

# Magnetic Field Effect on Sensitized Photocrosslinking Reaction of Azidomethylated Polystyrene

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## Synopsis

Characteristic curves for the lithographic sensitivity of poly(styrene-co-vinylbenzyl azide) (AZMS) with such sensitizers as 2,4-diisopentylthioxanthone (DITX), 2-chlorothioxanthone (CTX), and Michler's ketone were measured in the absence and in the presence of a magnetic field. By the application of a magnetic field, the gel fraction increases, and efficiency of photocrosslink formation increases by  $\approx 45\%$  (at 0.1 T) for AZMS/DITX and by  $\approx 20\%$  (at 0.5 T) for AZMS/CTX. Sensitization mechanism was investigated by measuring UV, IR, and FT-IR spectra, and AZMS is found to be sensitized effectively by DITX and CTX through the triplet energy transfer, and to produce the triplet nitrene as an intermediate. The magnetic field effect on photocrosslink formation of AZMS/sensitizer systems is interpreted in terms of the radical pair model of intermediate species which is formed by hydrogen abstraction of the nitrene from the sensitizer.

## INTRODUCTION

In recent microlithography, resist polymers are required to show high resolution and high sensitivity to energy radiation such as UV light, X-ray, and electron beam as well as high resistivity to dry etching.<sup>1-3</sup> Under the energy radiation, negative-working resist polymer crosslinks to form a 3-dimensional network leading to insolubilization of the polymer in developing solvents. Hence, elucidation of crosslinking mechanism and/or sensitization mechanism in a thin film of a resist polymer is essential to improve the characteristics of the negative-working resist materials.<sup>3-6</sup>

In photocrosslinking, polymer radicals come together to form radical pairs as intermediates. External magnetic field effect on radical pairs in solutions and in micelles is well established with the aid of laser flash photolysis technique in such aromatic molecules as dibenzoyl peroxide,<sup>7,8</sup> benzophenone,<sup>9-11</sup> 1,3-diphenyl-2-propanone,<sup>12,13</sup> and xanthone.<sup>14</sup> In the solid film of resist polymer, magnetic field effect on photocrosslink formation was first studied by us for bromo- and chloromethylated polystyrene (BCMS) with 2,4-diisopentylthioxanthone (DITX) as a sensitizer<sup>15</sup>; the efficiency of photocrosslink formation for the BCMS/DITX system was found to increase by  $\approx 36\%$  by the application of a magnetic field of 70 mT.

In the present article, to extend the study of the magnetic field effect on the efficiency of photocrosslink formation of resist polymers and to elucidate the sensitization mechanism, one of the azidomethylated polystyrene, i.e., poly(styrene-co-vinylbenzyl azide) (AZMS) has been prepared, and the characteristic curve for the gel fraction, and UV, IR, and FT-IR spectra of some AZMS/sensitizer systems have been measured in the absence and in the presence of an external magnetic field.

## EXPERIMENTAL

### Materials

AZMS (Fig. 1) was prepared by copolymerization of styrene (7.88 g) with vinylbenzyl azide (VBA) (2.12 g) in deaerated benzene solution (50 mL) at 70°C for 30 h, 2,2'-azobisisobutyronitrile (0.35 g) being used as an initiator. AZMS was further purified by reprecipitation from ethanol. Molecular weight and composition of three batches of AZMS (AZMS 1-3) were determined by high speed liquid chromatograph (Toyo Soda HLC-802UR) and elementary analysis, and the results are listed in Table I. VBA was prepared by treating vinylbenzyl chloride (10 g) with sodium azide (7 g) in *N,N*-dimethylformamide (100 mL) for 2 h followed by extraction with and evaporation of diethyl ether. Thermal stability of AZMS was checked by differential scanning calorimetry (DSC) (Rigaku Denki CN-8085EI); AZMS is stable up to 200°C, and above 200°C the azido group begins to decompose.

Thioxanthone (TX) (Kanto G. R. grade), DITX (Toshin Kayacure), 2-chlorothioxanthone (CTX), and benzanthrone (BA) (Tokyo Kasei E.P. grade), and Michler's ketone [4,4'-bis-(dimethylamino)benzophenone] (MK) (Kanto E. P. grade) used as sensitizers were purified by repeated recrystallization from ethanol three times. TX and DITX were further purified by vacuum sublimation. Other sensitizers employed were used as supplied. *p*-Xylene (Kanto G. R. grade) used as a solvent was purified by fractional distillation.

