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Electro-optical effects of anchoring transition in polymer dispersed liquid crystal films

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Two groups of polymer dispersed liquid crystal films have been studied, one with a fixed cell thickness but varying liquid crystal (LC) concentrations and the other with a fixed LC concentration but varying cell thicknesses. A sudden decrease in transmittance with increasing temperature was observed for films whose LC domain sizes were comparable to their cell thicknesses. In particular, spontaneous alignment of LC directors was observed below $\sim 20^\circ\text{C}$ when LC domains were formed spanning the space between upper and lower indium tin oxide-coated glass substrates. With increasing temperature, this axially aligned homeotropic configuration changed gradually into homogeneous configurations. We believe that the sudden decrease in transmittance originated from the anchoring transition at the glass substrates and polymer walls. In addition, it has been found that the intensity of ultra violet irradiation has a strong effect on the director configuration of LC domains, and that the cure temperature affects the anchoring transition temperature significantly.

1. Introduction

Polymer dispersed liquid crystal (PDLC) systems have been studied extensively for their potential applications such as reflective displays, light windows, and holographic gratings [1–7]. PDLCs are usually composed of microdomains of liquid crystal dispersed in a polymer matrix. Generally, the final properties of PDLC films are affected by a number of parameters including LC domain size, shape, density, director configuration, and anchoring properties [8–12].

In principle, the transmittance of PDLC films depends strongly on the difference between the effective refractive index of the LC domain, n_d , and the refractive index of the polymer matrix, n_p . Without application of an applied electric field, the LC director axis of each LC domain varies randomly, and light entering the film passes through a number of LC domains and polymer walls. Because the refractive index varies rapidly from domain to domain, the light is strongly scattered due to the refractive index mismatch. At high electric fields, n_d approaches the ordinary refractive index of the liquid crystal, n_o , and a high degree of transparency in PDLC films is achieved when n_d is equal to n_o . At intermediate fields, n_d depends strongly on the applied electric field and on the temperature, causing variation in transmittance of the PDLC films.

Recently, the effect of temperature on the electro-optical properties of PDLC films has been studied by several research groups. Amundson investigated optical transmittance as a function of applied voltage and temperature for PDLC films based on E7/NOA65 [9]; the films had a cell thickness of $18\ \mu\text{m}$ and droplet sizes on the order of $1\text{--}5\ \mu\text{m}$ in diameter. It was reported that the switching voltage decreased monotonically with increasing temperature. This observation is usually expected for typical PDLC films because the order parameter of the liquid crystal decreases with increasing temperature. Fuh *et al.* studied PDLC films based on E7/EPO305 which had a cell thickness of $56\ \mu\text{m}$ and droplet size on the order of $1\ \mu\text{m}$ in diameter [13]. Interestingly, they found that a plot of transmittance as a function of temperature displayed broad peaks, and explained their results by variation in the refractive indices of the polymer matrix and the LC droplets. Han also intensively studied PDLC films, based on TL213/PN393, with a thin walled foam-like morphology due to the high concentration of liquid crystal [14]. The author studied the optical transmittance of a PDLC film with 82.9 wt% TL213 which had a cell thickness of $13\ \mu\text{m}$ and a LC domain size of $\sim 10\ \mu\text{m}$ in diameter. It was found that the transmittance obtained at a fixed voltage decreased with increasing temperature above $\sim 43^\circ\text{C}$, independent of the driving frequency of the applied voltage. It was also reported that a plot of the transmission as a function of temperature exhibited

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corresponding peaks near 43°C, and that the shapes of the peaks depended markedly on the magnitude of the applied voltage. The experimental results were explained by conductivity effects due to free ions newly created at high temperatures.

