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Rotation of director during holographic recording in polymer-dispersed liquid crystals

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Rotation of director during holographic recording in polymer-dispersed liquid crystals

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The formation of holographic polymer-dispersed liquid crystals was investigated in detail. The director was found to rotate during holographic recording owing to the change in morphology and migration of liquid crystals. A strongly anisotropic grating was constructed with variable diffraction efficiency that can be controlled by applying an analogue voltage to the nematic liquid crystal. Effects of post-curing on electro-optical properties were also investigated and the switching characteristics were strongly affected by the intermediate layer between the polymer and liquid crystal.

Keywords: polymer-dispersed liquid crystal; holographic recording; director rotation

1. Introduction

The possibility of using polymer-dispersed liquid crystals (PDLCs) as recording media for thick, electrically switchable, holographic gratings makes them suitable for different applications in photonic devices (*1–39*). When an electric field is applied to the hologram via a pair of transparent electrodes, the natural orientation of liquid crystal (LC) droplets is changed, causing the refractive index modulation of the fringes to reduce and the hologram diffraction efficiency to drop to very low levels, effectively erasing the hologram. Sutherland *et al.* (*1*) reported electrical switching of the diffraction efficiency in volume Bragg gratings written holographically in PDLCs. Volume transmission holographic gratings in a photoreactive mixture containing nematic LC were recorded with 488 nm Ar ion laser light. The diffraction efficiency of the resulting holographic PDLC could be switched from a high value of about 50% to a value near zero for fields of about $11\,\mathrm{V\,\mu m^{-1}}$. Tondiglia *et al.* (*2*) reported storage and electrical switching for holographic image data in the same photoreactive mixture. The holographic transmission gratings showed fast response and relaxation times of 25 and 44 µs, respectively. Karasawa and Taketomi (*4*) investigated the effects of the material systems involved in the fabrication on the polarisation behaviour of a volume holographic grating. Depending on the kinds of materials used in the grating formation, the diffraction properties were such that either *p*- or *s*-polarised light was strongly diffracted, whereas light with the opposite polarisation was very weakly diffracted. The polarisation dependence of the diffraction properties were strongly affected by the type of photoreactive monomer and LC. Sutherland *et al.* (*9*) investigated the temporal evolution of an anisotropic reflection grating produced in a holographic PDLC film. They found that the grating is preceded in time by an isotropic concentration grating, and that the development of the anisotropic grating can be delayed until several seconds after laser exposure. Liu *et al.* (*12*) investigated the dependence of the diffraction efficiency on different monomers, LC content, exposure energy, and exposure temperature. They optimised fabrication conditions and obtained a highest diffraction efficiency of over 80%. Klosterman *et al.* (*19*) determined that the switching electric field of holographic PDLC could be reduced by adding a non-reactive surfactant-like molecule to the reactive syrup. A substantial lowering of the switching field has been attributed to a modification of the anchoring properties of the LC molecules and the polymer host. Liu *et al.* (*27*) intensively investigated the effects of the surfactants on the electro-optical properties of holographic PDLC Bragg gratings. The threshold electric field was drastically reduced from $13\,\mathrm{V\,\mu m^{-1}}$ to $2.3\,\mathrm{V\,\mu m^{-1}}$, while there were fast response and relaxation times of 60 and 80 µs, respectively. Woo *et al.* (*38*) also studied the effects of octanoic acid as the surfactant on morphology and the switching of holographic PDLC based on polyurethane acrylates. Droplet size decreases with an increase in the surfactant content, and this strongly affects the electro-optical properties of the holographic PDLC.

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The electro-optical properties of a holographic PDLC are strongly affected by the morphology and anchoring conditions. In this paper, a detailed investigation is reported of the diffraction properties and the morphology during the formation of a holographic PDLC. The direction of the LC director was found to rotate during the holographic recording. Additionally, the effects of uniform post exposure were investigated and the electro-optical properties were found to vary owing to changes in both morphology and anchoring conditions.

