



Effect of external electrical field on phase behavior and morphology development of polymer dispersed liquid crystal

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Abstract

Phase diagrams for mixtures of liquid crystal (LC)/monomer with and without an external electrical field applied have been established using polarized light microscope (PLM). The (isotropic + nematic) coexistent phase region and (isotropic + isotropic) phase boundary of LC/monomer mixtures were observed to shift upward to higher temperatures when the external electrical field exists. It was found that the electrical field applied during the cross-linking polymerization has a significant influence on the phase diagrams for the LC/polymer mixtures by rendering the coexistent phase regions shift upward to higher temperatures. The influence of the external electrical field on the processes of the isotropic–isotropic phase separation and liquid crystal ordering in PDLC formation has also been investigated. The results revealed that both the processes could be highly accelerated by the electrical field.

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Keywords: Polymer dispersed liquid crystal (PDLC); Phase behavior; Electrical field; Morphology; Phase separation; Liquid crystal ordering

1. Introduction

Polymer dispersed liquid crystal (PDLC) films are thin films composed of microdroplets of liquid crystalline material in a polymer matrix [1,2]. The polymer matrix basically provides thermal and mechanical stability, while the LC affords needed electro-optical properties. In the normal off state, a PDLC film is opaque and scatters light. When a sufficient voltage is applied, the PDLC film becomes transparent and allows light to pass [3–5]. Unlike conventional LC displays, the PDLC films are flexible and very easy to prepare. In addition, their light transmittance is much higher than

that of the more conventional twisted- or supertwisted-nematic devices, owing to the absence of polarizers [6]. Therefore, the PDLC films are candidates for several applications such as optic shutters, switchable windows and display devices [4,7].

For these reasons, PDLC has been the focus of investigation both theoretically and experimentally. Smith and coworkers have investigated the dependence of the optical performance of PDLC films on a variety of structural, electric-optical and thermal properties [8,9]. The report describes scanning electron microscope studies of film structure, measurements of voltage dependent film transmittance and light scattering, and calorimetric studies. These studies indicate that microdroplet formation in the films occurs as a result of phase separation. Koenig and coworkers have studied phase separation in the formation of PDLC films using rapid scan FTIR spectroscopy [10]. The films were made by photopolymerization of photo-curable matrix and

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liquid crystal mixtures. The studies show that the monomer conversion at phase separation decreases strongly with an increase in liquid crystal content, while the conversion required for phase separation increases with increasing temperature of polymerization. Kyu and coworkers have extensively investigated PDLC films made by photo-cure of photo-curable materials and nematic liquid crystals mixtures using light scattering, optical microscopy, and differential scanning calorimetry [11–14]. The studies reveal that photopolymerization of the LC/monomer mixtures results in phase separation due to instability caused by an increase in molecular weight and subsequent network formation, and the size of the phase separated LC droplet increases with LC concentration. Photopolymerization initiated in the isotropic phase gives uniformly distributed polygonal-shaped LC droplets in contrast to photopolymerization initiated in the two-phase region. Shen and Kyu also prescribed a theory [15] regarding the phase behavior of PDLC based on the Flory–Huggins theory [16] for the free energy of mixing of isotropic phase and Maier–Saupe theory [17,18] for phase transition of a liquid crystal. The calculated phase diagrams agree well with the experimental observations.

However, limited work has been reported upon the effect of external electrical field. Yang et al. have constructed a theory to predict the influence of the external field on the phase diagrams for the mixture of flexible polymers and liquid crystal [19]. The PDLC system considered is a mixture of LC with a non-cross-linked polymer which can be prepared by solvent induced phase separation method. The calculated results show that the external field promotes the alignments of the LC molecules, which strengthens the driving force for phase separation. Therefore, the binodal curves are shifted to higher temperatures and the spinodal curves are substantially changed. The effect of external field on the order parameter of the LC molecules, and the influence of the field intensity and polymer chain length on the phase diagram have been discussed. Recently, by using depolarized light intensity technique, a valuable method in following the phase transitions in LC polymers, author et al. have investigated the influence of the external electrical field on the phase separation kinetics of LC/monomer mixtures during the polymerization [20]. It was found that the phase separation process is accelerated and the clearing temperature of the formed PDLC film is raised by the electrical field. However, to author's knowledge, no experimental studies of the effect of the external electrical field on the phase diagrams of the mixtures of LC/monomer and LC/polymer have been reported so far. These investigations could be of great importance, which not only raises challenging questions addressing very basic aspects of LC research and provide a new way to investigate PDLC films academically, but also

brings useful information for the fabrication of PDLC films.

In the present work, phase diagrams for the mixtures of a multi-component nematic liquid crystal (TEB) and epoxy resins (E51) with and without an external electrical field applied were first established by means of polarized light microscope (PLM). Using the starting LC/monomer phase diagrams as a guide for thermally cured polymerization, a direct current electrical field was applied during the polymerization and the effect of the external electrical field on the phase diagram of the LC/polymer mixtures and the morphology of the formed PDLC film has been studied. The influence of the external electrical field on the kinetics of the isotropic–isotropic phase separation and liquid crystal ordering of the LC/monomer mixtures upon the polymerization has also been investigated.

