

# Synthesis of a Photo-Patternable Cross-linked Epoxy System Containing Photodegradable Carbonate Units for Deep UV Lithography

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**ABSTRACT:** Bis(2-(oxiran-2-ylmethyl)-1,3-dioxoisindolin-5-yl) carbonate and polymers containing 9-anthracenylmethylmethacrylate (AMMA), *p*-*tert*-butoxy styrene (PTBS), and methacrylic acid (MAA) monomeric units were synthesized with the aim of developing a novel photo-patternable cross-linked epoxy system. The oxirane groups in bis(2-(oxiran-2-ylmethyl)-1,3-dioxoisindolin-5-yl) carbonate were reacted with the carboxylic acid in the polymer to generate a cross-linked epoxy film, and the photo degradation of the cross-linked film was achieved through decomposition of the

carbonate groups in the cross-linked film by deep UV irradiation. Because the copolymer containing anthracene groups has relatively high reflective index and absorption at 248 nm, this cross-linked system can be applied to patternable bottom antireflective coating materials for deep UV lithography applications. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 114: 2093–2100, 2009

**Key words:** oxirane; carbonate; cross-linking; degradation; photoresist

## INTRODUCTION

Epoxy resins are used widely to increase the toughness of adhesives, inks, information storage systems, and lithographic materials because they form insoluble and infusible networks through cross-linking processes.<sup>1–3</sup> The insoluble and infusible properties of epoxy resins are valuable for protecting underlying coated materials on substrates particularly when further organic processes on this coating are needed. However, this intractability can be significantly disadvantageous when the network needs to be removed because harsh treatments, such as mechanical scratching or strong acid treatment methods must be applied. Several chemical processes have been developed to dissolve or decompose the cross-linked networks by incorporating some degradable functional groups.<sup>4–8</sup> For example, Sastri and Tesoro reported cross-linking systems containing a disulfide group that can be decomposed by a thiol treatment.<sup>9,10</sup> Buchwalter reported cycloaliphatic epoxy

systems with acetal that can be dissolved easily in an acid contained solution.<sup>11</sup> It is thought that if the cross-linked epoxy coating materials containing photo degradable functional groups are prepared, they can be used as coating materials which is stable for further organic coating processes and also can be patterned using a photolithography process.

For this purpose, a new cross-linked epoxy system was made, which was composed of a carbonate linkage as a photodegradable unit,<sup>12</sup> an anthracene group for antireflective chromophor,<sup>13</sup> and a protected hydroxyl group (Fig. 1). This system could be prepared by a reaction between the oxirane groups in bis(2-(oxiran-2-ylmethyl)-1,3-dioxoisindolin-5-yl) carbonate and the carboxylic acid groups in the polymers. The detailed synthetic procedures of these compounds and the photo degradation process of the epoxy system are discussed in this article. A preliminary experiment for a micron size pattern through deep UV irradiation was also performed for the possible future applications as a patternable bottom antireflective coating material.

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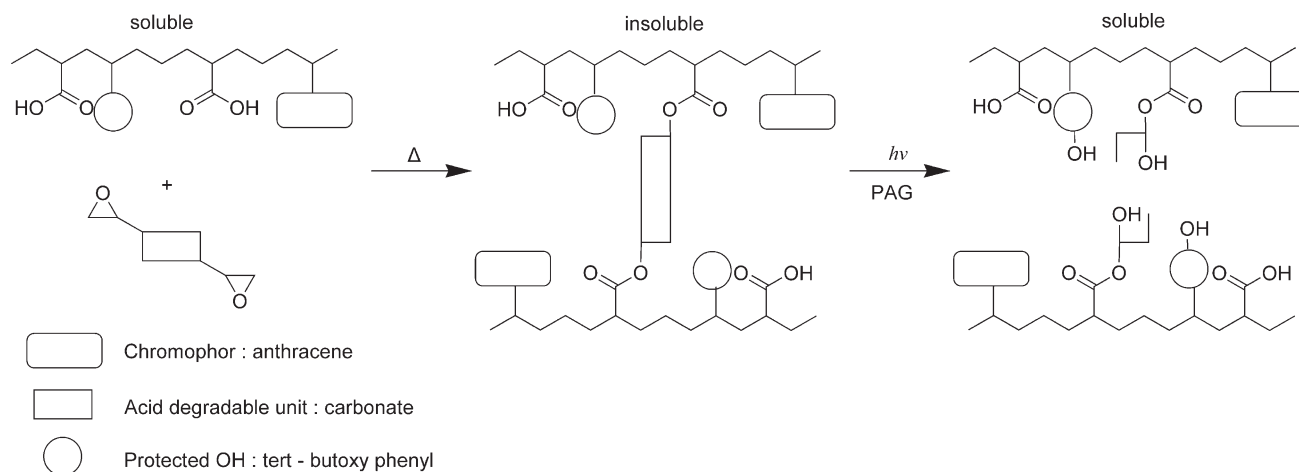
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## EXPERIMENTAL