2. Experiments

The nematic mixture pre-polymers used in this experiment were E7 (Merck Japan Co. Ltd) and NOA65 (Norland Products, Inc), respectively. The extraordinary and ordinary refractive indices of E7 are 1.736 and 1.511, respectively. The refractive index of the photo-cured NOA65 is 1.52, which is nearly equal to the ordinary refractive index of E7. A homogeneous pre-polymer mixture of 30 wt % E7 and 70 wt % NOA65 was sandwiched between two indium–tin–oxide (ITO)-coated glass substrates; 10 µm polyester film spacers maintained uniform film thickness. The above procedure yielded homogeneous films with good optical quality. Samples were exposed to a two-beam interference pattern created by a He–Cd laser (325 nm) with equal recording intensities of 13 mW. The illumination of two coherent laser beams creates an interference pattern with bright and dark regions. The grating constant was controlled by the crossing angle between the two writing beams and set to be 2.0 µm. The total exposure energy was controlled by the exposure time. The recorded gratings were monitored by a He–Ne laser 632.9 nm line, which was incident normal to the surface of the sample after passing through a half-wave plate to characterise the anisotropic holographic PDLC. The holographically-formed PDLC is a phase-separated LC/polymer system that provides an electrically switchable holographic recording. Electric field control of the diffraction efficiency was observed by the addition of 70 kHz rectangular voltage between the two ITO-coated glass substrates. The detector for monitoring the diffraction efficiency was interfaced to a personal computer or an oscilloscope for data acquisition and analysis. The samples were finally cured by ultra-violet (UV) light irradiation using an Hg lamp. To consider the effects of post-curing on the electro-optical properties, experiments were carried out both before and after post-curing.

3. Results and discussion

The holographic PDLC films were formed from a homogeneous mixture of pre-polymer and low-molar-mass nematic LC. As the polymer cures, the chemical potential of the system changes, increasing the miscibility gap between the LC and its host. The low-molar-mass LC therefore phase separates as a distinct micro-droplet phase, and larger phase-separated LC domains are generated. If the photopolymer system is used as host material, the degree of polymerisation can be controlled optically, and it is possible to record holographic gratings using the interference light. As phase separation and subsequent reorientation of LC advance, an anisotropic holographic grating builds up and its diffraction properties can be probed with a polarised laser beam. It is necessary to determine whether the recorded grating is a thin (Raman–Nath) or volume (Bragg) grating. For this purpose, the following well-known parameter can be used (*41*):

$$Q = \frac{2\pi d\lambda}{\Lambda^2 n}, \tag{1}$$

where λ is the wavelength of the light, n is the index of refraction, d is the thickness of the grating and Λ is the grating constant. Judging from the value of the parameter $Q<1.0$ under our experimental conditions, we were dealing with a thin grating, i.e. Raman–Nath diffraction. A nematic LC is composed of rod-like molecules, which tend to align with their molecular long axis along a preferred direction known as the director. The director in the phase-separated LC domains is expected to align in accordance with the morphology and anchoring conditions, and resulting holographic gratings show strong optical anisotropy. Additionally, the diffraction efficiency of the holographic PDLC can be electrically controllable because the director of the LC can be controlled by the addition of an electric field across the holographic PDLC film. The light diffraction results from the periodic mismatch of the refractive indices of the LC domain and polymer matrix. If the ordinary refractive index of the LC is matched with the refractive index of the polymer binder, the light diffraction disappears and the films are transparent for normally incident light.

We first observed the morphology of the holographic PDLC films in both the presence and absence of post-curing with varying total exposure time; the resulting photographs are shown in Figure 1. Figures 1(a)–1(d) show the samples before post-curing, whereas Figures 1(e)–1(h) show the samples after post-curing. In polymerisation-induced phase separation, the initial mixture is composed of photo-curable monomers and LC, and the separation is

