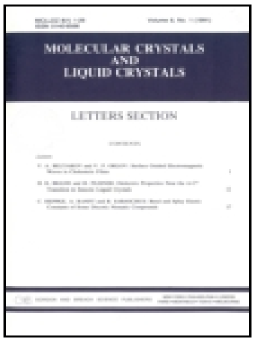

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Photo-Thermal Modulation of Cholesteric Liquid Crystals with a Dual Circularly Polarized Light Reflection Band

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In this study, we developed a cholesteric liquid crystal (Ch-LCs) composite with a dual (both right- and left-) circularly polarized light reflection band by utilizing a polymer template method, where a glassy Ch-LCs oligomer (GCLCO) with left-handed helical structure containing azobenzene dopant (AD) was infiltrated into the polymer template with right-handed helical structure. Photo-optical, thermal as well as glass-forming properties of the Ch-LCs film with a dual circularly polarized light reflection band were investigated, trans–cis (E–Z) photoisomerization of the AD in the GCLCO induced pitch length shortenings in response to the formation of the Z isomers by 365 nm ultraviolet (UV) irradiation, resulting that the reflection band of left-handed circularly polarized component of light in the Ch-LCs composite after UV irradiation exhibited a blue shift. What is more, the changed reflected color could be fixed in Ch glassy solid by rapid cooling from their Ch temperatures to below glass transition temperature *(T_g), in which the photo and thermal isomerization of the AD could not influence the Ch pitch any longer and the color was stably stored. This technique has great applications such as tunable lasers, optical sensors, and security technology.*

Keywords Azobenzene dopant; cholesteric liquid crystal; circularly polarized light reflecting; glassy chiral oligomer; photo-thermal switching

Introduction

Polymer/liquid crystals (LCs) composite systems have provided a new field of LCs science and technology [1–4]. In the polymer stabilized cholesteric LCs (PSCLCs), a desired macroscopic orientation of Ch-LCs directors can be stabilized or frozen by the cross-linked network, in which PSCLCs are formed by photo-polymerization of a relatively small amount of a photo-reactive monomers dissolved in the nonreactive Ch-LCs [5–11]. The characteristic helical structure of the Ch-LCs impart unique optical properties such as reflecting circularly polarized component of light, the reflected wavelength of Ch-LCs is generated by the photonic band gaps (PBGs) from the periodical helical orientation. The pitch p is defined as the distance in which Ch-LCs molecules rotate 360° along its helical axis, and for light propagating along the helical axis, $\lambda_0 = p \times n$, where λ_0 is the

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wavelength of the maximum reflection or the middle of selective reflection band and *n* is the average refractive index $n = (ne + no)/2$. The extraordinary and ordinary indices of the refraction are denoted by *ne* and *no* respectively. The PBGs width of a conventional Ch-LCs is equal to $p \times \Delta n$ and is proportional to the anisotropy of refractive indices $\Delta n = ne - no$. Within the bandwidth, light with the same handedness as that of the Ch-LCs will be circularly reflected, while the component with opposite handedness will be circularly transmitted [12,13]. Also the helical structure of Ch-LC is extremely sensitive to external influences such as temperature, pressure, and electric field and to the chemical modification of the molecules [14–26].

Among these, the change in the helical pitch caused by photochemical reactions in the Ch-LCs is especially of interest [27–45]. Azobenzene is a typical photochromic compound, which changes its molecular shape upon photoirradiation. It possesses a rod-like shape in a *trans* (*E*)-form while a *cis* (*Z*)-form exhibits a bent shape. Photoinduced *E-Z* isomerization in the azobenzene compound doped in Ch-LCs was found to cause either an elongation or shortening of the Ch-LCs pitch. Reversible switching of the PBGs was demonstrated in Ch-LCs doped with various azobenzene derivatives by UV exposure [28–31]. Sackmann reported the first example of a photomodulation of Ch-LCs and found that the helical pitch of the LCs could be altered by *E-Z* photoisomerization of azobenzene and stilbene derivatives, in which photoirradiation of the Ch-LCs results in the change in the reflection color due to the ratio of isomers of the azobenzene moieties [28]. Tamaoki *et al.* have demonstrated that the changes in the helical pitch and the corresponding reflection colors of the glass-forming Ch-LCs doped with azobenzene derivatives were driven by UV/Visible in the visible spectrum [29–31].