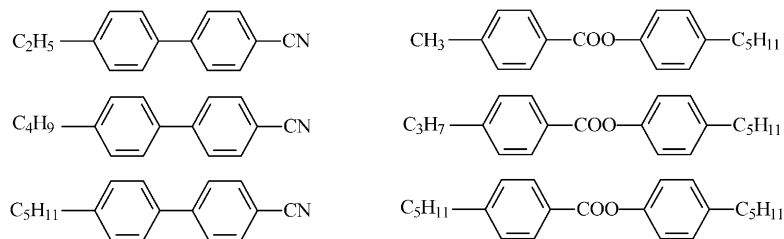
2. Experimental

2.1. Materials

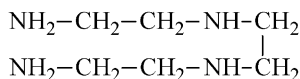
A multi-component eutectic liquid crystal (TEB) ($T_{KN} = -20$ °C, $T_{NI} = 62$ °C, $\epsilon_{\parallel} = 14.9$, $\Delta\epsilon = 9.4$, $n_e = 1.6925$, $\Delta n = 0.1703$) was purchased from Tsinghua Yawang LC Materials Co. Ltd. The matrix material: bisphenol A epoxy resin (E51) with the dielectric constant $\epsilon = 4.0$ and the refractive index $n = 1.5762$ was purchased from Shanghai Resin Plant and the chemical formula is available in the literature [20]. A multifunctional and amine-based cure agent was purchased from Shanghai Liyi Science and Technology Development Co. Ltd. The chemical compositions of the TEB liquid crystal and cure agent are shown in Scheme 1.

2.2. Sample preparations and measurements

TEB/E51 mixtures were prepared by weighting appropriate amounts of TEB and E51 in small vials, which were then mixed thoroughly. After heated above the clearing point of the LC, the samples were sandwiched between two conductive glass plates separated by a spacer with thickness of 35 μm . When the samples were naturally cooled down to room temperature, they were mounted on the hot stage of a polarizing microscope for the phase transition temperature measurements. The equilibrium TEB/uncured E51 mixtures have a good temporal stability. For the preparation of the TEB/cured E51 mixtures, the pre-mixtures of matrix material (E51) and LC (TEB) were carefully weighted in small vials, hand mixed for several minutes. After then, the cure agent was added to the mixtures, which were further thoroughly mixed at room temperature for 1 min. The mixtures were sandwiched



Liquid Crystal (TEB)



Cure agent

Scheme 1. Chemical compositions of the TEB liquid crystal and cure agent.

between two conductive glass plates separated by a spacer with thickness of 35 μm . The prepared samples were immediately put into an oven, in which cross-linking polymerizations were carried out. The polymerization temperature is 60 $^\circ\text{C}$ unless indicated otherwise. After polymerizations, the samples were naturally cooled down to room temperature for PLM observations.

In the case of studying the effect of the external field, a direct current electrical field with intensity ranging from 1.0 to 5.0 $\text{V}/\mu\text{m}$ was applied across the LC/E51 mixtures, LC/E51/cure agent mixtures during the polymerization and subsequent transition temperature measurements of the formed PDLC films. The electrical field direction is perpendicular to the sample plane. The samples with higher TEB contents cured under electrical field can appear more transparent than those cured without an electrical field due to the enhanced alignments of the liquid crystals in the PDLC film.

PLM experiments were carried out using a Leitz-Ortholux II polarized light microscope with hot stage. The phase transition temperatures of the mixtures of LC/monomer and LC/polymer were all detected by PLM during heating or cooling the samples on the hot stage with a programmable temperature controller. The heating or cooling rate is 1 $^\circ\text{C}/\text{min}$. Temporal evolution of the morphologies for the mixtures of LC/monomer during the polymerization was also followed by PLM. The induction times of the isotropic–isotropic phase separation and liquid crystal ordering of the LC/monomer mixtures during the polymerization are designated as the times when the appearances of the isotropic droplet and liquid crystal texture were first noticed under uncrossed polarizers and crossed polarizers, respectively.

3. Results and discussion

3.1. Effect of external electrical field on phase diagrams of TEB/uncured E51 mixtures

Fig. 1 illustrates experimental phase diagrams of LC (TEB)/monomer (E51) mixtures with and without an external electrical field applied. The phase diagram is of the upper critical solution temperature (UCST) overlapping with an isotropic + nematic coexistence region. It shows strong resemblance to those reported for other mixtures of nematic LC and monomer [13,14]. The phase diagram reveals isotropic liquid (*I*), isotropic liquid mainly containing monomer + isotropic liquid mainly containing LC ($I_M + I_{LC}$), isotropic liquid mainly containing monomer + nematic ($I_M + N$) and pure

