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Preparation and Photo- and Thermal-Curing Properties of Copolymers Applied in Negative-Type Photoresists

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A series of monomers was polymerized and used as polymer binders in negative-type photoresists. The thermal properties of the polymer binders and the mechanical properties of their patterns were then studied. We used two four-component polymer binders: one consisting of methacrylic acid, styrene, benzyl methacrylate, and glycidal methacrylate, and another consisting of methacrylic acid, styrene, isobornyl methacrylate, and phenylmaleimide. Diallyl monoglycidyl isocyanurate (DA-MGIC) or monoallyl diglycidyl isocyanurate (MA-DGIC) was then reacted with the formed polymer binder to create a novel polymer binder with photo- and thermal-curing properties. The results showed that the thermal decomposition temperature (T_d) of the polymer binders increased when the DA-MGIC or MA-DGIC monomer was used in the binder; this was presumably due to the photo-curing, thermal-curing, and inter-penetration network characteristics of the polymer binders. Elastic recovery and compression of the patterns of the photoresist were measured using a nanoindenter. The results showed that the patterns exhibited excellent mechanical properties. The patterns were observed with a scanning electron microscope. The taper angle of the patterns became vertical due to the increased intensity of the inter-penetration network, which was revealed by the excellent inhibition of the pattern.

Keywords Binder; elastic recovery; photo-curing; photoresist; thermal properties

Introduction

The dramatic growth of thin-film transistor liquid crystal displays (TFT-LCD) has been supported by a strong demand for flat-panel displays in applications such as TVs, monitors, and notebooks. In accordance with the demands for new-generation TFT-LCD technology, improvements in the performance of a color filter and a liquid crystal layer are concerned with the contrast, brightness, color saturation, uniformity, response time, and hardness [1,2]. In the conventional process, spacer beads (plastic or inorganic fine particles) agglomerate and distort the contrast ratio and lead to light leakage. Therefore, a photoresist (a column fixed by photo lithography) plays an important role in controlling the thickness and uniformity of the color filter and the TFT array substrate.

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Typically, a negative-type photoresist consists of a polymer binder, a polyfunctional monomer (or cross-linker), a photo initiator, a solvent, and additives [2–4]. The mechanism how a negative-type photoresist works is that when it is exposed to photo irradiation, the exposed area cannot subsequently be removed by an alkaline developer. Thus, after development, the desired pattern is formed on the substrate. The main function of the photoresist is to separate the TFT array and color filter substrate so as to lead liquid crystal drops to fill defined gaps (approximately 3–5 μm). Therefore, the photoresist is necessary for excellent mechanical properties, i.e., elastic recovery >70% and surface hardness >3H. The damage being caused by external force is avoided. Properties of acrylic copolymers such as their photosensitivity [5], thermal stability [6–12], pattern resolution [9,13], glass transition temperature [14], nanosilica-modified characteristics [8] and physical characteristics [15] have been investigated in negative-type photoresists. Properties and models of epoxy group–carboxylic acid reaction [16–18] have also been investigated. However, the preparation and photo- and thermal-curing properties of copolymers applied in negative-type photoresists have not been studied sufficiently. In this study, we examined these characteristics of the novel polymer binders. We also examined their influence on the mechanical properties and pattern profiles of the photoresists.

Experiment

Materials

Materials used in this experiment include methacrylic acid (MAA), styrene (STY), benzyl methacrylate (BzMA), glycidal methacrylate (GMA), isobornyl methacrylate (IBMA), phenylmaleimide (PMI), dipentaerythritol hexa-acrylate (DPHA), 2,2′-azobisisobutyronitrile (AIBN), 1-dodecanethiol (DT), potassium hydroxide (KOH), tetrahydrofuran (THF), n-hexane, hydroquinone (HQ), and triphenylphosphine (TPP). All of them were reagent-grade and purchased from Aldrich. Propylene glycol monomethyl ether acetate (PGMEA, DOW Chemical), 2-benzyl-2-dimethylamino-1-(4-morpholinophenyl)butanone (I-369, Ciba-Geigy), isopropyl thioxanthone (ITX, Ciba-Geigy), R-08 (Dainippon Ink and Chemicals), diallyl monoglycidyl isocyanurate (DA-MGIC, SHIKOKU Chemical), and monoallyl diglycidyl isocyanurate (MA-DGIC, SHIKOKU Chemical) were used as received.

Preparation of Binders

Polymer binders were synthesized by free-radical polymerization of four kinds of monomer. In a four-necked flask, the solvent (PGMEA, 50 wt%) was preheated and stirred up to 75°C under a nitrogenous atmosphere. The four kinds of monomer (MAA:STY:BzMA:GMA = 32:20:20:28 and MAA:STY:IBMA:PMI = 35:22:22:22, ca. 27 wt%) were premixed with the solvent (PGMEA, 20 wt%), an initiator (AIBN, 2 wt%), and a chain transfer agent (DT, 1 wt%) at room temperature for 30 min., and then, the monomer solutions were slowly poured into the flask by microinjection for 4 h at 75°C. Finally, the solutions were kept aside for 2 h at 75°C after microinjection. The polymer binders were cooled down to room temperature. Subsequently, the formed binders were premixed with an inhibitor (HQ, 0.1 wt%), a catalyst (TPP, 0.2 wt%), and an isocyanurate monomer (DA-MGIC or MA-DGIC, ca. 4–12 wt%) at room temperature for 30 min., and then, the solutions were poured into the flask for 8 h at 80°C. The compositions of the polymer binders are listed in Table 1.

