Polymer Journal, Vol. 39, No. 3, pp. 245–251 (2007)
©2007 The Society of Polymer Science, Japan

Photosensitive Poly(benzoxazole) Based on Poly(*o*-hydroxy amide), Dissolution Inhibitor, Thermoacid Generator, and Photoacid Generator

Tomohito OGURA, Ko-ta YAMAGUCHI, Yuji SHIBASAKI, and Mitsuru UEDA†

Department of Organic & Polymeric Materials, Graduate School of Science & Engineering, Tokyo Institute of Technology, 2-12-1-H120 O-okayama, Meguro-ku, Tokyo 152-8552, Japan

(Received November 28, 2006; Accepted December 23, 2006; Published February 9, 2007)

ABSTRACT: A positive-type photosensitive polybenzoxazole (PSPBO) based on a poly(*o*-hydroxy amide) (PHA), a dissolution inhibitor (DI) 9,9-bis(4-*tert*-butoxycarbonyloxyphenyl)fluorene (*t*-BocBHF), a thermoacid generator (TAG) isopropyl *p*-toluenesufonate (ITS), and a photoacid generator (PAG) (5-propylsulfonyloxyimino-5*H*-thiophene-2-ylidene)-(2-methylphenyl)acetonitrile (PTMA) has been developed. ITS was easily prepared by reaction of *p*-toluenesulfonyl chloride with isopropyl alcohol. The dissolution behavior of the PSPBO system was studied in relation to PTMA loadings, and post exposure baking (PEB) temperature and time. The PSPBO consisting of PHA (73 wt %), *t*-BocBHF (18 wt %), ITS (7.5 wt %), and PTMA (1.5 wt %) exhibited a sensitivity of 33 mJ/cm^2 and a contrast of 5.1 when exposed to 365 nm light (*i*-line) and developed with an aqueous alkaline developer, 2.38 wt % tetramethylammonium hydroxide solution (TMAHaq)/2.5 wt % *iso*-propanol (*i*-PrOH). A clear positive image with 3 µm features was produced by contact-printing and converted into a poly(benzoxazole) (PBO) pattern upon heating at 250 °C for 20 min. Thus, the ITS is effective as a TAG for improving the sensitivity and low temperature cyclization of PHA into the PBO as well to meet practical requirement in the industry. [doi:10.1295/polymj.PJ2006174]
KEY WORDS Functional Polymers / Polymer Synthesis / Gel / Tetrahydrofuran / Infrared Spectroscopy /

Photosensitive polybenzoxazoles (PSPBOs)[1–5] are used as buffer coatings to protect bare chips from stresses induced by fillers or thermal mismatches between a passivation layer and molding materials. These compounds possess high mechanical strength, thermal stability, and low dielectric constants. They have been developed to simplify processes in which a precursor of polybenzoxazoles (PBOs), poly(*o*-hydroxy amide) (PHA), is used as a polymer matrix. The phenolic hydroxyl groups in PHA promote solubility of 2.38 wt % tetramethylammonium hydroxide solution (TMAHaq), used as the aqueous alkaline developer. Previously, PHA obtained from 4,4′-(hexafluoroisopropylidene)bis(*o*-aminophenol) (6FAP) and 4,4′-oxybis(benzoic acid) derivatives was used because of its high transparency at a wavelength of 365 nm (*i*-line). A conventional PSPBO was formulated by adding the photo-sensitizer diazonaphthoquinone (DNQ) to PHA.[6]

The sensitivity of photopolymers is important in the design of photoresist materials. The conventional PSPBO based on DNQ, however, possesses low sensitivity (>100 mJ/cm^2) and a strong absorbance at 365 nm, and is difficult to use for thick patterns. To overcome these problems, a chemically amplified technique was introduced to develop PSPBOs coupled with a photoacid generator (PAG),[7] for which 9,9-bis(4-*tert*-butoxycarbonyloxyphenyl)fluorene (*t*-BocBHF) was used as a dissolution inhibitor (DI).[8] This three-component, positive-type, alkaline-developable PSPBO showed the sensitivity and contrast of 34 mJ/cm^2 and 5.8, respectively with *i*-line exposure, to produce a 20 µm image in 10 µm thick film. A positive image of PHA is converted into that of PBO by thermal treatment at 350 °C. This high-temperature process is not applicable to conventional electronic applications containing one or more thermally unstable organic components, such as glass-epoxy resin or glass-bis(maleimide)-triazine resins that may be present in a built-up or package board. Thus, a significant decrease in the cyclization temperature of PHA allows versatile development of a PSPBO system, producing more useful applications in the microelectronics industry.