As an extension of his work, the author studied PDLC films with relatively lower TL213 concentrations [15]. These films had a cell thickness of 13 μm and LC domain size on the order of 1 μm in diameter. It was interesting that the PDLC film with 80 wt% TL213 did not exhibit an abrupt decrease in transmittance with increasing temperature, as was observed for the PDLC film with 82.9 wt% TL213. Based on measurements on the transmittances and decay times, it was tentatively concluded that the sudden decrease in transmittance might originate from the temperature-dependent nematic to isotropic (N–I) phase transition under the PDLC environment. On the other hand, Fujisawa *et al.* have studied the electro-optical properties of polymer network liquid crystals (PNLCs) [16], which are different from PDLCs in morphology. While LC domains are surrounded by a polymer material and are isolated from each other in PDLCs, a three-dimensional polymer network is formed in continuous LC layers in PNLCs. Similar dependences of the transmittance and the decay time on temperature were obtained and explained in terms of the anchoring transition at polymer walls. Amundson *et al.* have also studied electro-optic effects due to anchoring transitions in PDLC films made from TL205 (EM industries) and a monomer mixture [17]. On heating the film, they found a sudden and dramatic change in the microscopic observations and attributed it to the homeotropic to homogeneous anchoring transition. It was also demonstrated that the anchoring transition temperature could be controlled by modifying the polymer side group and by mixing side groups.

In more recent studies, we found evidence that the rapid decrease in transmittance with increasing temperature observed for our PDLC systems could be associated with the anchoring transition of LC molecules at glass substrates and polymer walls. Furthermore, it was found that change in the optical transmittance associated with the anchoring transition was closely related to the PDLC cell thickness and LC domain size. Motivated largely by this experimental evidence, we have prepared and investigated two distinctive groups of PDLC films for a more detailed understanding.

2. Experimental

A variety of methods have been developed to fabricate PDLC films. One of the most widely used schemes in the fabrication of PDLC films is polymerization-induced

phase separation (PIPS), which was pioneered by Doane and other workers [1, 18, 19]. In this scheme, a homogeneous mixture of a liquid crystal and a prepolymer containing an appropriate amount of initiator is first prepared. Polymerization is then induced through the application of heat or ultra violet (UV) irradiation, depending on the type of initiator. As a result of the polymerization, the liquid crystal separates from the polymer matrix and forms diverse patterns of LC domains, depending on the preparation conditions.

In the present work, the PIPS method was employed to prepare PDLC films from a commercially available liquid crystal mixture (TL213, Merck) and a UV-curable prepolymer material (PN393, Merck). The N–I transition temperature for TL213 is 87°C. For sample preparation, we first fabricated empty PDLC cells and introduced homogeneous mixtures of TL213 and PN393 into a narrow gap between two indium tin oxide (ITO) glass plates by using capillary filling. The cell gap was controlled by using appropriate sizes of glass beads. The films were then cured by UV irradiation from a mercury arc lamp (Model 66033 and 68810, Oriel). The UV intensity was varied in the range 40–200 mW cm⁻², and the polymerization temperature was controlled in the range of 20–55°C. The driving signal of the applied voltage was sinusoidal at 1 kHz and the applied voltage was in root-mean-square (rms) voltage, unless otherwise specified. The fabricated films had a thin-walled foam-like morphology so that LC domains were separated by thin polymer walls. LC domain sizes were on the order of 1–12 μm in diameter, depending on the preparation conditions.

The electro-optical properties of the PDLC films were measured with a He-Ne laser and a power meter (Model 70260 and 70261, Oriel). The sample temperature was controlled within ±0.5 K by using a Janis Cryostat (Model VPF-475) with a temperature controller (Model 330, Lake Shore). The PDLC morphology was studied using an optical microscope equipped with a camera (Model CH40, Olympus). The temperature of the PDLC films for microscopic studies was controlled using an INSTEC hot stage (Model BS60 with optional 95°C limit).

3. Results and discussion

In the present work, we were most concerned about the effects of the cell thickness and the LC domain size on the abruptly decreasing transmittance with increasing temperature. Accordingly, we have prepared two distinctive groups of PDLC films and studied the effects of temperature on their electro-optical properties. For one group, the cell thickness was fixed at 13 μm and the

LC contents varied (75.9, 78.3, 80, 81.3 and 82.9 wt%). For the other group, the LC content was fixed at 82.2 wt% LC and the cell thickness varied (3.25, 6, 13, 50 and 100 μm). It should be noted that LC content here refers to TL213 content. Transmittance figures were normalized as 0% at 0 V_{rms} and 100% at saturation voltage.

