prepolymer mixture at a fixed composition of prepolymer/LC = 1.5/8.5 by weight, together with a photoinitiator viz. 1-hydroxy cyclohexyl phenyl ketone (HCPK) [28]. The reactive mixtures were mechanically mixed thoroughly before they were filled into the ITO coated cell by capillary action. Cell thickness was adjusted to 14.5 μm using glass bead spacers. The mixtures were then cured using a UV lamp (100 MW/cm^2, 365 nm) for 3 min.

2.3. Contact angle

Contact angles of the films with LC drop were measured using a contact angle meter (ATAGO contact angle meter) at room temperature.

2.4. Morphology and thermal property

Morphology of the films was studied using a scanning electron microscopy(SEM, Jeol JSM820). UV cured cells were first fractured in liquid nitrogen, followed by extraction of LC in methanol for 24 h, and were cleaned in a ultrasonic cleaner. Samples were sputtered with gold before viewing under SEM. Thermal property of the films was determined using a DSC (Du Pont 1090) at a heating rate of 10°C/min.

2.5. Electro-optic measurements [29,30]

The UV cured PNLC films sandwiched between two ITO coated cells were placed normal to the direction of collimated beam of He/Ne laser (wavelength of 632.8 nm). When the effects of temperature were measured, the cells were placed in a heating stage. Transmitted light intensity without any polarizer was measured with a photodiode. The output from a function generator was amplified and used to drive the shutter. The drive signal and the response of the photodiode were monitored with a digital storage oscilloscope (Hitachi VC-6023). The distance between the cell and photodiode was about 300 mm. The lined up facilities, controlled by a laboratory computer, were turned up for about an hour before the data were taken.

3. Results and discussion

3.1. Effect of diisocyanate structure

The only difference in this series is the type of diisocyanate incorporated in PU, viz. flexible aliphatic diisocyanate (HDI, A1), rigid and bulky cycloaliphatic diisocyanate (IPDI, A2), and rigid and cohesive aromatic diisocyanate (TDI, A3).

Fig. 1 shows the SEM morphology of this films where a subtle structural difference gives a significantly different film morphology. The LC domain size is order of 5–6 μm with A1, several μm with A2, and approximately order of 1 μm with A3. The difference in domain size seems due to the different chemical affinity between polymer and LC, together with the chain rigidity of PUA. Greater chemical affinity of aromatic diisocyanate (TDI) with aromatic type LC (E7:biphenyl and terphenyl compounds) should give smaller phase separations. Greater chemical affinity of LC with A3 is seen from the contact angle measurement (Table 2). With greater chain rigidity of PUAs containing TDI and

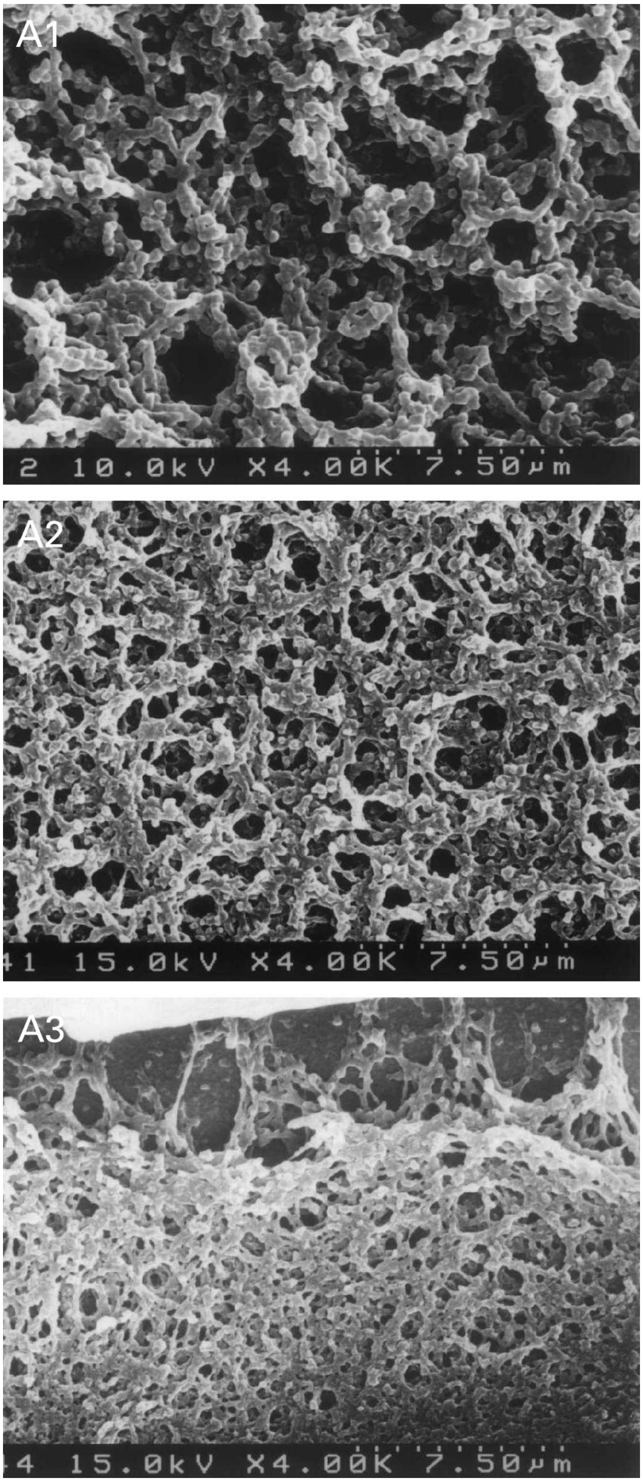


Fig. 1. SEM morphologies of PNLC vs. diisocyanate structure of PU blocks (Series A).

