

# A Novel Amide–Imide Copolymer as a Matrix Resin for Ultraviolet Photoresists: Preparation, Properties, and Application

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**ABSTRACT:** The matrix resin as the backbone material plays a crucial role in the performance of a photoresist. In this study, a novel amide–imide copolymer, poly[*N*-(*p*-hydroxyphenyl) methacrylamide-*co*-*N*-phenylmaleimide], was successfully prepared. This copolymer was very close to 1 : 1 (molar ratio) in composition and was predominately alternating in structure during the copolymerization with an equimolar monomer feed ratio. It had good solubility in organic solvents and good film-forming characteristic, and it was also soluble in a basic aqueous solution. Differential scanning calorimetry and thermogravimetric analyses showed that it had a glass-transition temperature

at about 290°C and excellent thermostability. Photolithographic experiments indicated that the UV photoresist formulated with this copolymer as a matrix resin, diazonaphthoquinone sulfonate as a photosensitizer, a solvent, and other additives had a resolution of about 1 μm, a contrast of 2.83, and a sensitivity of 28 mJ/cm<sup>2</sup>. This photoresist had good plasma etching resistance and could endure 260°C for 30 min without thermal deformation of the lithographic images. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 117: 3715–3721, 2010

**Key words:** copolymerization; matrix; photoresists

## INTRODUCTION

A photoresist is an etching-resistant film whose solubility in a development solution can be changed by the irradiation of UV, deep UV, electron beams, ion beams, and X-rays. It is a key and fundamental material for the lithographic technique in microelectronics manufacturing systems. As a rule, a photoresist is composed of a matrix resin, a photosensitizer (a photoactive compound), a solvent, and other additives. The matrix resin plays a crucial role in the performance of the photoresist, and it is the backbone of the photoresist.

At this time, novolac resin polymers are widely used as the matrix resins of g-line (436 nm), i-line (365 nm), and even deep-UV (248 nm) photoresists. However, the continuing drive for miniaturization in the microelectronics manufacturing industry is pushing novolac-based photoresist systems to their performance limits.<sup>1–3</sup> The glass-transition temperature ( $T_g$ ) of novolac is comparatively low and usually

ranges between 70 and 120°C, depending on the molecular weight,<sup>4,5</sup> so the thermostability of novolac resins is deficient. Modern advanced fabrication techniques, such as ion implantation, for multilayer photoresist systems require images that are stable to 200°C and above. Furthermore, in chemically amplified photoresist systems, the high- $T_g$  properties of the matrix resin can reduce the mobility of the catalyst (proton) during the postexposure bake of lithography, which improves the resolution of the photoresist.

Thus, some other phenolic polymers have been studied as replacements for and/or additives to novolac photoresists. Examples include poly(*p*-hydroxystyrene),<sup>6</sup> poly(alkyl-substituted *p*-hydroxystyrene),<sup>7–9</sup> and novolac–polyhydroxystyrene copolymer,<sup>10</sup> the  $T_g$ 's of which are usually from 160 to 180°C, depending on the molecular weights and purities. However, the synthesis and polymerization of their monomers are unattractive. Turner et al.<sup>11</sup> reported that poly[*N*-(*p*-hydroxyphenyl) maleimide-*co*-olefins] were high- $T_g$  base-soluble copolymers, which could be formulated into aqueous-developable positive photoresists, and the images were resistant to thermal deformation at 200°C and above. Chiang and Lu<sup>12</sup> demonstrated that poly[*N*-(*p*-hydroxyphenyl) maleimide-

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*co*-(*p*-trimethylsilyl) styrene] also possessed these properties and could be used as a matrix resin for high- $T_g$  UV photoresists.

In this article, a novel matrix resin of an amide-imide copolymer is reported. This copolymer was prepared conveniently by the solution free-radical copolymerization of *N*-(*p*-hydroxyphenyl) methacrylamide (NHMA) and *N*-phenylmaleimide (NPMI). It had good solubility in organic solvents, good film-forming characteristics, good hydrophilicity, excellent thermostability ( $T_g \approx 290^\circ\text{C}$ ), and good plasma-etching resistance. In addition, it retained solubility in a base aqueous solution and could be formulated with a diazonaphthoquinone sulfonate (DNS) to effect the near-UV photoresist behavior when applied as a UV photoresist system.