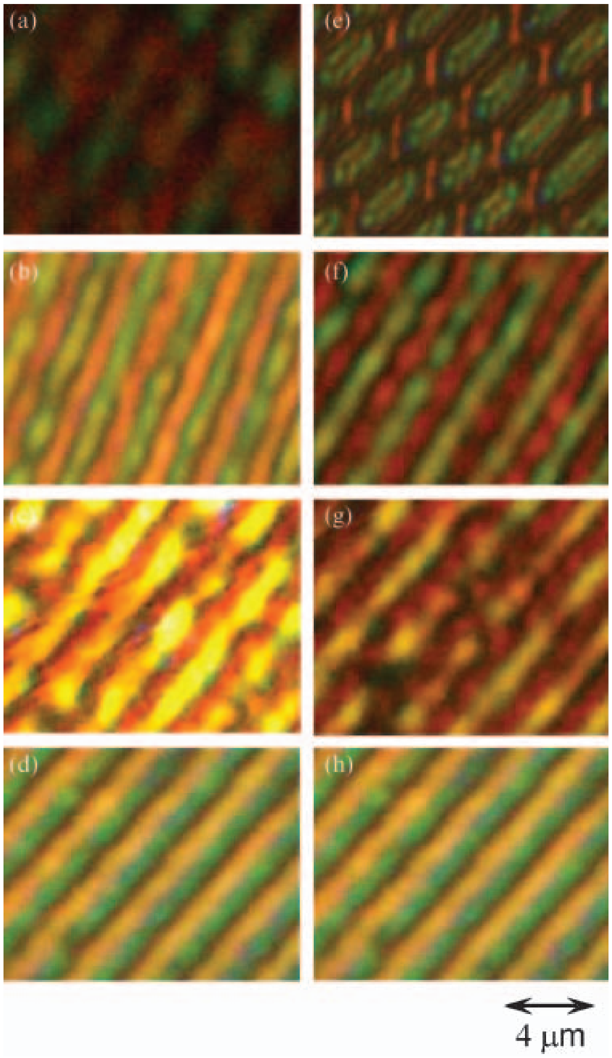

Figure 1. Morphology of holographic PDLC before (a–d) and after post-curing (e–h). The exposure times are (a, e) 0.5 s, (b, f) 1.0 s, (c, g) 1.5 s and (d, h) 2.0 s.

generated by photopolymerising the monomers. Using two interfering beams in the experiment, the phase separation between the matrix binder and LC is driven by the fringe pattern. Moreover, if the concentration of the nucleated droplets is optically controlled by the field gradient of the interference fringe, a droplet grating is generated, the properties and patterning of which depend on the degree of polymerisation and excitation conditions. Additionally, the photo-curable monomers are expected to be dominantly polymerised in the bright region and the phase-separated LC molecules should migrate from the bright region to the dark region. As shown in Figures 1(a) and 1(e), independent LC droplets were generated at the beginning of the grating formation. It should be noted that the boundary between the LC domain and matrix was more clearly observed after post-curing, as shown in Figure 1(e). We attribute this behaviour to photo-curable monomer that remains in the sample for low UV exposure energy before post-curing. A sufficiently high amount of the monomer can affect the microscope observation. For mid-range UV exposure energy, the LC domains grew and became connected, producing a "worm-like" LC domain structure in which the line width of the grating was periodically modulated, as shown in Figures 1(b), 1(c), 1(f) and 1(g). Finally, holographic gratings with straight lines and spaces were fabricated for high UV exposure energy as shown in Figures 1 d and 1 h.

The mechanism of light-induced phase grating formation is linked with phase separation and reorientation of the nematic LC. Moreover, reorientation of the nematic director is associated with the molecular migration during the grating formation and the influence of the interface between the LC drop and matrix polymer. The reading beam is diffracted with an efficiency defined as the intensity ratio of the diffracted beam and the incoming reading beam. Due to the strong optical anisotropy of the aligned LC director, the diffraction efficiency is strongly dependent on the polarisation azimuth angle of the probe laser beam. Figure 2 shows the diffraction efficiency versus polarisation azimuth of the probe beam for varying UV exposure times. The repeated patterns of valleys and peaks with a typical separation of 90°, which can be seen in Figure 2, are