Table 1. Compositions of polymer binders

Group	Binders	MAA of binder Molar ratio	DA-MGIC	MA-DGIC
I	Binder A	1	0	0
	Binder A-MGIC(0.2)		0.2	
	Binder A-MGIC(0.4)		0.4	
	Binder A-MGIC(0.6)		0.6	
II	Binder A	1	0	0
	Binder A-DGIC(0.2)			0.2
	Binder A-DGIC(0.4)			0.4
	Binder A-DGIC(0.6)			0.6
III	Binder B	1	0	0
	Binder B-MGIC(0.2)		0.2	
	Binder B-MGIC(0.4)		0.4	
	Binder B-MGIC(0.6)		0.6	
IV	Binder B	1	0	0
	Binder B-DGIC(0.2)			0.2
	Binder B-DGIC(0.4)			0.4
	Binder B-DGIC(0.6)			0.6

To prepare samples for characterization of the thermal properties and chemical structure, the formed polymer binders were diluted with THF and then dropped into n-hexane to induce polymer precipitation. This was repeated twice. Finally, the polymer binders were separated by filtration and then dried in oven for 24 h at 35°C.

Preparation of Photoresists

Negative-type photoresists were prepared by mixing the formed polymer binders (ca. 15 wt%), a polyfunctional monomer (DPHA, 10 wt%), photoinitiators (I-369, 6 wt%, and ITX, 2 wt%), solvent (PGMEA, 67 wt%) and a surfactant (R-08, 500 ppm) at room temperature for 6 h, and then, the photoresists were filtered through a 2-μm filter.

Characterization of Polymer Binders and Photoresists

The molecular weight of the binder was measured by gel permeation chromatography (GPC, Waters, Pump 1515 and RI detector 2414). The acid value of the binder was obtained by a titration meter (BRAND, Burette Digital 111) using 0.1 N KOH solution. The viscosity of the binder was measured by a viscosity meter (Brookfield DV-E, Spindle No. S18). The glass transition temperature (T_g) of the binder was obtained using a differential scanning calorimeter (DSC, METTLER TOLEDO, DSC 823e) when the temperature was raised from 25°C to 250°C at a constant rate of 10°C/min under nitrogen flow. The decomposition temperature (T_d) of the binder was obtained using a thermal gravimetric analyzer (TGA, Perkin-Elmer, TAC 7/DX) when the temperature was raised from 25°C to 600°C at a constant rate of 10°C/min under nitrogen flow. ^{1}H-NMR spectrum was recorded using nuclear magnetic resonance (^{1}H-NMR, Varian, GEMINI 2000) in THF-d_8 solvent.

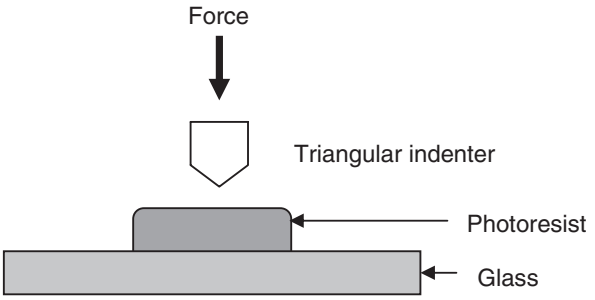


Figure 1. DUMH indenter.

The elastic recovery of the cylindrical pattern of the photoresist (diameter = 20–25 μm, height = 4–5 μm) was measured using a dynamic ultra micro hardness and micro compression tester (DUMH indenter, SHIMADZU DUH-W201S; Fig. 1) at a force of 10 mN, loading speed of 0.36 mN/s, and holding time of 5 s. The elastic recovery was calculated according to the following expression: elastic recovery (%) = $(D_1/D_2) \times 100\%$ (Fig. 2). The compression of the cylindrical pattern (diameter = 20–25 μm, height = 4–5 μm) was measured using a DUMH indenter, wherein the loading speed was 17.65 mN/s, and the holding time was 5 s. The compression of the pattern was calculated according to the inflection point of the curve (Fig. 3). The surface hardness of the films was assessed using an industrial pencil hardness test (MITSUBISHI Uni pencil). The thickness of the pattern was measured using a surface profiler (Taylor Hobson Form Talysurf series 2). The image of the pattern was observed by using a scanning electron microscope (SEM, Hitachi, S-4200).

