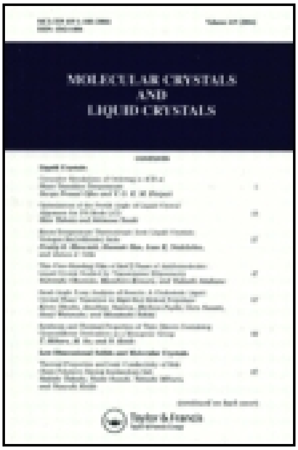

Molecular Crystals and Liquid Crystals Incorporating Nonlinear Optics
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl17

Photochemical Phase Transition Behaviors of Ternary Mixtures of Photochromic Azobenzene Derivative, Low Molecular Weight Liquid Crystal and Polymer Liquid Crystal

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Version of record first published: 04 Oct 2006.

To cite this article: T. Ikeda , T. Miyamoto , T. Sasaki , S. Kurihara & S. Tazuke (1990): Photochemical Phase Transition Behaviors of Ternary Mixtures of Photochromic Azobenzene Derivative, Low Molecular Weight Liquid Crystal and Polymer Liquid Crystal, Molecular Crystals and Liquid Crystals Incorporating Nonlinear Optics, 188:1, 235-250

To link to this article: http://dx.doi.org/10.1080/00268949008047820

Mol. Cryst. Liq. Cryst., 1990, Vol. 188, pp. 235–250
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Printed in the United States of America

Photochemical Phase Transition Behaviors of Ternary Mixtures of Photochromic Azobenzene Derivative, Low Molecular Weight Liquid Crystal and Polymer Liquid Crystal[§]

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(*Received April 3, 1990; in final form June 11, 1990*)

Ternary mixtures of a photochromic azobenzene guest, low molecular weight liquid crystal (LMWLC) and polymer liquid crystal (PLC) were used and their photochemical phase transition behaviors were explored with special Reference to the stability of the photochemically induced isotropic phase and the response to photoirradiation. It was found that the rate of the photochemical phase transition increased as the mole fraction of the LMWLC component increased, demonstrating that the response of the systems is improved in the presence of the LMWLC components. Furthermore, the stability of the photochemically stored image has been shown to be much improved in the presence of the PLC components. Results obtained in this study clearly indicate that the LMWLC/PLC mixtures are promising as hosts for the optical image recording devices.

Keywords: photochemical phase transition, azobenzene, photoisomerization, ternary mixture, polymer liquid crystal

INTRODUCTION

Low molecular weight liquid crystals (LMWLCs) have been widely used as active media in display devices. These display devices are essentially based on the electro-optic effect of LCs and require a matrix arrangement of transparent electrodes.[1] For improved resolution, laser-beam addressed display systems of LMWLCs have recently been developed.[1] These systems are based on the thermo-optic effects of

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[‡] Deceased on July 11, 1989.

[§] Part V of Effect of Structure of Photoresponsive Molecules on Photochemical Phase Transition of Liquid Crystals.

LCs and high resolution has been achieved by the use of the focused laser beam (< 10 μmϕ). However, the stability of the stored image is not satisfactory.[1]

The use of polymer liquid crystals (PLCs) as optical-image storage materials has become a topic of interest.[2] The advantages of PLCs over LMWLCs as image storage materials are: (1) because of the glass transition phenomenon (T_g), segmental motion of the polymer chains can be frozen-in, thus it is expected that the storage image can be kept stable below T_g for a long period; (2) good film-forming properties of the polymers expectedly enable cell-free performance of the image storage materials, which is evidently favored from an applicational viewpoint; (3) low fluidity of the polymers is favorable for the long-term stability of the stored image.

Shibaev *et al.*[3] and Coles and Simon[4] reported on laser-addressed PLC storage displays in which heat-mode writing was employed. Photon-mode image storage in the PLC was first reported by Eich and Wendorff as "holographic" optical storage.[5,6] In their system, photoisomerization of photochromic groups (azobenzene derivatives) incorporated into the PLCs induced "grating" in the PLC, which was a consequence of the change in the refractive index resulting from the isomerization.

In our research scheme on the photochemical phase transition behaviors of various liquid crystalline systems, which aims at the optical-image storage devices, we have explored the photochemical isothermal phase transition of various LMWLCs and PLCs triggered by photochemical reaction of only a minute amount of photoresponsive guests incorporated into the systems.[7-18] The working principle of our systems is *isothermal phase transition of LCs* at the irradiation site, thus we can expect a high contrast and a high signal-to-noise (S/N) ratio for the stored information.[19]

In a series of studies on the effect of the structure of photoresponsive molecules on the photochemical phase transition of LCs, we have revealed that azobenzene derivatives with mesogenic 4-cyanobiphenyl moieties in a single molecule act as effective trigger guest molecules both in the azo guest/LMWLC mixtures[15,17] and in the azo guest/PLC mixtures.[18] Furthermore, high stability of the stored information has been achieved in the azo guest/PLC host mixtures,[12] however, the response of these mixtures was found to be inferior to those of the azo guest/LMWLC mixtures.[18] From an applicational point of view, it is desired to provide the optical-image storage devices with both high stability of the stored information and quick response in the writing and read-out processes. One promising approach to solve this problem is to employ LMWLC/PLC mixtures as hosts in the photon-mode image recording systems. It is expected that in these systems LMWLC is responsible for the quick response and high stability of the stored information is achieved by the part of the PLC. In fact, the LMWLC/PLC mixtures have been demonstrated to work successfully in the electric-field-induced phase change of the systems.[20]

In the present study, 4-*n*-butyl-4′-methoxyazobenzene (BMAB) was used as a photoresponsive guest in the LMWLC/PLC mixtures and the photochemical phase transition behaviors of these ternary mixtures were investigated with special reference to such properties of the mixtures as the high stability of the photochemically induced phase and the quick response to photoirradiation. The choice of BMAB is based on the fact that this azobenzene derivative is mesogenic and can induce the photochemical phase transition of LCs quite effectively.

