Synthesis and Characterization of Negative-Type Polyamic Acid Ester with 1-Methacryloyloxy-2-Propanoate Group

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ABSTRACT: A negative-type photosensitive polyamic acid (PAA) was synthesized from cyclobutane-1,2,3,4-tetracarboxylic dianhydride and 2-(methacryloyloxy)ethyl 3,5diaminobenzoate in *N*-methyl-2-pyrrolidinone. Glycidyl methacrylate was added into the PAA solution to yield a photosensitive PAA ester (PAE) by the ring-opening esterification reaction of the carboxylic acid group in the PAA and glycidyl methacrylate. Esterification reactions were conducted with varying reaction temperatures and times. The typical PAE (PAE-C3) with a degree of esterification of 20% was used for a photosensitivity study. We investigated the effects of the postexposure baking temperature, amount of

photoinitiator, and exposure dose on the photosensitivity of PAE-C3. Furthermore, a photolithography evaluation was conducted on PAE-C3 in the presence of 1-[4-(phenylthio)-phenyl]-2-(*O*-benzoyloxime)-1,2-octanedione as a photoinitiator using a mercury lamp at a 365-nm wavelength. The resolution of the film with 2.0- μ m thickness was about 8 μ m. PAE-C3 cured at 250°C for 60 min was stable up to around 310°C in a nitrogen atmosphere. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 100: 2252–2258, 2006

Key words: photosensitive; polyamic acid ester; polyimide; esterification, glycidyl methacrylate

INTRODUCTION

Polyimides have been widely used in liquid crystal displays because of their many desirable properties such as excellent thermal stability, chemical resistance, and low dielectric constant.¹⁻⁶ In particular, photosensitive polyimides⁷ with direct patternability have been favored because they can be used to simplify complicated processes and to reduce the processing cost in the manufacture of thin-film transistor liquid crystal displays or integrated circuits.^{8–11} Therefore, many types of photosensitive polyimides have been developed during the past three decades. Kerwin and Goldrick first reported a photosensitive polyimide in 1971, which included polyamic acid (PAA) and sodium dichromate as a photoreactive group.¹² Commercially available photosensitive polyimides were reported by Rubner et al.¹³ They described a negative-type photosensitive polyimide precursor, in which a photoreactive methacryl group was introduced to the polymer side chain. In contrast, Yoda and Hiramoto reported that a photoreactive methacryl group was incorporated into a PAA by an ionic bond.¹⁴ Other PAAs were synthesized by the reaction of dianhydride and diamine having covalently bonded photoreactive groups.^{15,16} We recently synthesized a negative-type photosensitive PAA with good transmittance at 400 nm for liquid crystal display devices from cyclobutane-1,2,3,4-tetracarboxylic dianhydride (CBDA) and 2-(methacryloyloxy)ethyl 3,5-diaminobenzoate (MEDAB) containing a photosensitive group; we also studied its photosensitivity.¹⁷ As shown in our previous study, the photosensitive PAA showed a resolution of 25 μ m in a 2- μ m thickness film with an exposure dose of 200 mJ/cm² at 365 nm. However, the photolithographic process of the photosensitive PAA required postexposure baking at a high temperature of about 190°C to reduce the carboxylic acid content of the PAA.

In this study we synthesized a PAA ester (PAE) by the addition of a glycidyl methacrylate (GMA) group into the photosensitive PAA to control the content of carboxylic acid groups of the PAA without postexposure baking at high temperature and to increase the photosensitivity of the PAA. We also investigated several factors affecting the photosensitivity of the PAE, which were compared to those of the PAA reported in the previous study.