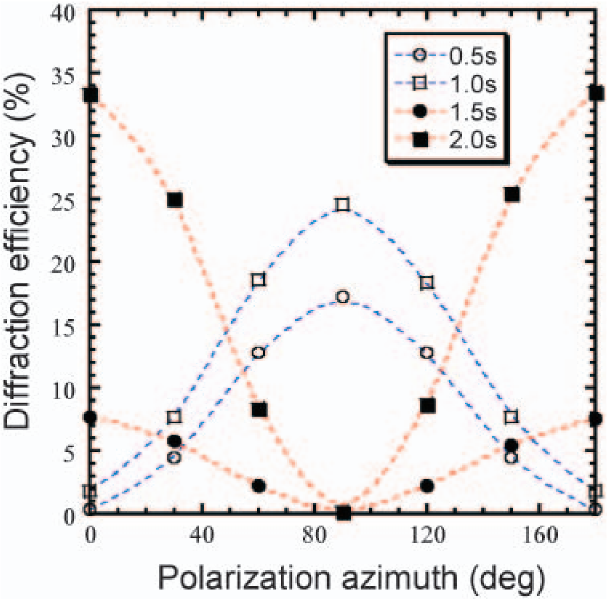

Figure 2. Diffraction efficiency versus polarisation azimuth of the probe light with varying exposure time. The polarisation azimuth is defined as the angle between the polarisation direction of the probe beam and grating vector of the holographic PDLC.

expected for a homogeneous aligned nematic LC in the resulting holographic PDLC. Care should be taken to ensure that if any reorientation of the nematic director takes place, it instantaneously follows the anisotropy of the holographic PDLC.

Considering the photographs shown in Figure 1 and the diffraction properties, the director of the nematic LC and grating formation mechanism are summarised in Figure 3. As shown in Figure 2, the director of the nematic LC rotates during holographic grating formation. At the beginning of the grating formation under 1.0 s of exposure time, the maximum diffraction efficiency is obtained when the polarisation azimuth of the probe beam is 90°. This means that, at the beginning of the grating formation for less than 1.0 s of exposure time, the director (**n**) of the nematic LC is perpendicular to the grating vector (**K**), as shown schematically in Figures 3(a) and 3(b). As shown in Figure 2, the director rotated over 1.5 s of exposure time and the minimum diffraction efficiency was obtained when the polarisation azimuth of the probe beam was 90°.

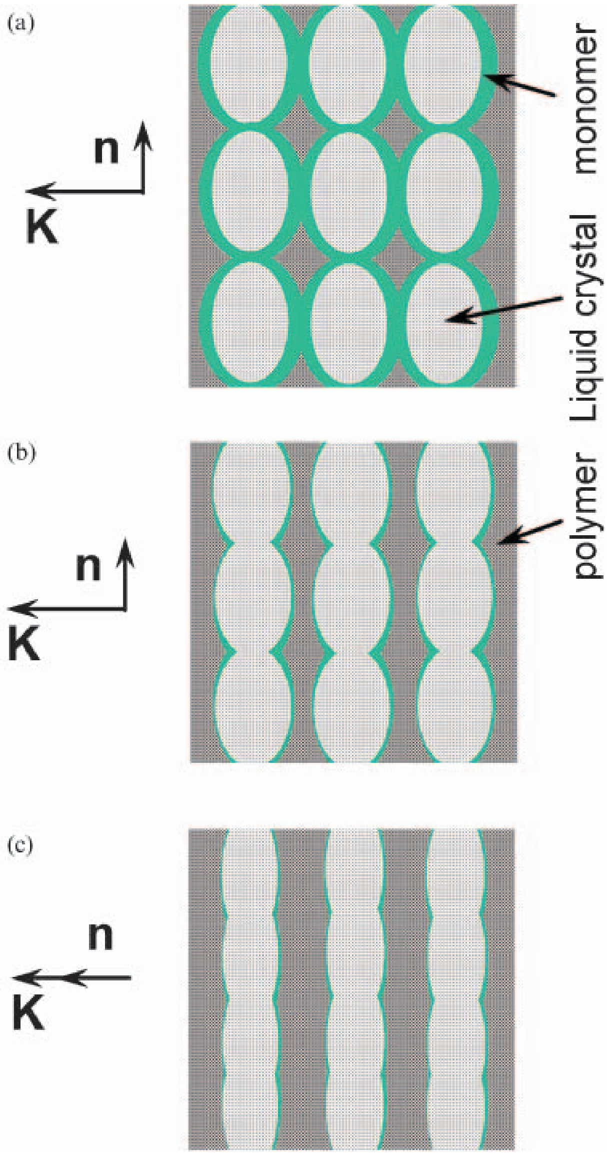

Figure 3. Schematic diagrams of the formation of holographic PDLC.

