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Light-scattering-mode optical switching and image storage in polymer/liquid crystal composite films by means of photochemical phase transition

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Abstract

Photochemical phase transition behavior triggered by photoisomerization of a guest azobenzene doped in nematic (N) liquid-crystalline (LC) domains was investigated in isotropic (I) acrylate polymers with alkyl side chains on their surfaces. These polymer/LC composite films were prepared by in situ thermal polymerization-induced phase separation method (thermal PIPS) from their starting mixtures containing a di- and a mono-functional acrylate monomer. Strong light-scattering states of the intrinsic composite films were transformed into complete transparent states by photochemical N–I phase transition of the LC domains within the polymer matrices that was induced by the *trans–cis* photoisomerization. Recovery process from the transparent state to the initial light-scattering state was strongly influenced by the alkyl side chains. While the composite film with short alkyl side chain acted as an all-optical switching material because the optical switching could be achieved reversibly and repeatedly between the two different optical states, the composite films with long alkyl side chain showed an ability of optical image storage with high contrast based on light-scattering mode. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Polymer/LC composite films; Photochemical phase transition; Memory effect

1. Introduction

Liquid crystals (LCs) are one of the most convenient materials to control light because they possess a large optical anisotropy and dielectric anisotropy, which are controllable by change in the alignment of the molecules with external stimulus such as electric field, heat, and light [1–3]. The control of light by light as the stimulus by the use of LC has become of interest as a potential technology for various photonic applications such as optical switching, optical image storage, and optical display [4–6]. To control LCs by light, many studies have shown that the use of azobenzene derivatives as a photoresponsive dopant is very effective [7–9]. It is well known that the azobenzene undergoes isomerization from *trans* to *cis* form under ultraviolet light irradiation, while the *cis* form can return to the *trans* form either photochemically or thermally. Such geometrical change could produce a concomitant change in physical and chemical properties not only in the azobenzene itself but also in the surrounding matrices. When the azobenzene is embedded in nematic (N) LC, the two isomers produce different environments, which are characterized by the two different molecular shapes. The *trans* form is favorable for the stabilization of the LC phase because it is a rod-shaped rigid molecule, while the *cis* isomer is bent and tends to destabilize the phase structure. Therefore, the ordered N phase is isothermally transformable into disordered I phase by the *trans–cis* photoisomerization of the guest azobenzene (photochemical N–I phase transition). The photochemical phase transition of LCs has been examined conveniently by the transmission- and reflection-mode analyses [4,10–14]. In the transmittance-mode analysis, the N–I photochemical phase transition, which results in disappearance of birefringence of the LC or loss of director field, can be evaluated easily through the change in transmittance of a probe light. This method needs essentially alignment of the LC and a pair of crossed polarizers with the LC sample between them. When the LC sample is in the N phase, the transmittance of the probe light is observed, whereas no transmitted light is

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detected for the sample in the photoinduced I state. Although the two modes are very useful for photochemical control of light by light, the loss of optical efficiency is unavoidable due to the use of the polarizes. If the control of light by light can be achieved in the absence of the polarizes, the optical efficiency of the photonic materials will be much improved.

From such viewpoint, the light-scattering-mode control of light was proposed and accomplished by employing a solid polymer matrix combined with the LC system in lieu of the function of the polarizers similar to a polymer-dispersed liquid crystals (PDLCs) [9]. PDLCs are an inhomogeneous composite film consisting of micrometer-sized low-molecular-weight LC domains or droplets dispersed in a solid polymer matrix [15–25]. In general, the light-scattering state is controlled through electrical control of ordering of the LC domains as in other LC devices. PDLCs with a memory function, which are potential for image storage and laser-addressed displays, were also reported by several researchers [26–32]. Especially, Kajiyama's group reported that polymer LC/low-molecular-weight LC composite systems containing a photoresponsive molecule provide light addressing and optical storage effect between light-scattering state and transparent state by applying electric and electromagnetic fields as stimulus [31,32]. In these composite films, optical information can be recorded due to reversible change in threshold frequency induced by isomerization of azobenzene. In the previous study, we reported all-optical control of the light scattering through the polymer/LC composite films based on the photochemical phase transition as a working principle [9]. In these composite films, photoirradiation at wavelengths to bring about the photoisomerization of the guest azobenzene resulted in reversible change in opaque light-scattering state and complete transparent state by the isothermal photochemical phase transition in the LC area at irradiated site.