EXPERIMENTAL

Materials

Maleic anhydride (99%, mp 54°C, Tokyo Kasei Kogyo Co., Ltd.), 3,5-dinitrobenzoyl chloride (98%, Acros Organics), and 2-hydroxyethyl methacrylate (95%, Tokyo

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Kasei Kogyo Co., Ltd.) were used without further purification for the synthesis of monomers. 1-Methyl-2-pyrrolidinone (NMP, 99%, bp 202°C, Kanto Chemical Co., Inc.), GMA (97%, Aldrich Chemical Co.), and triethylamine (98%, Kanto Chemical Co., Inc.) were used without further purification for the synthesis of the photosensitive PAE. The photoinitiator 1-[4-(phenylthio)phenyl]-2-(*O*-benzoyloxime) 1,2-octanedione (PPBO) was purchased from Ciba Specialty Chemicals Inc. and used as received.

Measurements

The ¹H-NMR spectra were taken on a Bruker AMX 300-MHz spectrometer with N,N-dimethylformamide- d_7 (DMF- d_7) and N,N-dimethylsulfoxide- d_6 $(DMSO-d_6)$ as solvents. The FTIR spectra were recorded on KBr pellets with a Bio-Rad Digilab Division FTS-165 spectrometer. Thermogravimetric analysis (TGA) was carried out on a TA Instruments 2950 apparatus at a heating rate of 10°C/min up to 800°C under a nitrogen atmosphere. Mass spectrometry and elemental analysis (EA) were carried out with a JEOL JMS-DX 303 mass spectrometer and a FISONS Instruments EA-1108, respectively. The film thickness was measured with a KLA-Tencor Alpha-Step 500 instrument. UV-visible spectra were recorded on quartz plates using a Scinco UV S-2100. The photoirradiation reaction was performed in the range of 365-400 nm using a 350-W high-pressure mercury lamp, and the exposure dose was measured at 365 nm. The surface profile and solution viscosity were measured with a JEOL JSM-6700F scanning electron microscope and a HAAKE VT 550 cone and plate viscometer, respectively.

Synthesis

Synthesis of CBDA

CBDA was prepared by a [2 + 2] cycloaddition reaction of maleic anhydride in chloroform according to a method previously reported.¹⁸ Yield: 40.0%. mp 112°C. Mass spectrometry m/z (relative intensity): 196 (M⁺). ¹H-NMR (DMF- d_7 , δ): 4.01 (s, cyclobutane, endo form), 4.13 (s, cyclobutane, exo form). ¹³C-NMR (DMF- d_7 , δ): 39.8 (cyclobutane, CH), 172.1 (C=O). ANAL. Calcd for C₈H₄O₆: C, 48.99%; H, 2.06%. Found: C, 48.30%; H, 2.07%.

Synthesis of 2-(methacryloyloxy)ethyl 3,5dinitrobenzoate (MEDNB)

MEDNB was prepared by the condensation of 3,5dinitrobenzoyl chloride (100.0 g, 0.43 mol) and 2-hydroxyethyl methacrylate (56.9 g, 0.43 mol) according to Kikkawa et al.'s method.¹⁵ Yield: 86%. ¹H-NMR (DMSO- d_6 , δ): 9.04 (t, aromatic, 1H), 8.89 (d, aromatic, 2H), 6.05 (d, vinyl, 1H), 5.70 (d, vinyl, 1H), 4.65 (m, methylene, 2H), 4.51 (m, methylene, 2H), 1.87 (s, methyl, 3H).

Synthesis of MEDAB

MEDAB was prepared by the reduction of MEDNB using hydrochloric acid and iron powder according to Kikkawa et al.' method.¹⁵ Yield: 59%. mp 93°C. ¹H-NMR (DMSO- d_6 , δ): 6.41 (d, aromatic, 2H), 6.02 (d, aromatic, 2H), 6.01 (d, vinyl, 1H), 5.68 (d, vinyl, 1H), 4.99 (d, amine protons, 4H), 4.42 (m, methylene, 2H), 4.36 (m, methylene, 2H), 1.85 (s, methyl, 3H). ANAL. Calcd for C₁₃H₁₆N₂O₄: C, 59.08%; H, 6.10%; N, 10.60%. Found: C, 59.38%; H, 6.20%; N, 10.70%.