Recently, sulfonic acids were found to work as effective catalysts for low-temperature cyclization of PHA.[9] This finding was applied to develop a low-temperature curable PSPBO coupled with a PAG [5-propylsulfonyloxyimino-5*H*-thiophene-2-ylidene-(2-methylphenyl)acetonitrile] (PTMA), for which DNQ or partially *tert*-butoxycarbonyl (*t*-Boc)-protected PHA was used as the DI or polymer matrix.[10,11] The former PSPBO, however, cannot be applied for thick patterns, and *t*-Boc groups must be introduced to PHA through

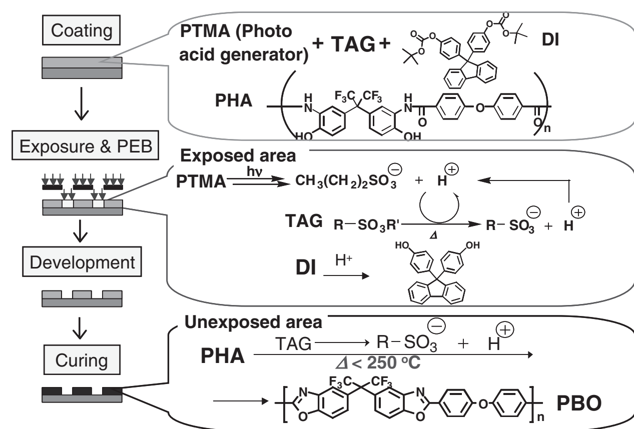
†To whom correspondence should be addressed (Tel/Fax: +81-3-5734-2127, E-mail: mueda@polymer.titech.ac.jp).

polymeric reactions in the latter PSPBO, which is troublesome. A more convenient method that is widely applicable and provides easy resist formulation involves a four-component system consisting of PHA, a PAG, a DI, and a thermoacid generator (TAG) based on acid-labile sulfonates.

For practical use of these acids, development of latent acid catalysts for low-temperature cyclization of PHA is more desirable. Latent catalysts are compounds stable under ambient conditions, which function only after specific external stimulation such as heating or photo-irradiation. Sulfonate derivatives are known to function as latent acid generators both by thermal treatment and photo irradiation.[12,13] Recently, Arimitsu *et al.* demonstrated a novel photoproliferation for a chemically amplified photoresist in order to improve the sensitivity.[14] We anticipated that such a latent thermo-sensitive acid catalyst will enhance both storage stability and processability of PHA solutions because of non-reactivity at storage temperatures, while the cyclization of PHA is promoted within a specific range of elevated temperatures.

This report describes the successful development of an alkaline-developable, chemically amplified positive-type PSPBO based on a PHA, DI (*t*-BocBHF), TAG (isopropyl *p*-toluenesufonate, ITS), and PAG (PTMA). A patterning process and subsequent low-temperature cyclization using PSPBO is shown in Scheme 1.

The formulation of PSPBO is simple, involving the addition of *t*-BocBHF, ITS, and PTMA to a PHA solution. The PSPBO solution is spin-coated and baked in the usual way. Then, the film is exposed to UV light to produce propanesulfonic acid from PTMA. Upon post-exposure bake (PEB) treatment of the PSPBO film, propanesulfonic acid catalyzes decomposition of ITS to yield propene and *p*-toluenesulfonic acid. These sulfonic acids induce a cascade of deprotection reactions of *t*-BocBHF to produce 9,9-bis(4-hydroxyphenyl)fluorene (BHF). The exposed and baked film then can be developed with 2.38 wt % TMAHaq to provide a positive image. The subsequent low-temperature cyclization into the PBO pattern at 250 °C proceeds with *p*-toluenesulfonic acid produced from thermal decomposition of ITS. As a consequence, the direct formulation of chemically amplified PSPBO simplifies the photolithographic process, and the following thermal treatment at 250 °C will promote PSPBO for a variety of applications.