In this study, a series of the polymer/LC composite films were prepared with acrylate-based solid matrices having various lengths of alkyl side chains and an NLC mixture containing an azobenzene derivative as a photoresponsive dopant by in situ thermal polymerization-induced phase separation (thermal PIPS) method. The photoresponsive behavior of these composite films was explored all-optically with special reference to the effect of the alkyl side chains extruding from the surfaces of the polymer matrices on the photoresponsive behavior.

2. Experimental

2.1. Materials

1,6-Hexanediol diacrylate (DA) and four monofunctional acrylates (*n*A, $n = 4, 6, 8, 12$) with different length of the alkyl group (denoted by *n*) were used as precursor materials to form the solid polymer matrices: butyl acrylate (4A), hexyl acrylate (6A), octyl acrylate (8A) and dodecyl acrylate (12A). 2,2-Azobis(isobutyronitrile) was used to initiate the polymerization. The difunctional acrylate introduces cross-links in the polymerized matrix and the monofunctional acrylates provide the cross-linked matrices with various lengths of alkyl chains extruding from the surface of the matrices, which are expected to affect significantly the anchoring behavior of the LCs. E7 (Merck Japan Ltd.), a four-component mixture of substituted cyanobiphenyls and a cyanoterphenyl, was used as a host nematic LC (NLC). This NLC shows an N phase between −20 and 61°C. We also used 4-butyl-4′-methoxyazobenzene (BMAB) as a guest photochromic molecule to impart the photoresponsiveness to the composite films. BMAB is reversibly transformed between the *trans* and *cis* form by irradiation with light corresponding to its π–π^* (around 360 nm) and n–π^* (around 420 nm) transition band. The *trans* isomer exhibits an N phase between 35 and 45°C, while the *cis* isomer shows no LC phase at any temperature.

2.2. Preparation of samples

The composite films were prepared from their homogeneous solutions of 14 wt% acrylate mixture (50 wt% DA + 48 wt% nA + 2 wt% AIBN) and 86 wt% LC mixture (87 wt% E7 + 13 wt% BMAB). The starting mixtures were injected into a glass cell separated with 13 μm silica spacer and then the samples were cured in a Mettler hot stage (Model FP-82 and FP-90) at 90°C over 90 min to ensure full cure. The prepared samples were termed *n*A/DA-CFs ($n = 4, 6, 8, 12$) or DA-CF without the monofunctional acrylate. DA-CF was prepared from the mixture of 14 wt% DA and 86 wt% LCs.

2.3. Optical measurement and SEM observation

The photoresponsive behavior of the composite films was investigated by using the same experimental setup as the previous report [9]. The setup consisted basically of a 500 W high-pressure mercury lamp with glass filters as a pumping light source, a He–Ne laser (633 nm) as a probe light source, and a photodiode. The photoirradiation of the samples was performed with 366 and >420 nm light, which correspond to the maximum absorbencies of the *trans*- and *cis*-azobenzene, respectively. The optical change of the samples induced by photoirradiation was followed by detecting intensity of the probe light transmitted through the samples with the photodiode. The intensity was automatically recorded with a microcomputer. We defined the 100% light transmittance as the intensity of the probe light in the absence of the samples. The optical texture of the composite films was observed under crossed nicols with a polarizing microscope (Olympus Model BH-2), and the light from the lamp of the microscope was filtered to avoid unnecessary exposure that is susceptible to photoisomerization of the azobenzene. The morphological observation of the solid polymer in the composite films