## EXPERIMENTAL

### Materials

The main materials, including maleic anhydride, *p*-aminophenol, aniline, acetic anhydride, *N,N*-dimethylformamide (DMF), acetyl acetate, toluene, triethylamine (TEA), tetrahydrofuran (THF), and azobisisobutyronitrile (AIBN), were analytically pure grade. AIBN was recrystallized from methanol before use. Methacryloyl chloride and nickel acetate were chemically pure.

### Monomer NHMA

We synthesized the monomer NHMA by referring to and improving the method in ref. 13. A 500-mL, three-neck, round-bottom flask equipped with a refluxing condenser, Teflon stirrer, and dropping funnel was charged with 250 mL of THF, 26.3 g (0.26 mol) of TEA, and 27.30 g (0.25 mol) of *p*-aminophenol. The refluxing condenser was connected to a gas-absorbing setup with a dilute sodium hydroxide (NaOH) aqueous solution as a hydrogen chloride absorbent. After *p*-aminophenol was dissolved, the system was cooled to a temperature below  $5^\circ\text{C}$  by ice water. A solution of 27.2 g (0.26 mol) of methacryloyl chloride in 50 mL of THF was dropped into it over a period of 30 min. After that, the system was kept reacting for 1 h at room temperature and then refluxed for 30 min. Once the reaction was complete (thin-layer chromatography indicated that the reaction was over), the system was cooled, filtered, and washed with cold THF. After the filtrate was distilled to remove the THF solvent, a colorless crystal NHMA was obtained and dried (38.0 g, 86% yield). The crude product was further purified by recrystallization from acetyl acetate.

mp =  $159\text{--}160^\circ\text{C}$ . FTIR (wave numbers,  $\text{cm}^{-1}$ ): 3286, 3206, 1646, 830.  $^1\text{H-NMR}$  (chemical shift, ppm): 1.988, 5.409, 5.780, 6.764–6.793, 7.511–7.540, 8.926. Mass spectrometry (MS) M + H: 178.2, M + Na: 200.0. ANAL.

Calcd. for  $\text{C}_{10}\text{H}_{11}\text{O}_2\text{N}$ : C, 67.78%; H, 6.26%; N, 7.90%. Found: C, 67.60%; H, 6.22%; N, 8.10%.

### Monomer NPMI

The monomer NPMI was prepared according to the method in refs. 14–16. A 500-mL, three-neck, round-bottom flask equipped with a refluxing condenser, dropping funnel, and Teflon stirrer was charged with 200 mL of acetone and 24.5 g (0.25 mol) of maleic anhydride. After the maleic anhydride was dissolved completely, a solution of 23.25 g (0.25 mol) of freshly distilled aniline in 50 mL of acetone was dropped slowly into the system with stirring and cooling by ice water. The system was heated and refluxed for 1 h. Then, the heating of the system was stopped, and the system was cooled for several minutes; 40 mL of TEA, 50 mL of acetic anhydride, and 3.0 g of nickel acetate were added. The mixture was kept heating and refluxing for another 4 h until the reaction was complete (thin-layer chromatography indicated that the reaction was over). At last, the mixture was cooled and poured into cold deionized water to separate out sediment. The sediment was filtered, washed with cold water, and dried. A yellow powder NPMI was obtained (38.9 g, 90% yield). The crude product was further purified by recrystallization from toluene.

mp =  $89\text{--}90^\circ\text{C}$ . FTIR (wave number,  $\text{cm}^{-1}$ ): 1713, 755, 696.