3.1. LC concentration dependence

Figures 1(a) and 1(b) show the dependence of light transmittance on temperature at various voltages for the 75.9 and 78.3 wt% PDLC films. For these films, the transmittance increased with increasing temperature at a fixed applied voltage. This observation is easily understood because the switching voltage required to reorient LC molecules usually decreases as the temperature increases. It is well known that reorientation fields for PDLC films vary by an order of magnitude, ranging from 0.5 to 10 $\text{V}\mu\text{m}^{-1}$ and are inversely proportional to the LC droplet size. In our case, average LC domain size was 1–2 μm and the reorientation field for maximum transmittance was $\sim 3.85 \text{V}\mu\text{m}^{-1}$. These results can be compared with experimental results obtained for PDLC films with higher LC contents. Figure 1(c) shows a plot of transmittance as a function of temperature at a given applied voltage. In figure 1(c), different voltages were used for the films with low LC content because these films had smaller LC domains and therefore needed higher applied voltages for reorientation of the LC molecules. Films with 81.3 and 82.9 wt% TL213 showed abruptly decreasing light transmittance above $\sim 43^\circ\text{C}$ with increasing temperature, while films with 75.9, 78.3 and 80 wt% TL213 showed no sudden decrease in transmittance. These results imply that the rapid decrease in transmittance above $\sim 43^\circ\text{C}$ depends significantly on the LC concentration; and the light scattering markedly increased for PDLC films with higher LC contents. This observation will be discussed in detail in the following paragraphs. It has been known that the sizes of LC domains in PDLC films based on TL213/PN393 are extremely sensitive to the LC concentration. In fact, the average LC domain size was observed to be smaller for films with lower concentrations of LC. For example, the average diameters of LC domains observed by polarizing microscopy were 1, 2, 2.5, 10 and 12 μm for 75.9, 78.3, 80, 81.3 and 82.9 wt% films, respectively. Therefore, the sudden decrease in transmittance observed for PDLC films with higher LC content can also be considered to arise from size effects of LC domains. It can be seen that the sudden decrease