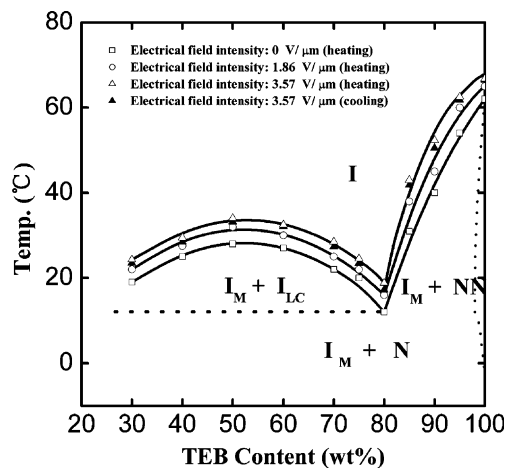


Fig. 1. Phase diagrams of TEB/uncured E51 mixtures under the external electrical field with various intensities.

nematic (N) phases. The ($I_M + I_{LC}$) binary phase exists when TEB content is approximately from 30 to 80 wt.%. Below the peritectic line (dotted horizontal line), isotropic liquid (I_M) and nematic (N) phases coexist. An illustrative dotted line based on Kyu's theory [15] divides the ($I_M + N$) region and pure nematic phase (N). Shown by (\blacktriangle) in Fig. 1 is a phase diagram obtained by cooling the samples. As it can be seen, the curve just shifts little bit downward compared with the one obtained on heating (shown by Δ). Under the present experimental condition employed, i.e. 1 °C/min, the difference in the results obtained on heating and cooling is small. The obtained phase diagrams are close to the true phase diagrams.

As viewed by the microscope, upon heating the 90/10 TEB/E51 mixture from a ($I_M + N$) coexistent phase region (20 °C) to an isotropic phase, the size of the nematic LC droplets decreases during heating the sample. Around 42 °C the nematic LC droplets disappear with the LC components completely dissolved in the monomers, indicating the transition from the ($I_M + N$) coexistence region to the (I) phase. Similarly, upon heating the 40/60 TEB/E51 mixture from a ($I_M + I_{LC}$) coexistent phase region to an isotropic phase, the isotropic droplets disappear around 26 °C, suggestive of a homogeneous isotropic solution of the LC molecules and monomers.

As Fig. 1 depicts, the external electrical field has a strong influence on the phase diagram. In the presence of the electrical field, the ($I_M + N$) \rightarrow (I) transition temperatures and ($I_M + I_{LC}$) phase boundary shift upward to higher temperatures. The effect of the external field on the phase transition temperatures for the mixtures of LC/monomer is displayed more explicitly in Fig. 2. Two results are shown for the mixtures with TEB content of 90 wt.% (a) and 50 wt.% (b), respectively. For the mixture with 90 wt.% TEB, it is observed that the

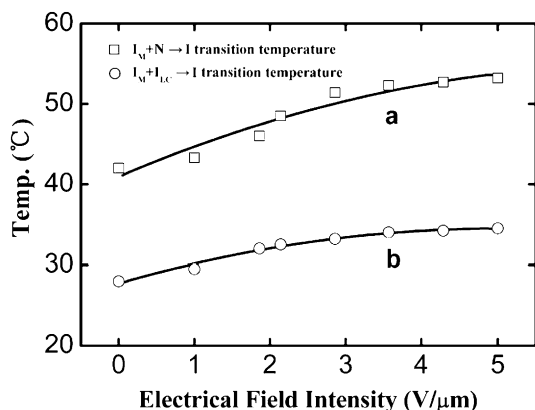


Fig. 2. Dependence of the phase transition temperatures on the electrical field intensity for the TEB/uncured E51 mixtures with TEB content of 90 wt.% (a) and 50 wt.% (b).

($I_M + N$) \rightarrow (I) transition temperature increases as the electrical field intensity increases. For instance, in the absence of an electrical field, the ($I_M + N$) \rightarrow (I) transition temperature is 42.0 °C. In the case of applying an electrical field with the intensity of 5 V/ μ m, the transition temperature becomes 53.2 °C. For the mixture with 50 wt.% TEB, an increase of the ($I_M + I_{LC}$) \rightarrow (I) transition temperature with increasing the electrical field intensity is also found. Furthermore, as it can be seen in Fig. 2, the effect of the electrical field on the ($I_M + N$) \rightarrow (I) transition is more pronounced than that on the ($I_M + I_{LC}$) \rightarrow (I) transition.

The upward shifting of the ($I_M + N$) \rightarrow (I) transition temperatures and ($I_M + I_{LC}$) phase boundary may be explained by the effect of the external electrical field on the alignments of the LC molecules. Due to the high dielectric anisotropy of the LC molecules [20,21], the external field may have an effect on the enhancement of the alignments of the LC molecules. The enhanced alignments of the LC molecules should decrease the entropy change of mixing, giving rise to a poor miscibility between the LC molecules and monomers. Therefore, the mixing temperature of the two components becomes higher, which results in the upward shift of the ($I_M + N$) coexistent phase region and ($I_M + I_{LC}$) phase boundary.