The exposure-development procedure was as follows: the photoresist was coated on a super-twisted nematic (STN) glass (75 mm × 75 mm) using a spin coater (500 rpm for 10 s) and then pre-baked at 90°C for 10 min in an oven. Subsequently, it was exposed to UV radiation (150 mJ/cm^2) through a contact mask (100-μm gap), and then, it was developed in 0.05 wt% KOH aqueous solution at 25°C for 2 min. Finally, the it was hard-baked at 230°C for 30 min in an oven.

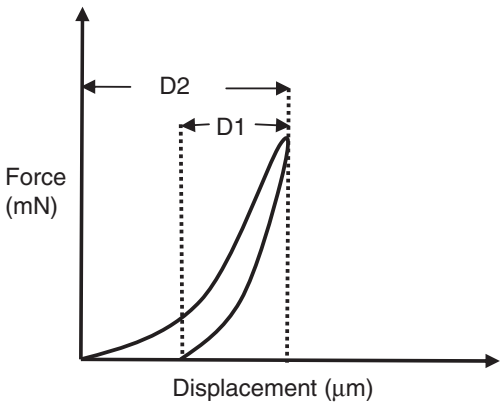


Figure 2. Elastic recovery graph.

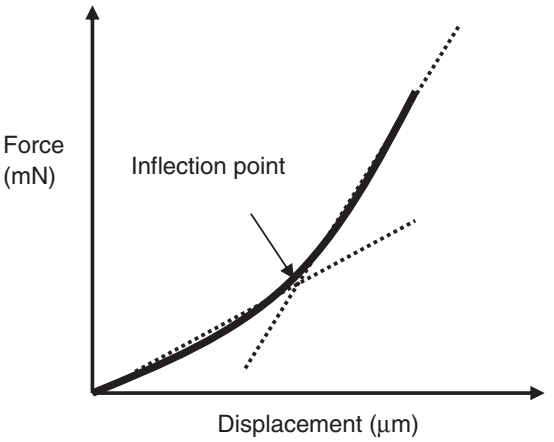


Figure 3. Compression graph.

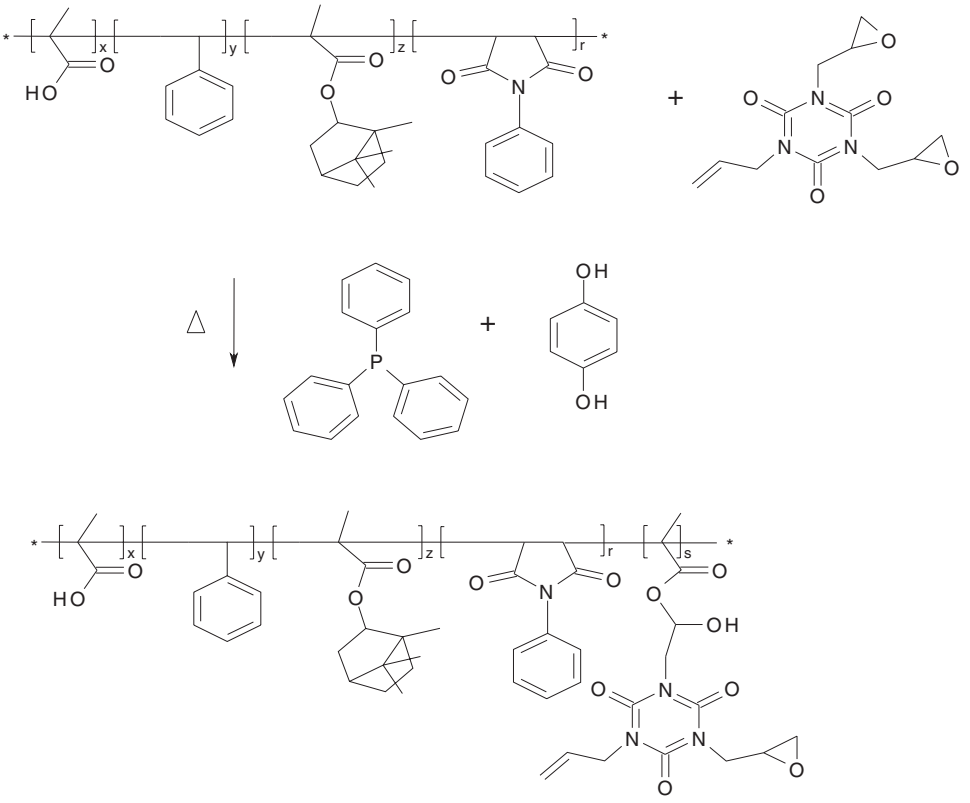
Results and Discussion

Typically, a negative-type photoresist consists of a polymer binder, a polyfunctional monomer (or cross-linker), a photo initiator, a solvent, and additives. The structure and characteristics of polymer binders, and the intensity of the inter-penetration network influence the mechanical properties of the photoresist. In this study, the characteristics of thepolymer binders are discussed. In addition, their influence on the mechanical properties and pattern profiles of the photoresist is discussed.