Scheme 1. Patterning process and subsequent low-temperature cyclization using positive-type PSPBO.

EXPERIMENTAL

Materials

The PHA derived from 6FAP and 4,4′-oxybis(benzoic acid) derivatives was prepared as described previously.[10] The number- and weight-average molecular weights (M_n and M_w) of PHA were 7,400 and 16,000 (M_w/M_n 2.2), respectively, as measured by gel permeation chromatography (GPC) with polystyrene standards. The photo-acid generator PTMA was donated by Ciba Specialty Chemicals and stored in a refrigerator. *t*-Boc BHF was prepared from BHF and di-*tert*-butyl dicarbonate.[8] 2-Methoxyethanol and other chemicals were used as received unless otherwise noted.

Synthesis of Isopropyl Methanesufonate (IMS).[15] Pyridine (8.15 mL, 100 mmol) was added dropwise over 15 min to a solution of isopropanol (3.83 mL, 50.0 mmol) and methanesulfonyl chloride (3.23 mL, 41.7 mmol) in dichloromethane (100 mL) at 0 °C. The resulting solution was stirred at 0 °C for 1.5 h, then at room temperature for 1.5 h, followed by dilution with dichloromethane (200 mL). The solution was washed successively with 1 M hydrochloric acid solution and aqueous 20% potassium hydrogen carbonate solution, dried over anhydrous magnesium sulfate, filtered, and concentrated at reduced pressure using a rotary evaporator to afford a oil which was distilled under reduced pressure to yield IMS (3.1 g, 53%) as a clear, colorless liquid. Infrared (IR) (KBr, ν, cm^{-1}): 1350 and 1176 cm^{-1} ($-SO_3-$). ^{1}H NMR ($CDCl_3$, δ, ppm): 4.98 (m, 1H, C$\underline{H}$(CH_3)$_2$), 3.02 (s, 3H,CH_3), 1.45 (d, 6H, C$\underline{H}$(C$\underline{H}_3$)$_2$).

Synthesis of Isopropyl p-Toluenesufonate (ITS).[16] This compound was prepared from isopropanol (8 mL, 104 mmol) and *p*-toluenesulfonyl chloride (8 mL, 104 mmol) as described above. The product was a clear, colorless liquid (5.50 g, 49%). IR (KBr, ν, cm^{-1}): 1361 and 1176 cm^{-1} ($-SO_3-$). ^{1}H NMR ($CDCl_3$, δ, ppm): 7.81 (d, 2H, ArH), 7.36 (d, 2H, ArH), 4.76 (m, 1H, C$\underline{H}$(CH_3)$_2$), 2.47 (s, 3H, CH_3), 1.29 (d, 6H, C$\underline{H}$(C$\underline{H}_3$)$_2$).

Thermal Decomposition of TAGs

0.5 g of IMS or ITS was placed in a flask, heated at 100, 125, and 150 °C under nitrogen for 1, 5, 10, and 20 min. The aliquot of each sample was transferred into a NMR tube, dissolved in $CDCl_3$, and the degree of the decomposition was determined from the integral ratio of the methyl group of IMS or ITS and that of the corresponding sulfonic acid.