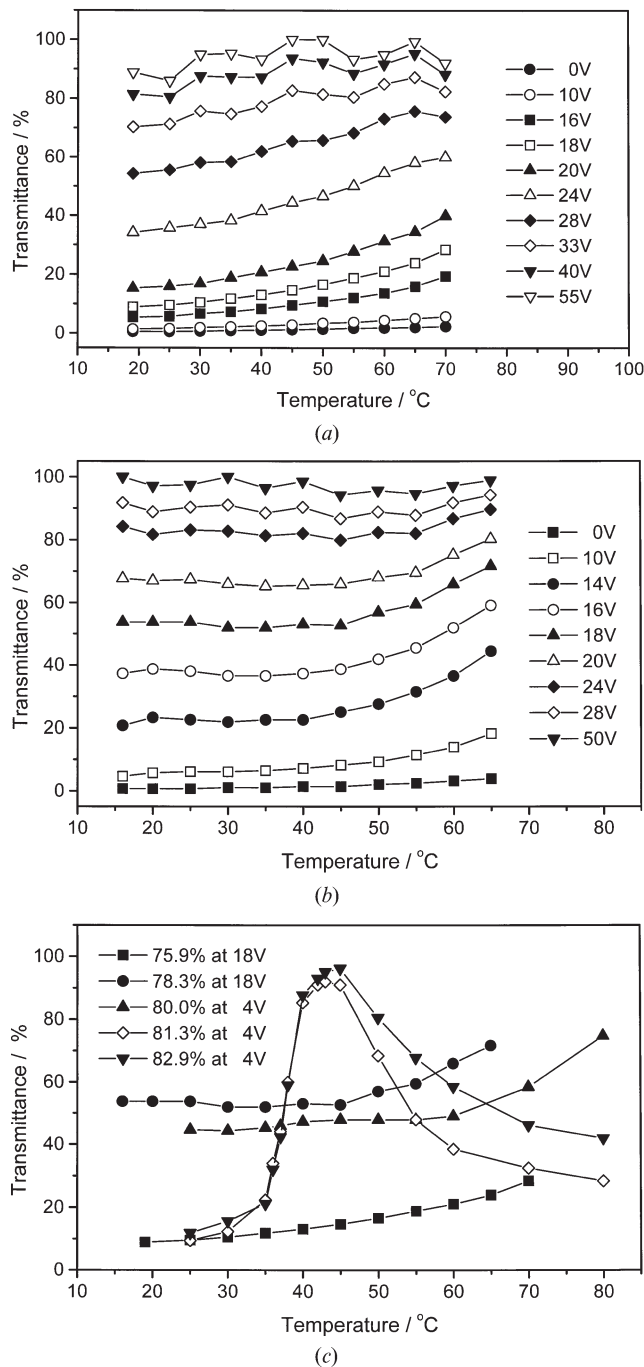


Figure 1. Transmittance versus temperature for PDLC films with a cell thickness of 13 μm containing (a) 75.9 wt% TL213 for various voltages, (b) 78.3 wt% TL213 for various voltages, and (c) different TL213 contents (75.9, 78.3, 80.0, 81.3 and 82.9 wt%).

in transmittance was observed for films whose LC domain sizes were comparable to the cell thickness of 13 μm .

3.2. Cell thickness dependence

Our study described in the previous section suggests that the ratio of LC domain size to cell thickness could be a critical factor for the sudden decrease in transmittance. For a better understanding of the relationship between LC domain size and cell thickness, we investigated PDLC films having the same LC domain size but different cell thicknesses. For this purpose, we fabricated five PDLC films with the same LC content of 82.2 wt% but diverse cell thicknesses (3.25, 6, 13, 50 and 100 μm). We examined optical microscope images of these films under crossed polarizers and found that they had a thin-walled foam-like morphology due to the high concentration of the liquid crystal. The average diameter of the LC domains was 11 μm for the films with cell thicknesses of 3.25 and 6 μm , and 9 μm for the films with cell thicknesses of 13, 50 and 100 μm .

Figures 2(a) and 2(b) show the dependences of the light transmittances on temperature at various voltages for the 6 and 50 μm PDLC films, respectively. The reorientation field for these films was $\sim 0.5 \text{ V} \mu\text{m}^{-1}$. In figure 2(a), the transmittance at zero external voltage for the 6 μm PDLC film was nearly constant below $\sim 20^\circ\text{C}$ and decreased abruptly at $\sim 20^\circ\text{C}$. The abrupt decrease in transmittance was hindered by an applied voltage. This is apparent because the decrease in the transmittance was steep at low voltages and became gradual with increasing voltage. It is evident that two competing forces are involved. One is the thermally driven force to destroy the initial LC director alignments; the other is the force driven by the applied electric field to restore the initial LC director alignments. This observation demonstrates that abrupt change in the transmittance resulted from the sudden modification of LC alignments of the LC molecules. In contrast, the transmittance for the 50 μm PDLC film showed a slightly increasing behaviour with increasing temperature. For summary, a plot of transmittance versus temperature for various films with different cell thicknesses is presented in figure 2(c). It is apparent from this figure that experimental results for films with smaller cell thicknesses (3.25, 6 μm) were quite distinct from those for films with larger cell thicknesses (50, 100 μm). In particular, it is interesting to note that the films with cell thicknesses of 3.25 and 6 μm were, even without application of an external electric field, transparent below $\sim 20^\circ\text{C}$. This implies that the director orientation for these films was spontaneously aligned along the direction normal to the ITO-coated glass substrates in the PDLC cells. A similar effect was observed for the film with a cell thickness of 13 μm at lower temperatures below $\sim 10^\circ\text{C}$ (not shown here). On