In the present study, we firstly report our investigations on the photo-optical, thermal as well as glass-forming characteristics of Ch-LCs film with a dual circularly polarized light reflection band, in which Ch-LCs solid film was derived from a polymer template with an infiltrated glassy Ch-LCs oligomer (GCLCO) containing azobenzene dopant (AD). The polymer template from the PSCLCs was formed by photopolymerizing LC monomers that were dispersed within a Ch-LC mixture, which initially defined the handedness and periodicity of the helical structure and then was subsequently removed as mentioned in our previous studies [21–26]. Herein, based on the memory effect of the polymer template and the photo/thermo responsive characteristics of the GCLCO with AD were introduced into the Ch-LCs system. Photo/thermal switching of the PBGs in the Ch-LCs with a dual circularly polarized light reflection band was investigated in detail, and the mechanism of the photo-optical, thermal as well as glass-forming properties on the UV/vis light and heat fields was demonstrated.

Experimental

Materials

In this study, nonreactive nematic LCs, SLC-1717 (20°C, 589 nm, $\Delta n = 0.201$; Slichem Liquid Crystal Material Co., Ltd.); photopolymerizable monomer, C6M; Glassy Ch-LCs oligomer (GCLCO), the molar ratio of chiral mesogen and achiral mesogen in the oligomer is 1.5:1, and the cholesteric phase appears between 189°C (isotropic temperature, T_i) and 52°C (glass transition temperature, T_g); Azobenzene dopant (AD, 4-hexyloxy-4′-ethoxyazobenzenes); Chiral dopant, R811 (Merck Co. Ltd); Photoinitiator, 2, 2-dimethoxy-2-phenyl-acetophenone (Irgacure 651, TCI Co. Ltd.); and Ultraviolet absorbing dye, (ULAD, 2-{2, 6-bis-[2-(4-dimethylamino-phenyl)-vinyl]-pyran-4-ylidene-malononitrile) were used. Nematic diacrylate monomer, C6M was synthesized

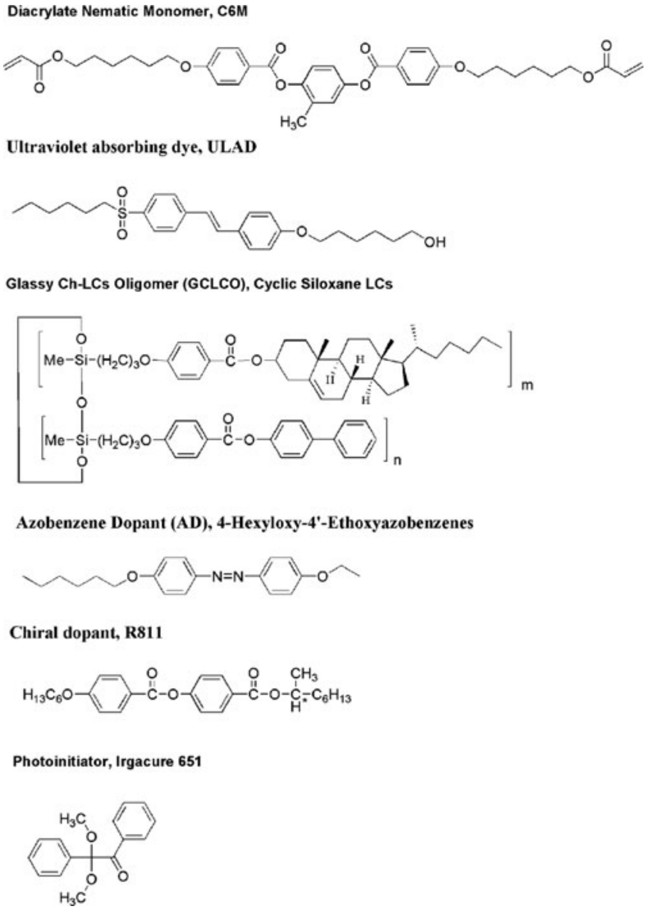

Figure 1. The chemical structures of the materials used.

according to the method suggested by D. J. Broer *et al.* [46]. GCLCO were prepared as described in previous papers [47, 48]. AD was synthesized according to the previous literatures [49, 50]. ULAD was lab-synthesized according to the earlier paper [51]. Figure 1 shows the chemical structures of C6M, GCLCO, R811, AD and Irgacure 651.