An initiator (AIBN), a chain transfer agent (DT), and four monomers (MAA:STY:BzMA:GMA = 32:20:20:28) were used to prepare polymer binder A; four other monomers (MAA:STY:IBMA:PMI = 35:22:22:22) were used to prepare polymer binder B. The isocyanurate monomer (DA-MGIC or MA-DGIC) was then reacted with the formed polymer binders to create a novel polymer binder with photo- (unsaturated double bond) and thermal-curing (epoxy group) properties. The structure and composition of the polymer binders are shown in Scheme 1 and Table 1, respectively.

Characterization of Polymer Binders

Measurement of Acid Value. The carboxylic acid of the MAA segment controls the acid value (AV) of a polymer binder. If the condensation reaction between the carboxylic acid of the polymer binder and the epoxy group of the isocyanurate monomer is successful, the AV of the polymer binder decreases compared with the original binder. In general, the AV of the polymer binders decreased when the molar ratio of the isocyanurate to MAA monomer increased from 0% to 60% (Table 2). For example, the AV of polymer binder A was 105.32 mg KOH per gram of solid binder, whereas the AV of another binder (binder A-MGIC(0.6)) was 82.79 mg KOH per gram of solid binder. Similarly, the results showed the same trend for binder A-DGIC, binder B-MGIC, and binder B-DGIC (Fig. 4). We also found that the AV of polymer binder B (133.63 mg KOH per gram of solid binder) was more than that of polymer binder A due to the higher MAA ratio of polymer binder B. We concluded that the epoxy group of the DA-MGIC or MA-DGIC monomer successfully reacted with the carboxylic acid of the MAA segment of the polymer binder in the presence of a catalyst (TPP) and an inhibitor (HQ) in this study.



Scheme 1. Preparation of polymer binder B with MA-DGIC monomer.

Table 2. Properties of polymer binders

Group	Binders	Mw	PDI (polydispersity index)	Viscosity (cP)	Acid value (mg KOH/g of binder)	T_g (°C)	T_d – 10% (°C)
I	Binder A	10,576	2.57	68.3	105.32	ND	319.1
	Binder A-MGIC(0.2)	14,717	3.29	69.6	95.60		343.9
	Binder A-MGIC(0.4)	14,865	3.25	68.9	84.50		348.1
	Binder A-MGIC(0.6)	14,155	3.21	68.5	82.79		344.8
II	Binder A	10,576	2.57	68.3	105.3	ND	319.1
	Binder A-DGIC(0.2)	14,522	3.16	71.1	94.39		344.3
	Binder A-DGIC(0.4)	14,683	3.21	72.6	83.97		340.5
	Binder A-DGIC(0.6)	14,331	3.14	70.4	78.04		345.4
III	Binder B	10,077	2.45	50.5	133.63	171.0	246.4
	Binder B-MGIC(0.2)	10,207	2.44	50.0	118.45	172.9	257.3
	Binder B-MGIC(0.4)	10,763	2.70	51.7	106.48	179.9	262.7
	Binder B-MGIC(0.6)	10,605	2.60	52.4	96.43	173.6	260.6
IV	Binder B	10,077	2.45	50.5	133.63	171.0	246.4
	Binder B-DGIC(0.2)	11,085	2.77	58.3	113.13	168.1	253.4
	Binder B-DGIC(0.4)	11,572	3.00	69.0	99.02	170.5	262.1
	Binder B-DGIC(0.6)	12,116	2.93	73.0	85.84	179.0	272.2

Note. ND: Not detected.

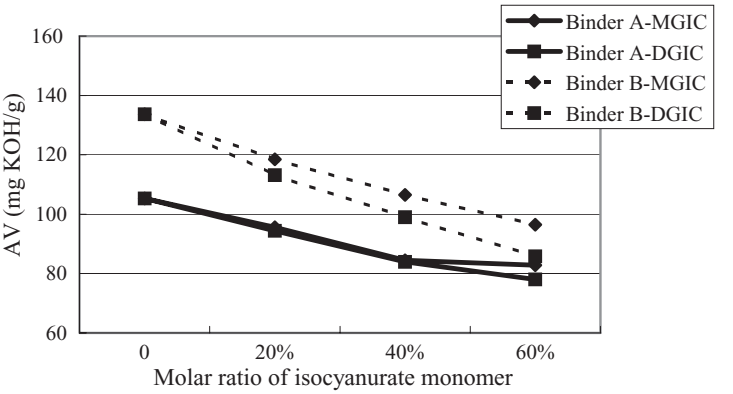


Figure 4. Effect of the molar ratio of isocyanurate to MAA monomer of binders on acid value.

Glass Transition Temperature. DSC was used to measure the glass transition temperature (T_g) of the polymer binders. The experiments were divided into four groups; these experiments were performed to investigate the dependence of the polymer binders' characteristics on the ratio of isocyanurate monomers, which is shown in Table 2. The results for Groups I and II (binder A series) showed that the T_g of the polymer binders could not be assessed for polymer binder A series (poly-MAA–STY–BzMA–GMA) synthesized by free-radical polymerization.