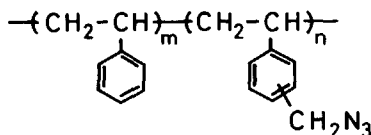


Fig. 1. Chemical structure of poly(styrene-co-vinylbenzyl azide) (AZMS).

TABLE I  
Ratio of Monomeric Unit of VBA (%), Weight Average Molecular Weight ( $\overline{M}_w$ ), and Polydispersity ( $\overline{M}_w/\overline{M}_n$ ) of the AZMS Polymer

	Ratio of VBA	$\overline{M}_w$	$\overline{M}_w/\overline{M}_n$
AZMS1	14.7	12900	2.33
AZMS2	14.7	12200	2.26
AZMS3	14.9	15000	3.12

### Measurements

*p*-Xylene solution of AZMS (15 wt %) and a sensitizer (0.2–2.5 wt %) was spin-coated on a Si wafer and prebaked at 80°C for 10 min. The thin ( $\approx 0.5 \mu\text{m}$ ) film of photosensitive material thus obtained on a Si wafer was placed in an electromagnet (JEOL JES-ME-1X) or in a permanent magnet (Sumitomo Special Metals Coop.), and irradiated with a 450 W medium pressure mercury lamp (Ushio UM452) through a UV31, UV33, or UV35 filter in argon atmosphere. After developing with 2-ethoxyethanol/isopentylacetate (58:42) followed by rinsing with *i*-propanol and post-baking at 80°C for 10 min, residual film thickness  $t$  was measured with an interferometer (Nikon surface finish microscope). Compared with the initial film thickness  $t_0$ , the gel fraction  $G(=t/t_0)$  was calculated as a function of incident energy to obtain the characteristic curve for the sensitivity.

UV absorption spectra of thin (1–3  $\mu\text{m}$ ) films of AZMS/sensitizer systems on quartz plates were measured with a Hitachi 200-20 recording spectrophotometer, and IR spectra, with a Hitachi 295-10 infrared spectrophotometer. FT-IR spectra of AZMS/sensitizer systems coated on a Si wafer or in KBr pellets were measured with a Nicolet 5DXB FTIR spectrophotometer.

## RESULTS AND DISCUSSION

### Magnetic Field Effect on Lithographic Sensitivity

Characteristic curves for the sensitivity of AZMS1/TX, AZMS1/DITX, AZMS1/CTX, and AZMS1/MK, and of AZMS2/11-chloro-6-hydroxy-benzanthrone (CHBA), AZMS2/9,10-dimethylanthracene (DMA), AZMS2/BA, and AZMS2/anthraquinone (AQ) irradiated with a 450 W medium pressure mercury lamp through a UV33 filter [except for the case of AQ(UV31) and of BA(UV35)] are shown in Figure 2. Lithographic sensitivity for the negative-working photoresist<sup>3</sup> is defined to be the incident energy at which the gel formation initiates ( $D_g^i$ ) and/or the gel fraction reaches 0.5 ( $D_g^{0.5}$ ), and the

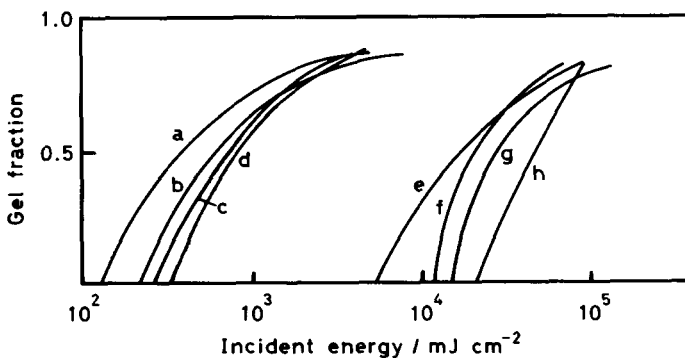


Fig. 2. Characteristic curves for the sensitivity of AZMS1 with (a) TX, (b) DITX, (c) CTX, and (d) MK as sensitizers, and those of AZMS2 with (e) CHBA, (f) DMA, (g) BA, and (h) AQ under the light irradiation with a medium pressure mercury lamp through a UV33 filter for (a)–(f), a UV35 filter for (g), and a UV31 filter for (h).