Degree of Cyclization from PHA into PBO

The PHA or the photosensitive polymer film spin-cast on a silicon wafer was baked at 100 °C for 5 min as a mimic of PEB. The thickness of each film was approximately 1.2 µm. The film was heated on a hotplate at a series of temperatures from 150 to 250 °C for 15 min each. The PBO film was prepared by heating at a temperature up to 350 °C; the film was held at that temperature for 1 h under nitrogen as a reference. Absorption intensities on the IR spectrum at 1045 cm^{-1} (A_{1045}) assignable to C–O stretch of benzoxazole group and at 609 cm^{-1} (A_{609}) assignable to silicon wafer as invariable standard was obtained, and the degree of cyclization was determined using the following eq 1,[11]

$$\text{Cyclization [\%]} = (A_{1045}/A_{609[\text{samp}]} - A_{1045}/A_{609[\text{init}]}) / (A_{1045}/A_{609[\text{PBO}]} - A_{1045}/A_{609[\text{init}]}) \times 100, \quad (1)$$

where the subscripts between the brackets following A_{1045}/A_{609} indicate the state of the film; *e.g.*, [samp] is the polymer sample at each heating temperature level (150–250 °C); [init] is initially pre-baked polymer film at 110 °C in air; [PBO] is the fully cured PBO at 350 °C for 1 h under nitrogen.

Dissolution Rate

DI, TAG, and PTMA were added to a PHA solution in cyclohexanone to construct a photosensitive polymer. The polymer film was spin-cast from the solution (15 wt % concentration) on a silicon wafer and pre-baked at 100 °C for 5 min, then exposed to a filtered super-high-pressure mercury lamp at 365 nm (*i*-line), followed by post-exposure baking at 80–140 °C for 3 min. The exposed film was developed with 2.38 wt % TMAHaq/2.5 wt % *iso*-propyl alcohol (*i*-PrOH) at 25 °C to determine dissolution rate (Å/sec).

Photosensitivity

The polymer film of 1.2-µm thickness on a silicon wafer was exposed to light at a wavelength of 365 nm, developed with 2.38 wt % TMAHaq/2.5 wt % *i*-PrOH at 25 °C for 33 s, and rinsed in water. A characteristic curve was obtained by plotting normalized film thickness as a function of exposure dose (mJ/cm^2). The image exposure was obtained as a contact print.

Measurements

IR spectra were obtained using a Horiba FT-720 spectrophotometer. The ^{1}H NMR spectra were recorded on a Bruker GPX300 (300 MHz) spectrometer. Number- and weight-averaged molecular weights (M_n and M_w) were determined by GPC using a Hitachi LaChrom with two polystyrene gel columns (TSK GELs; GMH_{HR}-M) at 40 °C in tetrahydrofuran (THF) at a flow rate of 1.0 mL/min, calibrated with polystyrene standards. The film thickness on silicon wafers was measured with a Veeco Instrument Dektak[3] surface profiler. Scanning electron microscope (SEM) photos were taken with a Technex Lab Tiny SEM 1540 instrument with 15 kV accelerating voltage for imaging. Pt/Pd was spattered on a film prior to obtaining the SEM pictures.

RESULTS AND DISCUSSION

Synthesis of TAG

TAG is required for compatibility with PHA and good thermal stability during pre-baking treatment. It decomposes near 150 °C to produce a sulfonic acid. While TAG decomposes easily with a photo-generated acid, the sensitivity of PSPBO is improved with a small amount of PAG. To satisfy these requirements for TAG, its design was based on sulfonate compounds able to decompose thermally with the production of stable carbocation species, giving olefins and sulfonic acids. TAGs, IMS, and ITS were prepared by reaction of isopropyl alcohol with methanesulfonyl chloride and *p*-toluenesulfonyl chloride in the presence of pyridine in dichloromethane.

Thermal Decomposition of TAGs

The relation between the decomposition percentage of TAGs and heating time at each temperature is shown in Figures 1 and 2. The thermal decomposition behavior of TAGs was followed by ^{1}H NMR spectroscopy. IMS is stable at 100 °C, and decomposes completely upon thermal treatment at 125 °C for

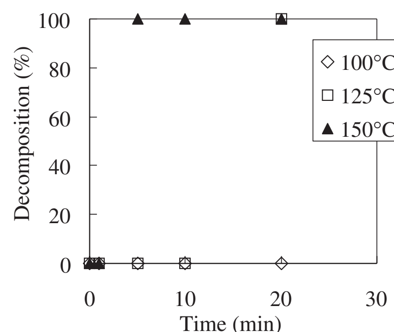


Figure 1. Relationship between the decomposition percentage of IMS and the heating time at each temperature.