3.2. Effect of external electrical field on phase diagrams of TEB/cured E51 mixtures

Fig. 3 shows the effect of the external electrical field on the phase diagram of TEB/cured E51 mixtures, for

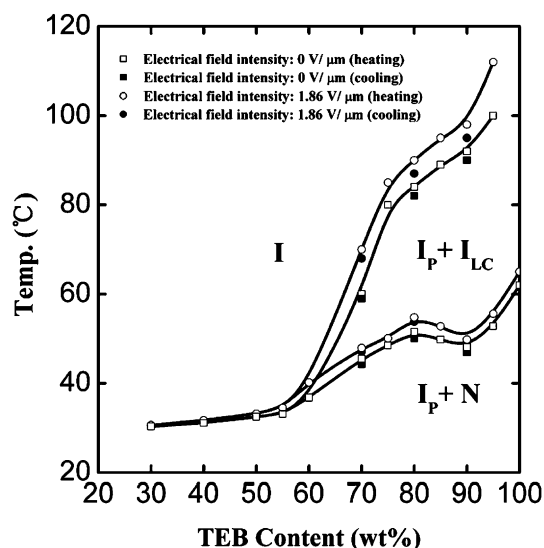


Fig. 3. Phase diagrams of TEB/cured E51 mixtures under the external electrical field with various intensities. Polymerizations were carried out at 60 °C.

which the electrical field was applied during the polymerization. The phase diagram exhibits various regions such as isotropic swollen network (I), isotropic network mainly containing polymer+isotropic liquid mainly containing LC ($I_P + I_{LC}$), isotropic network + nematic ($I_P + N$). In the present work, it is noted that the ($I_P + I_{LC}$) coexistence region was observed for the cured system with TEB content from 60 to 100 wt.%. When the content of TEB is below 60 wt.%, two phase areas were discerned: isotropic swollen network (I) and isotropic network + nematic coexistence regions ($I_P + N$). Fig. 3 also demonstrates some experimental data of the phase transition temperatures obtained on cooling. As it can be seen, the phase transition temperatures gained through cooling are little bit lower than those obtained on heating. However, the difference in the two results is small, indicating the obtained phase diagrams are close to the true phase diagrams.

Comparing the phase diagrams of LC/monomer and LC/polymer mixtures in the absence of the electrical field in Figs. 1 and 3, it is found that the polymerization exerts a pronounced influence. The curing polymerization reduces the miscibility between the LC molecules and reacting polymers. Consequently, the coexistence curves move to higher temperatures as can be seen in Figs. 1 and 3. It should be noted that the polymer network could not be dissolved in the anisotropic solvent. As argued by Nwabunma and Kyu [12], the isotropic LC molecules are presumably dissolved in the network through swelling upon heating. The LC droplets can reform in the network through deswelling during the cooling process. Taking the 80/20 TEB/E51 mixture as an example, at 100 °C the system is considered to be in the isotropic swollen network. Upon cooling to 80 °C, deswelling takes place and some isotropic LC droplets appear within the swollen network. With continued cooling to 40 °C, the swollen network deswells further leading to a liquid crystal ordering and the system enters the phase region where the isotropic network and nematic phase coexist. The application of the external field during the polymerization does not change the general feature of the polymerization influence on the phase diagram. But the coexistent phase regions move upward to higher temperatures corresponding to those of the mixtures without an electrical field applied.

More detailed information regarding the electrical field influence is shown in Fig. 4 in which the phase transition temperatures of TEB/cured E51 mixtures are plotted against the electrical field intensity. Results are shown for the mixtures with TEB content of 55 wt.% (a) and 75 wt.% (b). As it can be seen, the ($I_P + N$) \rightarrow (I), ($I_P + N$) \rightarrow ($I_P + I_{LC}$), and ($I_P + I_{LC}$) \rightarrow (I) transition temperatures increase with increasing the electrical field intensity. For example, in Fig. 4(a) the ($I_P + N$) \rightarrow (I) transition temperature is 33.1 °C when the electrical field is absent. When the electrical field intensity is increased