Experimental Cells

In order to induce a planar orientation of LC molecules, the inner surfaces of indium tin oxide coated (ITO) glass cells were coated with a 3.0 wt% polyvinyl alcohol (PVA) aqueous solution. The deposited film was dried at 80°C for 30 min and subsequently rubbed with a textile cloth under a pressure of 0.02 N/cm^2 along one direction. PET (polyethylene terephthalate) films of 12 ± 2 μm thickness were used as the cell spacers. Especially, the samples of the GCLCO containing 0–4 wt% of *E*-AD were filled into the LC cells at 120 C to obtain thin films in the *Ch* phase. All the mixtures showed only Ch phase, which was confirmed by DSC measurements and POM observations. After the films give the *Ch* phase with reflected color, they were annealed by ice cubes (ca. 0°C) to obtain glassy solid films

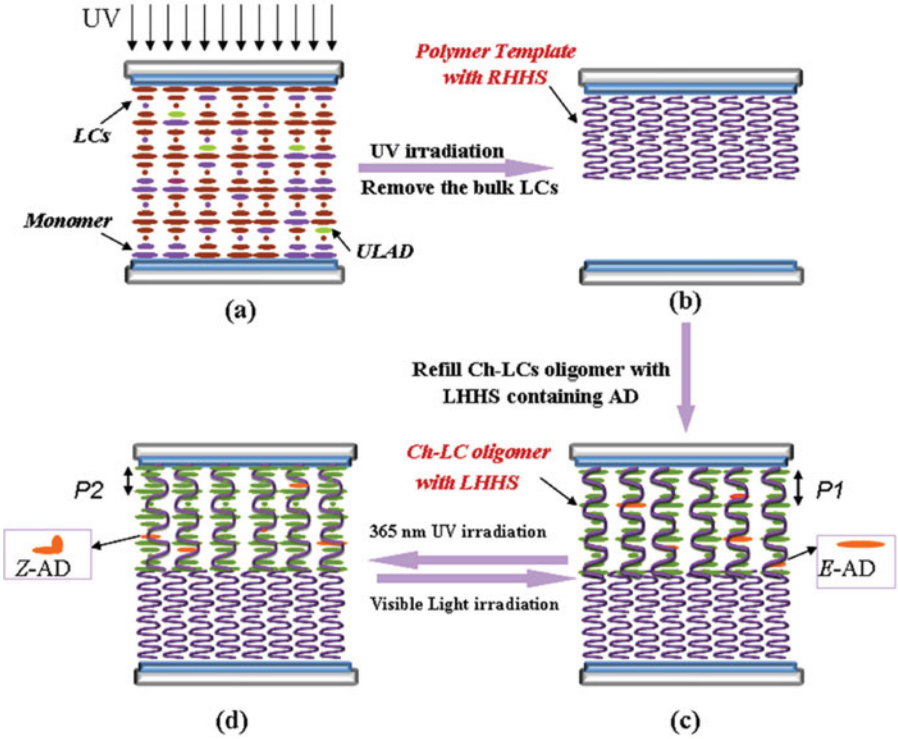

Figure 2. Illustration of the fabrication procedure of the Ch-LCs film.

retaining the reflected color of the *Ch* phase. The reflection bands were measured at room temperature for these glassy solid films.

Preparation of Ch-LCs Film with a Dual Circularly Polarized Light Reflection Band

The Ch-LCs film with a dual circularly polarized light reflection band was prepared by carrying out the following procedure. At first, the cell containing mixture 1 with right-handed helical structure (RHHS) were irradiated with UV light (365 nm, 1.0 mW/cm^2) for 30 min for polymerization purposes as shown in Fig. 2(a), in which the weight ratio of mixture 1 (wt%) is SLC-1717/R811/C6M/ULAD/Irg651 = 60.0/23.5/15.0/1.2/0.3. Following that, the cells were immersed in cyclohexane for about 48 h and later in tetrahydrofuran for 20 min to remove the nonreactive LCs. After that the cells were kept in vacuum chamber at 60°C for about 3 h. Thus the polymer template with a RHHS was obtained as shown in Fig. 2(b). Finally, the cell containing the polymer template was refilled with GCLCO/4 wt%AD mixture with a left-handed helical structure (LHHS) at 120°C as shown in Fig. 2(c), which is called cell A. At this temperature, the GCLCO/4wt%AD mixture was in *Ch* phase and rather fluid, so that they can move and diffuse to the polymer template. When the cell A was irradiated by 365 nm UV light (20 mW/cm^2) with different irradiation time, the pitch length and the following reflection color in the Ch-LCs cells changes due to the *E*-*Z* photoisomerization of AD as shown in Fig. 2(d). Then they were quenched to room temperature in a glassy state.