One possible explanation for these observations is suggested by focusing on the following two mechanisms for the reorientation of the nematic director in the holographic PDLC. One is reorientation of a grating-like wavy surface (Berreman's theory) (*42*), and the other is migration-induced reorientation during the phase separation and grating formation. The interfacial orientation of the director is also determined by the difference between the anisotropic surface free energy of the LC and the interfacial free energy of the matrix. If there is residual pre-polymer at the interface between the LC droplet and photocured polymer, as shown schematically in Figure 3, the interfacial free energy might be dependent on the UV exposure time and affect the director of the nematic LC. We investigated the effects of the residual pre-polymer on the reorientation of LC director by observing the change in post-curing and confirmed that the director did not change after the UV post-curing. This means the LC director is not affected by the change in anchoring interaction during UV post-curing. At the beginning of the grating formation, the director (**n**) of the nematic LC is perpendicular to the grating vector (**K**), as shown schematically in Figures 3(a) and 3(b). This means Berrman's effect is dominant for exposure times less than 1.0 s and the LC molecules align on a grating-like wavy surface. In the discussion of the effects of the UV exposure time on the optical anisotropy of the holographic PDLC, we find the director rotated for exposure times of more than 1.5 s, as shown in Figure 2 and the director (**n**) of the nematic LC was parallel to the grating vector (**K**), as shown schematically in Figure 3(c). According to the above-mentioned considerations, the LC molecules reorient to follow the migration, which causes an enhancement of the optical anisotropy of the holographic PDLC as grating formation completes.

A holographically-formed PDLC is a phase-separated LC/polymer system that provides an electrically switchable holographic recording. At a certain strength of the electric field applied to the holographic PDLC, diffraction spots disappeared because the LC director was homeotropically aligned over the threshold voltage and the ordinary index of LC was matched with the polymer matrix. The electro-optical properties of holographic PDLC should be strongly affected by the morphology and anchoring of the molecules. To measure the driving voltage, the electric field was increased and the diffraction efficiencies were monitored, as shown in Figure 4. Additionally, to measure the response time, the electric field was turned-on and turned-off, and the diffraction intensity was monitored by the digitising oscilloscope, as shown in Figure 5. The root mean square voltage (V_{rms}) is defined as a half value of the peak-to-peak voltage of the square wave output. Figure 4 shows the applied voltage dependence of diffraction efficiency of the holographic PDLC with exposure times of 1.0 s (sample_1s) and 2.0 s (sample_2s). The threshold voltage (V_{90}) and saturated voltage (V_{10}) are defined as the voltages that attain 90% and 10% of the diffraction efficiency at the OFF-state, respectively. As estimated from the data described in Figure 4, V_{90} and V_{10} were around $6.0V_{rms}$ and $15.0V_{rms}$, respectively, for sample_1s before post-curing, whereas they are around $9.5V_{rms}$ and $19.0V_{rms}$, respectively, for sample_2s before post-curing.