Synthesis of PAEs

MEDAB (26.4 g, 0.1 mol) was added to NMP (184 g) in the presence of hydroquinone (3 mg, 0.03 mmol) as a radical polymerization inhibitor, and CBDA (19.6 g, 0.1 mol) was slowly added into the mixture. The mixture was stirred for 24 h under a nitrogen atmosphere in an ice bath to give a PAA solution (20 wt % solid content). The resulting viscous solution was poured into excess methanol, and the PAA was isolated by filtration. The isolated PAA powder was washed several times with water and methanol and then dried at 100°C for 12 h in a vacuum.

A typical procedure for the synthesis of a PAE solution from the photosensitive PAA solution is as follows. GMA (0.426 g, 3 mmol) and triethylamine (15 mg, 0.15 mmol) as a catalyst were added to the photosensitive PAA (6.9 g) solution corresponding to a carboxylic acid group (6 mmol). Then, the mixture was stirred for 24 h under a nitrogen atmosphere. This reaction was carried out at 40, 60, 70, and 80°C and yielded PAE-A, PAE-B, PAE-C, and PAE-D solutions, respectively.

Moreover, as shown later, the esterification reaction was carried out at 70°C with reaction times from 3 to 24 h to give respective PAE-C1–PAE-C4 solutions. PAE powder was also obtained by a method similar to that of the PAA. The structural identification of the PAA and the PAE was confirmed by ¹H-NMR spectroscopy, as shown in Figure 1.

Photolithographic process

Change of postexposure baking condition

The PAE-C3 solution synthesized at 70°C for 12 h was used in this study. In order to select a postexposure baking temperature, the PAE-C3 solution containing 3 wt % PPBO to the PAA was spin coated on silicone wafers and prebaked at 90°C for 2 min. After exposure



Chemical shift (δ :ppm)

Figure 1 ¹H-NMR spectra of (a) polyamic acid and (b) polyamic acid ester PAE-C3.

with a dose of 200 mJ/cm², the wafers were postexposure baked at each temperature (110°C, 120°C, 130°C and 140°C) for 10 min and then immersed in a 2.38% tetramethylammonium hydroxide (TMAH) aqueous solution for 5 s. The film thickness was measured before and after immersiong in this aqueous solution.

Change of PPBO amount

The photosensitivity of PAE-C3 was studied by changing the amount of PPBO (1–5 wt %). PAE-C3 solutions containing various amounts of PPBO (1, 2, 3, 4, and 5 wt %) to the PAA were spin coated on silicone wafers. Then, the following steps were performed: they were prebaked at 90°C for 2 min, photoirradiated with an exposure dose of 200 mJ/cm², postexposure baked at 120°C for 10 min, and immersed in 2.38% TMAH for 5 s. The film thickness was measured before and after immersing in the 2.38% TMAH aqueous solution.

Optimization of exposure dose

The photosensitivity of PAE-C3 was studied by changing the exposure dose (20–200 mJ/cm²). The PAE-C3 solution containing 2 wt % PPBO was spin coated on silicone wafers. They were prebaked at 90°C for 2 min, photoirradiated with an exposure dose of 20–200 mJ/ cm², postexposure baked at 120°C for 10 min, and immersed in 2.38% TMAH for 5 s. The film thickness was measured before and after being immersed in the TMAH solution.

Determination of normalized film thickness

The normalized film thickness (d_1/d_0) was determined by dividing the remaining thickness (d_1) of the exposed film after development by the film thickness before development (d_0) . Lithographic imaging

The PAE-C3 solution containing 2 wt % PPBO was spin coated on silicon wafers and prebaked at 90°C for 2 min. The PAE-C3 film was exposed with a dose of 50 mJ/cm² at a 365-nm wavelength through a photomask having various pattern widths, followed by postexposure baking at 120°C for 10 min. The resulting film was developed by immersing it in 2.38% TMAH aqueous solution for 5 s at room temperature and then rinsing with deionized water several times. Finally, the patterned film was thermally cured at 250°C for 60 min in a convection oven.