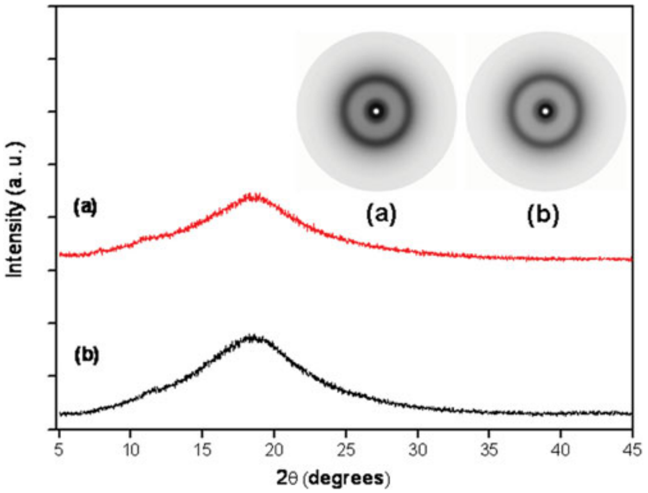

Figure 3. 1D-WAXD patterns and the corresponding typical 2D-WAXD patterns of the GCLCO, (a) at 120°C on the first heating and (b) at 15°C on the first cooling.

Measurements

The thermal behavior of the GCLCO and GCLCO/AD mixture was studied using a Pyris 1 DSC (PerkinElmer, Inc, USA). The temperature protocols for DSC measurements were as follows: firstly, heat the sample from room temperature to 200°C at a rate of 10°C/min; then cool down to 0°C at 10°C/min rate. The corresponding structure evolution for the GCLCO during heating and cooling was studied by *in-situ* wide-angle X-ray diffraction (WAXD), the wavelength of X-ray beam was 1.371 Å. 2D-WAXD patterns were collected continuously using a MarCCD X-ray detector (Rayonix, LLC, USA). The samples were observed using a polarizing light microscope (POM) (Leica, DM2500P) with a heating stage (LTS 420). The optical images were recorded using Linksys 2.43 software. The transmission spectra were obtained by UV/vis spectrophotometer (Hitachi, U-3010) at normal incidence. The transmittance of a blank cell was normalized to 100%. In general, λ_M and $\Delta\lambda$ are defined as the minimum wavelength of the transmitted light and the bandwidth at half-height of the peak, respectively. The reflection spectra were obtained by fiber spectrometer (Avantes, AvaSpec-2048). The morphology of the polymer network was observed by scanning electron microscopy (SEM) (Hitachi S-4700). The samples for SEM studies were prepared according to the method described in the previous study [52].

Results and Discussion

As mentioned above, GCLCO is cyclic siloxane side chain LC oligomer, it exhibits a stable Ch phase and is equally rather fluid when GCLCO is heated to about 120°C at 40°C/min rate which is above its glassy temperature (T_g). Followed that GCLCO was cooled to 15°C at 100°C/min rate, 1D-WAXD patterns and the corresponding typical 2D-WAXD patterns (corrected for air scattering and beam fluctuations) of the GCLCO was shown in Fig. 3, typical 2D-diffraction patterns of a Ch phase was observed at 120°C on the first heating and at 15°C on the first cooling, and 1D-WAXD patterns also suggested that the sample does not contain any crystal. This means that the molecular arrangement (Ch state) can be stored by this quenching process due to the "freezing" of the chain segments of