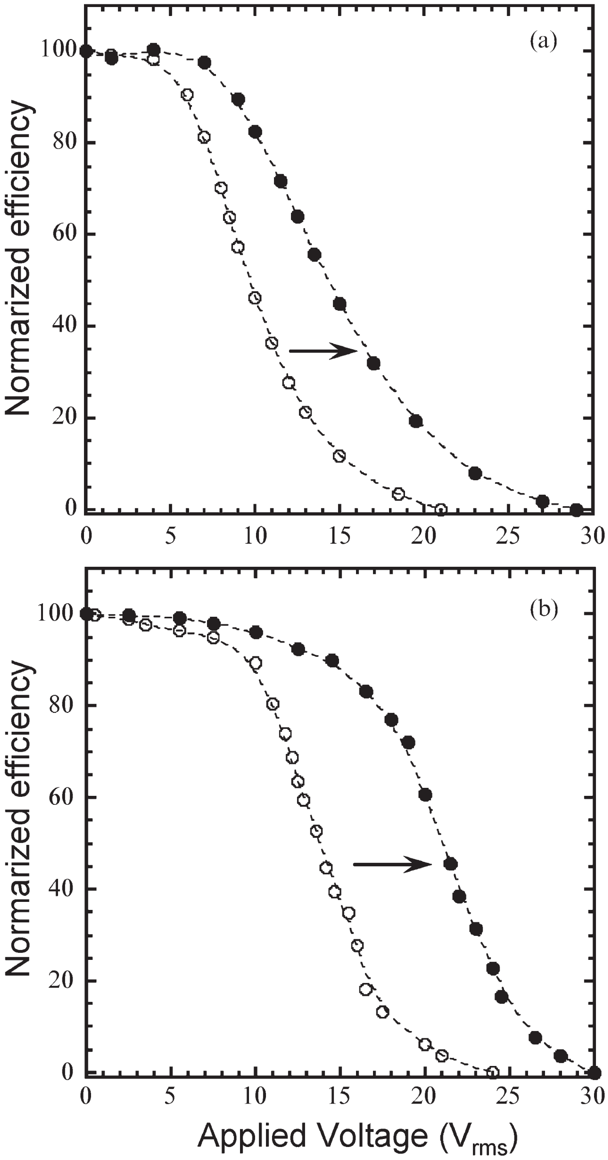

Figure 4. Diffraction efficiency versus applied voltage for holographic PDLC with (a) 1.0 s exposure time and (b) 2.0 s exposure time. Open circles denote the data before post-curing, whereas closed circles denote the data after post-curing.

Wu *et al.* (*43*) formulated the relation between the electro-optical properties and the shape and size of the LC droplet in the PDLC. They elucidated that the driving voltage was high in the case of a small LC droplet,

$$E_{th} = \frac{1}{a}\left|\frac{K\left(l^2-1\right)}{\Delta\varepsilon}\right|^{1/2}, \qquad (2)$$

where a and b are the lengths of the semi-major and semi-minor axes of the LC droplet, respectively, $l=a/b$, K is an elastic constant of the LC and $\Delta\varepsilon$ is the dielectric anisotropy. Fuh and Ko (*44*) measured the distribution of size and spacing of LC droplets of the PDLC films cured with various tungsten-halogen light intensities. They concluded that the droplet size was small in the composite films cured with a high curing light intensity and the PDLC films with large LC droplets had a low driving voltage. In our case, worm-like LC droplets in sample_1s, as shown in Figure 1(b), might give smaller l in comparison with sample_2s and the resulting driving voltage of the sample_1s was low according to equation (2). Moreover, the post-curing changed the driving voltage and switching time for both samples, as shown in Figures 4 and 5. These effects of the post-curing on the electro-optical properties cannot be entirely explained by the change in droplet shape or size. The results suggest the electro-optical properties were affected by the change in the boundary conditions at the interface between the LC and the matrix due to the polymerisation of the residual pre-polymer during post-curing. Liu *et al.* (*27*) investigated the effects of the surfactant on the electro-optical properties of holographic PDLC. When phase separation occurred in the homogeneous mixture, the surfactant formed an intermediate layer between the polymer and LC. The anchoring energy at the interface of the LC and surfactant became weaker than at the interface of the LC and polymer without surfactant. They concluded the decrease in anchoring strength effectively lowered the driving voltage of the holographic PDLC. Similar effects of the intermediate layer on the electro-optic properties of PDLC have been observed in a previous study