EXPERIMENTAL

Figure 1 shows the structures of the photochromic azobenzene guest, LMWLCs and PLCs as well as their abbreviations used in this study.

4-*n*-Pentyl-4′-cyanobiphenyl (5CB) and 4-*n*-pentoxy-4′-cyanobiphenyl (5OCB) were purchased from Merk Co. and used without further purification. 4-Hexyloxyphenyl-4′-ethyl-cyclohexane-carboxylate (ECH 206) was obtained from Fuji Pigment Co., Ltd. and used without further purification. Their phase transition temperatures are as follows: 5CB (K24N35I), 5OCB (K48N68I) and ECH206 (K22N46I). A polyacrylate with mesogenic phenyl benzoates in the side chain (PAPB3) and those with mesogenic cyanobihenyls in the side chain (PACB3 and PACB6) were

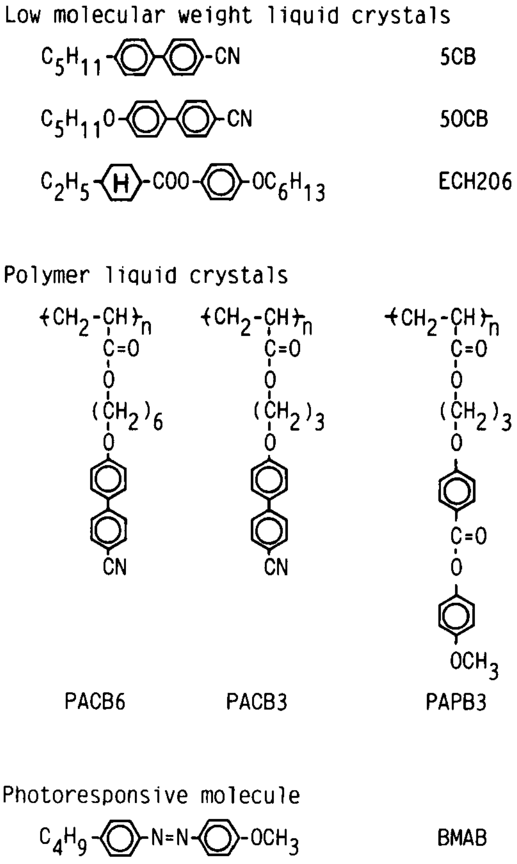

FIGURE 1 Structure of a photochromic azobenzene guest (BMAB), low molecular weight liquid crystals (LMWLCs) and polymer liquid crystals (PLCs) used in this study and their abbreviations.

prepared and purified as reported previously.[11,16] Synthesis and purification of 4-*n*-butyl-4′-methoxyazobenzene (BMAB) was conducted by the method already reported.[7] The number-average molecular weight (M_n), weight-average molecular weight (M_w) and the polydispersity index (M_w/M_n) of the PLC samples used in this study are listed in Table I.

LC behavior and phase transition behavior were studied on an Olympus model BHSP polarizing microscope equipped with a Mettler hot stage model FP-80 and FP-82. The thermodynamic properties and the glass transition temperature (T_g) of the mixtures were determined with a differential scanning calorimeter (DSC; SEIKO I&E SSC-5000) at a heating rate of 10°C/min. At least four scans were performed for each sample to check reproducibility.

The photochemical phase transition behaviors of BMAB/LMWLC/PLC mixtures were investigated by means of the apparatus already reported.[12,13] Samples (the LMWLC/PLC mixtures containing 5 mol% of BMAB) were prepared by casting the mixture solutions in chloroform onto glass plates. The mixture films were then dried under reduced pressure and their LC behavior was examined with a polarizing microscope after annealing in a thermostat at temperatures where the mixture films showed an LC phase. The sample mixtures thus prepared were placed in a thermostated block and irradiated with a monochromatic light from a JASCO CRM-FA irradiator.

RESULTS AND DISCUSSION

Thermotropic properties of the ternary mixtures

The liquid crystalline to isotropic (I) phase transition temperature (T_C) and the glass transition temperature (T_g) of the ternary mixtures were determined by microscopic observation and DSC measurements, respectively. Although the LC phase was observed in every mixture in the polarizing microscope, clear endothermic peaks due to the LC to I phase transition were undetectable in DSC in most of

TABLE I

Molecular weight and polydispersity index of PLCs used in this study.[a]

PLC	M_n	M_w	M_w/M_n
PAPB3	2900	9900	3.4
PACB3	6800	8600	1.3
PACB6	4000	5800	1.5

[a] Determined by GPC using polystyrene as standard.