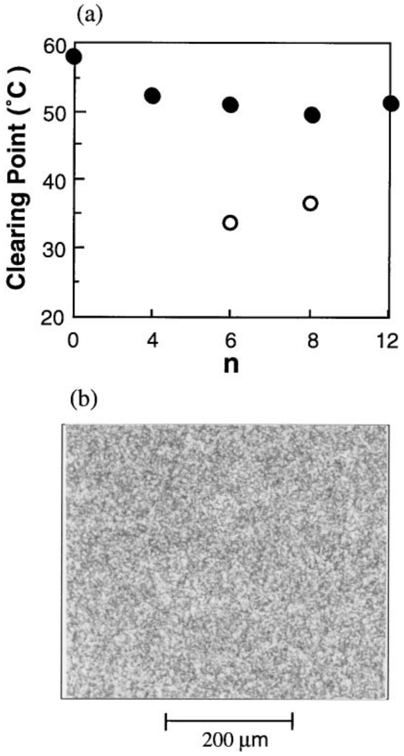

Fig. 1. Clearing points of the composites before (○) and after (●) thermal polymerization at 90°C for 90 min (a) and the optical texture of 12A/DA-CF observed between crossed polarizers at room temperature (b). The pre-homogeneous mixtures of DA-CF, 4A/DA-CF and 12A/DA-CF showed I phase. The optical textures of the other composite films were similar to that of (b).

was performed with a scanning electron microscope (SEM), JEOL JSM 35 CF. The specimen preparation was as follows: the cured samples were immersed in hexane until the low-molecular-weight LCs were entirely extracted to obtain the bare polymers, and then the cells were opened carefully after immersion in liquid nitrogen for a while.

3. Results and discussion

3.1. Thermal polymerization-induced phase separation

Fig. 1(a) shows clearing points of the starting mixtures and the resultant composite films. The clearing point was determined by monitoring the light intensity passed through the sample between the crossed polarizers of the polarizing microscope, and defined as the temperature at which the anisotropic texture disappeared entirely. The clearing points are plotted in Fig. 1(a) as a function of the number of the carbon atoms in the side chains of *n*A in the composite films. Before polymerization, the mixtures exhibited the N phase at room temperature (open symbols) or no LC phase at any temperature. However, new clearing points were observed around 50°C (closed symbols) in the composite films after polymerization. Fig. 1(b) shows the optical texture of 12A/DA-CF as a typical example of the texture after polymerization. The starting homogeneous mixture converted into the inhomogeneous composite film like *sandpaper*. This film presumably consists of the cross-linked polymer-rich I matrix and the LC-rich anisotropic domains. Similar textures were also observed in the other composite films. Although the unreacted monomers might have remained in the LC domains, we regarded the composite films with such texture as full-cured films because the clearing point no longer changed even after further polymerization. These results indicate that the polymerization well took place in the I fluid, and the LC molecules were separated from the mixture due to decrease in the mutual solubility as the molecular weight of the acrylate was increased by polymerization.

3.2. Observation of polymer matrix by SEM

In order to investigate the morphology of the composite films prepared, electron microscopy was employed. Fig. 2 shows the SEM micrographs of the bare polymers in the full-cured composite films. In situ polymerization of the monomers led to formation of polymer network that interconnected three-dimensionally. These micrographs suggest that the LC domains had existed as continuous cylindrical channels in the pores or interstices. The channels were distributed multiplicitly in submicrometer and micrometer scales. There is a distinct difference in the morphology of the composite films polymerized with a DA monomer and *n*A/DA monomers. The DA-CF consists of smaller channels in the network aggregated densely as nodular beads fused irregularly, whereas the *n*A/DA-CFs have relatively large channels in the less dense network.