IPDI, viscosity of polymer network increases and so coalescence of LC domains during UV curing becomes difficult, resulting in smaller LC domains. These two factors contribute to the morphology.

Fig. 2 shows that transmittance–voltage (*V*–*T*) relationships of this films. Threshold (V_{10}) and operating (V_{90}) voltages of these film decrease along A3 > A2 > A1, an order of increasing domain size implying that the electro-optic performance of the films is largely governed by the domain size which, however, can be controlled by the diisocyanate segment of the PU. The decrease of V_{10} and V_{90} along aromatic, cycloaliphatic, and aliphatic diisocyanate is directly related to the increased domain size. Regardless of the type of composite film, light scattering–light transmission transition occurs very sharply mainly due to the narrow LC domain size distribution or high film homogeneity. With the increase in domain size, initial transmittance viz. the off-state transmittance is increased due to the decreased number of scattering center.

Table 2
Contact angle of polymer films with LC drop (25°C)

Run	Contact angle (°)
A1	23.6
A2	20.4
A3	18.3
B1	20.4
B2	18.6
B3	16.6
C1	20.4
C2	19.3
C3	17.8
D1	20.4
D2	22.8
D3	25.2
E1	17.8
E2	19.1
E3	20.8

Fig. 3 shows the rise time (τ_R) and decay time (τ_D) of these films as a function of applied voltage. Rise time decreases asymptotically with applied voltage due to the rapid alignment of nematic directors along the field direction.

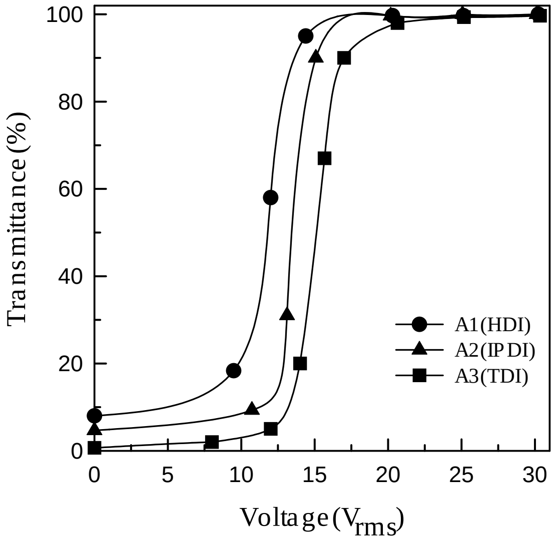


Fig. 2. Transmittance vs. applied voltage as a function of diisocyanate structure of PU block (Series A).

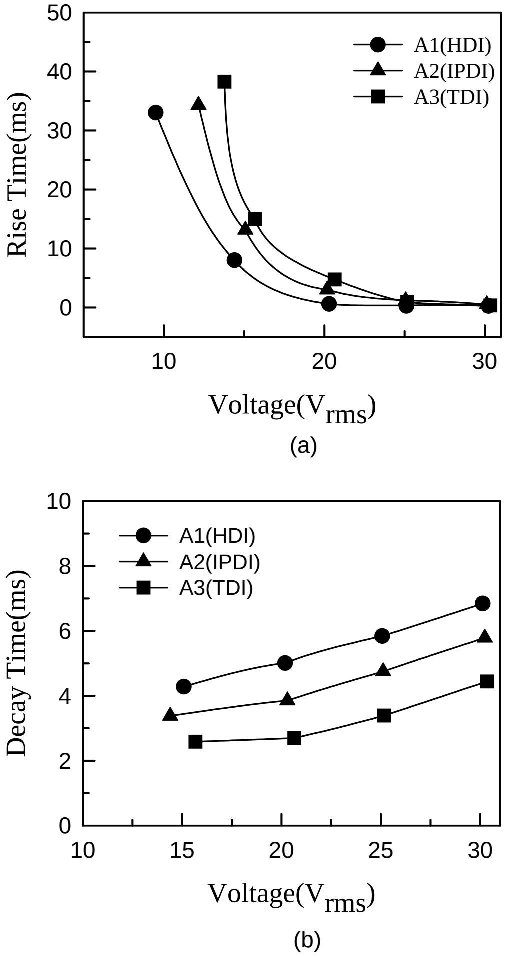


Fig. 3. (a) Rise time and (b) decay time of PNLC films vs. applied voltage as a function of diisocyanate structure of PU block (Series A).

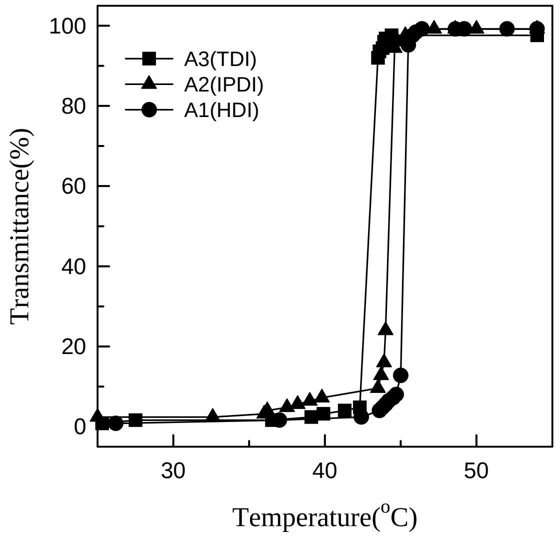


Fig. 4. Off-state transmittance vs. temperature as a function of diisocyanate structure of PU block (Series A).