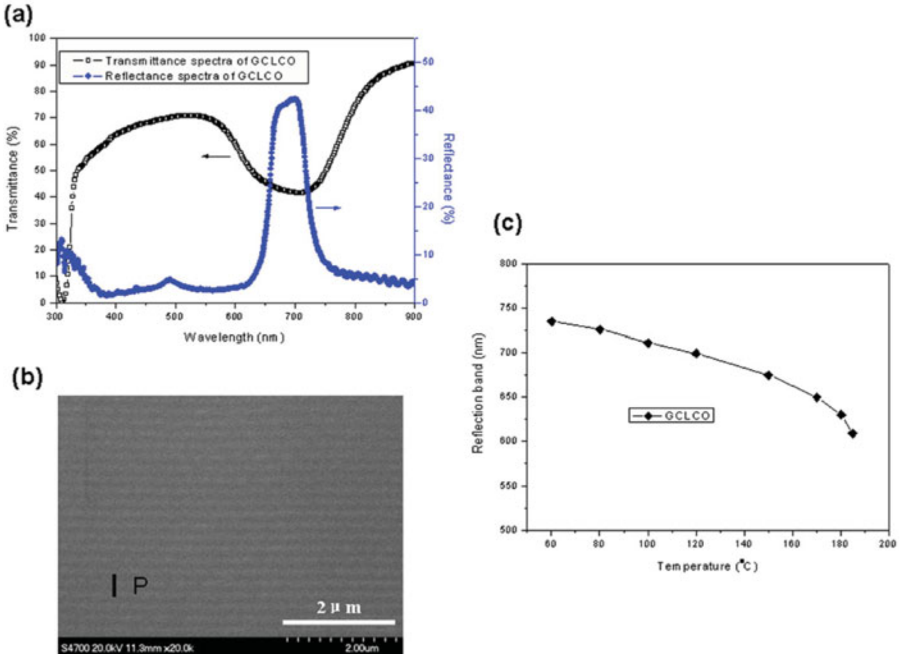

Figure 4. (a) Transmission and reflection spectra of the cell with GCLCO, the sample was prepared by heating the sample to 120°C followed by rapid cooling to glass state; (b) the corresponding SEM micrographs of the cross-section of the GCLCO; and (c) the temperature-dependence of the reflection spectra of the GCLCO.

glassy GCLCO below T_g. That is to say, such a cooling process prevents crystallization and preserves Ch structure. Figure 4(a) shows the transmittance and reflectance spectra of the GCLCO, the reflection band now occurs about 700 nm when the sample was heated to 120°C followed by rapid cooling to glass state. While the oriented planar texture of the GCLCO is restored by quenching, the cross-section of the material was investigated by SEM and pictures are taken as shown in Fig. 4(b), it can be seen that a uniform alternative dark and bright stripes with periodicity of the half-pitch was presented. It also suggested that Ch structure of the GCLCO was permanently stored by quenching the material to a glass state at room temperature. Figure. 4(c) shows the dependence of the spectra of the GCLCO on temperature, we see that the position of the reflection band has an obvious blue-shift with temperature. This indicates that the pitch length of the GCLCO decreases with temperature.

As is well known that, under normal room conditions, azobenzene materials are mostly in their elongated *E*-state [27]. A typical azobenzene molecule used in this study is azobenzene dopant (AD, 4-hexyloxy-4′-ethoxyazobenzenes) as shown in Fig. 1, which has an absorption maximum in the UV at 352 nm. By exposure to 365 nm UV light, the molecule undergoes a photoisomerization to the bent *Z*-state. For AD, the *Z*-state has absorption peaks in the visible at about 450 nm, with a significant reduction of the absorption at 352 nm (Fig. 5(a)). And relaxation back to the *E*-state is driven thermally and by exposure to light at the peak wavelengths of the *Z*-state. Herein, AD can be easily dissolved in the host GCLCO, which do not induce a particular change in the thermal properties of the

