Synthesis and Characterization of Poly(acrylate amic acid) and Its Application as Negative-Tone Photosensitive Polyimides

Chang-Hong Ho, Sheng Chang, Yu-Der Lee

Department of Chemical Engineering, National Tsing Hua University, Hsinchu 30013, Taiwan

ABSTRACT: The dianhydride monomer 3,3′,4,4′-benzophenone tetracarboxylic acid dianhydride and two diamine monomers, 4,4′-diamino-3,3′-biphenyldiol (HAB) and 2,4-diaminophenol dihydrochloride (DAP), were used to synthesize a series of poly(hydroxyl amic acid). Further functionalization by grafting acrylate groups yields the corresponding poly(acrylate amic acid) that underwent a crosslinking reaction on exposure to UV-light and was used as a negative-tone photosensitive polyimide (PSPI). The analysis of chemical composition and molecular weight of these poly(amic acid)s determined by nuclear magnetic resonance (NMR) spectroscopy, Fourier transform infrared spectroscopy, and gel permeation chromatography revealed that the molecular weight of the poly(hydroxyl amic acid) increased with the molar content of HAB in the feedstock, because HAB exhibited higher polymerization reactivity than DAP. Moreover, the degree of grafting acrylate groups onto poly(hydroxyl amic acid) was determined by ^{1}H-NMR spectroscopy. The photoresist was formulated by adding 2-benzyl-2-N,N-dimethylamino-1-(4-morpholinophenyl) butanone (IRG369) and isopropylthioxanthone as a photoinitiator, tetra(ethylene glycol) diacrylate as a crosslinker, and tribromomethyl phenyl sulfone as a photosensitizer. The PSPI precursor exhibited a photosensitivity of 200 mJ/cm^2 and a contrast of 1.78. A pattern with a resolution of 10 μm was observed in an optical micrograph after thermal imidization at 300°C. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 2395–2402, 2009

Key words: polyimide; lithography; photoresist; poly(hydroxyl amic acid); poly(acrylate amic acid)

INTRODUCTION

Polyimides (PIs) have been widely utilized in the field of electronics including buffer coatings, packages, passivation layers, alpha particle barriers, interlayer dielectrics, and wafer scale packages because of their outstanding thermal, mechanical, and electrical properties. Further modification of PIs with various functional groups largely expands their application ranges, such as in the fields of liquid crystal alignment layers, nonlinear optical polymers, and others.[1–7] In particular, photosensitive polyimide (PSPI) associated with easy pattern transfer has attracted great interest in the fields of integrated-circuits and MEMS because it can substantially reduce the number of microlithographic steps and simplify the complex procedures by direct exposure and development without the use of an additional photoresists.

PSPIs were first reported by Kerwin and Goldrick[8] in 1971 and are classified as positive or negative tone based on the difference of the dissolution rates of the exposed and the unexposed area. Most PSPIs used in today's microelectronic industries are negative-tone and those photoreactive groups, typically the methacrylate or acrylate groups, are attached to poly(amic acid) via the ionic bond formed by the acid-amine interaction[9,10] or through the ester bond[6,11,12] in the form of poly(amic ester), followed by an imidization of the PI precursor after the lithographic procedures. However, the conventional organo-soluble PSPIs are not employed in the practical application because the patterning processes for LSI manufacturing using an aqueous developer are presently practiced in terms of commercial value and environmental issues.

The hydroxyl- or carboxylic-containing PSPIs that could be developed with a base aqueous developer were proposed.[6,13–15] The pendant hydroxyl and carboxylic groups along the polymer chain allow further modification of the PI, such as cross-linking reaction or incorporation of photosensitive functional groups.[9,16,17] However, the introduction of the crosslinkable groups accompanied by the consumption of the hydrophilic groups would influence the hydrophilicity and lead to the decline in the solubility in aqueous alkaline developer. To our knowledge, few PSPIs containing photosensitive groups and carboxylic groups have been reported,[6,13,18–20] and the monomers utilized were prepared in laboratory scale for research, but are not practical for industry.

Hence, an alternative procedure of synthesis of PSPIs utilizing commercialized monomers was proposed in this work, which was easier than the molecular design method. Moreover, 4,4'-(hexafluroisopropylidene)-bis(o-aminophenol) was a halogen-containing diaminophenol derivative and was commonly utilized in the PI synthesis for various applications[4–6,14] because of the excellent solubility in organic solvent and optoelectronic properties. However, the toxic halogen might cause severe damage not only to the function of the electronic product but also to the environment if the discards are not treated properly.