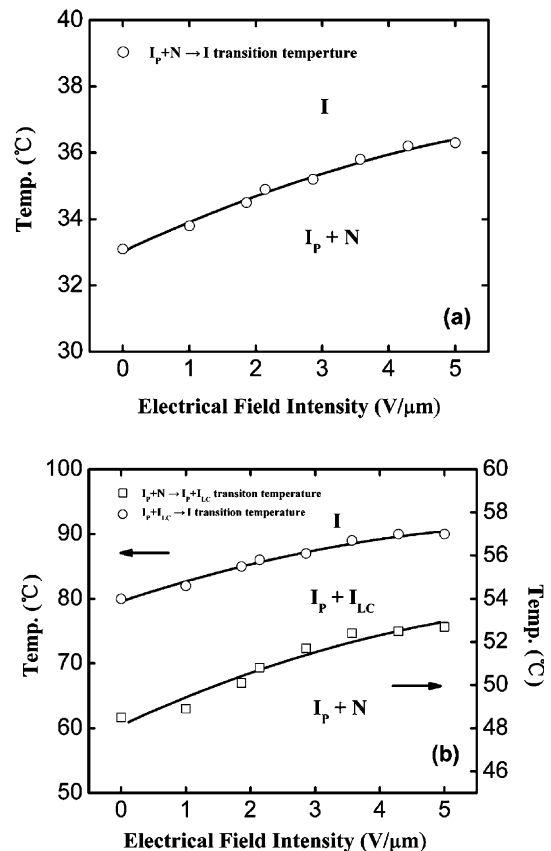


Fig. 4. Dependence of the phase transition temperatures on the electrical field intensity for the TEB/cured E51 mixtures with TEB content of 55 wt.% (a) and 75 wt.% (b). Polymerizations were carried out at 60 °C.

to 5 V/μm, the transition temperature becomes 36.3 °C. As for the mixture with 75 wt.% TEB shown in Fig. 4(b), a similar effect can be observed. The ($I_P + N$) \rightarrow ($I_P + I_{LC}$) and ($I_P + I_{LC}$) \rightarrow (I) transition temperatures increase, respectively, as the intensity of the electrical field is increased.

The influence of the external electrical field applied during the polymerization on the emergence morphologies was also examined. Fig. 5 shows PLM graphs of the morphologies for the mixtures with various TEB contents, which were polymerized in the absence or presence of an electrical field. Prepared without an electrical field, the LC domains are large and coarse, as can be seen in Fig. 5(a). When the film is prepared in the presence of an electrical field, the LC domains tend to be small and uniform, which is shown in Fig. 5(b). The uniform morphologies might be attributed to the faster phase separation induced by the electrical field (see following section). Owing to the faster phase separation during the polymerization, the growing LC domains are arrested by the polymer chains or network early, and the

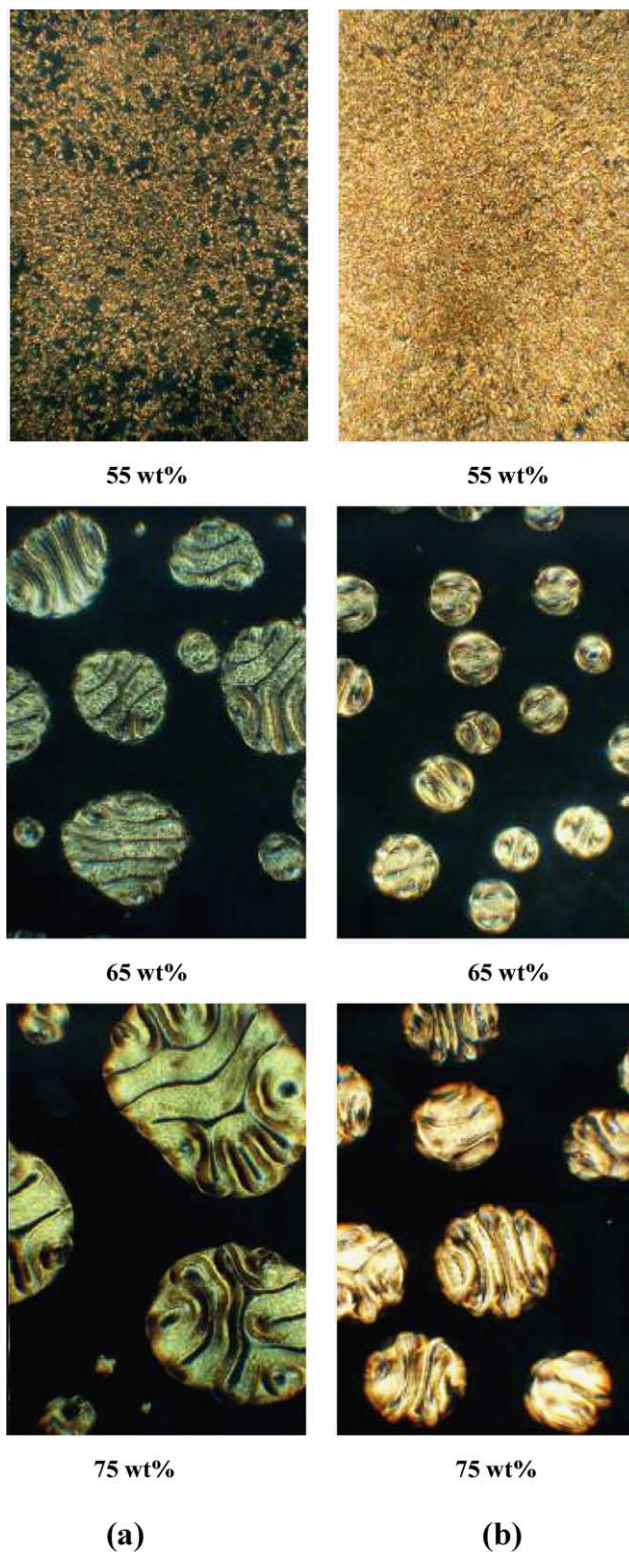


Fig. 5. Morphologies of the TEBCured E51 films with various TEB contents prepared under an electrical field with intensities of 0 V/μm (a) and 1.86 V/μm (b). Polymerizations were carried out at 60 °C (crossed polars 320×).