In Group III experiments (binder B series), the molar ratio of the DA-MGIC monomer was varied. The experiment results showed that the T_g of the polymer binders was between 171.0°C and 179.9°C when the molar ratio of DA-MGIC increased from 0% to 60% (Table 2). Therefore, the T_g of the polymer binders was independent of the DA-MGIC concentration for the range of values tested in this study. The Group IV results, where the MA-DGIC ratio was varied, also showed a similar trend (Fig. 5).

Thermal Decomposition Temperature. TGA was utilized to measure the thermal decomposition temperature (T_d) of the polymer binders. Figure 6 shows the TGA thermograms of Group I and II experiments, where the ratio of isocyanurate to MAA monomer was varied. The TGA curve shifted toward higher temperatures when the DA-MGIC or MA-DGIC monomer was reacted with polymer binder A. The temperature corresponding to a 10 wt% loss (T_d – 10%) was identified as the thermal decomposition temperature for easy comparison. The T_d – 10% value for polymer binder A was 319.1°C; the values for the other binders were greater than 340°C (Table 2). In addition, the T_d – 10% value was maximal when the DA-MGIC or MA-DGIC monomer was used in the binder; this was presumably due to the photo-curing, thermal-curing, and inter-penetration network characteristics of the isocyanurate structures. The Group III and IV results showed a similar trend for polymer binder B.

Characterization of Chemical Structure. We examined the ^{1}H-NMR spectrum of polymer binder A-MGIC(0.4) to characterize its chemical structure (Fig. 7). The peaks at 1.73 and 3.58 ppm were due to THF protons. The peaks between 7.0 and 7.2 ppm were due to the aromatic protons (STY and BzMA segment) of the polymer binder. The new peak that

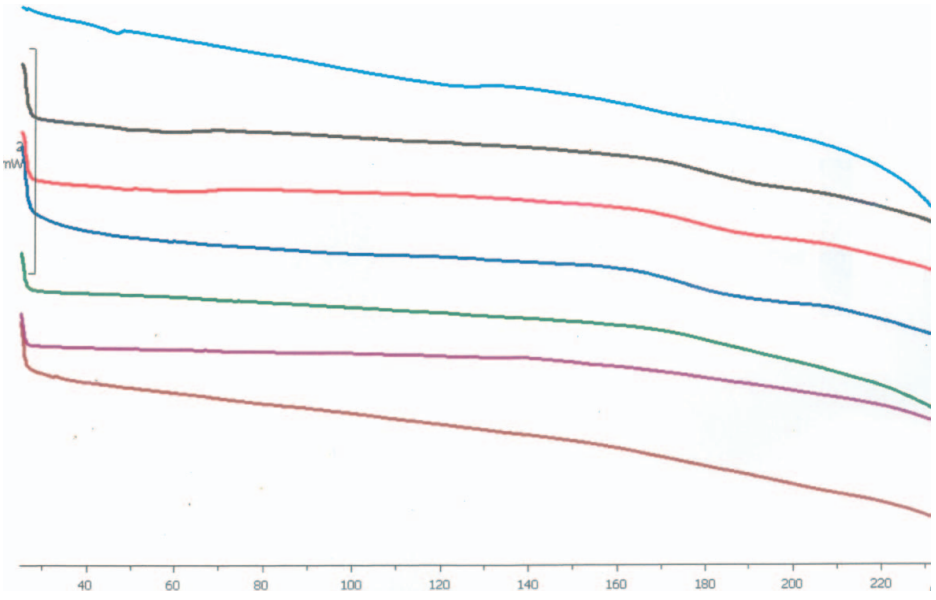


Figure 5. DSC thermograms of polymer binder B containing various molar ratios of isocyanurate monomer.

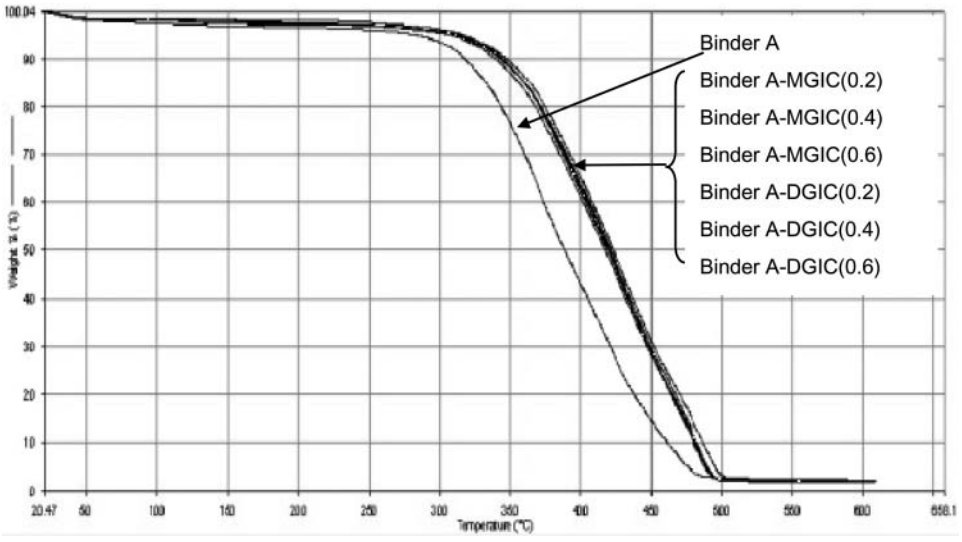


Figure 6. TGA thermograms of polymer binder A containing various molar ratios of isocyanurate monomer.