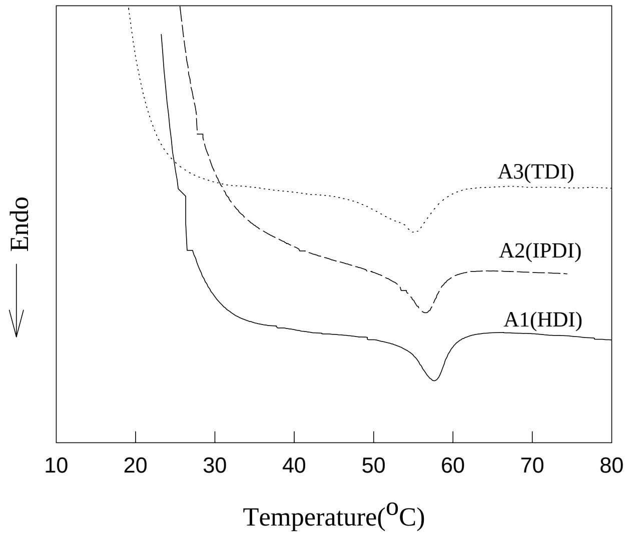


Fig. 5. DSC thermograms of the PNLC films with different diisocyanate structure of PU block (Series A).

Regarding the effect of diisocyanate type, HDI (A1) gives the quickest response to the electric field mainly due to the morphological effect of this film. In general smaller domains gives larger τ_R since they have to overcome more surface anchoring energy. The decay time is a response after switching off and is expected to be independent of the voltage. However our results show a small increase of τ_D with the increase in voltage. In this regard, Jain and Rout [31] proposed two stage relaxation depending on the type of film and field strength. At high applied voltage alignment of the LC molecules and bipolar axes along the field would complete. This involves much greater distortion of the director and in turn a greater restoring energy. So upon field removal, this state would first quickly return to an intermediate state where the molecular

Table 3
T_{NI} of LC in films

Run	T_{NI} (°C)	
	Off-state transmittance	DSC
A1	45.5	57.5
A2	44.6	56.5
A3	43.5	55.0
B1	44.6	56.5
B2	45.4	57.8
B3	46.2	58.5
C1	44.6	56.5
C2	44.0	56.0
C3	43.5	55.0
D1	44.6	56.5
D2	45.6	58.5
D3	46.4	59.0
E1	43.5	55.0
E2	45.5	57.0
E3	46.8	58.5

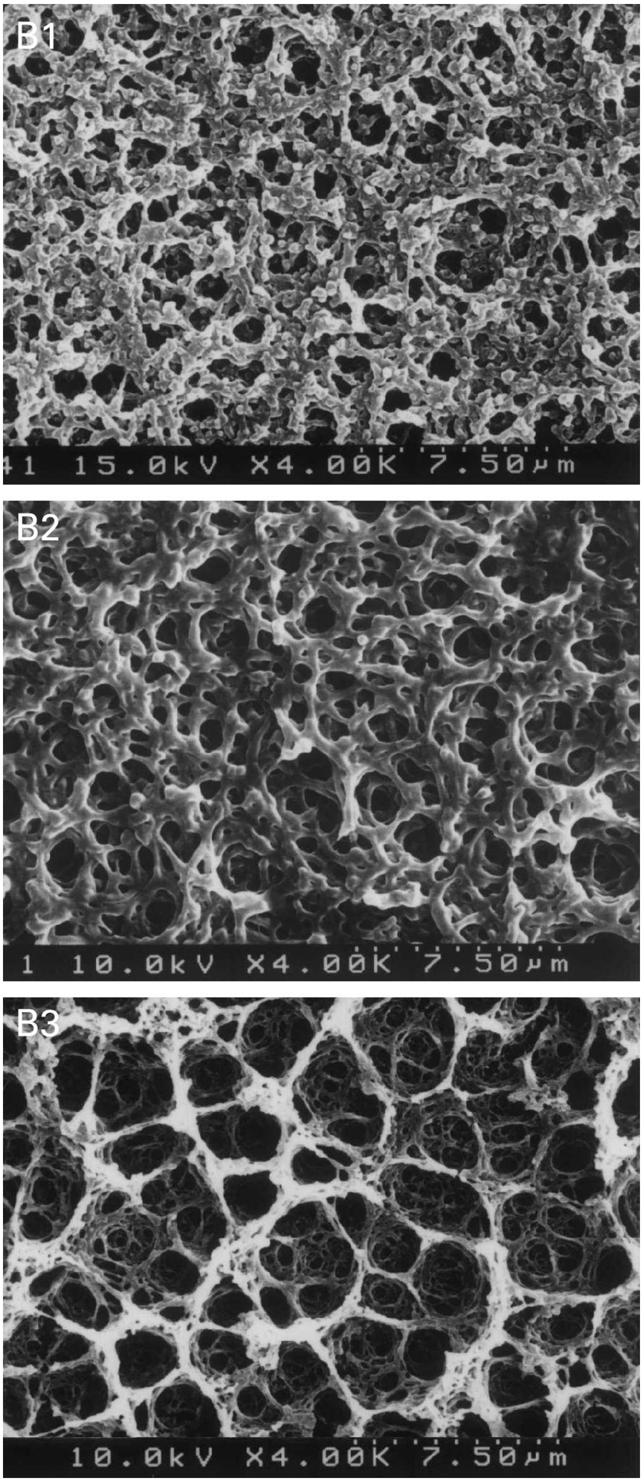


Fig. 6. SEM morphologies of PNLC vs. PU block length (Series B).