3.3. All-optical switching of composite films

The photoresponsiveness of the composite films was explored as change in transmittance of the probe light passed through the irradiated region when they were illuminated at wavelengths to bring about the photoisomerization of the guest azobenzene. Fig. 3 shows change in the transmittance measured simultaneously as a function of time when DA-CF (a) or 4A/DA-CF (b) was irradiated alternately at 366 nm (20 mW/cm^2) and at >420 nm at room temperature, respectively. In the initial states of the composite films, the transmittance of the probe light was very small because the light was scattered strongly by the samples. However, a very drastic change in transmittance occurred by irradiation at 366 nm. The transmittance increased and reached eventually each saturated state, at which the samples exhibited a clear transparent state. This change indicates that the photochemical N–I phase transition took place in the LC domains, which resulted from the *trans–cis* photoisomerization of the azobenzene.

In addition, the saturation state exhibited a completely transparent state, which was comparable with the transparency above their clearing points. Generally, the clearing

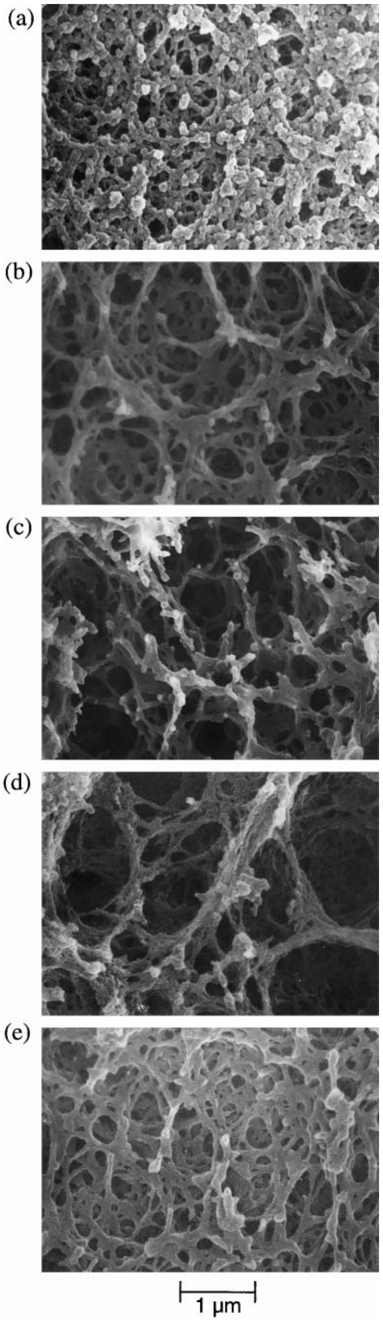

Fig. 2. SEM micrographs of the polymer network of DA-CF (a), 4A/DA-CF (b), 6A/DA-CF (c), 8A/DA-CF (d) and 12A/DA-CF (e).

point of the composite films is defined as a temperature at which the light scattering vanishes completely due to the thermal N–I phase transition of the LC domains. In the present study, the fact that the transparent states were obtained isothermally by irradiation at 366 nm means that the clearing temperature of the composite films was changed on photoirradiation. In fact, the inherent clearing point of 4A/DA-CF (52°C) was found to decrease over by 27°C on photoirradiation.

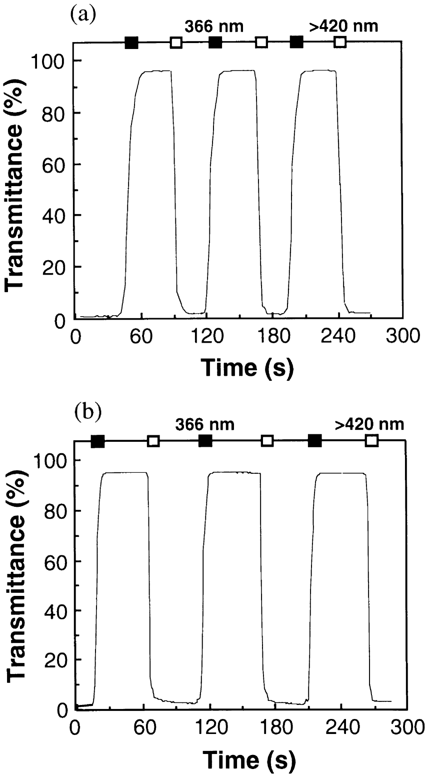

Fig. 3. All-optical switching in transmittance between opaque light-scattering states and clear transparent states of DA-CF (a) and 4A/DA-CF (b) induced by periodic irradiation at 366 nm (20 mW/cm^2) (■) and at >420 nm (□) at room temperature.