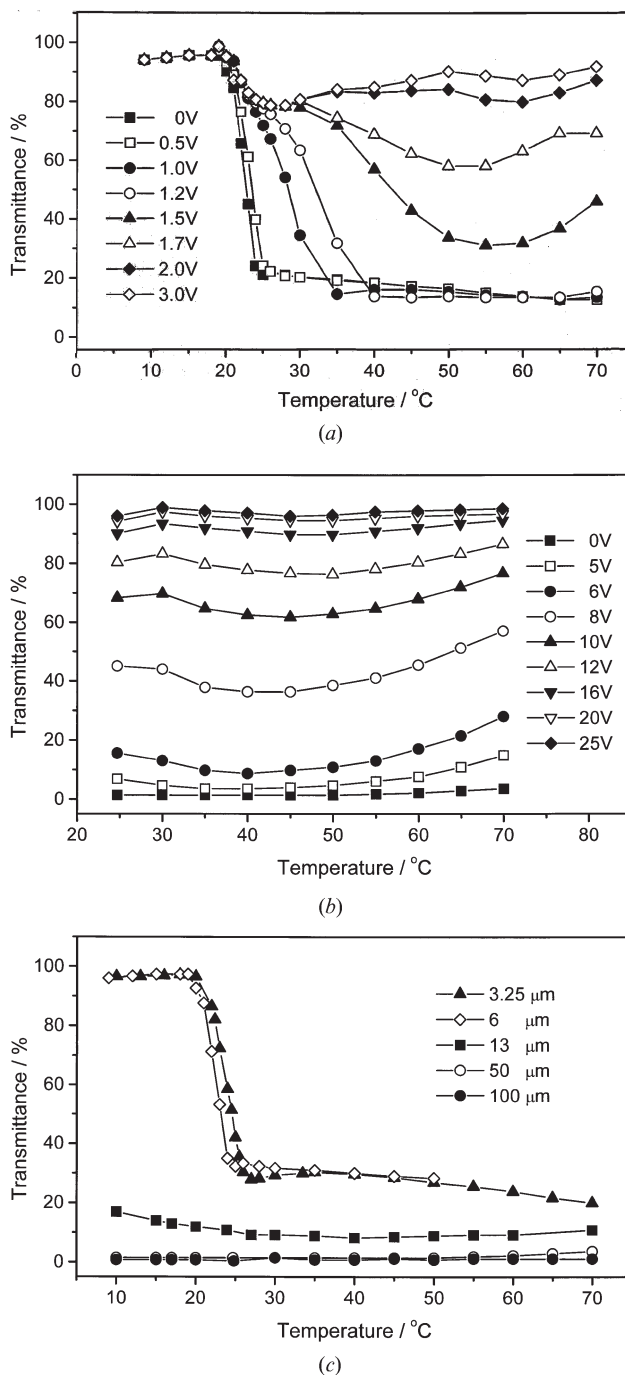


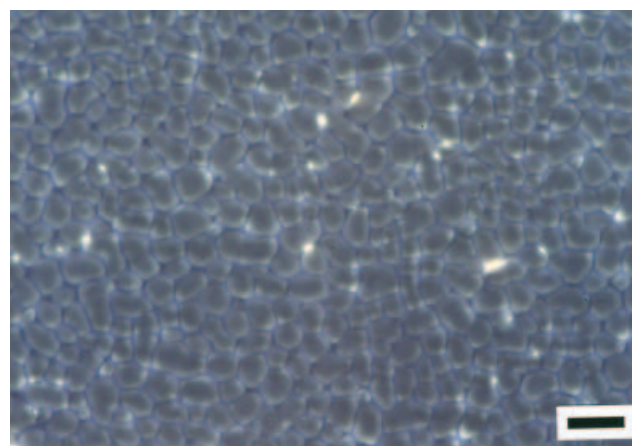
Figure 2. Transmittance versus temperature for PDLC films with 82.2 wt% TL213 and (a) a cell thickness of 6 μm , (b) a cell thickness of 50 μm , and (c) different cell thicknesses (3.25, 6, 13, 50 and 100 μm). In (c), transmittance was measured with no external voltage applied.

the other hand, electro-optical characteristics for the films with larger cell thicknesses (50, 100 μm) showed no spontaneously aligning behaviour, as shown in figure 2(c).