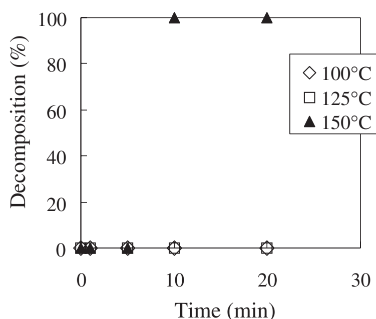


Figure 2. Relationship between the decomposition percentage of ITS and the heating time at each temperature.

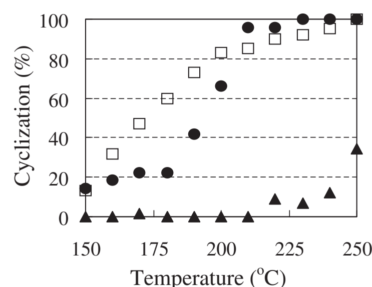


Figure 4. Degree of cyclization of PHA containing 10 wt % TAGs into PBO at different temperatures. IMS (□), ITS (●), and no-additive (▲).

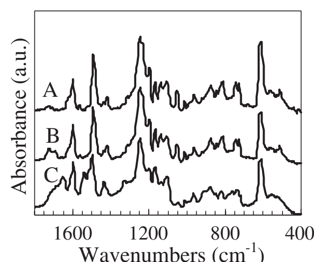


Figure 3. FT-IR spectra of PHA films containing 10 wt % IMS on a silicon wafer: (A) reference film baked at 350 °C for 1 h without IMS, (B) baked film at 250 °C for 15 min, (C) as-made film.

20 min. At 150 °C, complete decomposition occurred within 5 min. In contrast, ITS was more stable than IMS, and decomposed completely within 10 min at 150 °C. No mixture of TAGs and the corresponding sulfonic acids was observed in the ^{1}H NMR spectra, which suggests that the thermo-generated sulfonic acid promotes decomposition of TAGs.

Degree of Cyclization of PHA into PBO by IR Spectroscopy

The effect of TAGs on low-temperature cyclization of PHA to PBO was investigated using IR spectroscopy. The PHA solutions containing 10 wt % TAGs in 2-methoxyethanol were spin-coated on a silicon wafer, pre-baked at 100 °C for 5 min, and heated at each temperature for 15 min. A fully cured PBO film was prepared for each sample by increasing the temperature to 350 °C and then holding it for 1 h under nitrogen. IR spectra of the film are shown in Figure 3 after pre-baking and heating at 250 or 350 °C (as the reference). At a temperature of 250 °C or higher, the spectra exhibit characteristic absorption bands at 1045 and 1620 cm^{-1} corresponding to a benzoxazole ring. A band at 1654 cm^{-1} due to the amide group and a band near 3400 cm^{-1} due to the hydroxyl group of PHA disappear. Eventually, the IR spectrum obtained after thermal treatment at 250 °C for 15 min is identical to that of a fully cured PBO film (350 °C for 1 h). Thus, PHA is readily converted to the corresponding PBO in the presence of 10 wt % TAG. The degree of cyclization in the presence of ITS is less than that using IMS in the temperature range of 150–200 °C, but becomes greater than that using IMS above 210 °C (Figure 4). This behavior may be explained by high mobility at low temperatures and easy volatilization at higher temperatures of IMS compared to ITS. Based on these results, ITS was selected as the TAG for further investigation. The mechanism of acid-catalyzed cyclization involved sulfonic acid acceleration of the phenolic hydroxyl group attack on an amide carbonyl unit through protonation of the oxygen of the amide group. The degree of cyclization was calculated from the eq 1 as described in the Experimental section and is shown in Figure 4. An absorbance band at 1045 cm^{-1} (A_{1045}) assignable to C–O stretching of a benzoxazole group occurred in the IR spectrum upon an increase in temperature, while the band at 609 cm^{-1} (A_{609}) assignable to the silicon wafer was used as a non-interactive standard.