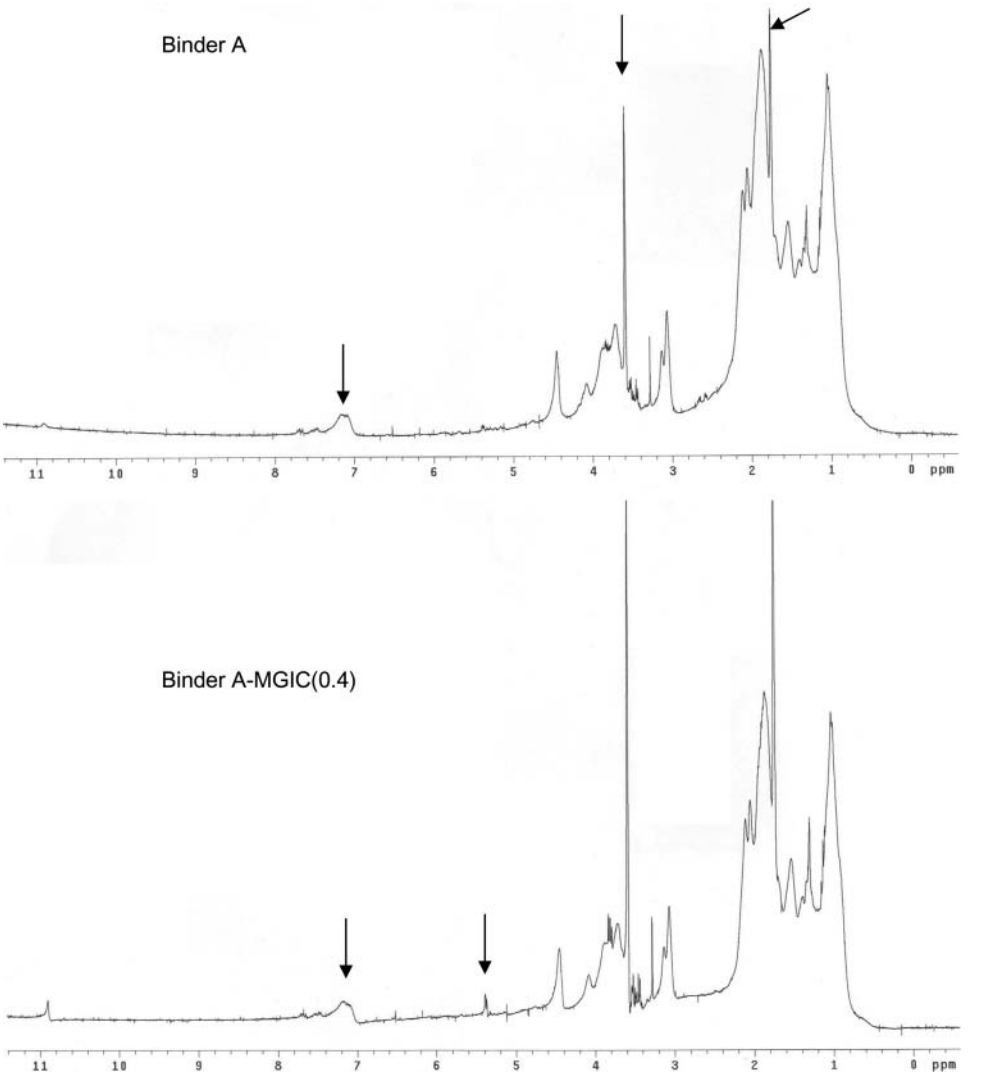


Figure 7. ^{1}H-NMR spectra of binder A-MGIC(0.4).

appeared around 5.2 ppm was due to the olefinic protons of the allyl end groups of the novel polymer binder [19,20]. The spectrum suggests that the epoxy group of the DA-MGIC monomer and the carboxylic acid of the polymer binder were successfully linked together to create a photo-curable polymer binder through condensation reaction.