### Copolymerization of NHMA and NPMI

A polymerization bottle was charged with 7.1 g (0.04 mol) of NHMA, 6.9 g (0.04 mol) of NPMI, 100 mL of DMF, and 0.12 g of AIBN. The bottle and contents were purged with high-purity argon. Then, the bottle was sealed, and the copolymerization was carried out in a  $65^\circ\text{C}$  bath for 48 h. We separated the copolymer by dropping the polymerization solution into deionized water. After drying *in vacuo* at  $60^\circ\text{C}$  for 48 h, 13.3 g (95% yield) of poly[*N*-(*p*-hydroxyphenyl) methacrylamide-*co*-*N*-phenylmaleimide] [poly(NHMA-*co*-NPMI)] was obtained. Gel permeation chromatography analysis in THF gave a number-average molecular weight ( $M_n$ ) of  $1.855 \times 10^4$ , a weight-average molecular weight ( $M_w$ ) of  $3.210 \times 10^4$ , and a molecular weight distribution of 1.730.

ANAL. Calcd for  $\text{C}_{20}\text{H}_{18}\text{O}_4\text{N}_2$  (1 : 1 structure): C, 68.56%; H, 5.18%; N, 8.00%. Found: C, 68.42%; H, 5.21%; N, 8.20%.

### Formulation and processing of the UV photoresist

A photoresist solution was formulated by the dissolution of 3.0 g of poly(NHMA-*co*-NPMI) (see previous discussion) and 0.3 g of the photosensitizer DNS

in a mixture of 5 g of propylene glycol monomethyl ether acetate, 5 g of ethyl lactate, and 1 g of  $\gamma$ -butyrolactone. DNS was prepared by the reaction of diazonaphthoquinone sulfochloride with 2,3,4,4'-tetrahydroxybenzophenone at a molar ratio of 3 : 1.

This photoresist was filtered through a 0.25- $\mu\text{m}$  Teflon filter and spin-coated at 4500 rpm on a 4-in. silicon (Si) wafer with a spinner. The film was prebaked on a hot plate at 100°C for 3 min to yield a film about 1  $\mu\text{m}$  thick and exposed with an MA6 dual-contact aligner (Suss MicroTec Inc., Germany; light source: 350-W high-pressure mercury lamp, wavelength = 365 nm, power density  $\approx$  1.8 mW/cm<sup>2</sup>). After exposure, development was carried out in a 0.2 wt % dilute NaOH solution for 15 s, and the sample was rinsed in deionized water for 10 s. The developed film was postbaked on a hot plate at 120°C for 2 min. The film thickness was measured with a Dektak II Profilometer (Veeco Instruments Inc., USA).

#### FTIR, <sup>1</sup>H-NMR, MS, elemental analyses, thermal analyses, plasma etching resistance, and imaging evaluation

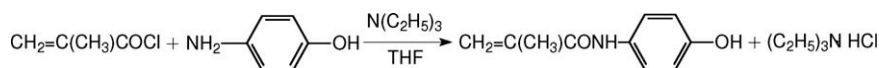
FTIR, <sup>1</sup>H-NMR, and MS spectra were obtained on an FTIR spectrophotometer (EQUINOX 55, Bruker Corporation, Germany) from KBr pellets, a Mercury VX-300 (Varian Inc., USA) with tetramethylsilane as an internal standard and deuterated acetone as solvent, and an Agilent 1100 LC/MSD (liquid chromatography/mass selective detector) system, respectively. Elemental analyses were determined on a Vario EL III elemental analyzer (Elementar Corporation, Germany) with acetanilide as the standard. Thermal analyses were per-

formed on a PerkinElmer DSC-7 and TGA-7 thermal analyzer (PerkinElmer Inc., USA) by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) evaluation, respectively. Plasma etching resistance was tested with a model ME-3A multifunctional magnetically enhanced reactive ion etching machine (China Academy Microelectronics Center, China; CHF<sub>3</sub>: 20 ccm, SF<sub>6</sub>: 80 ccm, O<sub>2</sub>: 5 ccm; sources: 50 W for 5 min and 100 W for 3 min). The quality of image in the photoresist patterns was evaluated with a JSM5510LV scanning electron microscope (JEOL, Japan), a WYKO NT1100 optical profiling system (Veeco Instruments Inc., USA), and a model COIC MA1000 optical microscope (Chongqing Optical & Electrical Instrument Co., Ltd., China). The thermal resistance of the patterns was examined with a 260°C hot plate in the atmospheric environment.