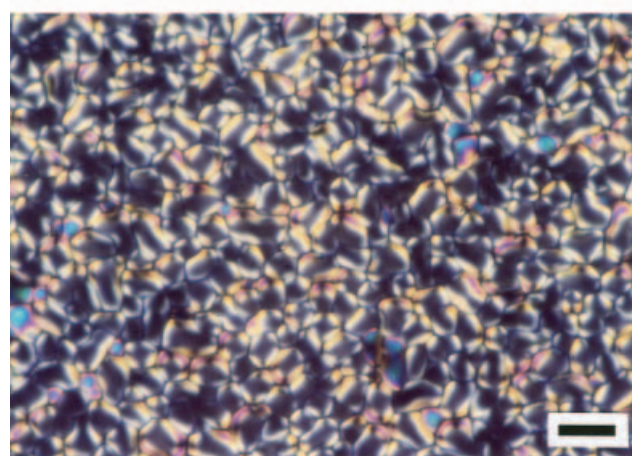
In order to investigate evolution of the directional configurations as a function of temperature, we examined films with an optical microscope at varying temperatures and applied voltages. Figure 3 shows photomicrographs of the 6 μm film between crossed polarizers. Similar images were observed for the 3.25 μm film (not shown here). Figure 3(a) shows an optical micrograph obtained at 14°C. Most LC domains and the vertical polymer walls appeared dark, while the boundaries near the vertical polymer walls were dimly bright between crossed polarizers. This observation indicates that the director orientation is axially aligned except for the boundaries of the polymer walls; see figure 4(a). In other words, the LC directors in the dark region were almost completely aligned along the direction normal to the ITO-coated glass substrates in the PDLC cell. In contrast, the dimness near the vertical polymer walls means that the LC director configuration at the boundaries is perpendicular to the polymer walls (homeotropic). A schematic illustration of the axially aligned director configuration is shown in figure 4(a).

As the temperature was increased to 22°C, this configuration (appearing dark) changed gradually into the homogeneous configuration (appearing bright), as shown in figure 3(b). It is interesting to note that a change in appearance from dark to bright started first in the vicinity of vertical polymer walls, and spread into the central region of the individual LC domains during the configurational transition of the LC director. This is evident in figure 3(b) from the observation that bright border regions near the vertical polymer walls expanded, while dark central regions shrank proportionally. With further heating, dark central regions associated with the axial configuration of LC domains eventually changed into two bright configurations of different appearance, as shown in figure 3(c).

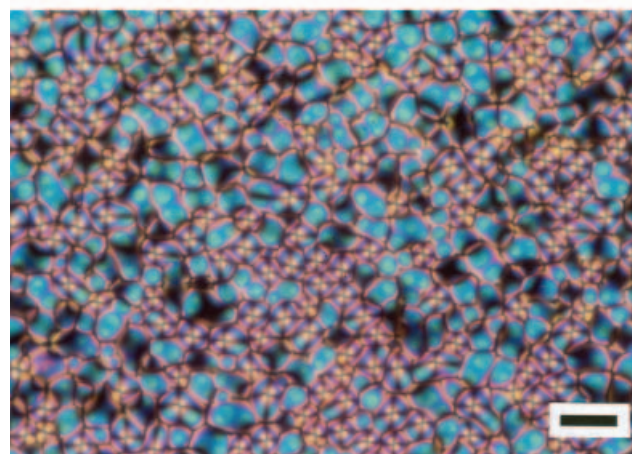
In figure 3(c), one can observe yellowish domains with a cross at the centre (type A), and cyan domains (type B). To investigate the director configurations, these domains were observed between crossed polarizers with increasing applied voltage. The microscopic view of an individual domain appeared darker gradually in both types with increasing applied voltage, which resulted from the increase of axial alignments in the individual LC domain. In addition, we studied in-plane birefringence by rotating the films while observing under crossed polarizers. For type A domains, the overall microscopic view remained unchanged except for a rotating cross at the centre of each LC domain as the film was rotated, indicating that type A has a radial director configuration in the central region with a cylindrical symmetry. A schematic illustration of this



(a)



(b)



(c)

Figure 3. Optical micrographs (400 \times) of a film with 82.2 wt % TL213 and a cell thickness of 6 μm between crossed polarizers. The images were obtained at (a) 14°C, (b) 25.5°C, and (c) 35°C. The scale bar corresponds to 20 μm .

radial director configuration is shown in figure 4(b). For type B domains, the individual domain changed

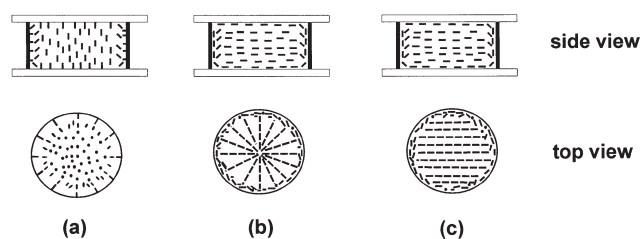


Figure 4. A schematic illustration of (a) the axially aligned director configuration below $\sim 20^\circ\text{C}$, (b) the radial director configuration, and (c) the planar director configuration.