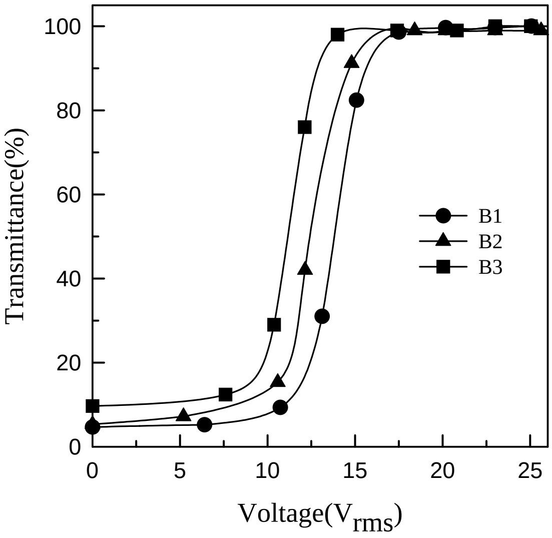


Fig. 7. Transmittance vs. applied voltage of the PNLC film as a function of PU block lengths (Series B).

alignment in the LC droplet in the off-state is essentially bipolar with the axis still remains oriented along the field direction. Eventually, the orientations of the bipolar axis become random. This relaxation proceeds slowly, especially for spherical droplets since there is little difference in the elastic deformation energy between the intermediate state and this final relaxed state [4,32]. Although the two stage relaxation was originally proposed for PDLC morphology, it should be applicable for PNLC morphology as well. Regarding the effect of diisocyanate, TDI (A3) gives the quickest, and HDI (A1) gives the slowest decay times, in accordance with their domain size. Generally, smaller domain gives smaller τ_D due to the higher elastic energy of deformed state.

Fig. 4 shows the off-state transmittance as a function of temperature during heating. The off-state transmittance shows an abrupt increase in transmittance from opaque to transparent state at a certain temperature. The transition is caused by several factors such as the decrease of birefringence, decrease of refractive index mismatch, and increase of mutual solubility. The transition temperature is closely related to the T_{NI} which may be either depressed due to the impurity effect of polymer compounds, or raised due to the preferential dissolution of light component of the LC mixture into polymer [17,33]. Since the LC being used in our experiment is a nematic mixture of different molecular weights, lower molecular weight compounds may preferentially be dissolved in polymer, contributing to an increase in T_{NI}. However, the T_{NI} of LC (61°C) is decreased in the films, and the factors contributing to depress the T_{NI} is more significant with an increasing order of A1 (HDI) $<$ A2(IPDI) $<$ A3(TDI). Solubility of PUA in LC seems maximum with A3 due to the great chemical affinity between the LC and polymer.

The decrease of T_{NI} is also seen from the DSC thermogram (Fig. 5), where the order of decrease is the same with the off-state transmittance measurement (Fig. 4). However, the decrease of T_{NI} in film is more significant and sensitive to the type of diisocyanate in terms of off-state transmittance than DSC measurements. The decrease of T_{NI} in films are tabulated in Table 3, together with other structural variables to follow.

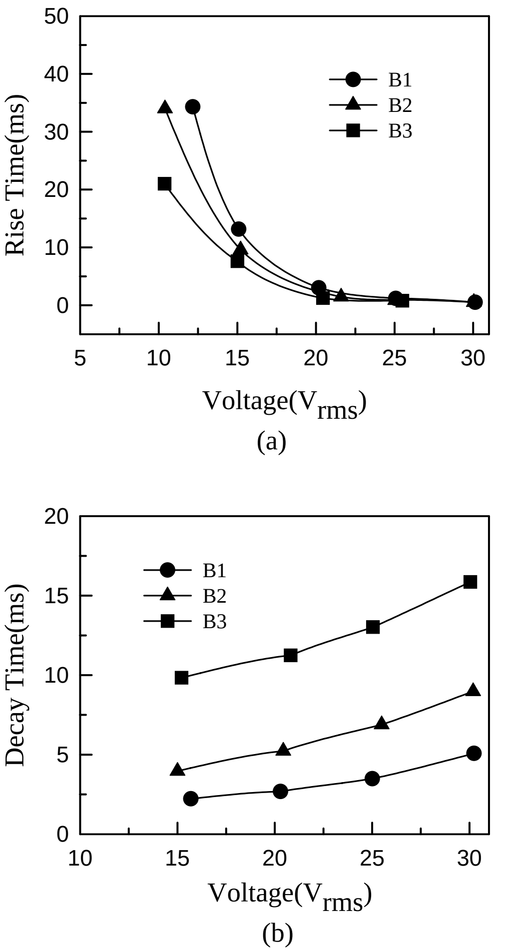


Fig. 8. (a) Rise time and (b) decay time of the PNLC films vs. applied voltage as a function of PU block lengths (Series B).

3.2. Effect of oligomer molecular weight

Fig. 6 shows the SEM micrographs of the films prepared with different prepolymer molecular weight (M_p). M_p calculated from the formulations are, respectively, 2666, 3888, and 5110 for B1, B2, and B3. It is clearly seen that the LC domain significantly increases in size with increasing M_p. Since the prepolymers were encapped with HEA and irradiated by UV to build up acrylate blocks, M_p corresponds to the molecular weight between crosslinks (M_c). So, the LCs are presumably entrapped between crosslinks and larger M_c should give larger LC domains. In contrast, the increase of urethane groups with increasing M_p contributes to the increased polymer–LC interaction (see the contact angle at Table 2), leading to the decrease in LC domain size. It seems that M_c essentially controls the LC domain size whereas the chemical composition of the PUA modifies the interfaces between polymer and LC. This is an advantage of using PU prepolymer to control the scale of phase separation in UV curing. It is also expected that B1 exerts stronger interfacial interactions with LC since it has more of hydrophilic urethane groups than the other two.