TABLE II  
Sensitivities  $D_g^i$  and  $D_g^{0.5}$ , and Quantum Efficiency for Photocrosslink Formation,  $\phi$ , for Some AZMS/Sensitizer Systems in the Absence and in the Presence of a Magnetic Field

	Content of sensitizer (wt %)	$H$ (T)	$D_g^i$ ( $J\ cm^{-2}$ )	$D_g^{0.5}$ ( $J\ cm^{-2}$ )	$\phi$
AZMS1/DITX	16.7	0	0.22	0.64	0.088
		0.1	0.15	0.47	0.13
AZMS3/DITX	16.7	0	0.065	0.17	0.26
		0.1	0.045	0.12	0.37
AZMS1/CTX	6.4	0	0.26	0.78	0.11
		0.5	0.21	0.65	0.14
AZMS1/TX	9.0	0	0.13	0.40	0.20
AZMS1/MK	4.0	0	0.33	0.84	0.041
AZMS2/CHBA	5.0	0	5.4	17	0.0033
AZMS2/DMA	10.0	0	11	21	0.0012
AZMS2/BA	5.0	0	15	29	0.0017
AZMS2/AQ	1.5	0	19	37	0.011

results are listed in Table II. The sensitivity of AZMS1 without any sensitizer is over  $150\ J/cm^2$  under the same experimental condition, and practically cannot be gelled. TX and its derivatives and MK are excellent sensitizers for the AZMS polymer. The dependence of sensitivity on different batches of the AZMS polymer was investigated between AZMS1 and AZMS3; AZMS3/DITX (16.7 wt %) shows the higher sensitivity than AZMS1/DITX (16.7 wt %) as listed in Table II.

Magnetic field effect on the characteristic curves of AZMS3/DITX (16.7 wt %) and AZMS1/CTX (6.4 wt %) are shown in Figure 3. By the application of a magnetic field, sensitivity,  $D_g^{0.5}$  increases by  $\approx 45\%$  (from 170 to 120  $mJ/cm^2$ ) for AZMS3/DITX at 0.1 T, and by  $\approx 20\%$  (from 780 to 650  $mJ/cm^2$ ) for AZMS1/CTX at 0.5 T.

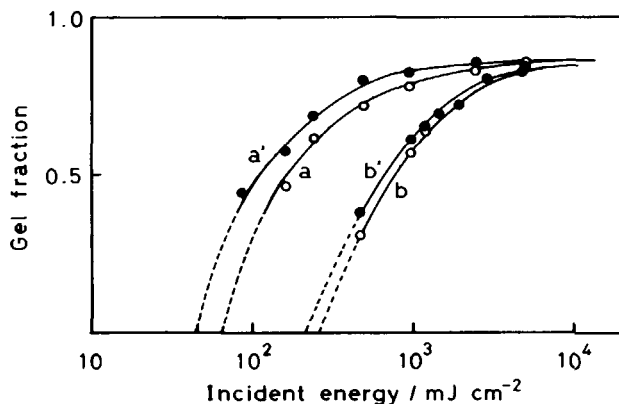


Fig. 3. Magnetic field effect on the characteristic curves for the sensitivity of AZMS3/DITX (curves a and a') and of AZMS1/CTX (curves b and b'). Magnetic field is 0 T for curves a and b, 0.1 T for curve a', and 0.5 T for curve b'.

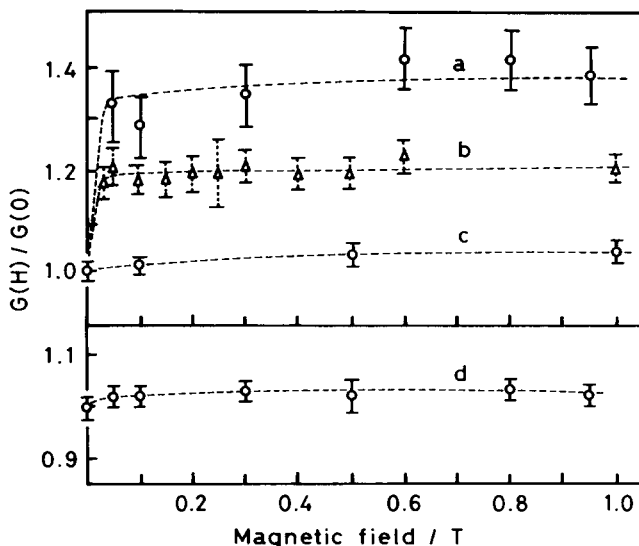


Fig. 4. Magnetic field dependence of the gel fractions of (a) AZMS1/DITX, (b) AZMS3/DITX, (c) AZMS1/CTX, and (d) AZMS1/MK at incident energies of (a) 484, (b) 162, (c) 1210, and (d) 484  $\text{mJ}/\text{cm}^2$ .