In this study, an aqueous based-soluble photosensitive poly(acrylate amic acid) was prepared by the esterification of the corresponding poly(hydroxyl amic acid), which was synthesized by the condensation of dianhydride monomers with an equivalent ratio of hydroxyl-diamine monomers whose molar composition was altered, with acryloyl chloride. The chemical structures, compositions, and the degree of grafting of the photoreactive groups were determined by the ^{1}H-NMR and FTIR spectroscopy. The molecular weight of the poly(amic acid) was measurement by gel permeation chromatography. The polymerization reactivity of the two hydroxyl-containing diamine groups was discussed. The solubility of the poly(acrylate amic acid) in the base aqueous solution was examined. The influence on the chemical composition of the PSPI and on the photolithographic properties, including photosensitivity, contrast, and resolution, was also investigated.

MATERIALS AND METHODS

Materials

4,4'-Diamino-3,3'-biphenyldiol (HAB) was purchased from Chriskev (Lenexa, KS). Isopropylthioxanthone (ITX) was purchased from Fluka (Milwaukee, WI). Tribromomethyl phenyl sulfone (TBPS) was obtained from TCI (Tokyo, Japan). Tetramethyl ammonium hydroxide (TMAH; 25 wt %), 2,4-diaminophenol dihydrochloride (DAP), and tetra(ethylene glycol) diacrylate (TEGDA) were obtained from Lancaster (Ward Hill, MA). 2-Benzyl-2-N,N-dimethylamino-1-(4-morpholinophenyl) butanone (IRG369) was obtained from Aldrich (Milwaukee, WI). All aforementioned reagents were used as received without further purification. Pyridine, triethylamine (Et$_3$N), and N,N-dimethylacetamide (DMAc), purchased from Tedia (Fairfield, OH), were dried with anhydrous calcium hydride and then distilled. These dehydrated reagents were stored in vials packed with molecular sieves (4A, Aldrich) in an inert atmosphere. 3,3',4,4'-Benzophenone tetracarboxylic acid dianhydride (BTDA) purchased from Chriskev was recrystallized from acetic anhydride, dried under reduced pressure at 120°C, and then stored in a nitrogen atmosphere.

Synthesis of poly(hydroxy amic acid) copolymer

A representative example of poly(hydroxyl amic acid) (PHAA) copolymer synthesis was described as follows: monomer-grade diamines, DAP (0.9851 g, 5 mmol) and HAB (1.0812 g, 5 mmol), were introduced to a dry nitrogen-flushed three-necked round-bottom flask. After the diamines had completely dissolved in anhydrous DMAc (20 wt % total monomer concentration) with 10 mmol dehydrated pyridine at 0°C, an equimolar amount of dianhydride monomers BTDA (3.2223 g, 10 mmol) was added to the solution. After the reaction had proceeded for 16 h, the viscous PHAA solution was precipitated in a methanol/deionized (DI) water mixture with a volume ratio of 1/1, and further purification by soxhlet extractor with a methanol/DI water mixture for 24 h was accomplished, followed by drying at 100°C under reduced pressure for at least 16 h.

Synthesis of poly(acrylate amic acid) copolymer

A representative example of synthesizing poly(acrylate amic acid) copolymer (PAAA) was described as follows. To a 100-mL three-necked round-bottom flask equipped with an addition funnel and a nitrogen inlet were added 2 g PHAA, 18 g anhydrous DMAc, and 2 g anhydrous triethylamine. Three times, a molar excess of acryloyl chloride solution in DMAc was added to the polymer solution dropwise at 0°C within 3 h after complete dissolution of the PHAA; the solution was stirred for another 18 h below 10°C. The Et$_3$N HCl salt, which made the product solution cloudy, was removed by vacuum filtration and the clear filtrate was precipitated in methanol. The collected precipitant, PAAA, was further purified by using a soxhlet extractor with HPLC-grade methanol and then dried at 100°C under reduced pressure for at least 16 h.