## RESULTS AND DISCUSSION

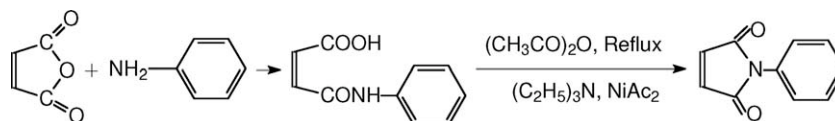
### Preparation and structure of the monomers and poly(NHMA-co-NPMI)

In this study, NHMA was successfully synthesized by the reaction of methacryloyl chloride and *p*-aminophenol with TEA as an antacid [eq. (1)]. Its structure was verified by the data analyses of its FTIR, <sup>1</sup>H-NMR, and MS spectra. However, Patai et al.<sup>13</sup> pointed out that the reaction of methacryloyl chloride and *p*-aminophenol gave only the amide ester (4-methacrylamidophenol methacrylate). This was probably because the antacid (e.g., TEA, pyridine) was not added to the reaction system during Patai et al.'s preparation:



(1)

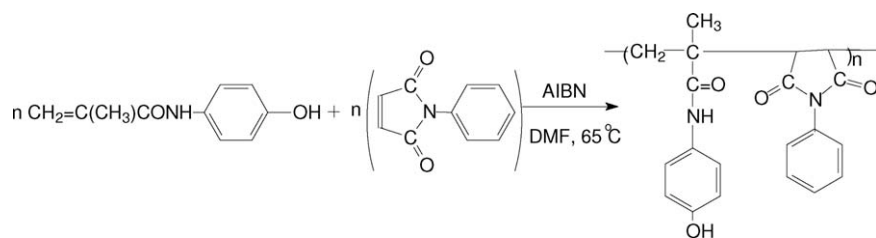
NPMI has been prepared and verified in detail in many previous studies [eq. (2)]:<sup>14-16</sup>



(2)

Poly(NHMA-co-NPMI) was obtained from the direct solution free-radical copolymerization of the monomers NHMA and NPMI, illustrated in eq. (3). This was very convenient for the preparation of poly(NHMA-co-NPMI). In its FTIR spectrum

(Fig. 1), the peak of the broad absorption band of hydroxyl groups was at 3301 cm<sup>-1</sup> (hydrogen bond). The peaks at 1775, 1700, and 1653 cm<sup>-1</sup> corresponded to the vibration absorption of bond C=O:



(3)

In this study, an equimolar monomer feed ratio was used to copolymerize NHMA with NPMI. The resulting copolymer was very close to 1 : 1 (molar ratio) in composition and predominately alternating in structure, as determined by elemental analyses. This was attributed to the different polarities of the polymerization double bonds, that is, slightly electron rich in the methacrylamide and badly electron poor in the maleimide. Other classic behaviors were well documented for similar systems, such as styrene and *N*-(*p*-hydroxyphenyl) maleimide (NHMI),<sup>17</sup> styrene and *N*-(*p*-acetoxystyryl)maleimide,<sup>11</sup> and *p*-trimethylsilylstyrene and NHMI.<sup>12</sup>

### Properties of the copolymer

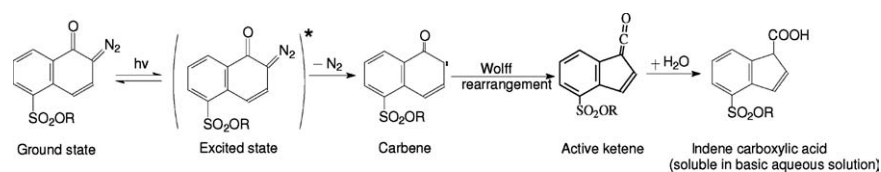
Experiments showed that poly(NHMA-*co*-NPMI) was soluble in a basic aqueous solution (e.g., NaOH and tetramethylammonium hydroxide) because of the existence of hydroxyl groups; this was very convenient to the development process of a UV-positive photoresist. Furthermore, poly(NHMA-*co*-NPMI) could be well dissolved in many organic solvents (e.g., DMF, dioxane, THF, propylene glycol monomethyl ether acetate, ethyl lactate), and this resulted in homogeneous polymer solutions. When these solutions were spin-coated on the Si wafer, uniform films were formed. So, poly(NHMA-*co*-NPMI) had good film-forming characteristics.