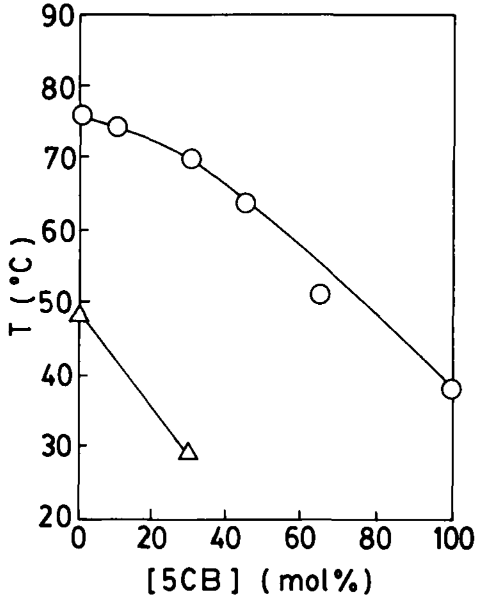

FIGURE 2 Phase transition temperatures (T_C) and glass transition temperatures (T_g) of the BMAB/5CB/PAPB3 mixtures as a function of the mole fraction of 5CB. (○), T_C; (△), T_g. The concentration of BMAB was kept at 5 mol%.

the mixtures and only detectable in BMAB/5CB/PACB6. In Figures 2–4 are shown T_C (○) and T_g (△) of the BMAB/5CB/PAPB3, BMAB/5CB/PACB3 and BMAB/5CB/PACB6 mixtures as a function of the mole fraction of the LMWLC components, respectively. In these phase diagrams, the mole fraction of BMAB was kept constant (5 mol%). In all ternary mixtures possessing 5CB as a LMWLC component, both T_C and T_g decreased rather monotonically as the mole fraction of 5CB increased. However, the appearance of the phenomenon of the glass transition as a shift of the base line toward endothermic direction in DSC thermograms depended strongly on the PLC component of the mixtures. Namely, in the BMAB/5CB/PAPB3 and BMAB/5CB/PACB3 mixtures, the glass transition was detected at the mole fraction of 5CB up to 30 mol% and 40 mol%, respectively, while in the BMAB/5CB/PACB6 mixtures it was observed even at the mole fraction as high as 60 mol%. The monotonical decrease in T_g may be interpreted as the LMWLC component acting as a plasticizer in the mixtures. A similar role of BMAB has been confirmed in the BMAB/PAPB3 mixtures.[11]

In relation to the stability of the photochemically induced I phase, which is closely related to the stability of the stored information in the photon-mode optical recording systems, it has been revealed that T_g plays a significant role.[12] The induced I phase by photoirradiation in the BMAB/PAPB3 mixture could be kept without change for more than one year by keeping the irradiated sample below T_g of the mixture.[12] It seems very convenient, therefore, that the sample mixtures possess T_g around room temperature or a little higher than room temperature. From this

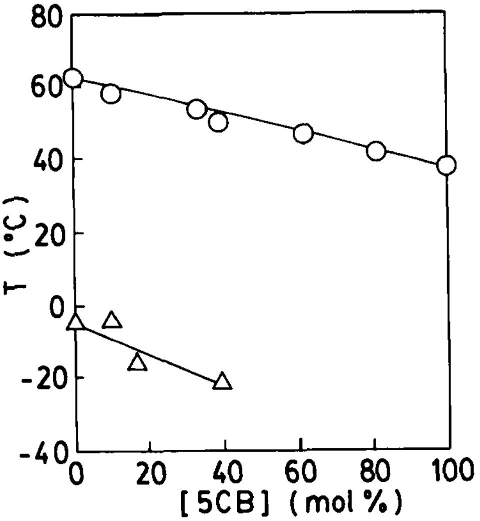

FIGURE 3 Phase transition temperatures (T_C) and glass transition temperatures (T_g) of the BMAB/5CB/PACB3 mixtures as a function of the mole fraction of 5CB. (○), T_C; (△), T_g. The concentration of BMAB was kept at 5 mol%.

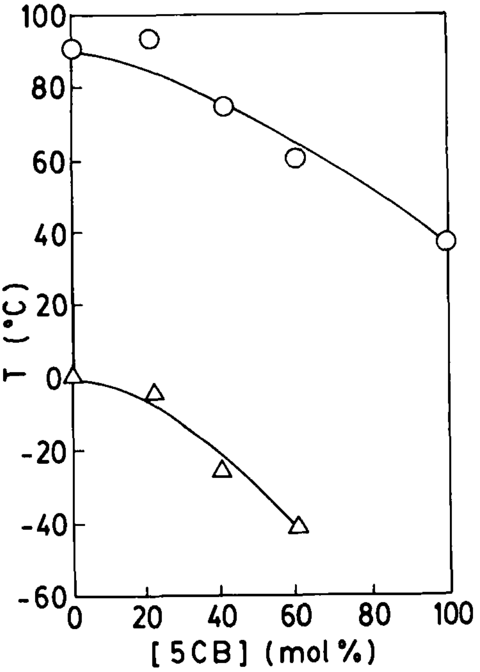

FIGURE 4 Phase transition temperatures (T_C) and glass transition temperatures (T_g) of the BMAB/5CB/PACB6 mixtures as a function of the mole fraction of 5CB. (○), T_C; (△), T_g. The concentration of BMAB was kept at 5 mol%.

standpoint, the BMAB/5CB/PAPB3 mixtures are better than the other mixtures, since the BMAB/5CB/PAPB3 mixtures showed T_g of around 30°C even at the mole fraction of the LMWLC component of 30 mol%.