The transparent states induced by the photochemical N–I phase transition could return to the initial light-scattering states by the I–N phase transition induced by thermal or photochemical *cis–trans* back-isomerization of the guest azobenzene. However, it took more than 1 h at room temperature that the transparent states were transformed to the initial light-scattering states thermally because of the slow rate of the thermal *cis–trans* isomerization of the azobenzene at room temperature. Therefore, photoirradiation at >420 nm was necessary to restore efficiently the initial light-scattering state as the light is very effective to accelerate the *cis–trans* back-isomerization. When irradiated at >420 nm, the transparent samples were immediately returned to the initial light-scattering state. These results demonstrate that all-optical switching in transmittance could be performed repeatedly between opaque light-scattering states and clear transparent states by alternate irradiation of the composite films at 366 nm and at >420 nm.

3.4. Optical memory effect of composite films

Fig. 4 shows the photoresponsive behavior of the other composite films prepared from the starting mixtures containing various monofunctional acrylates when they were photoirradiated under the same conditions as those of DA-CF. Fig. 4(a) shows the responsive behavior on photoirradiation at 366 nm. In all composite films, the initial

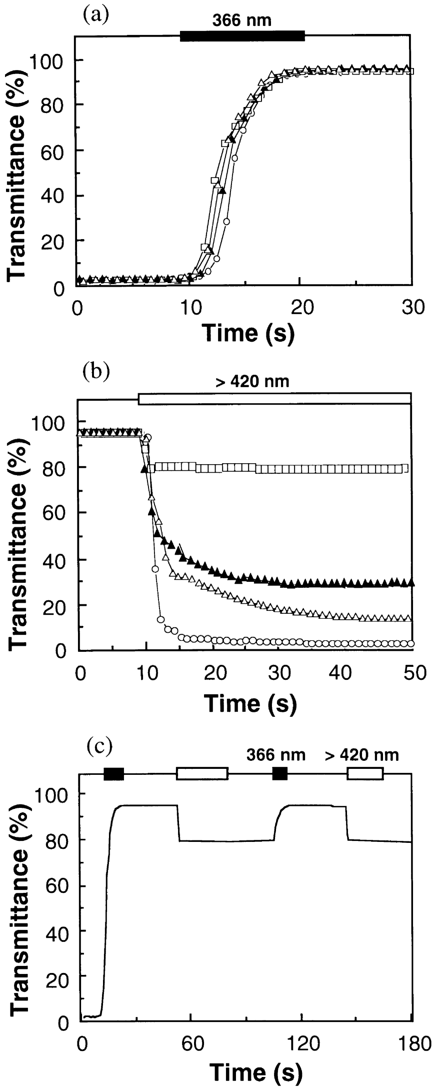

Fig. 4. Photoresponsive behavior of the composite films with various alkyl side chains on irradiation at 366 nm (20 mW/cm^2) (a) and at >420 nm (b), and optical behavior of 12A/DA-CF induced by alternating irradiation at 366 nm and at >420 nm (c): ○, 4A/DA-CF; △, 6A/DA-CF; ▲, 8A/DA-CF; □, 12A/DA-CF.

light-scattering powers decreased with photoirradiation, and they were finally transformed into a transparent state after roughly 10 s. Although each composite film having an alkyl brush of a different length on the polymeric matrices, the photoirradiation resulted not only in a similar response but also in an identical state in transparency with DA-CF without the side chains. These results clearly indicate that the photochemical N–I phase transition occurred efficiently in the LC domains surrounded by the polymeric matrices and the side chains of the matrices imparted no appreciable influences to the photoresponsive behavior.