The DSC and TGA curves (curve 1) of poly(NHMA-*co*-NPMI) are shown in Figure 2. This indicated that its starting decomposition temperature

was about 330°C, lower than that of the commonly used poly(styrene-*alt*-NHMI) (curve 2,  $M_n = 1.153 \times 10^4$ , decomposition temperature  $\approx 350^\circ\text{C}$ ). However, its  $T_g$  was about 290°C, which was higher than that of poly(styrene-*alt*-NHMI) ( $T_g \approx 255^\circ\text{C}$ ).<sup>17</sup> This difference was more than 30°C. Both  $T_g$ 's were far bigger than those of polystyrene ( $T_g = 100^\circ\text{C}$ ) and poly(styrene-*alt*-NPMI) ( $T_g = 225^\circ\text{C}$ ). It is well known that the upper limit temperatures of polymers are their  $T_g$ 's, so poly(NHMA-*co*-NPMI) had a better thermostability. This was attributed to its special structure. In this copolymer, each repeat unit contained an amide and an imide structure, respectively, in which nitrogen atoms contained lone electron pairs of *p* orbitals and could form *p*- $\pi$  conjugations with benzene rings and carbonyl groups. This increased the conjugation degree and chain-stiffening effect. What is more, the hydroxyl group enhanced the polarity of this copolymer. All played important roles in the high thermal resistance of poly(NHMA-*co*-NPMI). As expected, poly(NHMA-*co*-NPMI) as a matrix resin for the photoresists endured a higher microelectronic processing temperature than other polymers, for example, polystyrene, novolac resin, poly(styrene-*alt*-NPMI), and poly(styrene-*alt*-NHMI).

Figure 3 shows the plot of  $T_g$  versus  $M_n$  for poly(NHMA-*co*-NPMI). It illustrates that the  $T_g$  of this copolymer increased with  $M_n$  and then plateaued in the  $M_n$  range 20,000–30,000. Above this range, the change in  $T_g$  was very slow, and it remained a plateau.

### Properties of the photoresist



(4)



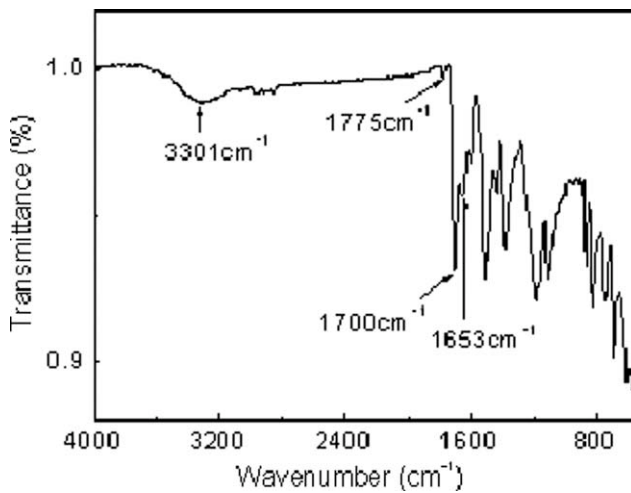


Figure 1 FTIR spectrum of poly(NHMA-co-NPMI).