Efficiency of photocrosslink formation,  $\phi$ , is evaluated according to the following equation<sup>16</sup>:

$$\phi = rd/2.303E_g D \overline{M}_w$$

where  $E_g$  is the gel point exposure ( $\text{einstein}/\text{cm}^2$ ),  $D$  is the optical density (i.e., absorbance) of the AZMS/sensitizer film of thickness  $r$  and of specific gravity  $d$ , and  $\overline{M}_w$  is the weight average molecular weight of AZMS. With the use of a UV33 (or UV31 for AQ and UV35 for BA) filter,  $D_g^i$  was measured (or estimated). Considering the absorption spectrum of the sample film and the spectral energy distribution of the mercury lamp,  $E_g \times D$  value is evaluated to be  $6.5 \times 10^{-8}$   $\text{einstein}/\text{cm}^2$  for the film of  $1.7 \mu\text{m}$  thickness of AZMS1/DITX as an example, and  $\phi$  is determined to be 0.088 assuming  $d \approx 1$ . The results for other AZMS/sensitizer systems thus calculated are tabulated in Table II. By the application of a magnetic field, photocrosslinking efficiency increases by  $\approx 45\%$  for AZMS1/DITX and AZMS3/DITX at 0.1 T and by  $\approx 20\%$  for AZMS1/CTX at 0.5 T.

The magnetic field dependence of the gel fraction  $G(H)$  of AZMS1/DITX, AZMS3/DITX, and AZMS1/CTX at a fixed incident energy (484, 162, and 1210  $\text{mJ}/\text{cm}^2$ , respectively) are plotted in Figure 4. Probable error for the mean value of  $G(H)$  is evaluated from a set of the experimental  $G(H)$  values, and the corresponding error of  $G(H)/G(0)$  is shown in Figure 4 by a vertical line. The gel fractions of both AZMS1/DITX and of AZMS3/DITX increase as the magnetic field increases up to 0.05 T. The magnetic field effect on the gel fractions saturates at  $\approx 0.05$  T to be  $G(H)/G(0) = 1.35$  and 1.2, respectively.

Magnetic field effect on the gel fraction of AZMS was also measured by the use of other sensitizers such as TX, MK, BA, CHBA, DMA, and AQ. Among them, the magnetic field effect was confirmed only with AZMS1/MK (10 wt %); the magnetic field dependence of  $G(H)/G(0)$  at incident energy of 484 mJ/cm<sup>2</sup> shows a slight increase ( $\approx 3\%$ ) by the application of a magnetic field between 0.05 and 0.95 T as shown in Figure 4. In the other AZMS/sensitizer

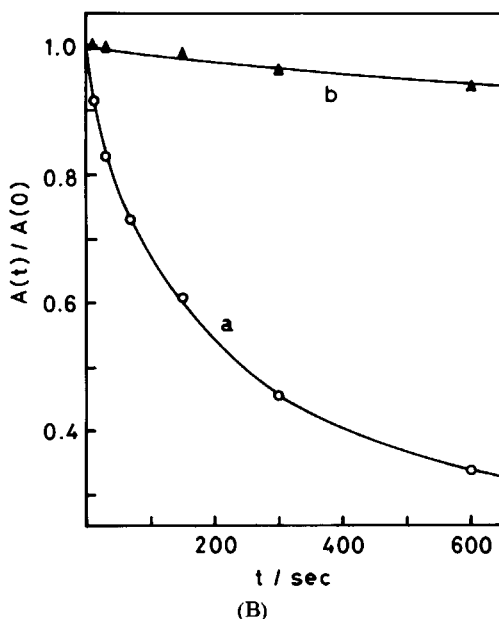
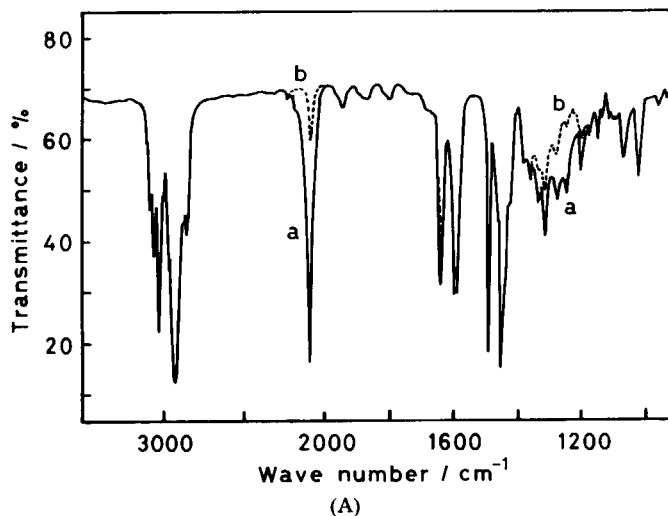