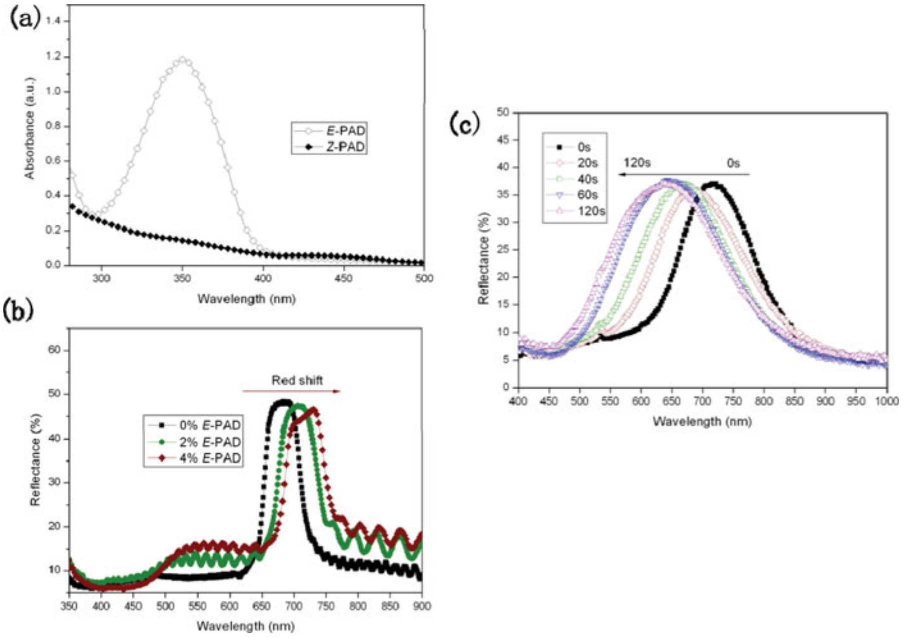

Figure 5. (a) Absorption spectra of AD for the dark (*E*-dominated) and UV-exposed (*Z*-dominated) states; (b) the change in reflection band as a function of the concentration of AD; (c) the change in reflection band as a function of the irradiation time of the GCLCO with 4 wt% AD, the sample was prepared by rapid cooling to the glassy solid state after UV irradiation at 365 nm with different irradiation time at 120°C.

GCLCO (except T_g of the GCLCO was lowered a little as the AD was added into the system) since AD is LC molecule as described in previous papers and its concentration is low in the system [50]. For our present result, the addition of *E*-AD to the host GCLCO made the pitch remarkably longer with an increase AD' concentration as shown in Fig. 5(b), it is likely that an achiral azobenzene has an opposite twist power compared with that of the pure GCLCO. This could be due to that a decrease in concentration in chiral molecules in a given volume, induced by the incorporation of the nonchiral "solvent" molecules (AD). The change in the reflection band by the *E*–*Z* photoisomerization of the GCLCO containing 4% AD was also investigated. While maintaining Ch phase at 120°C, the cell with the GCLCO containing 4% *E*-AD was irradiated with a 365 nm UV light (20 mW/cm^2) from a high pressure mercury lamp passed through appropriate filters. After the UV irradiation, the cell was rapidly cooled to the glassy solid state that maintained the Ch-LC reflected color. Depending on the UV irradiation time, the reflected band was changed as shown in Fig. 5(c), it is obvious that the photoisomerization of AD from the *E* to *Z* isomer by UV irradiation induced λ_{max} to become lower values and the reflected color had a blue-shift. This could be due to that the *Z*-AD disordered the molecular arrangement of the Ch-LCs phase because of their bent structures and the pitch changed in response to such a state of disorder. In this situation, the disordered sense led to the reduction of the pitch of the Ch-LCs molecules, which might be ascribed to that the thermal behavior of the GCLCO shows $(dP/dT) < 0$ as mentioned above in this study [32].

Similarly with that of our previous study [21–26], a polymer template with a RHHS was achieved by photo-polymerizing C6M monomers in PSCLCs which initially defined

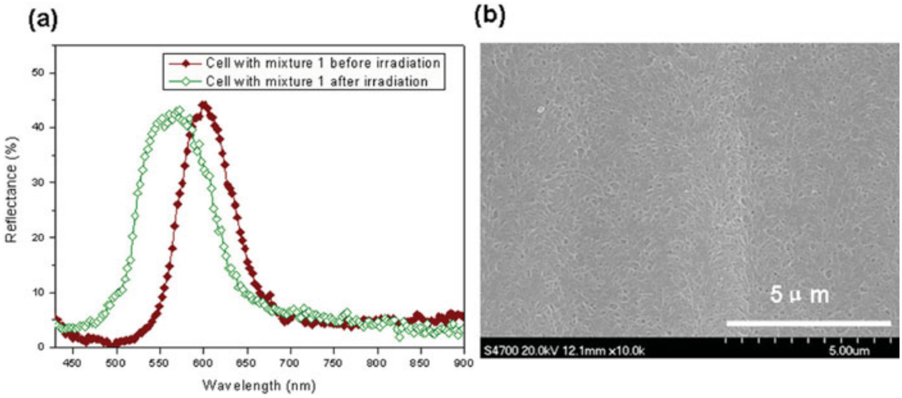

Figure 6. The reflection spectra of cell with mixture 1 before and after UV irradiation; (b) SEM micrograph of the surface morphology of the cell with mixture 1 after UV irradiation.