Poly(NHMA-co-NPMI) itself was not sensitive to near-UV irradiation, but the incorporation of DNS did effect the sensitivity to the near-UV region. The photoresist formulated by this copolymer and DNS

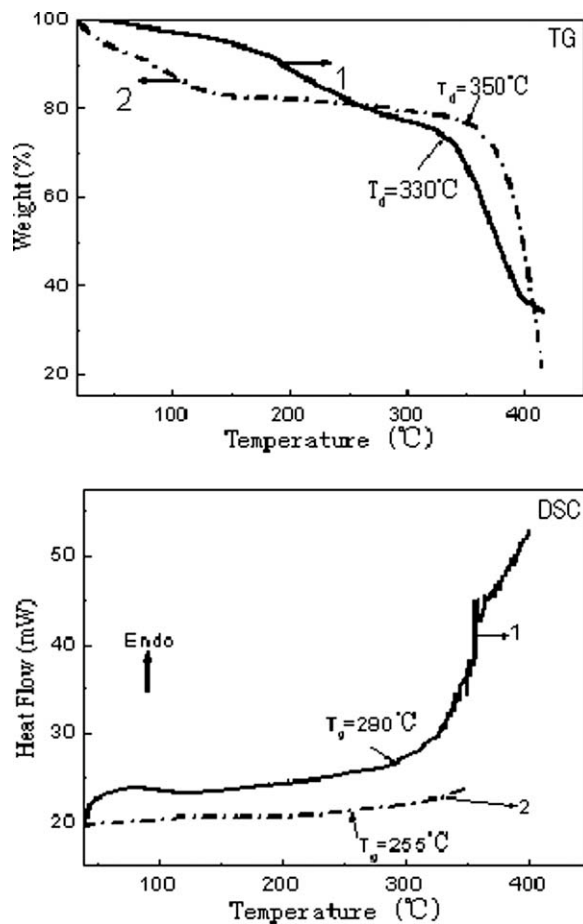


Figure 2 TGA and DSC curves of (1) poly(NHMA-co-NPMI) (1 : 1 composition) and (2) poly(styrene-alt-NHMI) (1 : 1 composition).

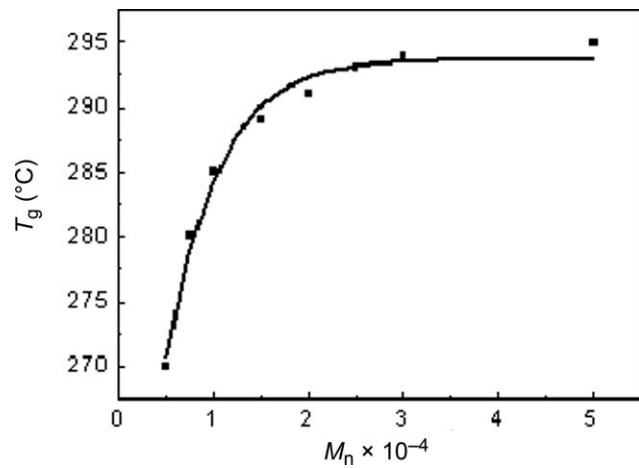


Figure 3 Plot of  $T_g$  versus  $M_n$  for poly(NHMA-co-NPMI).

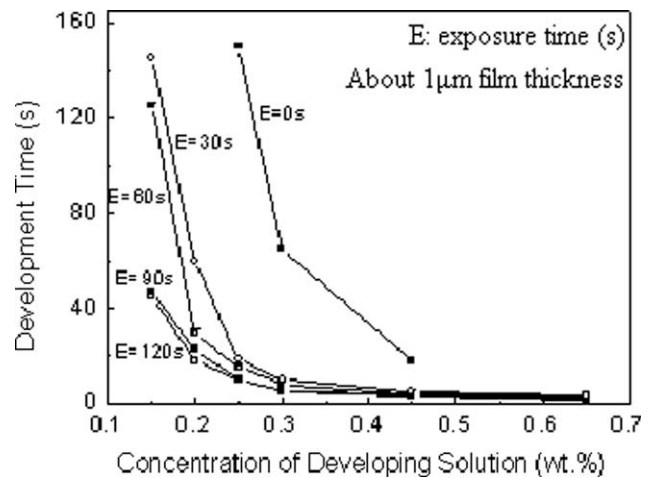


Figure 4 Relationship between the development time and concentration of the developing solution.