Characterization

The chemical structures of the poly(amic acids), including PHAA and PAAA, were confirmed by nuclear magnetic resonance (^{1}H-NMR) and Fourier transform infrared spectrometer (FTIR). All of the ^{1}H-NMR spectra were recorded on Varian Unityinova 500 NMR Spectrometer at 500 MHz, and d_6-DMSO was used as solvent. The FTIR spectra were obtained by 16 scans in the range of 4000–400 cm^{-1} with a resolution of 2 cm^{-1}. The inherent viscosities of the poly(amic acid) were measured at 30°C by using a Capillary Ubbelohde viscometer. The molecular weight of the poly(amic acid) was determined by a size exclusion chromatography (SEC) with

DMAc containing 0.05 wt % $LiNO_3$ as the eluent at a constant flow rate of 0.8 mL/min; polystyrene was used as the standard to construct a calibration curve. Transmission UV–vis spectra were obtained on a Hitachi U3300 UV–Vis spectrophotometer.

The film thickness of the PSPI coated on a glassy substrate was measured by using an α-step instrument (Tencor-P10). The lithographic patterns were observed by using an optical microscopy.

Preparation of poly(amic acid) film and thermal conversion to PI

A 20 wt % poly(amic acid) solution in DMAc was prepared, and the polymer solution was spin-coated on a 2.5×2.5 cm^2 glass plate using a spin coater. The wet film was dried at 170°C in an nitrogen atmosphere for 30 min and this poly(amic acid) gel film was then converted to the PI at 300°C for 2 h via thermal imidization in the nitrogen atmosphere.

Preparation of photoresist formula and lithographic evaluation

The representative photoresist formulation was prepared as follows: 1 g PAAA, 40 mg photo-initiator comprising equal weights of IRG369 and ITX, 200 mg photo-crosslinker (TEGDA), and 110 mg photosensitizer (TPBPS) were entirely dissolved in 3 g DMAc as solvent. The viscous solution that was filtered through a 1.2-μm nylon filter before use was spin-coated on a 2.5×2.5 cm^2 glass plate and soft-baked at 100°C for 8 min in sequence; the gel-film with thickness of 6 μm was exposed to a broadband UV light by using a single-side Mask Aligner. The pattern was developed with a DMAc/TMAH (2.38 wt %) mixture with a volume ratio of 1/1 at room temperature, and the development time that was needed to completely remove the unexposed film depended on the chemical composition of the co-PHAA. It was then hard-baked at 170°C for 30 min to remove the solvent that was trapped in the resin. The photosensitivity and contrast, representing the performance of a photoresist, were determined by plotting a characteristic exposure curve of normalized thickness against the exposed does. With respect to the resolution of a PSPI, each patterned specimen that was composed of poly(amic acid) was cured to generate PI via thermal imidization at 300°C for 2 h; the pattern was then observed by using an optical microscope.

RESULTS AND DISCUSSION

Synthesis and characterization of poly(hydroxyl amic acid)

An alternative method of preparing the PSPI containing hydrophilic carboxylic acid groups and photo-crosslinkable was proposed, and Scheme 1 presents a flow chart of the synthesis of the PSPI: photosensitive groups were grafted onto PHAA, which was prepared by polycondensation of the dianhydrides and two diamines including HAB and DAP, via a acrylation reaction. After UV-light exposure, the crosslinked poly(amic acid) was thermally cured to generate the PI.

Diaminophenol derivatives are not sufficiently stable and the oxidation of the free amine to the nitroso and nitro groups makes the purification, synthesis, and monomer storage difficult. For the oxidation stability consideration, diaminophenol was generally protected by acid and was presented in the diamine-acid salt state.[21,22] However, an adequate base was necessary to convert the inactive salt-type compound to the corresponding neutral diamine, showing polymerization reactivity.[23] In this work, pyridine was utilized to generate a free mono-substituted diaminophenol *in situ* in the synthesis of poly(hydroxyl amic acid).

The asymmetric DAP monomer showing low polymerization reactivity yielded the BTDA-DAP PHAA with a low inherent viscosity of 0.12 dL/g. Consequently, a co-monomer HAB diamine that yielded the corresponding BTDA-HAB PHAA with an inherent viscosity of 0.78 dL/g was exploited to increase the molecular weight of the co-poly(hydroxyl amic acid) (co-PHAA) and to adjust the chemical composition of the co-PHAA.