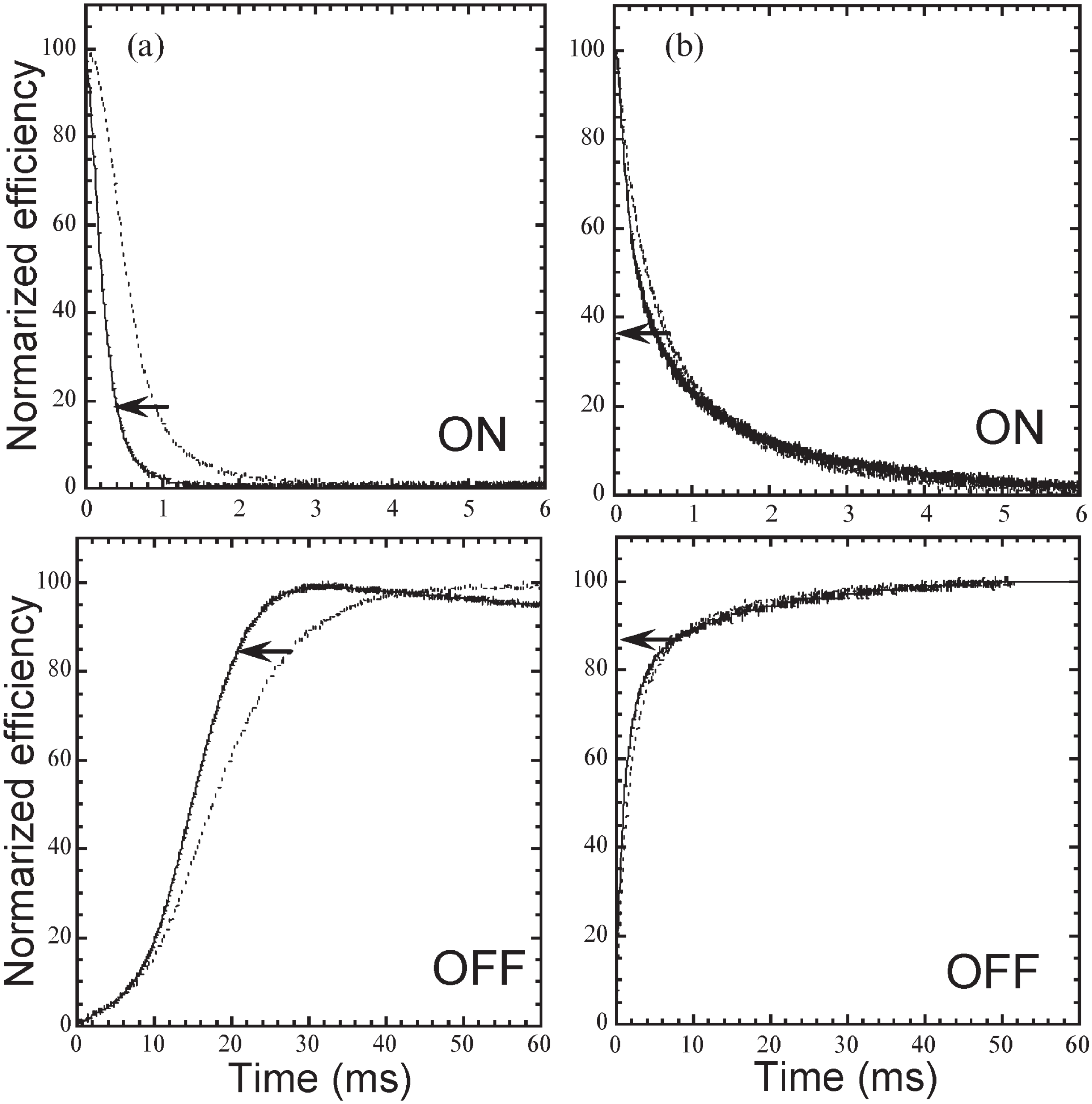

Figure 5. Switching behaviour of the holographic PDLC with (a) 1.0 s exposure time and (b) 2.0 s exposure time. Dotted curves represent the data before post-curing, whereas solid curves represent the data after post-curing.

(*45*). PDLC films were prepared by casting poly(vinyl alcohol)/LC emulsion with pre-polymer and post-curing. An intermediate layer consisting of the photocured polymer reduced the anchoring strength and lowered the driving voltage. In the present study, the residual monomer before post-curing works as the intermediate layer and lows the driving voltage, as shown in Figure 4. A more satisfying explanation of these phenomena in future work would require additional information regarding the switching mechanism, and in particular about the anchoring of the molecules.

4. Conclusions

We have studied in detail the diffraction properties and the morphology during formation of a holographic PDLC and found that the direction of the LC director rotated during the holographic recording. These phenomena are explained by focusing on two mechanisms of the reorientation of the nematic director in the holographic PDLC. One is the reorientation of a grating-like wavy surface, and the other is the migration-induced reorientation during phase separation and grating formation. The holographically-formed PDLC was a phase-separated LC/polymer system that provided an electrically switchable holographic recording. Electro-optical properties of holographic PDLC were dependent on the exposure time and also strongly affected by post-curing. These effects of the post-curing on the electro-optical properties cannot be entirely explained by the change in droplet shape or size. The results suggest that electro-optical properties were affected by the change in boundary conditions at the interface between the LC and matrix due to the polymerisation of the residual pre-polymer during post-curing.

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