Measurement of thermal property

The thermal property was measured for the PAE-C3 synthesized at 70°C for 12 h. The PAE-C3 solution containing 2 wt % PPBO was spin coated on two glass plates (25×25 mm) to give two thin films. One film was treated with the following steps: it was prebaked at 90°C for 2 min, photoirradiated with an exposure dose of 50 mJ/cm², and baked at 120°C for 10 min. The other film was treated with the following steps: it was prebaked at 90°C for 2 min, photoirradiated with an exposure dose of 50 mJ/cm², baked at 120°C for 10 min. The other film was treated with the following steps: it was prebaked at 90°C for 2 min, photoirradiated with an exposure dose of 50 mJ/cm², baked at 120°C for 10 min, and cured at 250°C for 60 min. The thermal stability of the two films was investigated with TGA at a heating rate of 10°C/min under a nitrogen atmosphere.

RESULTS AND DISCUSSION

Synthesis of monomers

Alicyclic dianhydride (CBDA) was synthesized by the [2 + 2] cycloaddition of maleic anhydride as shown in Scheme 1(a), and the yield was 40%. Moreover, the chemical structure of CBDA was identified by EA and ¹H-NMR spectroscopy. ANAL Calcd for $C_8H_4O_6$: C,



Scheme 1 The synthesis of monomers (a) cyclobutane-1,2,3,4-tetracarboxylic dianhydride and (b) 2-(methacryloyloxy)ethyl 3,5-diaminobenzoate.

48.99%; H, 2.06%. Found: C, 48.30%; H, 2.07%. ¹H-NMR (DMF- d_7 , δ): 3.89–4.01 (m, cyclobutane, endo form), 4.13 (s, cyclobutane, exo form). MEDAB containing methacrylate moieties was synthesized as shown in Scheme 1(b), and the yield was 59%. The structure of MEDAB was identified by ¹H-NMR spectroscopy. ¹H-NMR (DMSO- d_6 , δ): 6.41 (d, aromatic, 2H), 6.02 (d, aromatic, 2H), 6.01 (d, vinyl, 1H), 5.68 (d, vinyl, 1H), 4.99 (d, amine protons, 4H), 4.42 (m, methylene, 2H), 4.36 (m, methylene, 2H), 1.85 (s, methyl, 3H).

Synthesis of PAEs

Scheme 2 shows the synthetic route of the PAA and PAEs. In the first step, the photosensitive PAA was synthesized from CBDA and MEDAB in NMP. The structural identification of the representative PAA was confirmed by ¹H-NMR spectroscopy, as shown in Figure 1. ¹H-NMR (DMSO- d_{67} δ): 12.7–12.3 (b, carboxylic acid protons, 6H), 10.7-10.3 (m, amide protons, 6H), 8.4-7.9 (m, phenyl ring, 14H), 6.1-6.0 (d, vinyl, 4H), 5.8-5.6 (d, vinyl, 4H). In the second step, the PAEs (PAE-A–D and PAE-C1–C4) were synthesized by ring-opening esterification of the PAA and GMA with varying reaction temperatures and times. As shown in Tables I and II, the degree of esterification increased with increasing reaction temperature and time but the solution viscosity decreased. This is considered as the PAA being depolymerized at a high reaction temperature.¹⁹ The degree of esterification of the PAE-C3 synthesized at 70°C for 12 h was about 20%, which was calculated from the comparison of the proton peak due to the alcohol at 5.2 ppm with the proton

peak due to the aromatic group at 7.9 ppm in the ¹H-NMR spectrum. The structural identification of PAE-C3 was confirmed by comparing it with PAA in the ¹H-NMR spectra, as shown in Figure 1. The proton peak of the alcohol generated by ring-opening esterification of carboxylic acid and GMA newly appeared at 5.2 ppm, and the peak intensity of the carboxylic acid group at 12.7–12.3 ppm was decreased by esterification. Therefore, we confirmed that PAE-C3 was synthesized. A photolithographic evaluation was performed with PAE-C3, which showed a relatively high degree of esterification and a low decrease of the solution viscosity.