Figure 1(a) presents the IR spectrum of the co-PHAA, which included that characteristic peaks of the C=O of the carboxylic acid and the amide group at 1720 and 1660 cm^{-1}, respectively, as well as the OH absorption of carboxylic acid at 3100–3400 cm^{-1}. Figure 2(a) displays the ^{1}H-NMR spectrum of the co-PHAA, and the signals corresponding to the constitutive protons were marked, indicating that main absorptions were assigned to the amide groups at 9.8 ppm, the hydroxyl groups from HAB and DAP moieties at 10.2 and 10.4 ppm, the aromatic ring of the dianhydride at 8.0–8.2 ppm, and that of diamines at 7.0–8.0 ppm, respectively. However, the absorption from HAB and DAP was not able to be resolved precisely due to the asymmetric structure of poly(amic acid).

The chemical composition of the co-PHAA was estimated from the ^{1}H-NMR spectrum and determined by the following formula,

$$R_H(\%) = \frac{A_H/2}{A_H/2 + A_D} \times 100\%$$

$$R_D(\%) = (1 - R_H) \times 100\%$$

where R_H (%) and R_D (%) represent the molar content of the HAB and DAP units in a co-PHAA, and
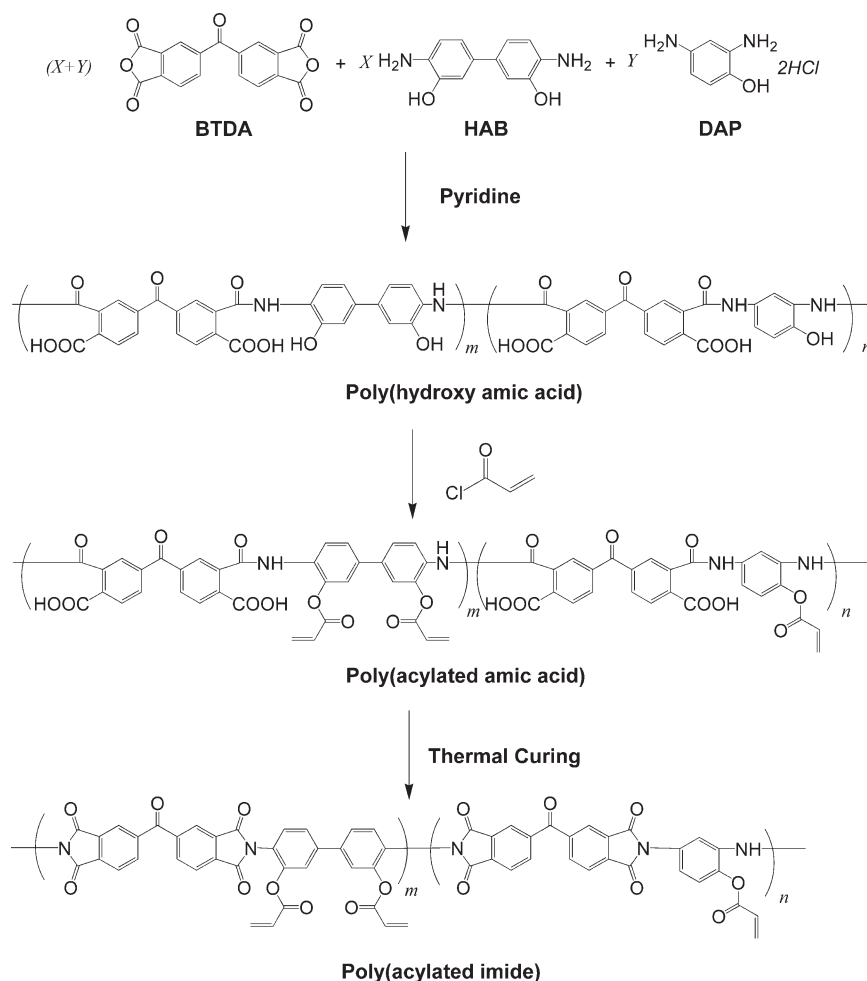
Scheme 1   Protocol of synthesis of PSPI.