Intriguing optical behavior was observed in the photoinduced recovery process to the initial states. Fig. 4(b) shows the optical behavior of the composite films on irradiation at > 420 nm. It was found that the composite films showed different photoresponsive behavior from that of 4A/DA-CF and DA-CF. Despite the photoirradiation at >420 nm which was very effective to transform the transparent composite films into light-scattering state, the initial light-scattering states could not be obtained in the films with $n =$ 6, 8 and 12. Further, the transmittance remained unchanged at some levels (i.e. memorized states) even if the photoirradiation at >420 nm was continued or ceased. In particular, 12A/DA-CF was characteristic of a highly memorized state with 80% in transmittance, whereas light scattering was predominant in the other composite films after irradiation at >420 nm.

To investigate the relationship between the alkyl side chains and the photoresponsive behavior in further detail, another series of 12A/DA-CF with various compositional ratios of 12A to DA were prepared under the same conditions as the above samples. The compositional ratio with respect to the LCs was kept the same. Fig. 5 shows the three different optical states which are characterized by the initial state (closed symbols) and the photoinduced optical states (open symbols). The horizontal axis indicates the ratio of 12A to DA (12A/DA, wt%/wt%) in the acrylate mixture. At any ratio of 12A to DA, the composite films showed strong light-scattering states before photoirradiation (closed circles) and they were transformed into the complete transparent states (open circles) by photoirradiation at 366 nm as shown in Fig. 4(a). These results indicate that the side chain does not affect significantly the photochemical N–I phase transition in the LC domains at any ratio of 12A/DA.

When the transparent films were irradiated at >420 nm, the transmittance of the probe light decreased due to increase in light-scattering power on photoirradiation. However, the transmittance no longer decreased below some levels even on prolonged irradiation (open triangles). It was observed that the level of the transmittance depended on the amount of 12A and increased as the ratio of 12A/DA increased as shown in Fig. 5. From these results, it seems reasonable to assume that the photoresponsive behavior in

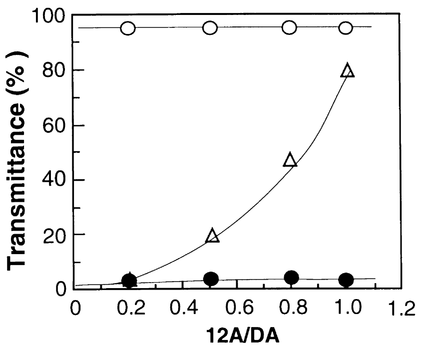

Fig. 5. Photoinduced changes in transmittance of 12A/DA-CF prepared from the homogeneous mixtures containing various compositional ratios of 12A to DA (12A/DA, wt%/wt%): ●, initial state before irradiation; ○, transparent state induced by irradiation of the initial film at 366 nm; △, memorized state induced by irradiation of the transparent film at >420 nm.