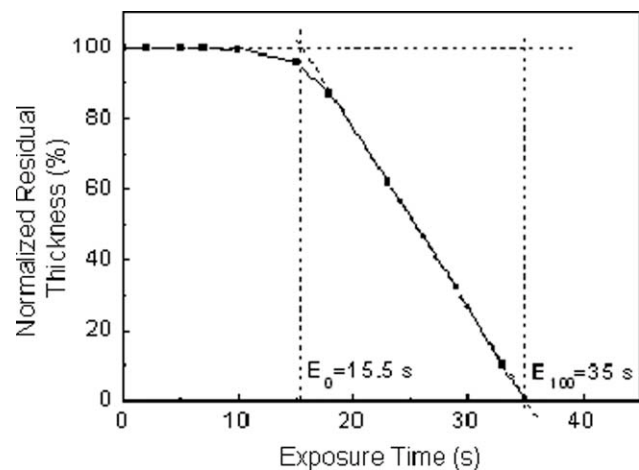
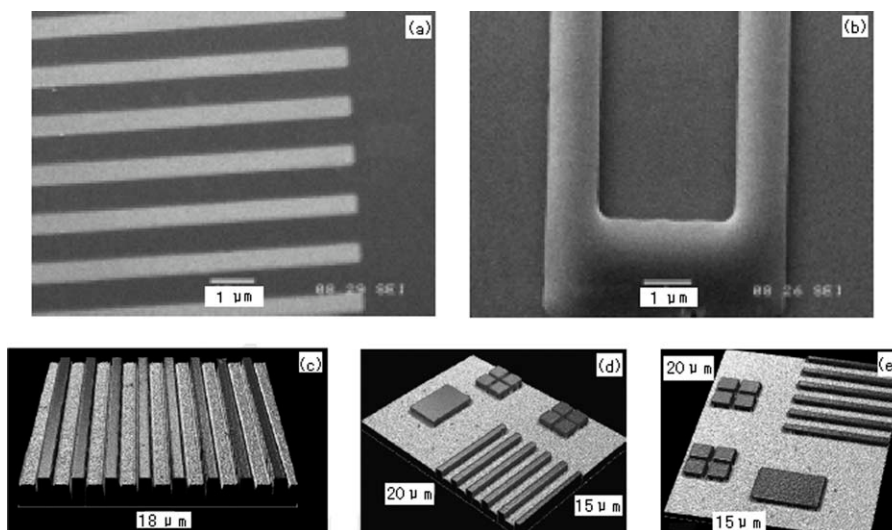


Figure 5 Photographic sensitivity curve of the photoresist (spin coating at 4500 rpm, prebaking at 100°C for 3 min, no postexposure baking, development for 15 s in 0.2 wt % NaOH aqueous solution, 10 s of rinsing in deionized water, and postbaking at 120°C for 2 min).



**Figure 6** (a,b) Scanning electron microscopy and (c–e) optical profile photographs of photolithographic images.

was positive working,<sup>18,19</sup> whose photochemical reaction mechanism is listed in eq. (4). After exposure, it could be developed in a cheap NaOH aqueous solution. Figure 4 shows the relationship between the development time and the concentration of developing solution when an about 1 μm thick photoresist film underwent different exposure times ( $E$ 's = 0, 30, 60, 90, and 120 s). This indicates that this photoresist had a larger contrast when the concentration of the developing solution was 0.2 wt %. So the 0.2 wt % NaOH aqueous solution was chosen as the developing reagent.