A_H and A_D denote the areas of the peaks at 10.2 and 10.4 ppm, respectively. The molecular weight of the co-PHAAs was determined by SEC, in which the eluent had to be doped with some salt, such as LiNO$_3$, because the intermolecular polyelectrolyte effect would affect the molecular conformation of the co-PHAA in DMAc, leading to over measurement of the molecular weight of the PI precursors.[7,24,25] Table I presents the detailed results of the molecular weight, the yield, and the chemical composition of various co-PHAAs. The co-PHAAs were partially dissolved in the hot methanol, and refluxed MeOH/DI water used in the purification of the crude product would remove the lower M_w copolymer, which gives a narrow PDI comparable to that from chain polymerization. The increase of the molecular weight and the yield of the co-PHAA with the HAB concentration in the feedstock and the practical HAB composition exceeding the feedstock content elucidated that the polymerization reactivity of the HAB was higher than that of the DAP monomers.
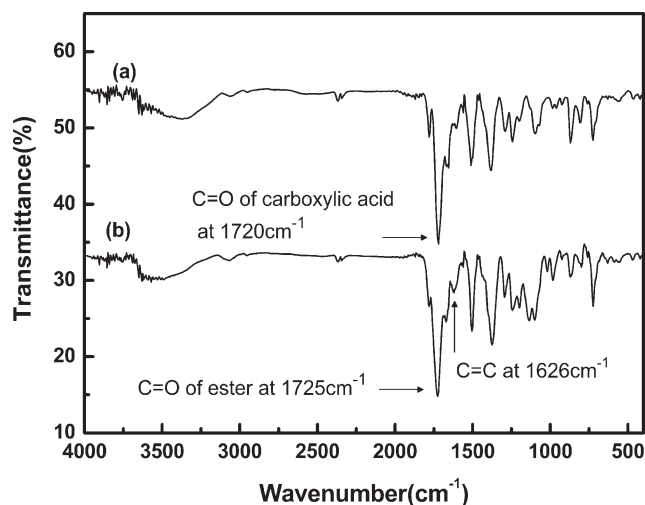

Figure 1   FTIR spectra of poly(amic acid): (a) poly(hydroxyl amic acid); (b) poly(acrylate amic acid).
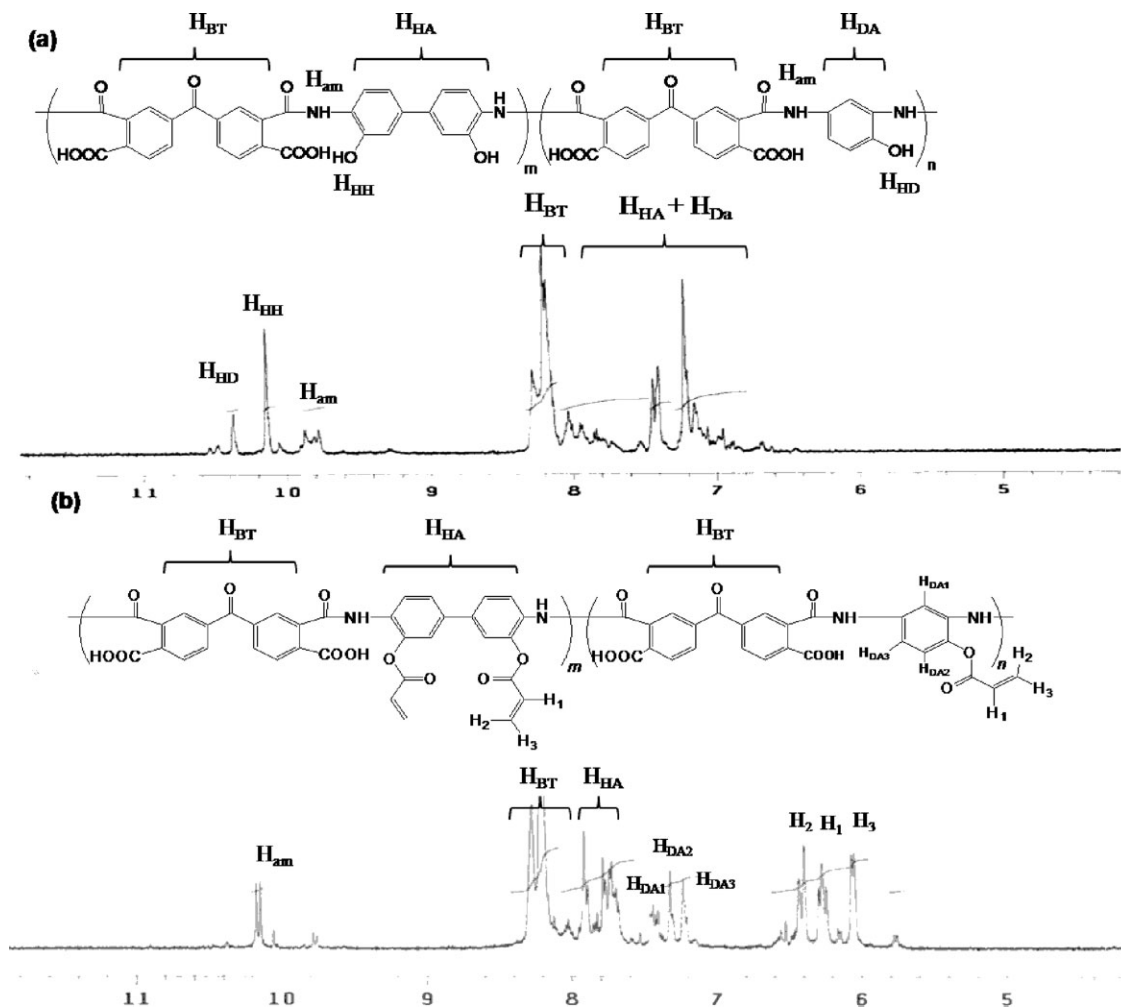
Figure 2 H-NMR spectrum of the poly(amic acid): (a) poly(hydroxyl amic acid); (b) poly(acrylate amic acid).