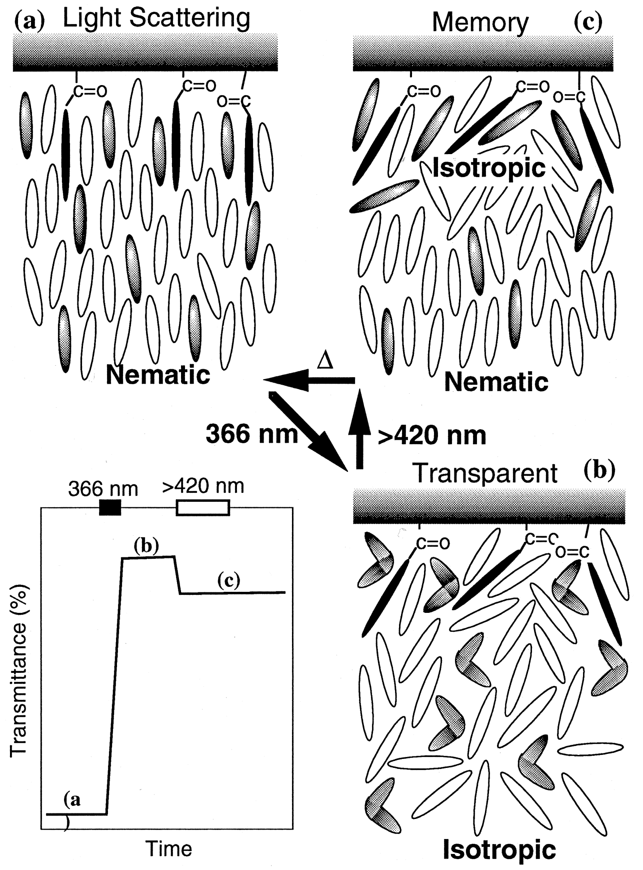

Fig. 6. Schematic model for the optical effect in the composite films with the alkyl side chains on surface of the polymer matrix. Upon irradiation at 366 nm, the ordered N phase in the initial light-scattering state (a) is transformed into disordered I phase in the transparent state (b) induced by *trans–cis* photoisomerization of guest azobenzene molecules. When the transparent film in (b) is irradiated at >420 nm, the N phase is recovered in the bulk due to *cis–trans* photoisomerization of the azobenzene molecule, but not in the surface region because of strong anchoring effect of the long alkyl side chains (c). The memorized state is transformed into the initial ordered N phase (a) in the light-scattering state by thermal treatment above the clearing point and then cooling down.

the recovery and the memorized states was strongly affected by the alkyl side chains on the polymer matrix surfaces.

3.5. Mechanism of memory effect

Here we need to discuss why the high-transmittance state was maintained in the composite films with long alkyl side chains, despite photoirradiation at >420 nm. The above experimental results led to the model in Fig. 6 for the photoresponsive behavior and the memory effect. In the present composite films, the changes in transmittance result from two processes, although it is difficult to separate them under steady-state irradiation because they occur simultaneously and the composites are a complex system. The first process is the photoisomerization of the doped azobenzene molecules, which is followed by reorientation of the surrounding molecules.

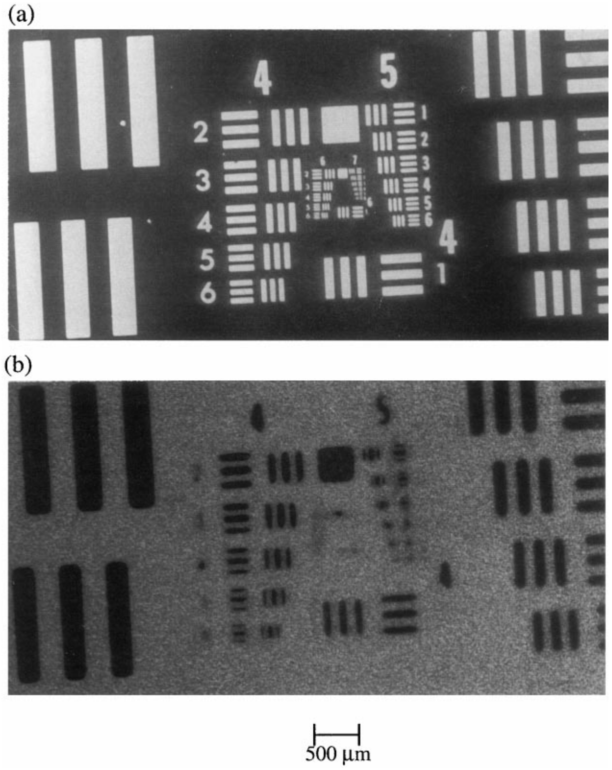

Fig. 7. Optically recorded image in 12A/DA-CF (b). The sample was covered with a negative photomask (a) and irradiated with 366 nm light at room temperature.