Lithographic Evaluation of the PHA/DI/TAG/PAG System

The PHA matrix polymer was prepared from 6FAP and 4,4′-oxybis(benzoyl chloride) according to a method reported previously.[9] The M_n and M_w of PHA were 7,400 and 16,000, respectively.

Preliminary optimization studies of processing conditions and composition ratios of DI, TAG, and PAG to PHA for formulation of new PSPBOs were conducted using *t*-BocBHF and PTMA as the DI and a PAG, respectively, according to a previous report.[8] Films were obtained by spin-casting a diluted solution of PHA, DI, TAG, and PTMA in cyclohexanone on a silicon wafer, and then pre-baked at 100 °C for 5 min in air. This photosensitive polymer film was irradiated with UV light (100 mJ/cm^2) at 365 nm (*i*-line) using a filtered super-high-pressure mercury lamp, baked after exposure at a set temperature for 3 min, and developed with TMAHaq/2.5 wt % *i*-PrOH at 25 °C.

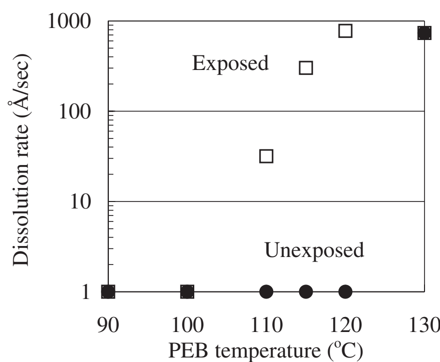


Figure 5. Effect of PEB temperature on dissolution rate for PHA/*t*-Boc BHF/ITS/PTMA (73/18/7.5/1.5 wt/wt/wt/wt) resist system in exposed (□) and unexposed (●) areas. The *i*-line exposure and PEB time were fixed at 100 mJ/cm^2 and 3 min, respectively.

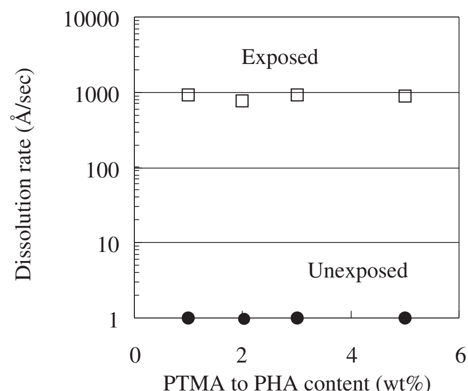


Figure 7. Effect of PTMA loading on PHA for dissolution rate for PHA/*t*-Boc BHF/ITS/PTMA resist system in exposed (□) and unexposed (●) areas. The *i*-line exposure, PEB temperature, and time were 100 mJ/cm^2, 120 °C, and 3 min, respectively.

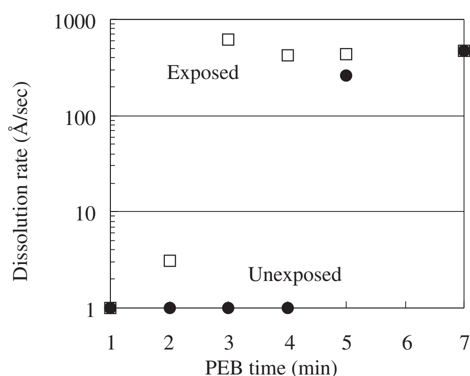


Figure 6. Effect of PEB time on dissolution rate for PHA/*t*-Boc BHF/ITS/PTMA (73/18/7.5/1.5 wt/wt/wt/wt) resist system in exposed (□) and unexposed (●) areas. The *i*-line exposure and PEB temperature were fixed at 100 mJ/cm^2 and 120 °C, respectively.

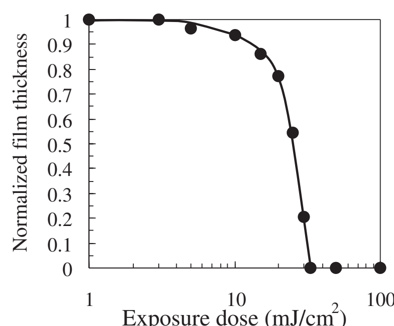


Figure 8. Characteristic photosensitive curve for the PHA/*t*-Boc BHF/ITS/PTMA (73/18/7.5/1.5 wt/wt/wt) resist system. The PEB temperature and time were 120 °C and 3 min, respectively.