Synthesis and characterization of poly(acrylate amic acid)

Poly(amic acid)s containing photo-polymerizable acrylate groups, PAAA, were prepared by the esterification of PHAA with acryloyl chloride under the catalysis of triethylamine. The FTIR spectrum of the PAAA shown in Figure 1(b) revealed a new absorption at 1626 cm^{-1} and a shift in carboxylic acid C=O absorption from 1720 cm^{-1} toward 1725 cm^{-1}, representing the ester groups absorption. The ^{1}H-NMR spectrum of poly-(acrylate amic acid) displayed in Figure 2(b) indicated that (1) the intensities of the hydroxyl groups from monomers of HAB and DAP at 10.2–10.4 ppm declined; (2) a set of splitting patterns in the range 6.0–6.8 ppm, like those of the acryloyl chloride (not shown here), was present; and (3) two doublet-splitting peaks at 9.8 and 10.2 ppm from the amide hydrogen were observed, indicating the acrylate groups were successfully attached to PHAA. In the reaction of grafting photosensitive groups onto PHAA, the reactivity of the hydroxyl groups of poly(hydroxyl amic acid) toward acryloyl chloride is higher than that of carboxylic acid. Further-

more, according to previous studies,[26,27] the reaction of hydroxyl (amic acid) with an acid chloride in the presence of triethylamine gives a major product of hydroxyl imide or hydroxyl isoimide rather than hydroxyl amic ester, suggesting that the PSPI present in this study is associated with poly(acrylate amic acid).

The grafting degree (GD%) of the photo-reactive groups was defined as the proportion of hydroxyl groups in the co-PHAA that were converted into acrylate groups and was estimated by the following formula involving the ratio of M_p to M_{100}, that denotes the practical amount of acrylic groups grafted onto poly(hydroxyl amic acid) and the total amount of hydroxyl groups in poly(hydroxyl amic acid), respectively.

$$ \text{GD(\%)} = \frac{M_p}{M_{100}} \times 100\% = \frac{A_{ac}}{A_{an}} \times \frac{2R_H + 2R_D}{2R_H + R_D} \times 100\% $$

where A_{ac} and A_{an} in the formula represent the area of the ^{1}H-NMR signal of acrylate proton at 6.0–6.8 ppm and the dianhydride aromatic area at

HAB Molar Content, Molecular Weight of the Poly(hydroxyl amic acid), and the Grafting Degree of Acrylate Groups for the Various Poly(hydroxyl amic acid)

Entry	HAB Molar Composition		Yield of co-PHAA[a] (%)	Graft Ratio of Acrylate Groups (%)	Molecular Weight		
	Theor. Value (%)	Exp. Value (%)			M_w	M_n	PDI
BHD	50	56.7	66.1	89.1	19,746	14,319	1.37
BH2D	33	43.7	50.2	83.0	16,418	12,651	1.30
BH4D	25	27.1	43.7	68.6	15,045	11,234	1.26

[a] The yield of PHAA was defined as $(W_{pro} - W_{tot})/W_{tot} \times 100\%$, where W_{pro} and W_{tot} are the weight of the product and that of total monomer used in the PHAA synthesis, respectively.