Fig. 6(a) shows schematic illustration of alignment of NLC near the polymer wall with the alkyl side chains in the initial composite films. Multiple distribution of such configurations leads to light scattering because orientation of the optic axis varies randomly from domain to domain by the irregular polymer surfaces. The *trans–cis* isomerization of the azobenzene guest molecules by photoirradiation at 366 nm results in the transparent composite films due to the N–I phase transition that eliminates the director fields (Fig. 6(b)).

After photoirradiation at >420 nm, we assume that there are two distinct domains in the fluid LC channels in the polymer matrices with respect to the reorientation from the induced I phase to the N phase: the I domains at the center of the channels are free to change quickly to the ordered N phase concomitant with light scattering on irradiation at >420 nm, but the I domains near the polymer wall are constrained to reorient along the alkyl side chains which are still in the disordered state. The photoinduced memory states shown in Fig. 6(c) may be characterized by coexistence of the two phases: the N domains at the center and the I domains near the alkyl side chains within the fluid channels. In fact, the two domain sizes in the memorized states are very important to confirm the effect of the side chains on the memorized states. Unfortunately, it is difficult to define the actual sizes of the two different phases due to the complex structure of the polymer matrices and the multi-sized LC channels. The different recovery behavior between the composite films with or without side chain chains as shown in Figs. 3 and 4, however, obviously indicates that the memorized states resulted from the presence of the side chains. The transmittance of the memorized states presumably depends on the volume fraction of the I domains remaining at the surfaces of the polymer walls, which is also strongly dependent on the length of the alkyl side chains extruding from the surfaces. It seems reasonable that the highest transmittance in the memorized states was observed in 12A/DA-CF that possessed the longest side chains among the monofunctional acrylates examined in the present study.

3.6. Image storage experiments

Such memory effect can be applied to optical image storage. Image storage by the use of a photomask was performed in 12A/DA-CF at room temperature. The sample was covered directly with a negative photomask and irradiated at 366 nm to induce the N–I phase transition in the LC domains. The stored image was examined under crossed nicols in the polarizing microscope. Photographs of the photomask and the stored image are shown in Fig. 7 (a) and (b), respectively. The black images in (b) correspond to the irradiated site (I phase). In fact, the stored image showed a high contrast with the opaque light scattering and the photoinduced clear transparent sites at direct observation. The transparently memorized image was maintained roughly for three months at room temperature, although the relaxation to the initial light-scattering state took place in several months. The recorded image was erasable effectively by cooling it down slowly after heating the sample above the clearing point at which all domains are I phase. Additionally, the resolution in the image storage can be improved further by eliminating experimental defects, such as thickness of glass substrates, poor spatial distribution of the Hg lamp beam, and distortion of the focus of the writing beam.

4. Conclusion

We have shown the photoresponsive behavior of the light-scattering composite films, which are composed of acrylate polymers with alkyl side chains and a low molecular weight NLC containing an azobenzene as a photochromic dopant. The photoresponsive behavior was strongly influenced by the monofunctional acrylate monomers used for the preparation of the composite films. Introduction of the side chains on the polymer matrix enabled not only the optical switching but also the optical image storage by controlling their lengths. In 4A/DA-CF with the short alkyl side chains, the reversible and repeatable changes between the light-scattering and clear transparent states could be achieved by alternating irradiation at 366 and >420 nm similar to DA-CF without side chains. Therefore, 4A/DA-CF and DA-CF acted as a haze-free all-optical switching material without polarizers. Such light-scattering mode driven by light may be potential for all-optical switching or optically controllable projection materials. It was confirmed that the photoresponsive behavior in the recovery process was significantly affected by the alkyl side chains. Particularly, 12A/DA-CF with the long alkyl side chain showed the memorized transparent state induced by irradiation at 366 nm. So the light-scattering image with a high contrast could be stored in the composite film through a very simple manner of photo-irradiation and could be rewritten after erasing thermally. Finally, the memory function of the present system needs to be investigated further in order to enhance long-term stability of the recorded information.

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