LC domains have less chance to move and collide with each other, which results in small and uniform LC domains. A closer examination of the morphologies of the 55/45, 65/35 and 75/25 TEB/E51 mixtures shown in Fig. 5 reveals that the LC contents in the initial mixtures have a marked influence on the emerged morphologies of the formed PDLC films. With the increase of the LC content

in the initial mixtures, the size of the phase separated LC droplets increases. The observations also demonstrate that within the experimental condition employed, the formed LC domains become smaller and more uniform when the intensity of the external electrical field is increased. The obtained uniform droplet morphologies are desirable for the electro-optical applications.

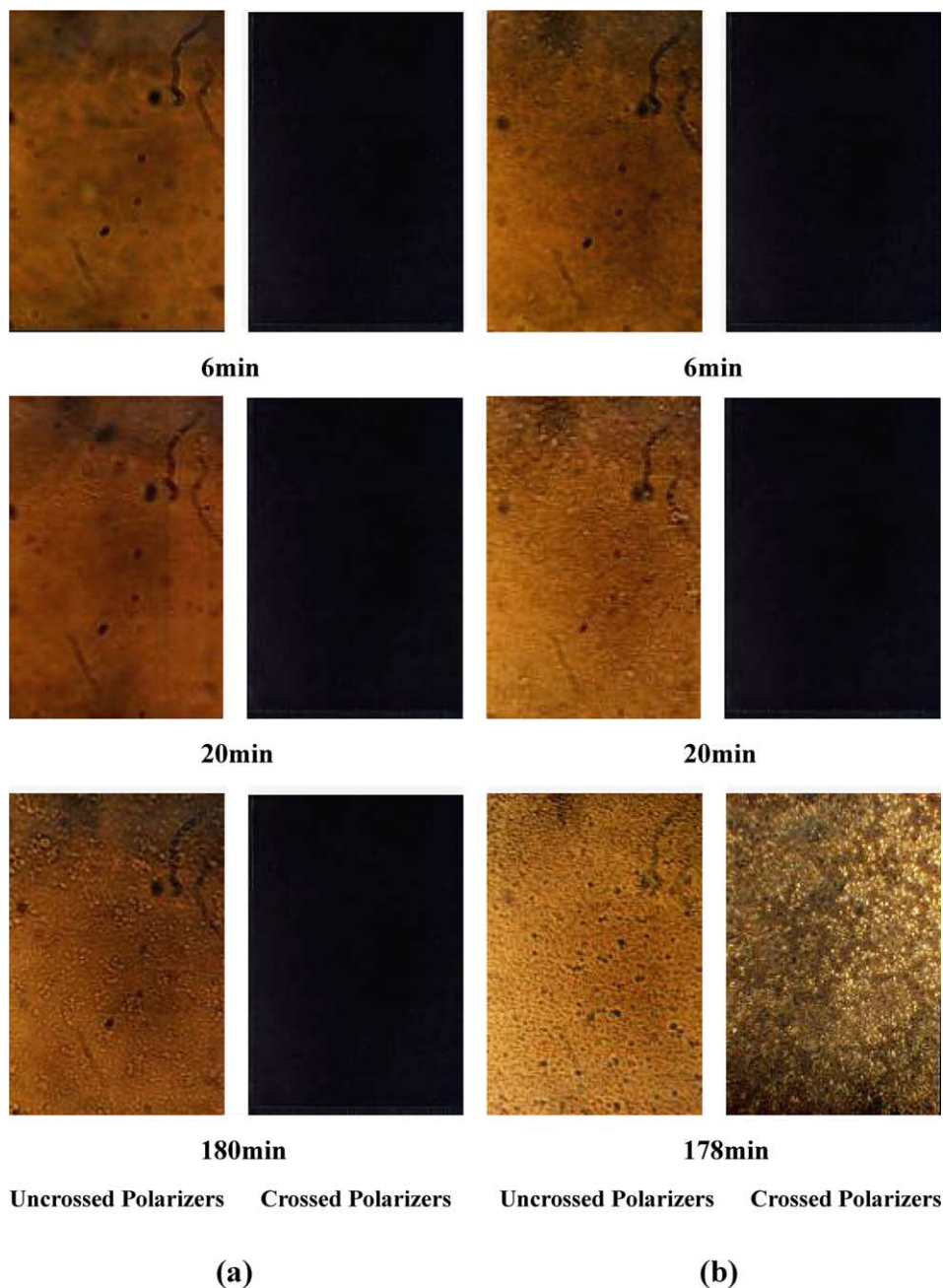


Fig. 6. Temporal evolution of the morphologies of TEB/E51 mixtures during the polymerization under an external electrical field with intensities of $0 \text{ V}/\mu\text{m}$ (a) and $1.86 \text{ V}/\mu\text{m}$ (b). The content of TEB in the mixtures is 75 wt.% (320 \times).