Fig. 5(A). IR spectra of the AZMS1/DITX (16.7 wt %) film on a NaCl plate irradiated with a medium pressure mercury lamp through a UV33 filter for (a) 0 and (b) 80 min in argon atmosphere. (B) Absorbance change  $A(t)/A(t=0)$  of (a) 2099 cm<sup>-1</sup> band and (b) 1645 cm<sup>-1</sup> band of the AZMS1/DITX (16.7 wt %) film on a NaCl plate under the light irradiation in argon atmosphere.

systems, film surface became rough after developing the film, and the gel fraction could not be measured so precisely as to draw any conclusion about the magnetic field effect.

### Sensitization Mechanism of AZMS / Sensitizer

Sensitization mechanism of the AZMS/sensitizer systems was investigated by measuring UV, IR, and FT-IR spectra. Figure 5(A) shows IR spectra of AZMS1/DITX film on a NaCl plate under the light irradiation through a UV33 filter in argon atmosphere. The 2099 and 1645  $\text{cm}^{-1}$  bands are assigned, respectively, to antisymmetric stretching vibration of the azido group in AZMS<sup>17-19</sup> and to the C=O stretching vibration of DITX.<sup>20-22</sup> In Figure 5(B), decreases in intensity of the 2099 and 1645  $\text{cm}^{-1}$  bands were plotted

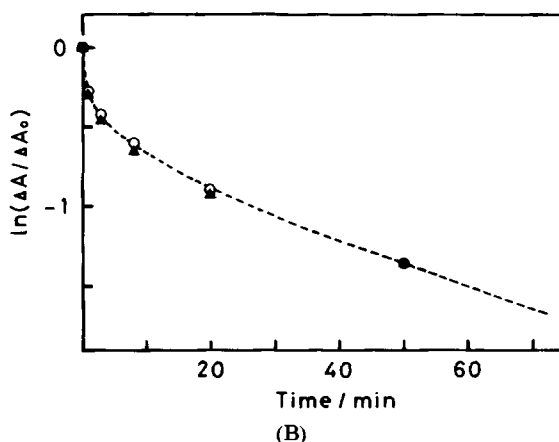
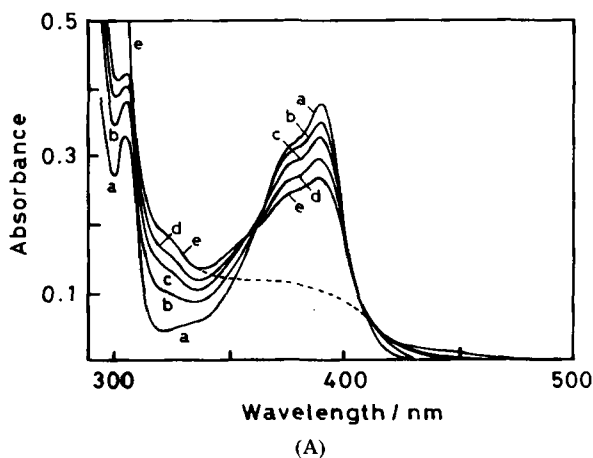