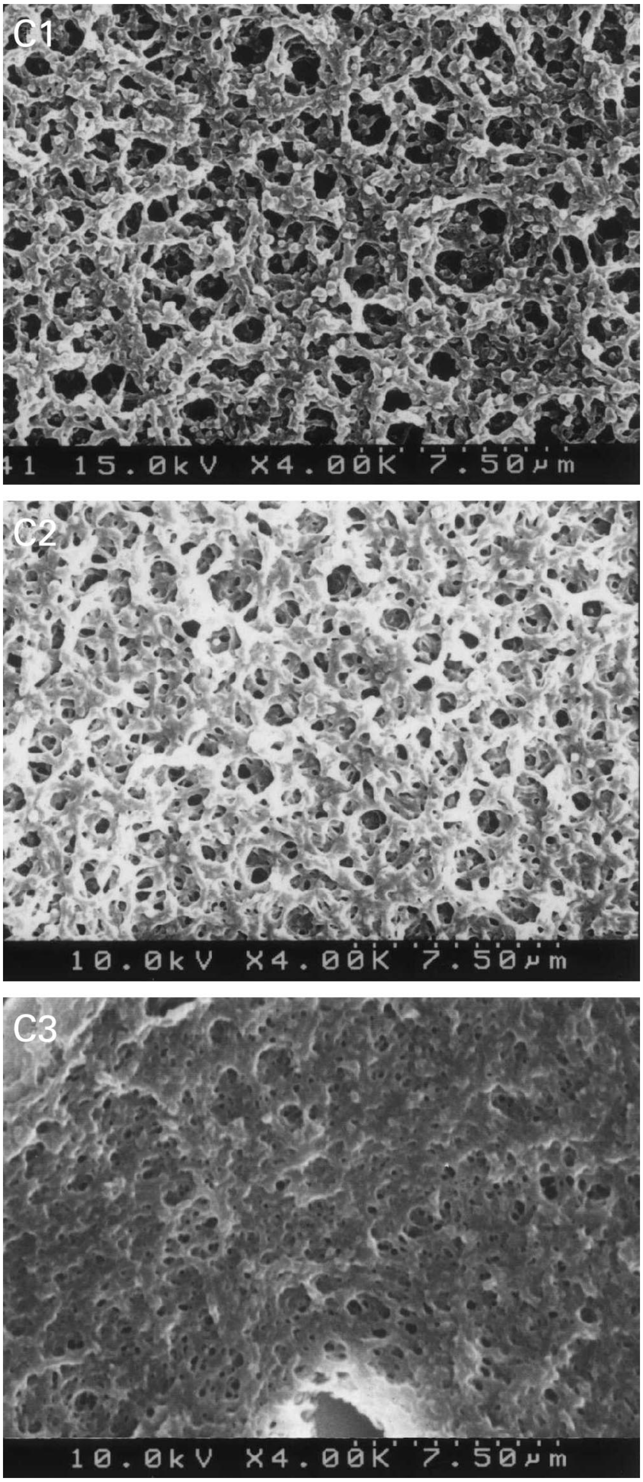


Fig. 9. SEM morphologies of PNLC vs. functionality number of PPG (Series C).

Fig. 7 shows the V–T relationships for the three types of films. It is seen that the curves move toward lower voltage with increasing M_p.

Fig. 8 shows τ_R and τ_D of these films. τ_R monotonically decreases with M_p and asymptotically with voltage, whereas τ_D increases with M_p and voltage. It is seen that M_p exerts a

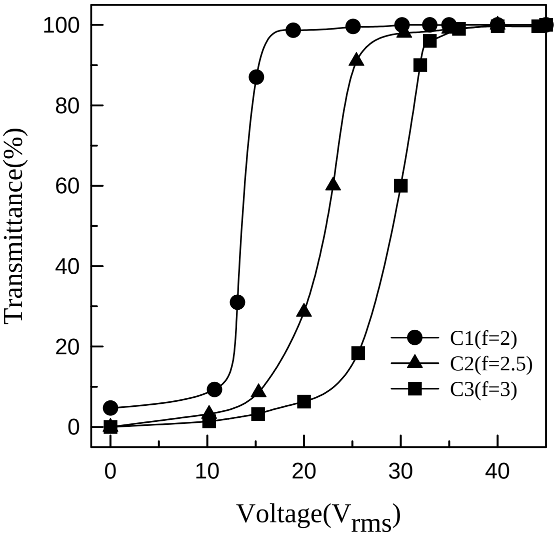


Fig. 10. Transmittance vs. applied voltage as a function of functionality number of PPG (Series C).

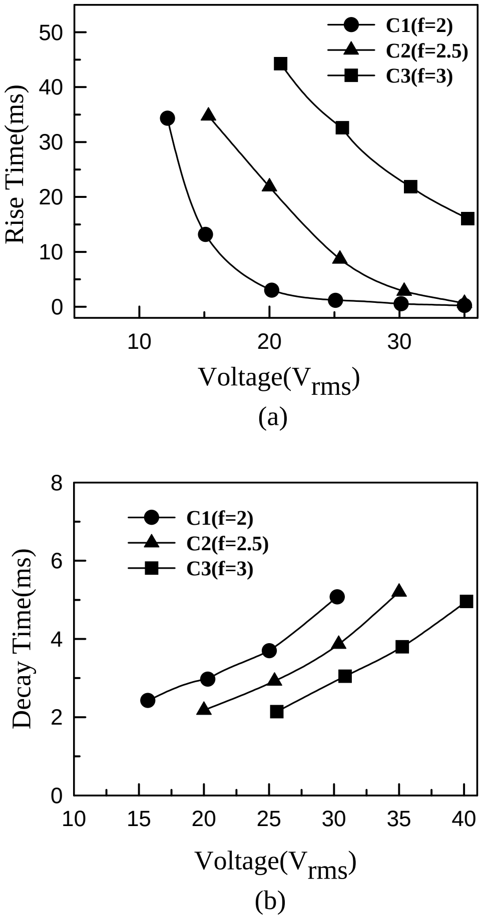


Fig. 11. (a) Rise time and (b) decay time of the PNLC films with different functionality number of PPG (Series C).