the periodicity or pitch (*P*) of the structure, then subsequently removed the nonreactive LCs from the system as shown in Fig. 2(b). Then a LHHS Ch-LCs material containing a GCLCO with *E*-AD was refilled into the above polymer template as shown in Fig. 2(c). As mentioned above, the glass-forming properties of the GCLCO and photoisomerization characteristics of the AD determined the optical properties of the Ch-LCs film in this study. Figure 6(a) shows the reflection spectra of cell with mixture 1 before and after irradiation, the reflection wavelength of the cell with mixture 1 before and after irradiation is about 605 and 560 nm, respectively, as shown in Fig. 6(a), it is obvious that the reflection wavelength of the cell had a blue-shift after irradiation as a result of polymer network formation in the bulk LCs. The helical pitch of the composites is believed to decrease slightly in relation to the volume shrinkage resulting from the reaction of polymerization and cross-linking [19, 20]. Figure 6(b) shows SEM photographs of the surface morphology of the cell with mixture 1 after irradiation, the helical structure of the polymer network formed in PSCLCs can clearly be imaged.

As mentioned in the experimental section, the LC cell with the polymer template was refilled with GCLCO/4wt%AD mixture with a LHHS at 120°C and was further cooled to the room temperature in a glassy state. Figure 7 shows the SEM image of the cross-section of the cell A from the above technique, it is obvious that the cell possesses two distinct regions (Region I and Region II) and two different pitches of P1 and P2 can be observed in the cell A, it means that a spatial segregation structure of right handedness (RH) and left handedness (LH) domains occurring through the thickness of the LC cell was obtained. Herein, the top layer (Region I) is the RH helical polymer template, the thickness of which was measured to be about 6 μm with a half of the cell thickness of around 12 μm. While the bottom layer (Region II) is GCLCO with LH helical structure containing AD, this suggested that ULAD played an important role in forming the above structure similar to that of our previous study [26].

Figure 8(a) shows the time-dependence of the reflection spectra of the cell A during irradiation, we can observe two reflection bands in the spectra of cell A as shown in Figure 8(a), in which the reflection wavelength of the L-CPL and R-CPL of the cell A is about 738 and 548 nm, respectively. This suggests that the difference in the pitch length between LHHS and RHHS of the composite. That is, either right handed circularly polarized light (R-CPL) or left handed circularly polarized light (L-CPL) were reflected within the

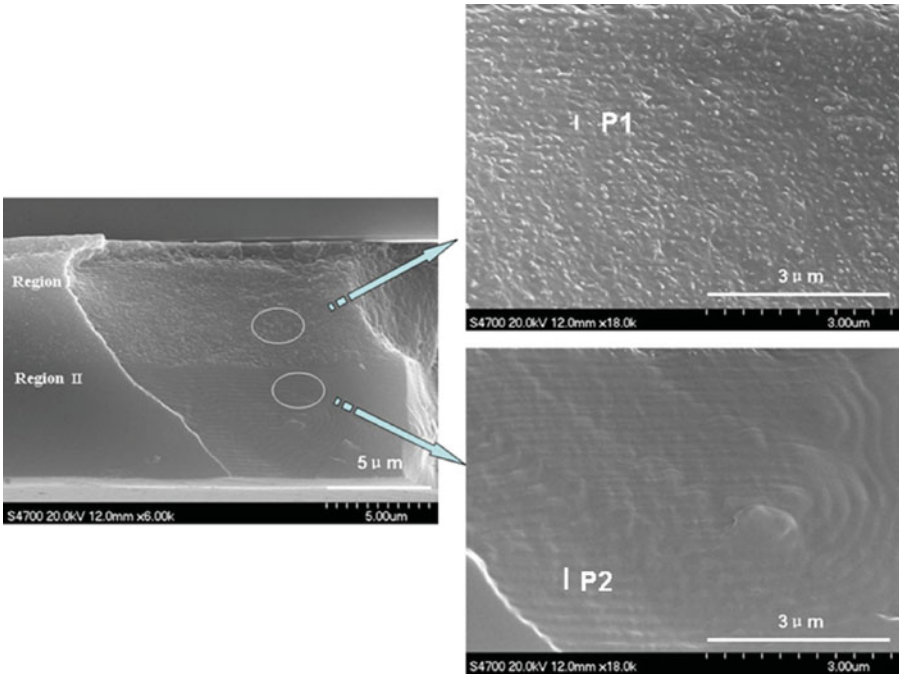

Figure 7. SEM image of the cross-section of the cell A.