Fig. 6(A). UV absorption spectra of the AZMS3/DITX film (of  $\approx 1.2 \mu\text{m}$  thickness) on a quartz plate irradiated with a medium pressure mercury lamp through a UV33 filter for (a) 0, (b) 1, (c) 8, (d) 50, and (e) 200 min. UV absorption spectrum of AZMS3/DITX after developing the film is shown by a broken line. (B) Dependence of absorbance,  $\Delta A (= A(t) - A(t = 200 \text{ min}))$ , at 390 nm on irradiation time in the absence (○) and in the presence (▲) of a magnetic field of 0.1 T.

against irradiation time; photochemical reaction (such as hydrogen abstraction) of DITX<sup>22</sup> is negligibly small, whereas the azido group in AZMS is rapidly decomposed. Because only the DITX molecule is effectively excited by the light longer than 330 nm, and because excited DITX effectively intersystemcrosses to the triplet state,<sup>21,23-26</sup> this result clearly suggests that the azido group in AZMS is decomposed effectively through the triplet energy transfer from DITX to AZMS.

In UV absorption spectra of AZMS3/DITX and AZMS1/CTX shown in Figures 6 and 7, photobleaching of the 390 nm bands of DITX and CTX was observed as in BCMS/DITX,<sup>15</sup> where photocrosslinking of BCMS proceeds through hydrogen abstraction of DITX in the triplet state.<sup>15,22</sup> Although the bleaching in Figures 6 and 7 is much less compared to the one in BCMS/DITX,

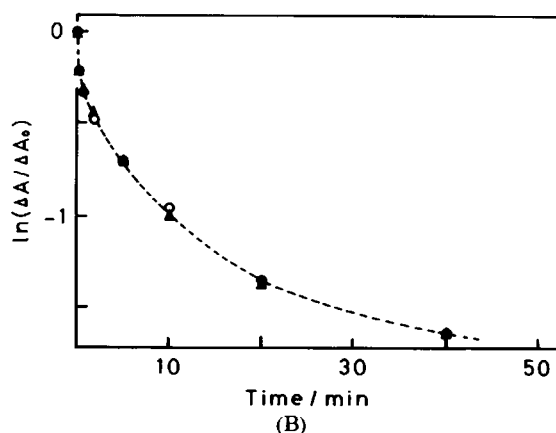
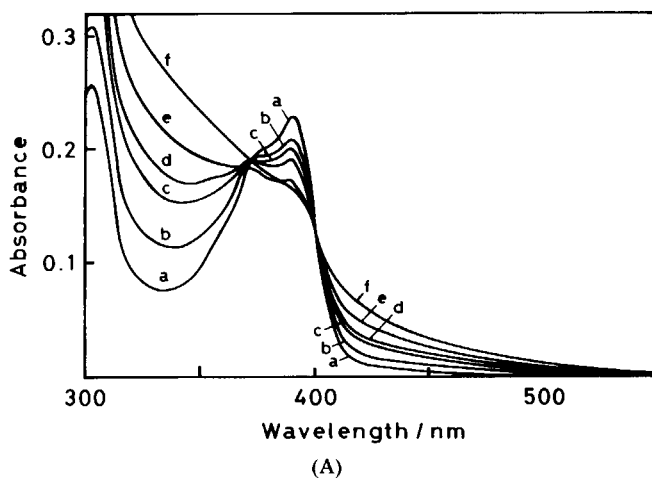


Fig. 7(A). UV absorption spectra of the AZMS1/CTX film (of  $\approx 1.1 \mu\text{m}$  thickness) on a quartz plate irradiated with a medium pressure mercury lamp through a UV33 filter for (a) 0, (b) 0.5, (c) 4, (d) 10, (e) 40, and (f) 160 min. (B) Dependence of absorbance,  $\Delta A (= A(t) - A(t = 160 \text{ min}))$ , at 391 nm on irradiation time in the absence (○) and in the presence (▲) of a magnetic field of 0.089 T.

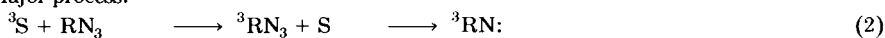


this result suggests that as a minor process the triplet sensitizer abstracts a hydrogen atom from AZMS.