Figure 5 shows the exposure response curve of this photoresist. It was observed that when the photoresist film was developed thoroughly (i.e., the normalized residual thickness was zero), a 35-s exposure duration was needed (i.e., the thoroughly-developed exposure time  $E_{100} = 35$  s). After extrapolation, the exposure duration was 15.5 s when the photochemical reaction of the photoresist was initiated (i.e., the exposure time initiating the photochemical reaction of the photoresist  $E_0 = 15.5$  s). The contrast ( $\gamma = 2.83$ ) of this photoresist was calculated

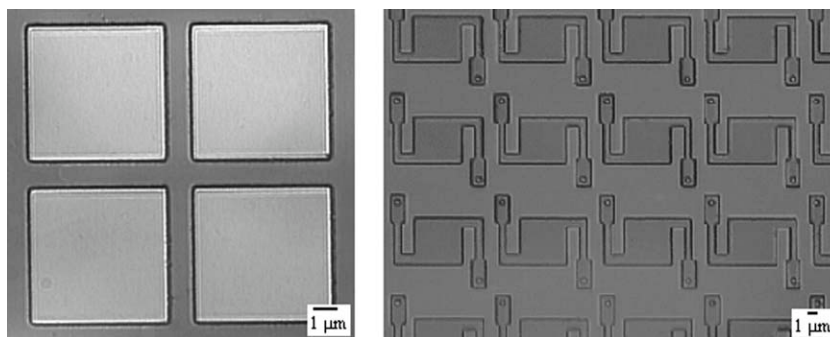
by eq. (5) and was larger than that ( $\gamma = 2.7$ ) of a UV photoresist with poly(styrene-*alt*-NHMI) as the matrix resin:

$$\gamma = [\log_{10}(E_{100}/E_0)]^{-1} = [\log_{10}(35/15.5)]^{-1} = 2.83 \quad (5)$$

The photographic sensitivity of the photoresist was about 28 mJ/cm<sup>2</sup>, as determined by a UV light energy integrator. This value was comparable to that of a commercial novolac photoresist.

### Photoimaging

After exposure, development, and postbaking of the photoresist, scanning electron microscopy [Fig. 6(a,b)] and optical profile [Fig. 6(c,d)] photographs of the photolithographic images were taken, as shown in Figure 6. The resolution of the line width and space in the patterns was about 1 μm, and the profiles of these lines were clear and steep.



**Figure 7** Optical microscope photographs of lithographic images after reactive ion etching.

Experiments showed that the images from the photoresist based on this high- $T_g$  copolymer had a markedly enhanced resistance to thermal flow, and no image distortion was observed after a thermal resistance test at 260°C for 30 min [Fig. 6(e)]. However, this excellent thermal resistance had little effect on the reflow of the standing wave. Generally speaking, the standing wave effect in the modern lithographic process is eliminated by back antireflective coatings and/or other techniques. Even if the reflow technique were used to eliminate the standing wave effect, the reflow temperature needed to be far below the  $T_g$  of the matrix resin. If this temperature were close to or exceeded the  $T_g$ , the thermal distortion of the lithographic images would be inevitable. This was not advisable.

Figure 7 displays the optical microscopy photographs of the lithographic images after reactive ion etching. These patterns still remained clear and intact. It verified that the images had high plasma-etching resistance because of the two benzene rings in each repeat unit of this copolymer structure.

### CONCLUSIONS

The monomer NHMA was successfully synthesized by the reaction of methacryloyl chloride and *p*-aminophenol with TEA as an antacid. Then, a novel imide-amide copolymer, poly(NHMA-*co*-NPMI), was conveniently prepared by solution free-radical copolymerization. Analysis and testing verified that the copolymer was very close to 1 : 1 (molar ratio) in composition and predominately alternating in structure during the copolymerization at an equimolar monomer feed ratio. Experiments showed that this copolymer had good solubility and good film-forming characteristics. It was soluble in a basic aqueous solution for the convenient development process of the UV photoresist. The thermal property curves demonstrated that this copolymer had outstanding thermostability with a  $T_g$  of about 290°C, which was higher than that of poly(styrene-*alt*-

NHMI). Photolithographic experiments indicated that the UV photoresist formulated with this copolymer as a matrix resin, DNS as a photosensitizer, a solvent, and other additives had a resolution of about 1  $\mu\text{m}$ , a contrast of 2.83, and a sensitivity of 28  $\text{mJ}/\text{cm}^2$ . This photoresist had excellent thermostability and good plasma-etching resistance.

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