In Figure 5 is shown the change in enthalpy (ΔH_C) and entropy (ΔS_C) at the LC to I phase transition of the BMAB/5CB/PACB6 mixtures as a function of the mole fraction of the LMWLC component. It is seen that both values exhibited maximum values at the composition of 5CB/PACB6 = 20/80 (mol/mol) and then gradually decreased with increasing the mole fraction of the LMWLC component. This behavior may be explicable in terms of the close packing of the mesogenic moieties in the mixture at this particular composition, since it is expected that the LMWLC components may act as stuffing in the mixture to result in the closely-packed state of mesogenic groups in the mixture.

Figure 6 shows the T_C and T_g of the BMAB/5OCB/PACB3 mixtures as a function of the mole fraction of 5OCB. In these mixtures, T_C of BMAB/PACB3 was very similar to that of BMAB/5OCB, thus T_C of the ternary mixtures did not change significantly with the mole fraction of the LMWLC component. Furthermore, T_g was observed in a mixture with the LMWLC component as high as 60 mol% as in the case of the BMAB/5CB/PACB6 mixtures.

It is worth mentioning here that we examined other combinations for the mixtures; BMAB/ECH206/PAPB3. We expected that both LMWLC and PLC components in this mixture possess a similar structure, thereby we would have ternary mixtures of BMAB/ECH206/PAPB3 well miscible with each other. However, the ECH206/PAPB3 showed phase separation at any compositional ratio as revealed by microscopic observation and DSC measurements. Therefore, we did not examine their photochemical phase transition behaviors.

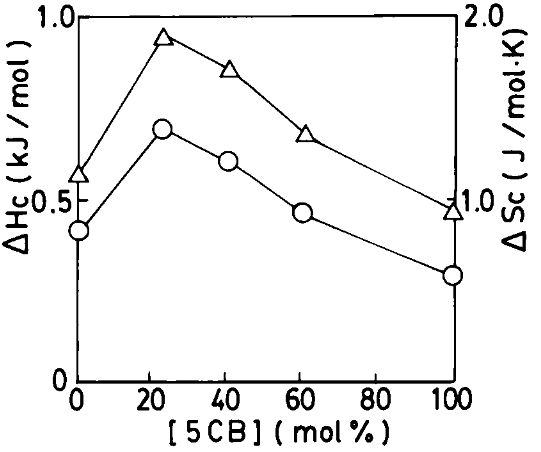

FIGURE 5 Change in enthalpy (ΔH_C) and entropy (ΔS_C) at the liquid crystalline to isotropic phase transition of the BMAB/5CB/PACB6 mixtures. (Δ), ΔH_C; (○), ΔS_C. The concentration of BMAB was kept at 5 mol%.

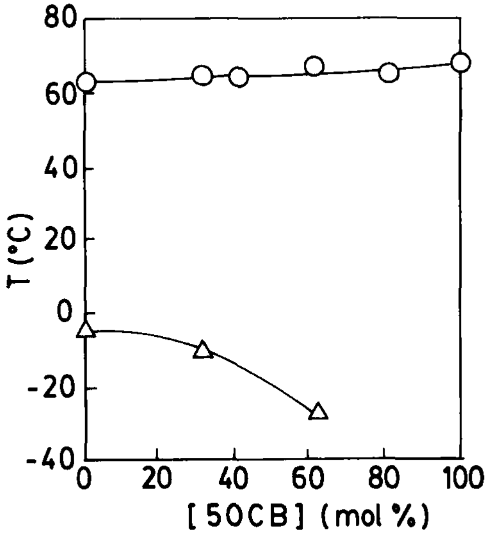

FIGURE 6 Phase transition temperatures (T_C) and glass transition temperatures (T_g) of the BMAB/5OCB/PACB3 mixtures as a function of the mole fraction of 5OCB. (○), T_C; (△), T_g. The concentration of BMAB was kept at 5 mol%.

Photochemical phase transition behaviors of the ternary mixtures

A typical example of the photochemical isothermal phase transition behaviors of the BMAB/LMWLC/PLC mixtures is shown in Figure 7 where a BMAB/5OCB/PACB3 mixture containing 5 mol% of BMAB was irradiated at 360 nm at the reduced temperature ($T_{red} = T/T_C$) of 0.99. In this figure, the change in transmittance of the linearly polarized light (633 nm), I_t, is shown as a function of irradiation time. The transmittance was measured through the crossed polarizers between which the sample was placed. It is clearly seen that photoirradiation induced the LC to I phase transition of the mixture isothermally, as demonstrated by the loss of birefringence ($I_t = 0$). In the course of the LC → I phase transition, the change in I_t is strange: it decreased initially, going up to a maximum value and then decreased again to $I_t = 0$. This behavior of I_t is explicable in terms of interference, which was thoroughly discussed in the previous paper.[12] As described in the preceding paper,[18] we defined the response time, τ, as the time required to reduce the transmittance, I_t, to 10% of the maximum value in order to discuss the photochemical phase transition behavior quantitatively. In the present study, all the photochemical phase transition behaviors of the ternary mixtures were evaluated at $T_{red} = 0.98$. Furthermore, because of high concentration of the photoresponsive dopant in the mixtures the change in absorbance on photoirradiation could not be measured reliably, so that in the present study the rates of photoisomerization were not determined.