Thermal properties

We compared the thermal properties of the photocured PAE-C3 before and after thermal treatment at 250°C for 60 min. As shown in Figure 2, the PAE-C3 cured at 250°C was stable up to around 310°C and its weight loss occurred with one step, which revealed that the imidization reaction of PAE-C3 was almost completed at 250°C for 60 min. In contrast, the PAE-C3 baked at 120°C exhibited a weight loss with two steps. The first weight loss occurred at around 130°C, which might be attributable to removal of the 1-methacryloyl-2-propanate group formed by decomposition of the ester linkage of PAE-C3 and vaporization of unreacted residual GMA. In the first weight loss region the weight loss was about 27%, which might be due to the amount of the GMA (24 wt %) added to the PAA and water formed by the imidization reaction. This result revealed that the GMA was removed and the imidiza-





Scheme 2 The synthesis of (a) polyamic acid and (b) polyamic acid ester.

tion reaction was completed during thermal treatment to yield a thermally stable polyimide film.

Degree of imidization of PAE

The degree of imidization of PAE-C3 was investigated after thermal treatment at 250°C for 60 min and compared to that of the PAE-C3 baked at 120°C as well as to the PAA. The degree of imidization was confirmed by FTIR spectroscopy.¹⁸ The degree of imidization (DI) was calculated using eq. (1),

TABLE I
Effects of Esterification Reaction Temperature on
Solution Viscosity and Degree of Esterification

Polymer solutions ^a	Esterification reaction temp. (°C)	Solution viscosity ^b (cP)	DOE ^c (%)
PAA	_	323	_
PAE-A	40	315	8
PAE-B	60	300	18
PAE-C	70	275	24
PAE-D	80	190	36

^a The reaction time was fixed at 24 h.

^b The solution viscosity was measured with a cone and plate viscometer.

^c The degree of esterification (DOE) was calculated by the comparison of an alcohol proton peak with an aromatic proton peak.

DI (%) =
$$\frac{\left(\frac{A_{1355}}{A_{1460}}\right) PAE-C3}{\left(\frac{A_{1355}}{A_{1460}}\right) PAA}$$
 (1)

where A_{1355} is the absorbance of the characteristic imide II band ($\nu_{C-N'}$ imide II) and A_{1460} is the absorbance of the aromatic ring stretching band (ν_{C-C}). A fully imidized sample was prepared from the PAA imidized at 250°C for 2 h, which was used as a reference film.

As the thermal treatment temperature increased, the intensity of the absorption band at 1355 versus 1460

 TABLE II

 Effects of Esterification Reaction Time on Solution

 Viscosity and Degree of Esterification

Polymer solutions ^a	Esterification reaction time(h)	Solution viscosity ^b (cP)	DOE ^c (%)
PAE-C1	3	327	10
PAE-C2	6	320	15
PAE-C3	12	315	20
PAE-C4	24	275	24

^a The reaction temperature was fixed to 70°C.

^b The solution viscosity was measured with a cone and plate viscometer.

^c The degree of esterification (DOE) was calculated by the comparison of an alcohol proton peak with an aromatic proton peak.



Figure 2 TGA curves of polyamic acid ester PAE-C3 (a) cured at 250°C for 60 min and (b) baked at 120°C for 10 min.

cm⁻¹ was increased. That is, the degree of imidization of PAE-C3 was increased from 7.1 to 89.8% by the thermal treatment, which means that curing at 250°C made PAE-C3 a thermally stable polyimide film.