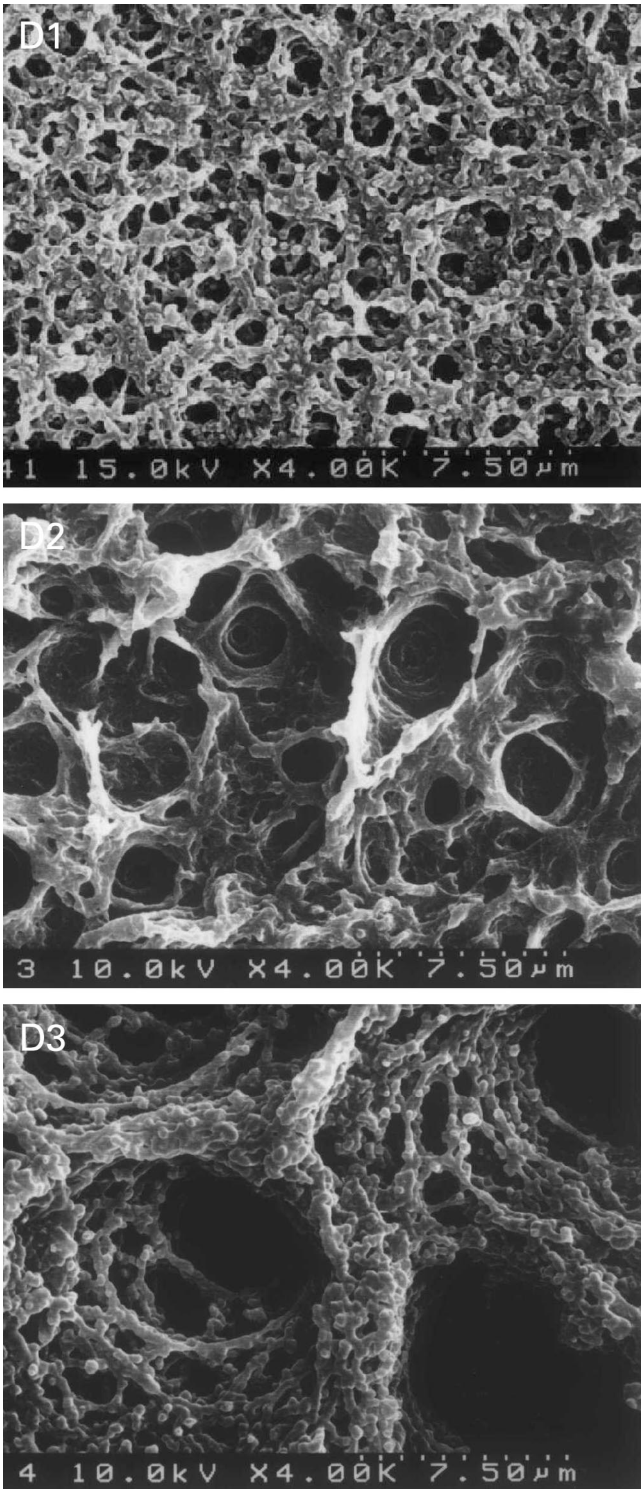


Fig. 12. SEM morphologies of PNLC vs. M_n of bifunctional PPG (Series D).

significant effect on τ_R and τ_D. It seems that the response times are very much governed by the domain size and polymer–LC interactions to some extent. Relatively long τ_D of B3 is due to the relatively low crosslinking density, providing less interfaces and little interactions with LC.

Off-state transmittance and DSC measurements again

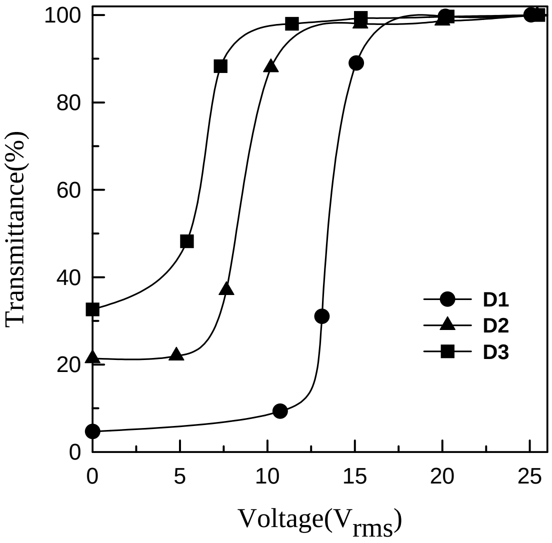


Fig. 13. Transmittance vs. applied voltage of the PNLC films with different M_n of bifunctional PPG (Series D).