8.0–8.2 ppm, respectively. The R_H and R_D are the molar content of moieties of HAB and DAP in co-PHAA discussed in the previous section. The results listed in Table I reveal that the degree of grafting acrylate groups to poly(amic acid) was higher than 80%, but, except for BH4D, implying that the number of the photosensitive groups in PAAA increased with the molar content of HAB units in PHAA.

Lithographic evaluation

Figure 3 plots a representative UV–vis spectrum of photosensitive poly(amic acid) in DMAc with a concentration of 1 mg/L at wavelengths of 250–800 nm, showing that the PAAA has a high transparency above 460 nm, and a strong absorption at 250–400 nm. The transparency of PAAA at 365 nm (i-line) is 91%. Thus, these photosensitive poly(amic acids) can be exposed by a i-line and g-line stepper or a broadband source.

The evaluation of solubility of PAAA in the aqueous base solution was made by dissolving the polymer in 2.38% of TMAH. Although the PAAA could be dissolved in the TMAH solution with a dissolu-

tion rate of 0.05 μm/s, the weak adhesion strength between PAAA and the glass substrate led to a delamination after a long developing time. Hence, a mixing solution of TMAH and DMAc that shows higher dissolution rate was utilized as a developer in this work to evaluate the lithographic properties of PAAA. The dissolution rate of these three poly(acrylate amic acids) was determined by measuring the decline in the film thickness at a given development time using an α-step instrument. The results shown in Figure 4 indicated that the BHD exhibited the slowest dissolution rate among the three specimens because of its high molecular weight showing low solubility and the low concentration of hydroxyl groups.

The formulated negative-tone PSPI photoresist, beside the poly(amic acid) resin, consisted of initiators, crosslinkers, and TBPS as photo-sensitizers.[6] Figure 5 plots the characteristic exposure curves of various PSPI and the corresponding properties were listed in Table II. Photosensitivity $(D^{0.5})$, which specifies the speed of how fast the photoresist responds to light, is defined as the exposure dose that retains 50% of the original film thickness after
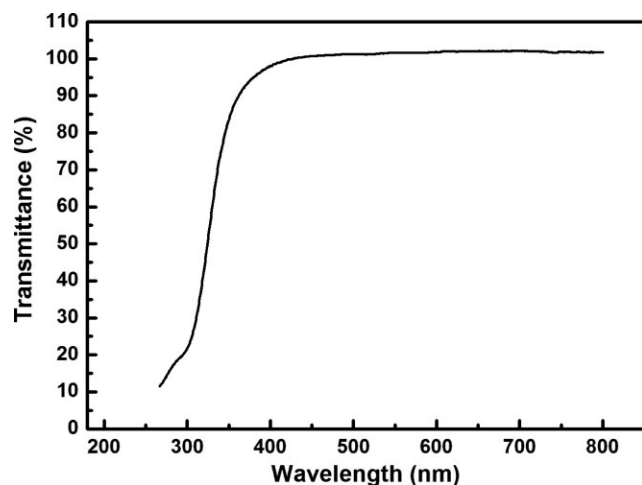

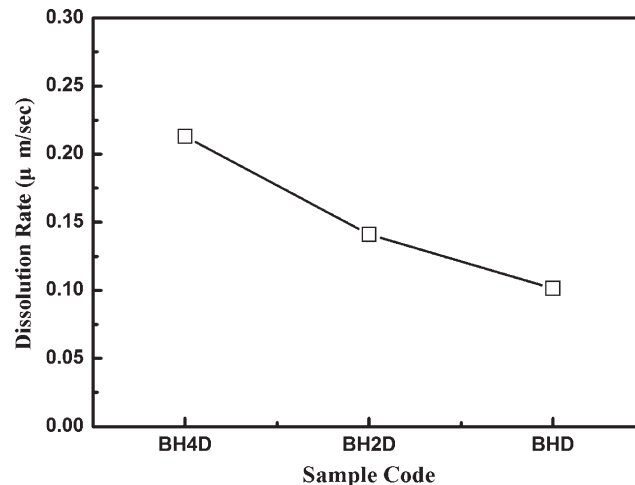**Figure 3** A representative UV–vis spectrum of poly(acrylate amic acid) in DMAc.