from cyan to relatively dark in colour, and a broad black cross at the centre appeared when the sample was rotated, see figure 3(c). Furthermore, the colour of the individual domain changed from cyan to dark and then dark to cyan alternately at every 45° rotation of the sample, indicating high in-plane birefringence. This observation strongly indicates that a type B domain has a director configuration, as illustrated schematically in figure 4(c). In summary, it is considered that variation in the director configuration with increasing temperature is associated with the anchoring transition at polymer walls.

All of the PDLC films discussed above were fabricated by the same process, and no surface treatment was performed on the ITO-coated glass substrates. Furthermore, the sizes of LC domains were about the same (9–11 μm) for all of the films. Therefore, it appears that differences in the cell thickness caused the distinct optical characteristics. More specifically, it is believed that a strong correlation exists between the sudden decrease in transmittance observed for the 3.25 and 6 μm films and the number of LC domains existing across the cell gap. It is thought that spontaneous alignment of the LC takes place when only a single LC domain is formed across the space between the upper and lower ITO-coated glass substrates, spanning the cell gap, see figure 4(a). In this case, LC domains were separated by thin polymer walls extending vertically from one substrate to the other. It is reasonable to assume that the substrates were also coated with the polymer, and that the glass substrates could play an important role in the spontaneous LC alignment. It is believed that LC molecules were anchored homeotropically to the glass substrates at low temperatures and the entire LC domain tended to align axially as a result of this anchoring force (except for boundaries near polymer walls), as shown in figure 4(a). With increasing temperature, variation in the director configuration, caused by the homeotropic to homogeneous anchoring transition, yielded a corresponding change in the transmittance, which was most clearly observed for

the 3.25 and 6 μm films. The phenomena observed in the 3.25 and 6 μm films can be used as the basis of optically and thermally switching light shutter valves.

A question can be raised as to why the abrupt decrease in transmittance due to the anchoring transition was not observed when a number of LC domains existed across the PDLC cell gap. The reason is not clear at this point. We think that the configurational change in the director vector resulting from the anchoring transition occurs only in LC domains which are in direct contact with the glass substrates. In this case, variation in transmittance, due to the anchoring transition in the LC domains near the glass substrates, can be easily screened by multiple scattering effects, because light entering the PDLC film should still pass through a number of LC domains and polymer walls inside the PDLC film. Therefore, the sudden decrease in transmittance was observed only for films whose cell thicknesses were comparable to or smaller than the LC domain size.

3.3. Curing condition dependence

In order to investigate how different curing conditions affect the optical characteristics of the PDLC films, we fabricated 3.25 and 6 μm PDLC films under different curing conditions. In the preparation of 3.25 μm films, we varied the intensity of UV irradiation while keeping the curing temperature at 50°C . Intensities of UV irradiation for the 3.25 μm -#1 and 3.25 μm -#2 films were 40 and 200 mW cm^{-2} , respectively. On the other hand, in preparation of 6 μm films, we varied the curing temperature while keeping the intensity of UV irradiation at 200 mW cm^{-2} . The cure temperatures for the 6 μm -#1 and 6 μm -#2 cells were 22°C and 50°C , respectively.

Figure 5 shows a plot of light transmittances as a function of temperature for these films. These results demonstrate that the optical characteristics are strongly dependent on the cure temperature as well as on the intensity of UV irradiation. The 3.25 μm -#1 film showed no observable anchoring transition down to 10°C , while the 3.25 μm -#2 film showed an anchoring transition at 20°C . This result demonstrates that UV intensity has a marked influence on the director configuration of LC domains. The photomicrographs of the 3.25 μm films under crossed polarizers (not presented here) showed that the two films had very similar morphological LC domain structure, but quite distinct director configurations. More specifically, the 3.25 μm -#1 film consisted of LC domains mainly with homogeneous director configuration, see figure 3(c), while the 3.25 μm -#2 film was composed of