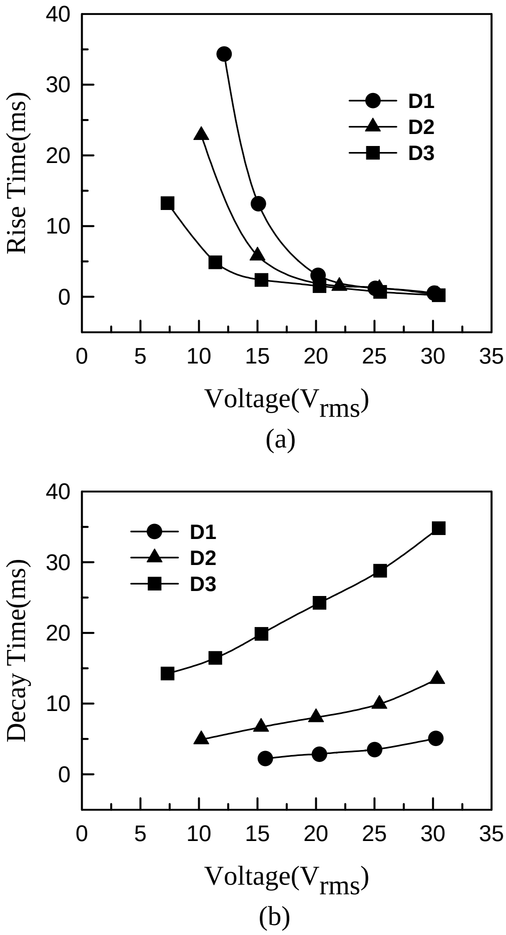


Fig. 14. (a) Rise time and (b) decay time of the PNLC films with different M_n of bifunctional PPG (Series D).

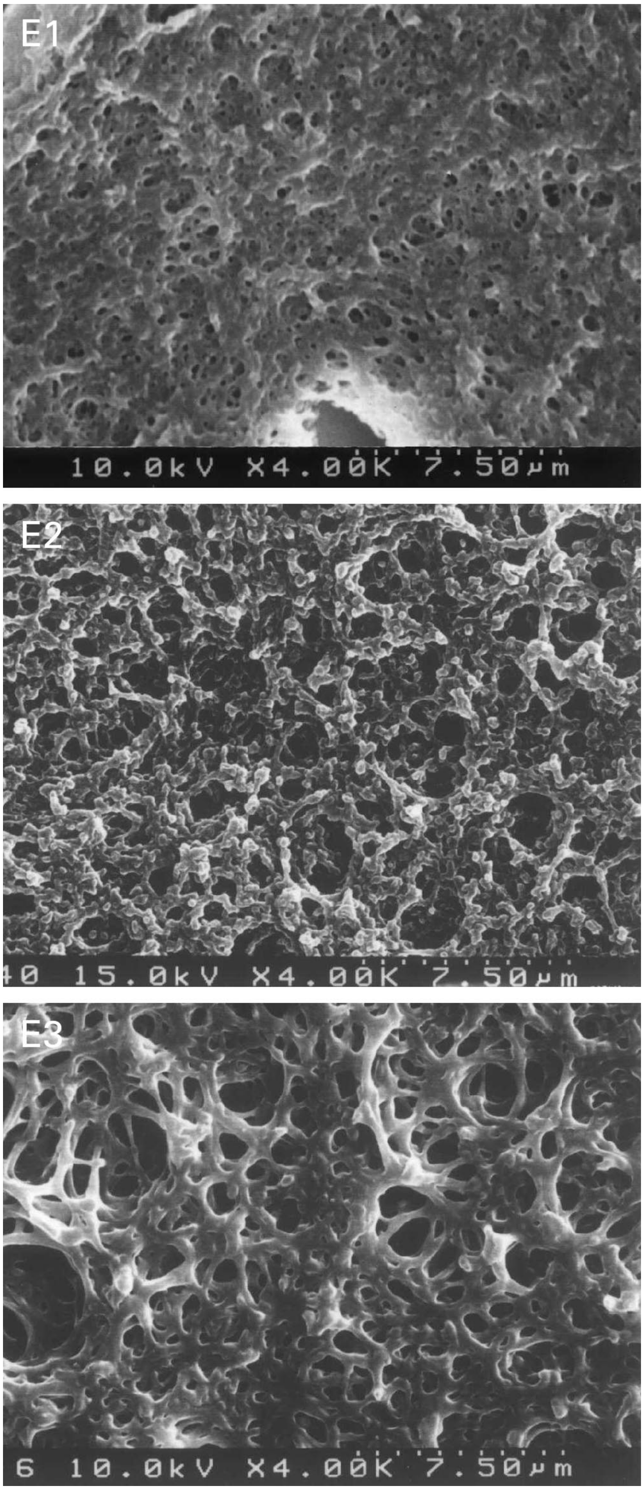


Fig. 15. SEM morphologies of PNLC films vs. M_n of trifunctional PPG (Series E).

showed that T_{NI} is decreased with the addition of PUA and the effect is more pronounced with the decrease in M_p (Table 3). The lowest T_{NI} with B1 is related to the greatest chemical interactions with B1 via polar interactions (Table 2) and greatest solubility of this prepolymer in LC.