In Figure 8 is shown the rates of the photochemical phase transition of the BMAB/5CB/PLC ternary mixtures containing 5 mol% of BMAB as a function of the mole fraction of 5CB. As described above, the rates of the photoisomerization of BMAB

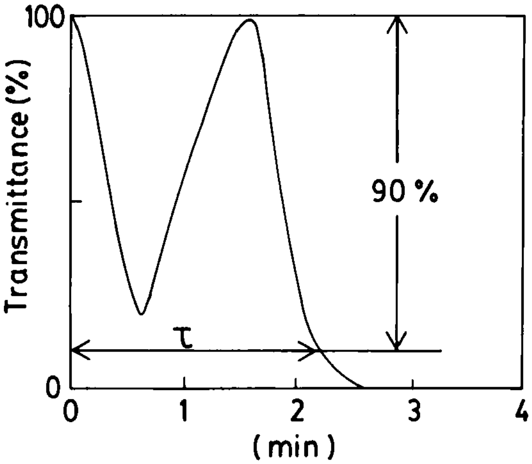

FIGURE 7 Photochemical phase transition behavior of the BMAB/5OCB/PACB3 mixture doped with 5 mol% of BMAB at T_{red} = 0.98. Photoirradiation was performed at 360 nm and the transmittance of a He-Ne laser (633 nm) observed between a pair of the crossed polarizers is plotted as a function of the irradiation time. The compositional ratio of 5OCB/PACB3 was 40/60 (mol/mol).

could not be determined in the present mixtures, thereby rates evaluated by the reciprocal of the response time (τ^{-1}) are shown in this figure. A general trend can be seen in that the rates of the photochemical phase transition increased as the mole fraction of the LMWLC component increased. Furthermore, the rates of the photochemical phase transition increased in the order of the BMAB/5CB/PAPB3 < BMAB/5CB/PACB3 < BMAB/5CB/PACB6 mixtures. It must be mentioned

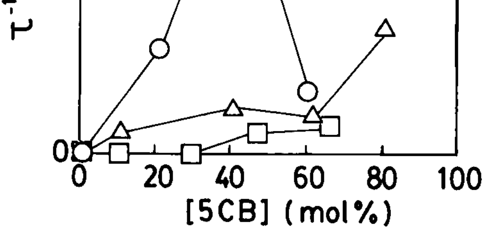

FIGURE 8 Rates of the photochemical phase transition of the BMAB/5CB/PLC mixtures as a function of the mole fraction of 5CB. (□), BMAB/5CB/PAPB3; (△), BMAB/5CB/PACB3; (○), BMAB/5CB/PACB6. The rate of the photochemical phase transition was evaluated by the reciprocal of the response time, τ^{-1}. Photoirradiation was performed at 360 nm at T_{red} = 0.98.

here that in the absence of the LMWLC component the isothermal phase transition of the mixtures could not be induced photochemically in any BMAB/PLC mixtures. This is a reasonable result on the basis of the previous results.[12,16]

The low rates of the photochemical phase transition observed for the BMAB/5CB/PAPB3 mixtures seem to be rationalized in view of the weak interaction between the cyanobipheny mesogens in the LMWLC component and the phenyl benzoate mesogens in the PLC component. It was shown in Part II and Part III of this series that 4-cyanobiphenyl moieties incorporated into the photoresponsive azobenzene guest molecules were effective in inducing the isothermal phase transition of the guest/host mixtures owing to the interaction between the mesogenic groups in the photoresponsive guests and the host mesogens; thus perturbation imposed in the form of photoisomerization of the guest molecules to the host mesogens acted quite effectively to induce the photochemical phase transition of the mixtures.[15,17] However, the effect of the structure of the photoresponsive guests was different depending on the structures of the guests and the host mesogens. In the mixtures of the azobenzene guests and the 5CB host, the rates of the photochemical phase transition were higher by approximately two order of magnitude than those of the azo guests and the ECH206 host, although the rates of photoisomerization of the azo dopants were similar in both mixtures. This was interpreted in terms of stronger interaction between the 4-cyanobiphenyl moiety in the azo guest and the 5CB host than the interaction associated with the 4-cyanobiphenyl moiety and ECH206. In a similar system, i.e., a strongly interacting system, the perturbation triggered by the photoisomerization is considered to be propagated to the whole system due to the strong interaction, thereby the photochemical phase transition is expected to take place effectively. The same concept seems to be applied to the present systems. Namely, because of presumably weak interaction between the phenyl benzoate mesogens in PAPB3 and 5CB, the perturbation in the form of the photoisomerization may not be propagated effectively in the BMAB/5CB/PAPB3 mixtures. On the other hand, in the BMAB/5CB/PACB3 and BMAB/5OCB/PACB3 mixtures both the LMWLC and PLC components possess the same mesogenic moieties, so that the perturbation may be propagated effectively to the whole system, leading to higher rates of the photochemical phase transition of the mixtures.