different reflection band by the cell A. The film characteristic that reflects both R-CPL and L-CPL is attributed to the polymer template has a characteristic of memorizing the helical structure of the initial PSCLCs as mentioned above [5–8]. With an increase of the irradiation time, it can be observed that the reflection band began to change gradually with the irradiation time ranging from 0 as 60 s. It is easy from these spectra to observe directly the reflection band of L-CPL had a blue-shift, the reflection band is around 670 nm after 60 s irradiation. Figure 8(b) shows the reflection wavelength of the cell A as a function of

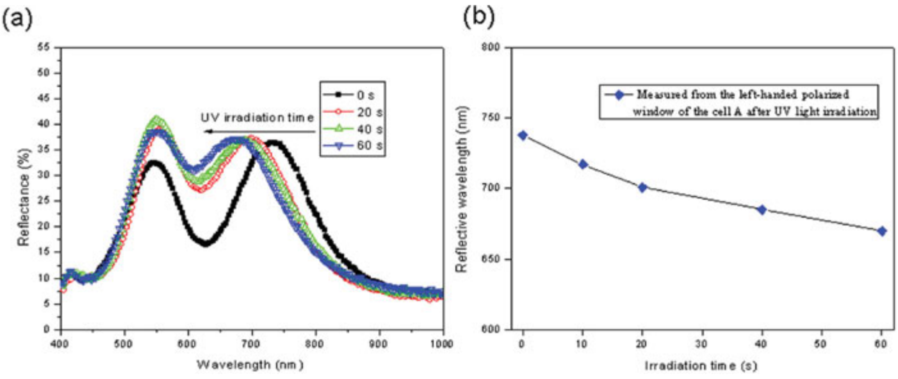

Figure 8. (a) The irradiation time dependence of the reflection spectra of the cell A during irradiation of 365 UV light at 120°C; (b) the reflection wavelength as a function of irradiation time measured from the left-handed polarized window of the cell A during irradiation of 365 UV light at 120°C.

irradiation time during 365 UV light irradiation at 120°C. It is obvious that the reflection band of the cell A measured by fiber spectrometer have a blue-shift from 738 to 670 nm with an increase of the UV irradiation time, which indicates that pitch length of the GCLCO containing 4 wt% of AD decreases with the irradiation time increasing. Herein, the color tuning for the cell A is mainly due to the variation of the HTP value/pitch length of the GCLCO induced by photo-isomerization from *E* state to *Z* state of the added AD as suggested above.

We note that if the cell A was obtained after 60 s irradiation with 365 nm light at 120°C and followed by rapid cooling to the glass state, no change in the reflection bands happened in the cell A, which indicates that the fixed Ch structure is not perturbed by the thermal isomerization of the AD. Furthermore, the reflection color disappeared when the cell A was reheated to T_i (above 190°C), and it also reverted to the initial state (two reflection bands) until the temperature drops to Ch temperature, because T_i completely resets the molecular arrangement of the Ch-LCs and the conformation of the AD to the initial state (*E*-form). Therefore this new Ch-LC material with a dual circularly polarized light reflection band is an effective material for storage of the information written by a photon state.

Conclusions

In summary, photo-thermal modulation of reflection characteristics and glass-forming properties in Ch-LCs with a dual circularly polarized light reflection band was investigated, in which the GCLCO/AD with LHHS was infiltrated into the polymer template with RHHS. It suggests that the L-CPL reflection band of the Ch-LCs composite derived from GCLCO with AD after 365 UV light irradiation exhibited a blue shift from 738 to 670 nm over the irradiation time ranging of 0–60 s. While, the R-CPL reflection band of Ch-LCs composite changed little. The reason for this is that photoisomerization from *E*- to *Z*-AD in the Ch phase induced consecutive pitch shortenings in response to the formation of the *Z* isomers upon UV irradiation. More importantly, the Ch-LCs composite in this study had a glassy state characteristic that maintain the Ch structure after rapid cooling from the Ch phase, and the changed pitch length followed by the changed reflection color was fixed in the glassy solid. This novel Ch-LCs composite can be considered as promising materials for photonic and optical applications.

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