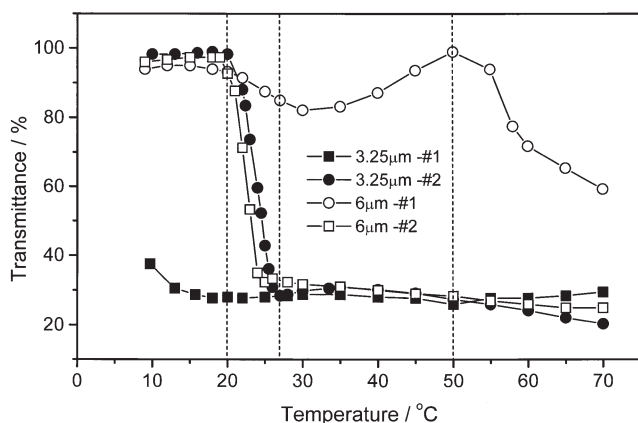


Figure 5. Transmittance versus temperature for the 3.25 and 6 μm PDLC films prepared under different curing conditions. The intensities of UV irradiation for the 3.25 μm -#1 and 3.25 μm -#2 films were 40 and 200 mW cm^{-2} , respectively, at a fixed curing temperature 50°C. The curing temperatures for the 6 μm -#1 and 6 μm -#2 films were 22 and 50°C, respectively, at a fixed intensity of UV irradiation of 200 mW cm^{-2} .

LC domains with homeotropic configuration, see figure 3(a).

The two 6 μm films cured at different temperatures showed another interesting feature. Figure 6 shows a photomicrograph of the 6 μm -#1 film at 17°C between crossed polarizers. It is worth noting that the morphological structure of LC domains of this film was quite different from that of the 6 μm -#2 film. The photomicrograph of the 6 μm -#2 film is not shown here but is almost identical with that shown in figure 3(a). Interestingly, the 6 μm -#1 film exhibited an anchoring transition (homeotropic to homogeneous) at 50°C, which is considerably higher than the 20°C for the 6 μm -#2 film, as seen in figure 5. It is believed that the observed change in the anchoring transition temperature originated from the difference in their morphologies.

4. Conclusions

We have studied two groups of PDLC films to understand the effect of temperature on their electro-optical properties. One group included PDLC films with a fixed cell thickness but varying LC concentrations; the other group had a fixed LC concentration but varying cell thicknesses.

From the study of LC concentration dependence, it was found that an abrupt decrease in transmittance took place in the films whose LC domain sizes were either comparable to or smaller than their cell thickness. Furthermore, the study of cell thickness dependence revealed that spontaneous alignment of LC directors occurred below 20°C, when LC domains

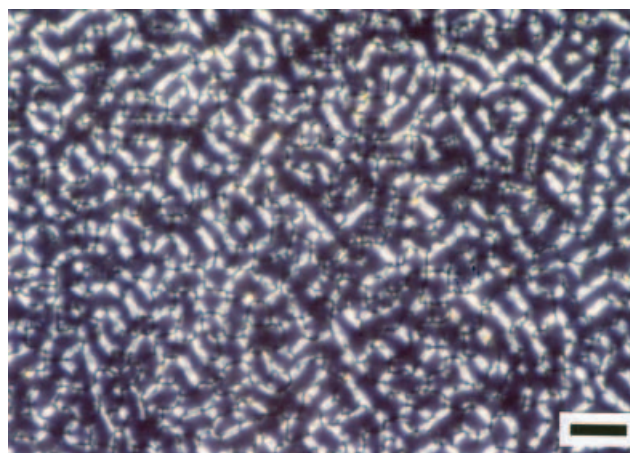


Figure 6. Photomicrograph (400 \times) of the 6 μm -#1 film at 17°C between crossed polarizers. The scale bar corresponds to 20 μm .

were formed spanning the space between upper and lower ITO-coated glass substrates. As the temperature was increased, this axially aligned homeotropic configuration changed gradually into the homogeneous configuration. We believe that the abrupt decrease in transmittance arose from the anchoring transition at glass substrates and polymer walls. In addition, it was observed that the intensity of UV irradiation had a strong effect on the director configuration of LC domains and that the curing temperature considerably affected the anchoring transition temperature.

Acknowledgements

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