Characterization of Patterns

Effect of Polymer Binder on Mechanical Behavior. Photoresists were prepared by adding suitable amounts of formed polymer binders and cross-linkers (DPHA, a polyfunctional monomer); the photoresist coating was then applied on a glass substrate using a

Table 3. Mechanical properties of photoresist

Group	Run no.	Binders	Elastic recovery (%)	Compression (mN)	Hardness (H)
I	A0	Binder A	72.0	200	3H
	A1	Binder A-MGIC(0.2)	80.4	300	4H
	A2	Binder A-MGIC(0.4)	79.6	350	4H
	A3	Binder A-MGIC(0.6)	80.2	300	4H
II	A0	Binder A	72.0	200	3H
	A4	Binder A-DGIC(0.2)	78.0	375	4H
	A5	Binder A-DGIC(0.4)	78.8	375	4H
	A6	Binder A-DGIC(0.6)	79.8	350	4H
III	B0	Binder B	70.2	175	3H
	B1	Binder B-MGIC(0.2)	75.5	175	3H
	B2	Binder B-MGIC(0.4)	72.6	175	4H
	B3	Binder B-MGIC(0.6)	73.1	175	4H
IV	B0	Binder B	70.2	175	3H
	B4	Binder B-DGIC(0.2)	76.3	175	3H
	B5	Binder B-DGIC(0.4)	73.7	175	4H
	B6	Binder B-DGIC(0.6)	74.7	175	4H

lithographic process. The thickness of the film was controlled between 4 μm and 5 μm, and the mechanical properties of the film were then measured. Their effects on the mechanical properties of the photoresist are shown in Table 3. In Group I and II experiments, moderate mechanical properties (elastic recovery = 72.0%) for the patterns were obtained by adding 15 wt% of polymer binder A to the photoresist. The elastic recovery was dramatically increased and maintained between 78.0% and 80.4% when the DA-MGIC or MA-DGIC monomer was added to polymer binder A (Fig. 8). In Group III

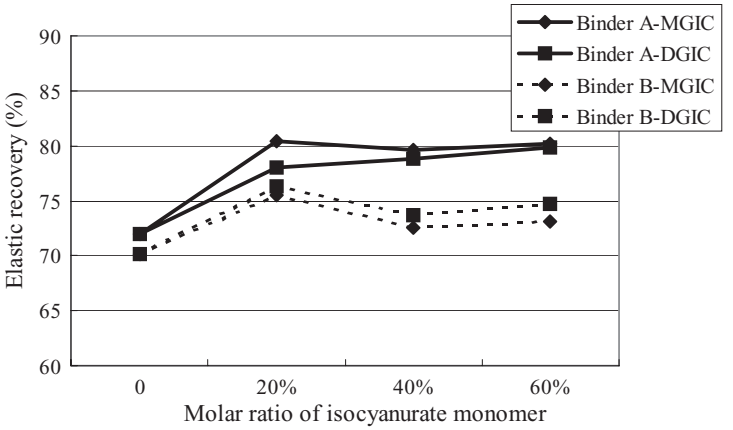


Figure 8. Effect of the molar ratio of isocyanurate to MAA monomer of binders on elastic recovery.

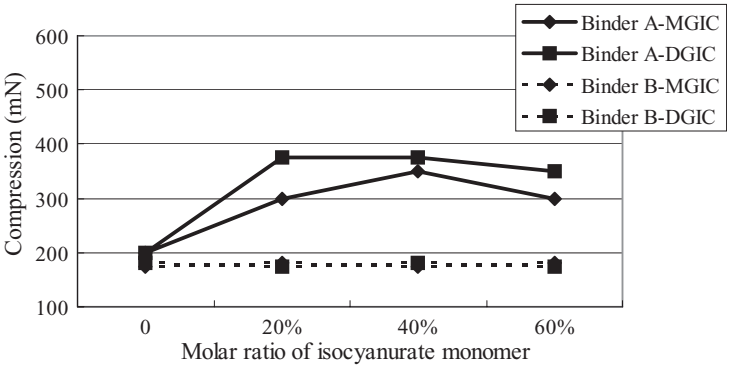


Figure 9. Effect of the molar ratio of isocyanurate to MAA monomer of binders on compression.

and IV experiments, moderate mechanical properties (elastic recovery = 70.2%) for the patterns were obtained by adding 15 wt% of polymer binder B to the photoresist. Subsequently, the elastic recovery was also increased and maintained between 72.6% and 76.3% when the DA-MGIC or MA-DGIC monomer was added to polymer binder B. We concluded that the superior mechanical properties of the photoresist were due to the photo-curing, thermal-curing, and inter-penetration network characteristics of the novel polymer binders.

We investigated the effects of on other mechanical properties (compression) of the patterns when adding 15 wt% of polymer binder A to the photoresist; the results showed that the compression of the patterns was 200 mN (Table 3). The compression of the patterns was dramatically increased and maintained between 300 mN and 375 mN when the DA-MGIC or MA-DGIC monomer was added to polymer binder A (Fig. 9). In summary, the good mechanical properties of the photoresist can be attributed to the photo-curing and inter-penetration network characteristics of the polymer binders.