3.3. Effect of external electrical field on morphology development and kinetics of phase separation and LC ordering in PDLC formation

It has been recognized that the final LC domain morphology depends not only on the thermodynamic phase equilibria of the LC/polymer mixtures, but also

strongly on the kinetics of phase separation and LC ordering as most polymer systems rarely reach an equilibrium state.

Fig. 6 presents a typical result for the temporal evolution of the domain morphologies for the TEB/monomer mixtures in the absence and presence of an electrical field during the polymerization. The polymerizations

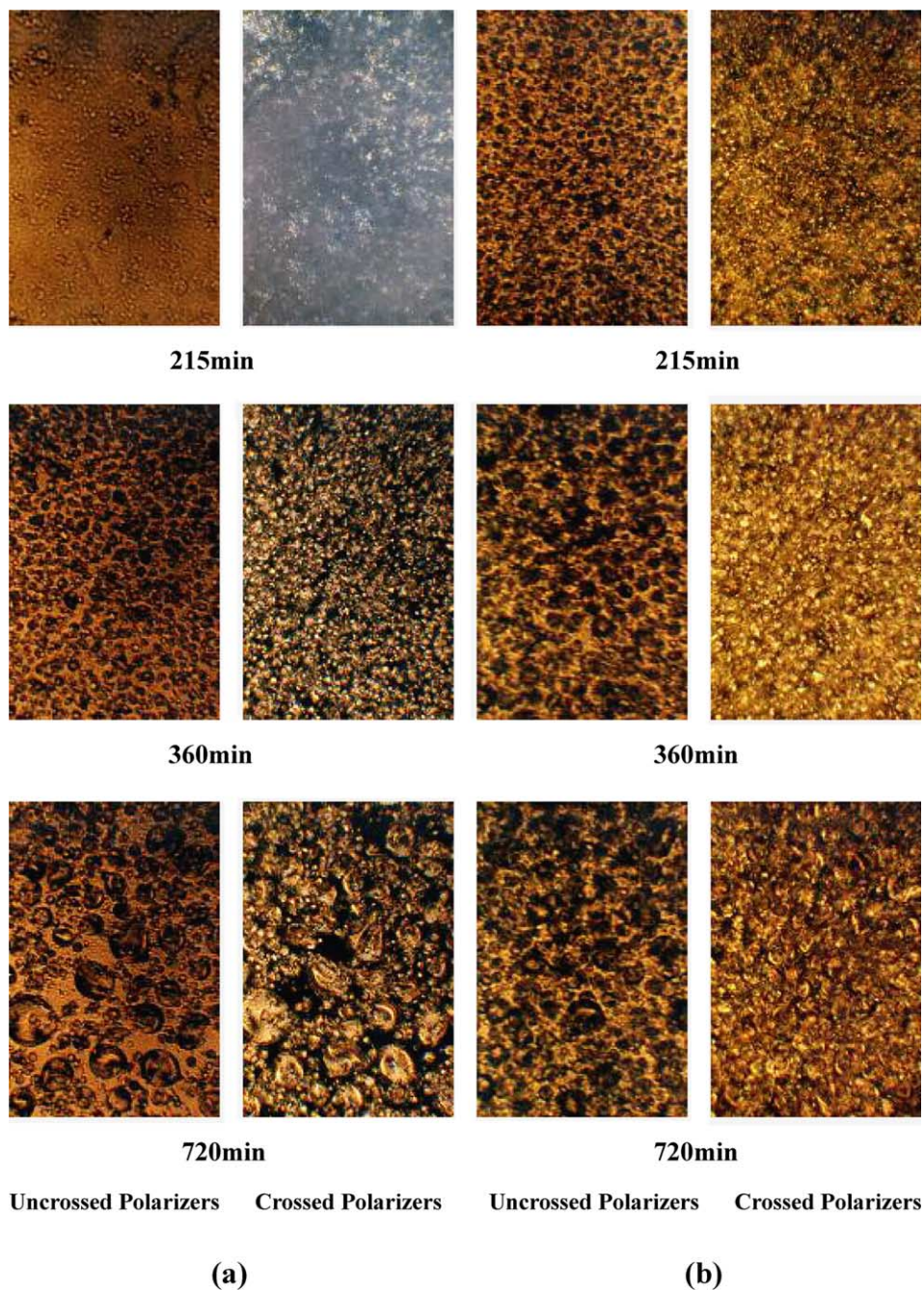


Fig. 6 (continued)

were carried out at 28 °C, where the initial states are in the isotropic phase. The TEB content in the mixtures is 75 wt.%. The PLM graphs shown in Fig. 6(a) are the observations for the system in the absence of an external electrical field. It is seen that nothing is observed for a short reaction time. After polymerizing for about 20 min, small isotropic droplets can be observed under uncrossed polarizers indicating that the isotropic–isotropic phase separation process takes place. When the polymerization advances for about 215 min, the liquid crystal ordering appears as viewed by PLM under crossed polarizers. After then, the nematic LC droplets grow until ultimately confined by the formed network of matrix materials. When an external electrical field with the intensity of 1.86 V/ μm is applied during the polymerization, it can be seen from the PLM graphs in the Fig. 6(b) that both the phase separation and LC ordering processes have been accelerated. The phase separation takes place just after polymerizing for about 6 min and the liquid crystal ordering occurs with the polymerization proceeding for about 178 min. The optical observations also demonstrate that it takes shorter time for both processes to be finished in the presence of the electrical field.