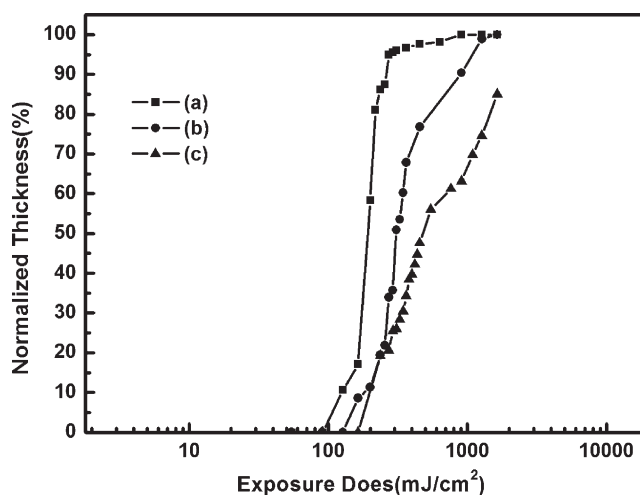**Figure 4** Dissolution rate of the photosensitive poly(amic acid).

Figure 5 Characteristic exposure curve for the PSPI: (a) BHD; (b) BH2D; (c) BH4D.

20μm

Figure 6 Optical micrograph of PSPI (BH2D) after development and thermal curing.

development, and the contrast is determined as the slope of the initial tangent to the characteristic curve. As can be evident, of these three PSPIs, BHD exhibited the highest photosensitivity (200 mJ/cm^2), which increased with the concentration of the acrylate groups attached to the poly(acrylate amic acid). Furthermore, the BH4D, which was the PSPI with the least photo-reactive group, exhibited a 20% film loss after development even though the exposure dose exceeded 1600 (mJ/cm^2) because BH4D cannot generate a full 3D network structure resistant to developer erosion at a reasonably exposed dose. In contrast, BHD containing the most acrylate groups were able to form a compact network structure that exhibited the largest difference between the dissolution in the exposed and the unexposed areas, yielding the highest contrast of 1.78 among the three PSPI.

Figure 6 presents the optical micrograph of the transformed pattern that was converted to the PI through thermal imidization. Table II presents in detail properties of the various PSPI, suggesting that the resolution for BHD that exhibited the best contrast property was worse than that for BH2D and BH4D, which had a 10 μm resolution, because BHD with high molecular weight was associated with a low dissolution rate, such that the nonexposed resins were not completely removed in a reasonable developing period, especially in the fine line region.

CONCLUSIONS

A series of the poly(acrylate amic acid)s were prepared by grafting acrylate groups onto poly(hydroxyl amic acid)s, which were synthesized by the polycondensation of BTDA and a mixture of HAB/DAP with various molar compositions. The HAB moieties in the poly(hydroxyl amic acid) and the degree of grafting of acrylate groups determined from the ^{1}H-NMR spectra indicated that the HAB moieties in the poly(hydroxyl amic acid) exceeded the feedstock content and that the conversion of hydroxyl to acrylate groups increased with number of HAB units in the PHAA, suggesting that HAB exhibited higher polymerization reactivity than DAP, and that the acrylate groups in the poly(acrylate amic acid) increased with the number of the HAB units in the poly(acrylate amic acid). BHD containing the most photo-cured groups had a photosensitivity of 200 mJ/cm^2 and a contrast of 1.78, which decreased as the concentration of the acrylic groups declined. BH2D and BH4D both showed a pattern resolution of 10 μm, but the low dissolution rate of BHD due to its high molecular weight prevented the unexposed resists from being completely stripped within a reasonable development time and made a worse resolution than BH2D and BH4D.

TABLE II
Lithographic Properties of Photosensitive Polyimide

Entry	Photosensitivity (mJ/cm^2)	Contrast	Resolution (μm)
BHD	200	1.78	–
BH2D	310	1.33	10
BH4D	540	0.81	10

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