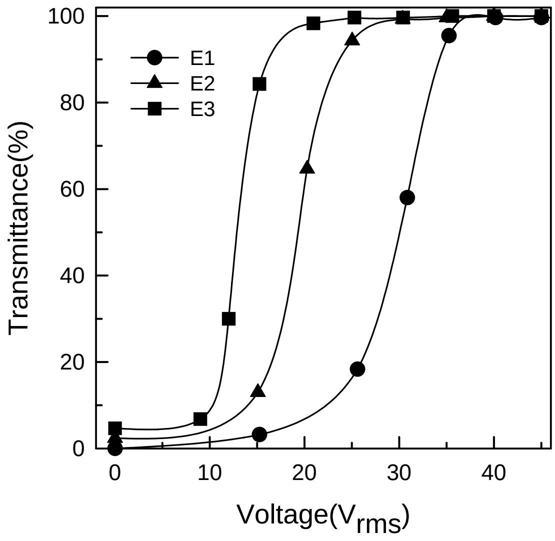
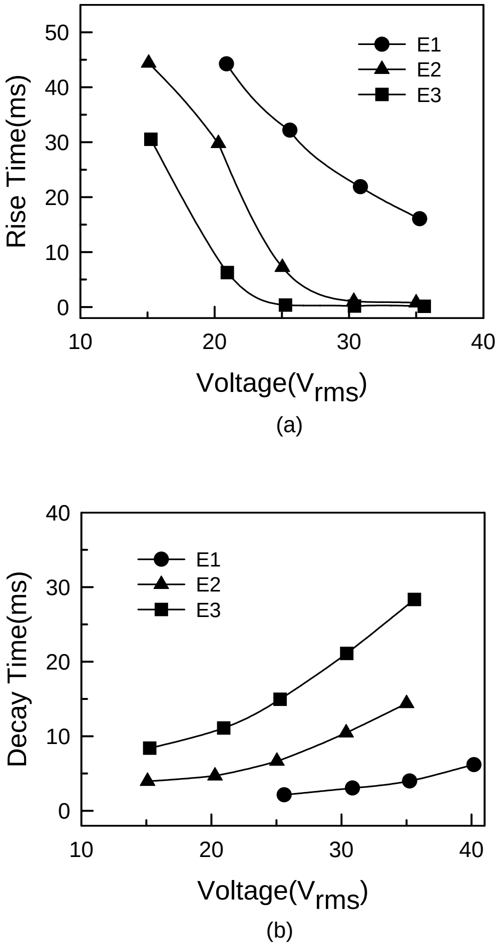


Fig. 16. Transmittance vs. applied voltage of the PNLC films with different M_n of trifunctional PPG (Series E).



(a)

(b)

Fig. 17. (a) Rise time and (b) decay time of PNLC films with different M_n of trifunctional PPG (Series E).

3.3. Effect of PPG functionality

Fig. 9 shows the SEM micrographs of the films which indicate that LC domain decreases in size with increasing functionality (*f*) of PPG. This is because the crosslinking density of PU domain increases with increasing *f*, and the molecular weight between the crosslinks effectively controls the LC domain size. The increased polymer–LC interactions via the increased number of hydrophilic urethane groups should also contribute to the decreased domain size.

As expected, the corresponding *V–T* curves moves toward higher voltage with increasing *f* of PPG (Fig. 10). This is a direct response of the decreased LC domain size as before. It should be noted that the off-state transmittance is about 5% with C1 due to the large LC domain size of this film. τ_R increases and τ_D decreases with increasing *f* (Fig. 11). At around the operating voltage, for example at 15 V for C1, τ_R is about fivefold of τ_D. Off-state transmittance variation with temperature and DSC thermograms also show that T_{NI} of LC slightly decreases in film (Table 3). However, the effect of increasing *f* is marginal.

3.4. Effect of molecular weight of PPG ($f = 2$)

Fig. 12 shows the SEM micrograph of the films as a function of bifunctional PPG molecular weight (M_n). Since the hydroxy terminated PUs were end capped with IPDI and subsequently with HEA and were subject to UV cure with acrylate monomer mixtures, the M_n of bifunctional PPG approximately corresponds to molecular weight between crosslinks, viz. M_c. Consequently, the LC domain size significantly increases with increasing M_n of PPG, and it is order of 10 μm with PPG3000. In addition, the increased length of hydrophobic aliphatic chain should also contribute to the increased domain size.

Direct response to the morphology is *V–T* curves of the films (Fig. 13). Off-state transmittance of the films with PPG1000 is about 5%, and it is over 20% with PPG2000, and 30% with PPG3000. Such a great off-state transmittance is due to the insufficient number of scattering centers which, in contrast, is due to the large LC domain size. With high off-state transmittance, small contrast ratio results.

τ_R decreases with domain size as well as with applied voltage, whereas τ_D increases with domain size (Fig. 14). Notably, τ_D is longer than τ_R with PPG 3000, and the two response times are very much the same with PPG 1000 and PPG 2000.

Off-state transmittance and DSC measurements (Table 3) showed that T_{NI} is decreased in films, and the decrease is more significant with lower M_n of PPG. This is presumably due to the smaller domain size, and larger polymer–LC interaction due to the increased number of hydrophilic urethane groups. Obviously, larger LC domain keeps its T_{NI} closer to its pure value.

3.5. Effect of molecular weight of PPG ($f = 3$)

LC domain also increases in size with M_n of trifunctional PPG (Fig. 15). However, the domain size is much smaller as compared with bifunctional PPG when compared at the same molecular weight. This is simply because the segment length between the crosslinks decreases with increasing f.

As before, V–T curve moves toward higher voltage with increasing the f of PPG (Fig. 16). However, the V_{10} and V_{90} of these films are higher, and the off-state transmittance is much lower than those of bifunctional PPGs. They are mainly due to the difference in domain size. Trifunctional PPG films give same voltage dependence of response times with bifunctional ones although these films gives the larger τ_R and smaller τ_D (Fig. 17).

Transition temperature measured from off-state transmittance decreases less with PPG3000 and more with PPG1000 due mainly to the LC domain size (Table 3). Essentially identical tendency is obtained with DSC measurement although DSC is virtually insensitive to the variation of T_{NI} in film.

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