In the BMAB/5CB/PACB6 mixtures, the rate of the photochemical phase transition was low at the compositional ratio of 5CB/PACB6 = 60/40 (mol/mol). Microscopic observation suggested phase separation in this mixture. Photoisomerization of BMAB in a LMWLC-rich domain expectedly acts effectively as a perturbation to induce the photochemical phase transition, however, in a PLC-rich domain the photochemical phase transition is retarded due to low mobility of the mesogens. Under the present experimental set-up, transmittance was measured across the mixture films, so that for such samples, as most of the area was in the induced I phase but only a small part was still in the LC phase, the photochemical phase transition would not be regarded as completed. The low rate of this mixture may be interpreted on this basis.

Figure 9 shows the rates of the photochemical phase transition of the BMAB/5OCB/PACB3 mixtures as a function of the mole fraction of the LMWLC com-

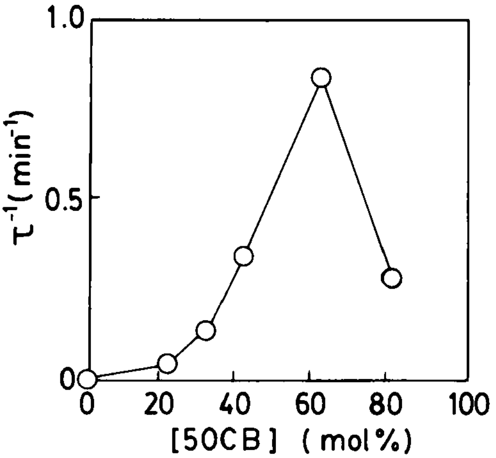

FIGURE 9 Rates of the photochemical phase transition of the BMAB/5OCB/PACB3 mixtures as a function of the mole fraction of 5OCB. The rate of the photochemical phase transition was evaluated by the reciprocal of the response time, τ^{-1}. Photoirradiation was performed at 360 nm at T_{red} = 0.98.

ponent. Study on these mixtures stemmed from the expectation that due to the presence of the oxygen atom in the LMWLC component intermesogenic interaction between the LMWLC and PLC components would be strengthened by dipole-dipole interaction, which would result in higher rates of the photochemical phase transition. In fact, the maximum rate was higher in these mixtures than that of a similar system, the BMAB/5CB/PACB3, however, the effect of the alkoxy group does not seem to be significant. In these mixtures, the rate was again low at a high compositional ratio of 5OCB to PACB3 (80/20) and this result is explicable in terms of the phase separation.

Effect of BMAB concentration

Figure 10 shows T_C and T_g of BMAB/5OCB/PACB3 mixtures as a function of the mole fraction of BMAB. Here, the composition of 5OCB to PACB3 was kept constant throughout (5OCB/PACB3 = 30/70 mol/mol) T_g decreased with increasing the concentration of doped BMAB and above 5 mol% the shift of the base line toward endothermic direction (T_g) could not be further observed. On the other hand, T_C was less affected by the concentration of the doped BMAB in these mixtures. Microscopic observation revealed that these mixtures apparently showed a homogeneous mixture macroscopically below the BMAB concentration of 20 mol%.

In Figure 11 are shown the rates of the photochemical phase transition of the BMAB/5OCB/PACB3 mixtures as a function of the mole fraction of BMAB, which were evaluated at T_{red} = 0.98. It is seen that the rate increased with the concentration of BMAB up to 5 mol%, and then decreased gradually. When the concentration of BMAB is high, the majority of the incident photons are absorbed at the surface layer of the mixtures and the phase transition takes place at the surface

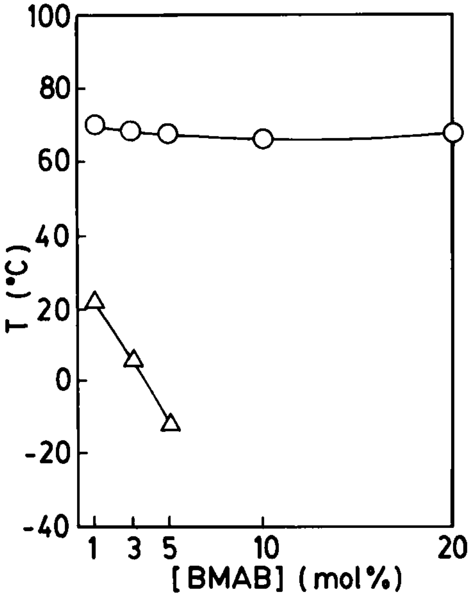

FIGURE 10 Phase transition temperatures (T_C) and glass transition temperatures (T_g) of the BMAB/5OCB/PACB3 mixtures as a function of the concentration of BMAB. (○), T_C; (△), T_g. The compositional ratio of 5OCB to PACB3 was kept at 5OCB/PACB3 = 30/70 mol/mol throughout.

layer. However, the rest of the mixture remains in the LC state and the mixture at this stage is regarded as the photochemical phase transition which has not been completed. Since the cis-form of BMAB has a low value of the extinction coefficient at 360 nm, the incident photons tend to penetrate more deeply as the trans-form of BMAB is consumed and the I phase is finally induced across the mixture films.