Post-exposure baking (PEB) temperature is crucial for chemically amplified resist systems because diffusion of the generated acids in the films is a key process. To clarify the dissolution behaviors of PSPBO films containing *t*-Boc BHF, ITS, and PTMA in both exposed and unexposed areas, the dissolution rate was estimated by the change in film thickness before and after development. As shown in Figure 5, a large difference in dissolution (dissolution contrast, DC) of approximately 800 times between the exposed and unexposed areas in 2.38 wt % TMAHaq/2.5 wt % *i*-PrOH was obtained for PEB at a temperature range of 120 °C, indicating that the photo-generated acid decomposes *t*-Boc BHF effectively to give BHF by PEB treatment. PEB temperatures greater than 130 °C may induce thermal decomposition of *t*-Boc BHF, resulting in greater solubility in 2.38 wt % TMAHaq/2.5 wt % *i*-PrOH.

The effect of PEB time on dissolution rate of the film was investigated as shown in Figure 6. PEB times of 3–4 min were adequate to produce a large DC between exposed and unexposed areas. Further PEB treatment may induce decomposition of TAG, resulting in no DC between the exposed and unexposed areas.

The effect of PTMA loading of PSPBO on the dissolution rate of the film is shown in Figure 7. Even with 1 wt % loading of PTMA to PSPBO, a large DC was obtained, indicating that the photo-generated acid catalyzes a cascade of ITS decomposition, followed by effective deprotection of *t*-Boc BHF by PEB.

Based on the studies involving PTMA loadings, and PEB temperature and time, a PSPBO consisting of PHA (73 wt %), *t*-Boc BHF (18 wt %), ITS (7.5 wt %), and PTMA (1.5 wt %) was formulated. The photosensitivity curve of resist films 1.2 µm thick is shown in Figure 8. The resist containing *t*-Boc BHF and ITS possessed outstanding sensitivity (D_0) of 33 mJ/cm^2, and good contrast (γ_0) of 5.1.

Figure 9 shows the scanning electron micrograph of a contact-printed image obtained with the system de-

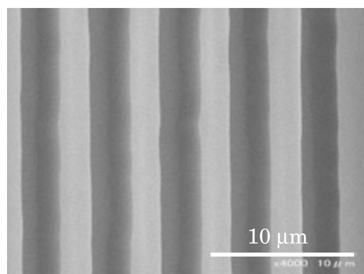


Figure 9. SEM image of positive-pattern 3-μm-thick film based on PHA/*t*-Boc BHF/ITS/PTMA (70.4/21.1/7.1/1.4 wt/wt/wt/wt).

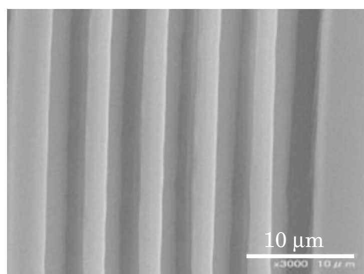


Figure 10. SEM image of PBO pattern cured at 250 °C for 20 min under nitrogen.

scribed: the resist layer was exposed to 50 mJ/cm^2, post-baked at 115 °C for 3 min, and developed with 2.38 wt % TMAHaq/2.5 wt % *i*-PrOH at 25 °C. A clear, positive pattern with an 3 μm feature was observed using a 3 μm thick film.

Low-Temperature Cyclization of PHA

The printed pattern was converted to a PBO pattern without deformation by heating at temperatures up to 150 °C for 10 min and 250 °C for 20 min under nitrogen (Figure 10). The formation of PBO was confirmed by IR spectroscopy, in which the characteristic oxazole ring absorption appeared at 1616 cm^{-1} and absorptions due to hydroxyl and carbonyl groups at 3300 and 1650 cm^{-1}, respectively, in the PHA spectrum disappeared.