Photosensitive properties

In this study we investigated several factors affecting the photosensitivity of the PAE.

Effect of postexposure baking temperature

We studied the effects of varying postexposure baking temperatures on the normalized film thickness of exposed and unexposed areas. Exposed areas of the film were not dissolved in films baked above 120°C and unexposed areas of the film were completely dissolved in films baked below 140°C, both of which are shown in Figure 3. Therefore, we selected postexposure baking temperatures of lower and broader ranges (120–140°C) than that of the photosensitive PAA (190°C).



Figure 3 The effect of the postexposure baking temperature on the normalized film thickness of polyamic acid ester PAE-C3 in (a) an exposed area and (b) an unexposed area.



Figure 4 The effect of the amount of photoinitiator on the normalized film thickness of polyamic acid ester PAE-C3.

Effect of amount of PPBO

Figure 4 shows the effect of the amount of PPBO on the normalized film thickness. The normalized film thickness increased with increasing amounts of PPBO. The PAE-C3 containing 2 wt % PPBO is considered to be the most suitable condition when exposed to a dose of 200 mJ/cm² and postexposure baked at 120°C. Therefore, PAE-C3 required a smaller amount of PPBO than the photosensitive PAA containing 3 wt % PPBO.

Effect of exposure dose

The photosensitivity of PAE-3 was studied with various exposure doses (20–200 mJ/cm²) that are shown in Figure 5. The normalized film thickness increased with increasing exposure dose, and the optimum exposure dose was 50 mJ/cm². Therefore, PAE-C3 showed better photosensitivity than photosensitive PAA, which was reported as 200 mJ/cm².¹⁹

Lithographic evaluation

In our previous study on a photosensitive PAA, the resolution of the pattern was about 25 μ m in 2.0 μ m



Figure 5 The effect of the exposure dose on the normalized film thickness of polyamic acid ester PAE-C3.





(b)

Figure 6 The pattern profile of polyamic acid ester PAE-C3 measured by (a) scanning electron microscopy and (b) an Alpha-Step 500; resolution = 8 μ m.

thick film. The photolithography evaluation was performed using the PAE-C3 solution containing 2 wt % PPBO under the following processing conditions optimized in this study: it was prebaked at 90°C for 2 min, photoirradiated with an exposure dose of 50 mJ/cm² at 365 nm, baked at 120°C for 10 min, and immersed for 5 s in 2.38% TMAH; then, the imidization reaction was performed at 250°C for 60 min. The pattern resolution of PAE-C3 was evaluated with an Alpha-Step 500 and scanning electron microscopy. Figure 6 shows the representative line-pattern profile. The pattern resolution of an about 2.0 μ m thick film was about 8 μ m. Therefore, the PAE-C3 showed better resolution than the photosensitive PAA.¹⁹

CONCLUSION

Negative-type photosensitive PAEs with a 1-methacryloyloxy-2-propanoate group were synthesized by ringopening esterification of PAA prepared from CBDA and MEDAB in NMP. The photosensitivity was studied for PAE-C3, which showed a high degree of esterification and a low decrease of the solution viscosity. We confirmed the optimum processing conditions as the following: prebaking at 90°C for 2 min, photoirradiating with an exposure dose of 50 mJ/cm² at 365 nm, baking at 120°C for 10 min, and immersing in 2.38% TMAH for 5 s; then, the imidization reaction was performed at 250°C for 60 min. Moreover, photolithographic evaluation of PAE-C3 was performed and its pattern resolution was 8 μ m in an approximate 2.0 μ m thick film.

In conclusion, the photosensitive PAE prepared in this study was found to be a negative-type photosensitive precursor with better photosensitivity compared to that of photosensitive PAA. It can also be applied as a material for liquid crystal display devices such as interlayer insulators, outer coatings, and column spacers, which require good photosensitivity at low exposure energy, high thermal stability, as well as good transmittance at 400 nm.

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