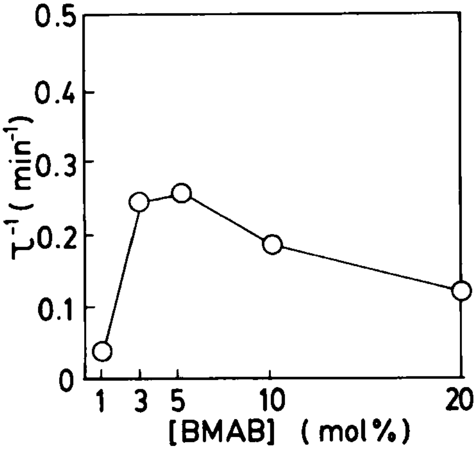

FIGURE 11 Rates of the photochemical phase transition of the BMAB/5OCB/PACB3 mixtures as a function of the concentration of BMAB. The rate of the photochemical phase transition was evaluated by the reciprocal of the response time, τ^{-1}. Photoirradiation was performed at 360 nm at T_{red} = 0.98. The compositional ratio of 5OCB to PACB3 was kept at 5OCB/PACB3 = 30/70 mol/mol throughout.

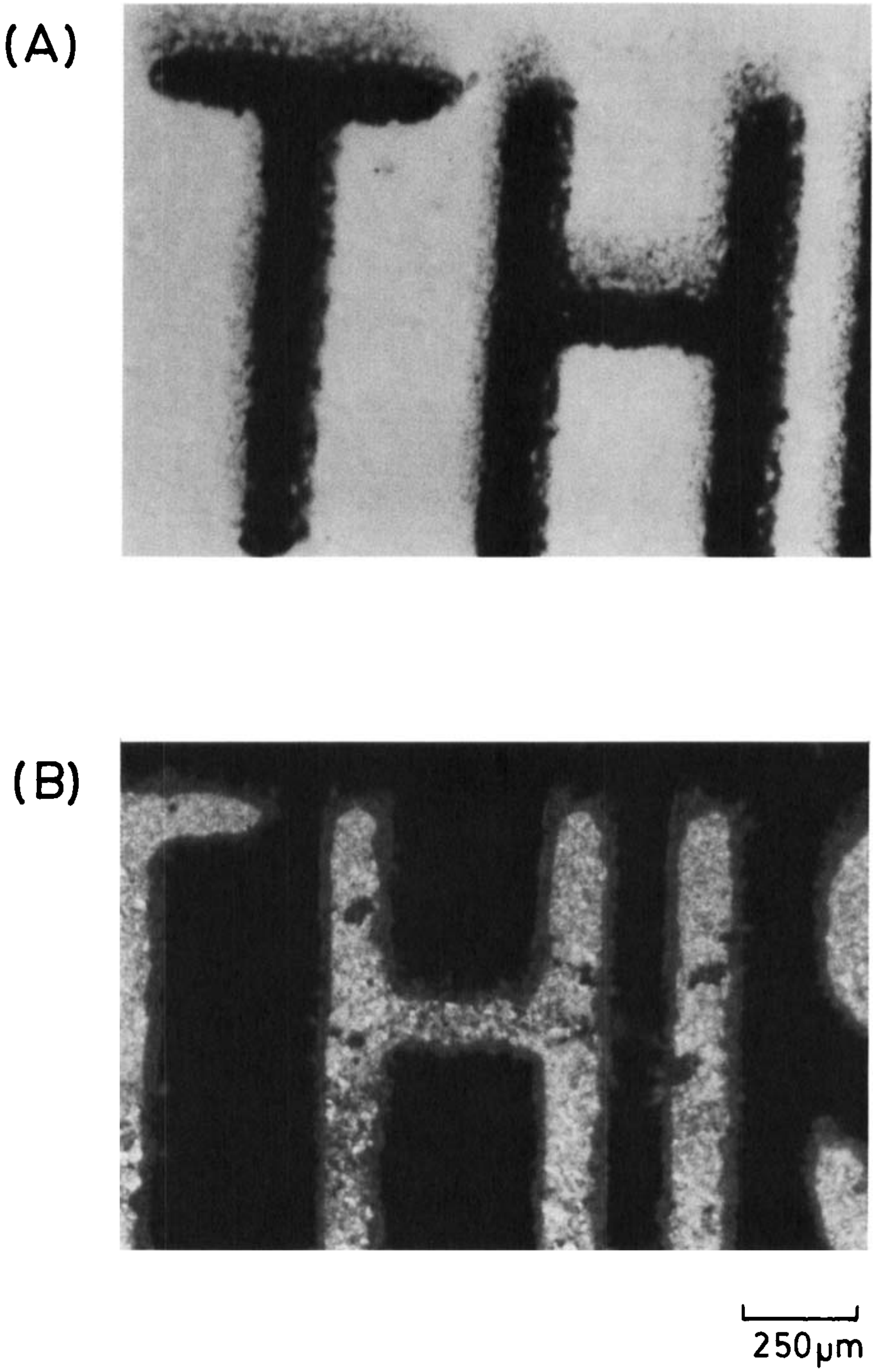

FIGURE 12 Photochemically recorded images in the BMAB/5OCB/PACB3 mixtures. (A, C), photomask; (B, D), stored image observed in the polarizing microscope. The sample was the mixture of 5OCB/PACB3 (30/70 mol/mol) containing 20 mol% of BMAB. Photoirradiation was performed at 360 nm at T_{red} = 0.98. See Color Plate IX.