The resist system was used to produce a thick pattern. The 20-μm image was made in 10 μm thick film by PEB at 115 °C for 3 min after exposure to 70 mJ/cm^2 of *i*-line, followed by developing with 2.38 wt % TMAHaq/2.5 wt % *i*-PrOH at 25 °C for 110 s. Adequate resolution and edge sharpness can be seen in Figure 11.

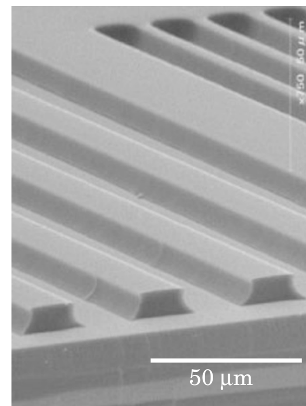


Figure 11. SEM image of positive-pattern 10-μm-thick film based on PHA/*t*-Boc BHF/ITS/PTMA (70.4/21.1/7.1/1.4 wt/wt/wt/wt).

CONCLUSION

Two TAGs (IMS and ITS), which improve the sensitivity and transparency at the *i*-line region of PSPBO, and accomplish low-temperature cyclization of PHA to PBO, were prepared from isopropyl alcohol and methanesulfonyl chloride or *p*-toluenesulfonyl chloride. ITS satisfied the requirements for TAG, and was used for formulation of a positive-type alkaline developable PSPBO based on a PHA, *t*-Boc BHF, and PTMA. The new resist system showed high sensitivity and contrast of 33 mJ/cm^2 and 5.1, respectively, with *i*-line exposure. Furthermore, the clear positive image after development was converted to a PBO image by low-temperature treatment. A thick pattern also was obtained with the PSPBO system. This new formulation method provides a more efficient and versatile process compared to the standard route that requires large exposure doses and high cyclization temperatures.

Acknowledgment. This work was financially supported by a Grant-in-Aid for Science Research (No. 18350059) from the Japanese Ministry of Education, Science, Sports, and Culture, which is gratefully acknowledged.

REFERENCES

1. R. Rubner, *Adv. Mater.*, **2**, 452 (1990).
2. H. Ahne, R. Rubner, and R. Sezi, *Appl. Surf. Sci.*, **106**, 311 (1996).
3. D. N. Khanna and W. H. Muller, *Polym. Eng. Sci.*, **29**, 954 (1989).
4. T. Banba, E. Takeuchi, A. Tokoh, and T. Takeda, *Proc. Electr. Compon. Cof IEEE.*, 564 (1991).
5. R. Rubner, *J. Photopolym. Sci. Technol.*, **17**, 685 (2004).
6. K. Fukukawa and M. Ueda, *Polym. J.*, **38**, 405 (2006).
7. K. Fukukawa, Y. Shibasaki, and M. Ueda, *Polym. J.*, **37**, 74 (2005).
8. T. Ogura and M. Ueda, *J. Polym. Sci., Part A: Polym. Chem.*, in press (2006).
9. F. Toyokawa, K. Fukukawa, Y. Shibasaki, and M. Ueda, *Chem. Lett.*, **33**, 1342 (2004).
10. F. Toyokawa, K. Fukukawa, Y. Shibasaki, and M. Ueda,

Polym. J., **37**, 517 (2005).
11. K. Fukukawa and M. Ueda, *Macromolecules*, **39**, 2100 (2006).
12. G. G. Barclay, D. R. Medeiros, and R. F. Sinta, *Chem. Mater.*, **7**, 1315 (1995).
13. S. Matuszczak, J. F. Cameron, J. M. J. Frechet, and C. G. Wilson, *J. Mater. Chem.*, **1**, 1045 (1991).
14. K. Arimitsu, K. Kudo, and K. Ichimura, *J. Am. Chem. Soc.*, **120**, 37 (1998).
15. P. N. Culshaw and J. C. Walton, *Tetrahedron Lett.*, **31**, 2457 (1990).
16. J. Granander, R. Sott, and G. Hilmersson, *Tetrahedron*, **58**, 4717 (2002).