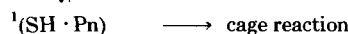
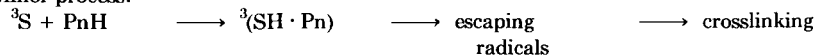
Based on the above results, photocrosslinking mechanism of the AZMS/sensitizer systems is summarized in Scheme 1 where both PnH and RN<sub>3</sub> stand for the AZMS polymer, and S and S'H, the sensitizer. Photocrosslinking of AZMS proceeds through azo coupling, insertion into C—H bond of nitrene, and recombination of free radicals which are generated through hydrogen abstraction by nitrene<sup>27</sup>:



Major process:



Minor process:



Scheme 1. Photocrosslinking Mechanism of AZMS/Sensitizer Systems

### Magnetic Field Effect on Photocrosslinking Efficiency

In order to investigate whether the light absorption is influenced by a magnetic field, the photobleaching of the 390 nm band in UV absorption spectra of the AZMS3/DITX and AZMS1/CTX films were measured under the light irradiation in the absence and in the presence of a magnetic field; the results are shown in Figures 6(B) and 7(B). The rates of the photobleaching of DITX and CTX are not affected by the application of a magnetic field of 0.1 and 0.089 T, respectively. Considering that the rate by which triplet sensitizer abstracts a hydrogen atom from polymer is not affected by the application of a magnetic field as is reported in BCMS/DITX,<sup>15</sup> the results in Figures 6(B) and 7(B) indicate that the light absorptivity of the sensitizer and the energy transfer rate from the triplet sensitizer to AZMS are not affected by the application of a magnetic field. Consequently, the magnetic field effect on the efficiency of photocrosslink formation is qualitatively understood according to radical pair model in Scheme 1. Triplet nitrene (<sup>3</sup>RN:) originating from AZMS in the triplet state (<sup>3</sup>RN<sub>3</sub>) abstracts a hydrogen atom from the AZMS polymer (PnH) or from the sensitizer (S'H) to form a triplet radical pair, <sup>3</sup>(RNH · Pn) or <sup>3</sup>(RNH · S'). The triplet radical pair can convert to the singlet radical pair which subsequently undergoes recombination to fulfill C—H

bond insertion or cage reaction, the latter of which does not induce crosslinking. The triplet radical pair can generate escaping free radicals, which eventually induce crosslinking of the polymer. By the application of a magnetic field, conversion rate between the triplet and the singlet radical pairs decreases, and the formation of the escaping free radicals from the triplet radical pairs increases, resulting in the increase of the efficiency of photocrosslink formation. From the fact that the magnetic field effect on the gel fraction of AZMS/DITX saturates at a low magnetic field ( $\approx 0.05$  T) (Fig. 4), it is suggested that hyperfine-induced triplet-singlet mixing of the radical pair is affected by the magnetic field.

The triplet nitrene intersystem crosses to the singlet nitrene. If the intersystemcrossing (ISC) rate between the two manifolds is decreased by the application of a magnetic field as in the case of radical pairs, chemical processes originating from the triplet nitrene proceed more efficiently. Because the triplet nitrene is much more reactive than the singlet nitrene, and crosslinking occurs more easily through the triplet nitrene than through the singlet nitrene, the net magnetic field effect on the ISC rate of the nitrene is expected to result in the increase of the efficiency of photocrosslink formation. Further study of the magnetic field effect on the ISC rate is in progress.

As the minor process, triplet sensitizer ( $^3S$ ) abstracts a hydrogen atom from the AZMS polymer (PnH) to form a triplet radical pair between the ketyl radical ( $\cdot SH$ ) and the polymer radical (Pn $\cdot$ ) (Scheme 1). The magnetic field effect on the triplet radical pair between the ketyl and the polymer radicals has been reported for BCMS/DITX in the previous paper<sup>15</sup>; the efficiency of photocrosslink formation increases by the application of a magnetic field.

## CONCLUSION

Lithographic sensitivity and efficiency for photocrosslink formation of AZMS/DITX, AZMS/CTX, and AZMS/MK are found to be increased by the application of a magnetic field of  $\approx 0.1$  T. AZMS is sensitized effectively by DITX and CTX through the triplet energy transfer, and generates the triplet nitrene. Photocrosslinking proceeds through the triplet radical pair which is formed by hydrogen abstraction of the nitrene from the sensitizer and the AZMS polymer. External magnetic field affects the ISC rate between the triplet and the singlet radical pairs, and increases the efficiency of photocrosslink formation of AZMS/DITX and AZMS/CTX.

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