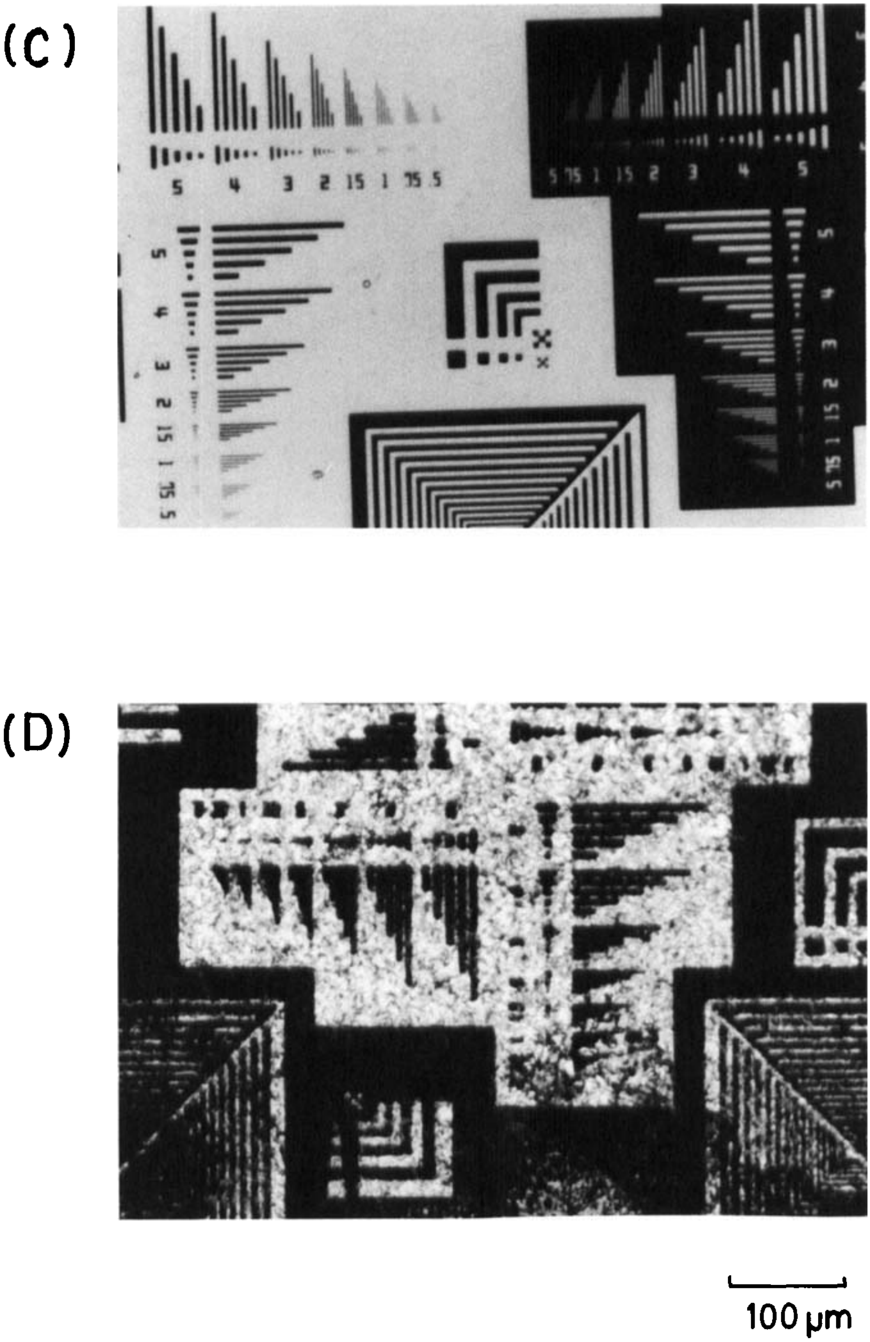

FIGURE 12 (*continued*)

See Color Plate X.

This may be one reason why the rate of the photochemical phase transition decreased when the concentration of BMAB was high.

Photochemical image storage

Image storage experiments by the use of a photomask were conducted on the mixtures of BMAB/5OCB/PACB3. Two types of photomasks were used, being placed directly on the mixtures, and the resulting samples were irradiated at 360 nm. After exposure, the stored image was examined in a polarizing microscope. Two examples of the stored image are shown in Figure 12. In these experiments, the 5OCB/PACB3 mixture (30/70 mol/mol) containing 20 mol% of BMAB was used as an image-storage material. Of note is the stability of the stored image. When the compositional ratio of the LMWLC was high, the stored image easily faded out probably due to the high mobility of the systems, however, at the compositional ratio of 5OCB/PACB3 = 30/70 the stored image could be kept very stable for a long period (at the present stage, more than two months).

CONCLUSION

In the present study, the ternary mixtures of BMAB/LMWLC/PLC were used to improve the response and their photochemical phase transition behaviors were examined for various combinations of LMWLC and PLC components. It was found that in any mixtures composed of BMAB and PLC alone the isothermal phase transition could not be induced photochemically while in the ternary mixtures the photochemical phase transition was induced on photoirradiation. Furthermore, with increasing the mole fraction of the LMWLC component the rate of the photochemical phase transition increased. These results clearly demonstrate that the presence of the LMWLC components significantly improves the response of the systems. Furthermore, the stability of the stored image has been much improved in these ternary systems owing to the presence of the PLC component. Thus, the present study has provided an unequivocal piece of evidence that the LMWLC/PLC mixtures are promising for optical-image recording devices. Although this is true basically, other combinations of LMWLC/PLC are to be examined for the optimum performance as image-storage materials from both the aspects of quick response and stability of the stored information.

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