Fig. 7 shows the induction times of the phase separation and LC ordering as a function of the electrical field intensity for the mixture with 75 wt.% TEB polymerized at various temperatures where all the initial states of the polymerizations are in the isotropic phase. Quantitative information regarding the effect of the external field is provided. As Fig. 7 depicts, both pro-

cesses could be accelerated by the electrical field. For instance, when the polymerization is carried out at 28 °C, in the absence of an electrical field, the induction times of the phase separation and LC ordering are 20 and 215 min, respectively. When an electrical field with the intensity of 2 V/ μm is applied, the induction times of the phase separation and LC ordering are decreased to 2 and 170 min, respectively. The results in Fig. 7 also show that within the experimental temperature range employed, the induction times of the phase separation and liquid crystal ordering become longer with increasing polymerization temperature. The electrical field has a stronger influence on the liquid crystal ordering than that on the isotropic–isotropic phase separation. By a separate experiment, the polymerization percentage of E51 monomer with and without external field has been determined by weighting the polymer network formed in the PDLC film. The results show evidence that the polymerization percentage of E51 monomer becomes higher when the systems are subjected to the electrical field.

The accelerated processes of the phase separation and LC ordering could be explained by the following reasons. First, the existence of an external electrical field could make the LC molecules easily align, giving rise to a smaller entropy change of mixing and a poor miscibility. Therefore, the immiscibility between the LC molecules and reacting polymers increases rapidly when the systems are subjected to an external electrical field. Under such a circumstance, the UCST curve would shift upward faster. When the UCST curve surpasses the reaction temperature, the phase separation takes place. The ordered LC molecules exclude the reacting polymer strongly from the LC-rich domain and the LC content might increase rapidly to a critical value for the LC phase formation [15,22]. Second, the presence of the external field may also increase the nucleation density and therefore produce a larger number of densities of small LC droplets, relative to the case where no external field exists, accelerating the ordering process and giving a more uniform morphology.

4. Conclusions

In the present work, we have established the phase diagrams for the mixtures of multi-component eutectic nematic liquid crystal (TEB) and monomer (E51) with and without an external electrical field applied. Based on the phase diagrams, the effects of the external electrical field on the phase diagrams and morphologies of TEB/cured E51 systems have been investigated. The influence of the applied electrical field on the processes of the isotropic–isotropic phase separation and LC ordering in PDLC formation has also been studied. The results are summarized as follows:

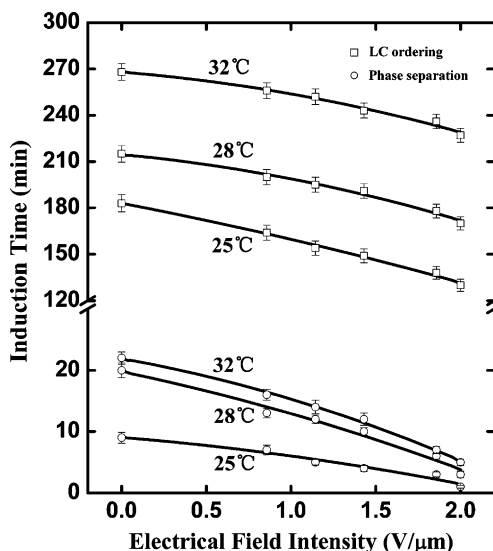


Fig. 7. Dependence of the induction times of the isotropic–isotropic phase separation and liquid crystal ordering processes on the electrical field intensity for the TEB/E51 mixtures with 75 wt.% TEB polymerized under various temperatures.

1. The applied electrical field renders the ($I_M + N$) coexistent phase region and ($I_M + I_{LC}$) phase boundary of the LC/monomer mixtures shift upward to higher temperatures. The ($I_M + N$) \rightarrow (I) and ($I_M + I_{LC}$) \rightarrow (I) transition temperatures of the LC/monomer mixtures increase as the applied electrical field intensity increases.
2. The electrical field applied during the cross-linking polymerization shifts the ($I_P + N$) coexistent phase region and ($I_P + I_{LC}$) phase boundary of the LC/polymer mixtures to higher temperatures. Such an effect was observed to be more pronounced when the electrical field intensity becomes higher.
3. The optical observations and the analyses of the induction times reveal that the applied electrical field during the polymerization can highly accelerate the processes of the isotropic–isotropic phase separation and LC ordering for the LC/monomer mixtures.

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