The photoresist coating was applied onto a glass substrate using a lithographic process. The thickness of the film was controlled between 4 μm and 5 μm, and the surface hardness of the film was then measured. The results showed that the surface hardness of the film was maintained at 3H when polymer binder A or B were added to the photoresist; the surface hardness of the film was enhanced to 4H when the DA-MGIC or MA-DGIC monomer was added to the polymer binder (Table 3). Therefore, an excellent surface hardness for the film can be obtained by introducing components such as DA-MGIC and MA-DGIC into the polymer binder.

Profile of Photoresist. The pattern profiles of the photoresist were observed by SEM (SEM diagrams), as shown in Fig. 10. Cylindrical patterns were only observed when polymer binder A or B was used (Run nos. A0 and B0). The taper angle for the patterns became vertical when the DA-MGIC or MA-DGIC monomer was added to the polymer binder (Run nos. A1–A6 and B1–B6). We concluded that this was due to the increased intensity of the inter-penetration network, as shown by the excellent inhibition of patterns.

Figure 10. SEM diagrams of photoresist of runs A0–A6 and B0–B6. *(Continued)*

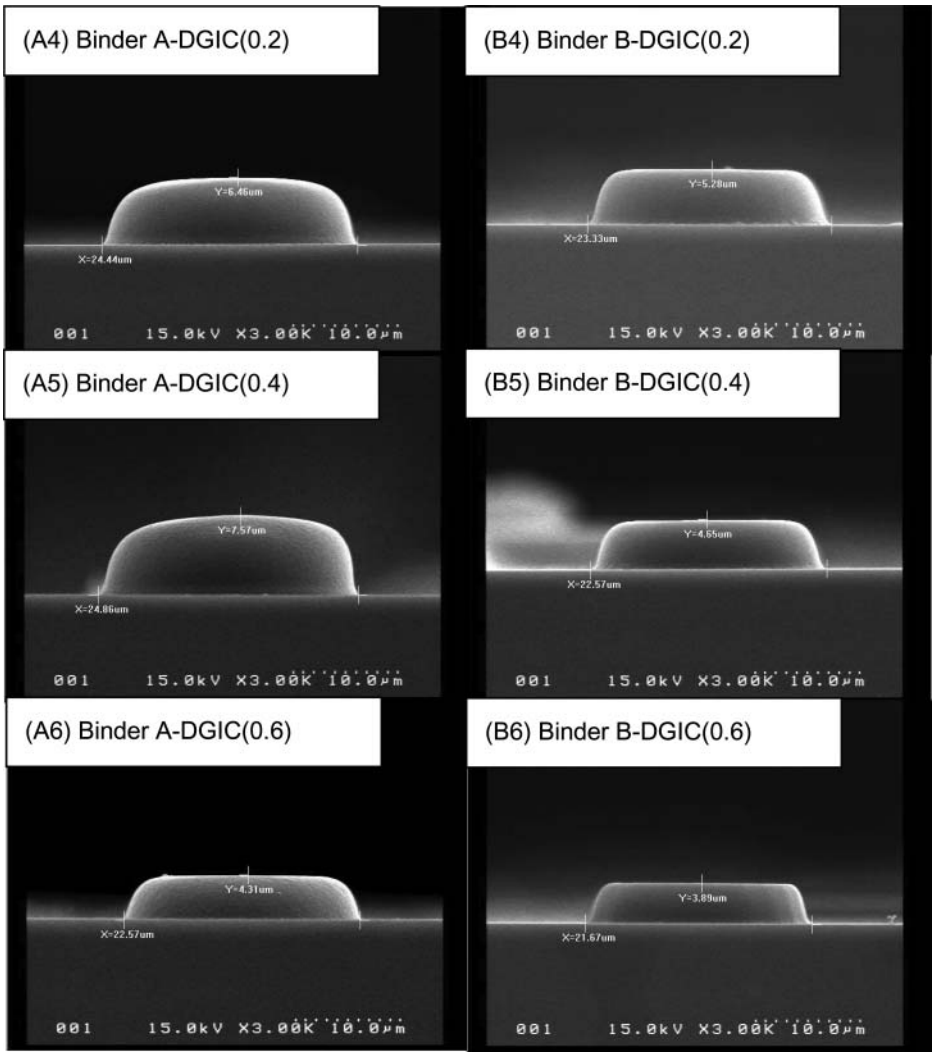


Figure 10. (Continued)

Conclusions

The four-component polymer binders A (MAA, STY, BzMA, and GMA) and B (MAA, STY, IBMA, and PMI) were synthesized by free-radical polymerization. DA-MGIC and MA-DGIC were then reacted with the formed polymer binders to create novel polymer binders with photo- and thermal-curing properties. The acid value, thermal behavior, and chemical structure of these novel polymer binders and the mechanical properties of the resulting patterns were investigated. The results showed that the thermal decomposition temperature (T_d) of the polymer binders increased when the DA-MGIC or MA-DGIC monomer was used in the binder. The elastic recovery and compression of the patterns were measured using a nanoindenter. The results showed that the patterns exhibited excellent mechanical properties due to the photo-curing, thermal-curing, and inter-penetration network characteristics of the novel polymer binders.

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