### Materials and instrumentations

9-Anthracene methanol, methacrylic acid (MAA), allylamine, *m*-chloroperbenzoic acid (*m*-CPBA),



**Figure 1** Structural change of cross-linker through the lithographic process.

triposgen and dicyclohexylcarbodiimide (DCC) were purchased from Aldrich. 4-Hydroxy phthalic acid and 4-dimethylaminopyridine was supplied by Acros and Fluka, respectively. *p*-*tert*-Butoxy styrene (PTBS) and Azobis(isobutyronitrile) (AIBN) were acquired from TCI. Photo acid generator (IRGACURE PAG 203) was obtained from Ciba specialty chemicals. Solvents, such as tetrahydrofuran (THF), chloroform, and propylene glycol methyl ether acetate (PGMEA) were sourced from Aldrich. THF and chloroform were distilled from sodium and calcium hydride under nitrogen atmosphere, respectively. All other reagents were used without further purification.

The  $^1\text{H}$  nuclear magnetic resonance (NMR) spectra were recorded in  $\text{CDCl}_3$  and  $\text{DMSO}-d_6$  on a 300 MHz Bruker DPX-300 spectrometer. Thermal analysis was performed on TA DSC2920 in a  $\text{N}_2$  atmosphere at heating and cooling rates of  $10^\circ\text{C}/\text{min}$ . The molecular weight of the polymer was measured by gel permeation chromatography (with Waters 2414 RI detector, Shodex LF-804 column and THF as the mobile phase). The absorbance of polymer film on a quartz plate was measured using Agilent 8453 ultraviolet/visible (UV/VIS) spectrophotometer. The Fourier transform infrared spectra (FT-IR) measurements were performed on a JASCO FT/IT-200 using a KBr pellet. The pattern formation experiments were performed using an Ushio UX-1100SM exposure system with a short pass filter (Asahi spectra, XUS0250).

#### Synthesis of 2-allyl-5-hydroxyisoindoline-1,3-dione (1)

A 250-mL round bottomed flask equipped with a Dean-Stark condenser was charged with 5 g (27.45 mmol) of 4-hydroxyphthalic acid and 4.1 mL (5.49 mmol) of allylamine in 100 mL of dry toluene. The

mixture was stirred for 24 h at  $130^\circ\text{C}$ , and then recrystallized by cooling to room temperature. The solvent was removed under vacuum to give a white powder in 81% yield.

$^1\text{H}$ -NMR ( $\text{CDCl}_3$ ):  $\delta$  (ppm) 4.26 (d, 2H,  $-\text{CH}_2-$ isoindolidone), 5.26 (q, 2H,  $\text{CH}_2=$ ), 5.81 (m, 1H,  $-\text{CH}=\text{CH}_2$ ), 6.64 (s, 1H, OH), 7.10 (d, 1H, phenyl), 7.29 (s, 1H, phenyl), 7.75 (d, 1H, phenyl).

#### Synthesis of bis(2-allyl-1,3-dioxisoindolin-5-yl) carbonate (2)

4.96 mL of triethylamine was added to a stirred solution of 3.62 g (17.82 mmol) of compound (1) in 100 mL of THF. A total of 0.88 g (2.97 mmol) of triposgen in 50 mL of THF was added dropwise at  $0^\circ\text{C}$ . The reaction mixture was stirred for 3 h at room temperature and the solvent was removed from the mixture. A yellow powder type mixture was dissolved in methylene chloride, and washed sequentially with an ammonium chloride 10 wt % aqueous solution and distilled water. The organic layer was dried over magnesium sulfate and the solvent was removed. The product was purified by recrystallization in methanol below  $0^\circ\text{C}$ . The yield was 72%.

$^1\text{H}$ -NMR ( $\text{CDCl}_3$ ):  $\delta$  (ppm) 4.26 (d, 4H,  $-\text{CH}_2-$ isoindolidone), 5.23 (q, 4H,  $\text{CH}_2=$ ), 5.86 (m, 2H,  $-\text{CH}=\text{CH}_2$ ), 7.64 (d, 2H, phenyl), 7.82 (s, 2H, phenyl), 7.96 (d, 2H, phenyl).

#### Synthesis of bis(2-(oxiran-2-ylmethyl)-1,3-dioxisoindolin-5-yl) carbonate (BOCA)

3.12 g (12.95 mmol) of *m*-chloroperbenzoic acid (*m*-CPBA) was dissolved in 50 mL chloroform containing 1.4 g (3.23 mmol) of compound (2). The resulting mixture was then heated under reflux for 48 h. The reaction mixture was filtered, washed with a sodium hydrogen carbonate 10 wt % aqueous solution, sodium sulfate 10 wt % aqueous solution respectively,

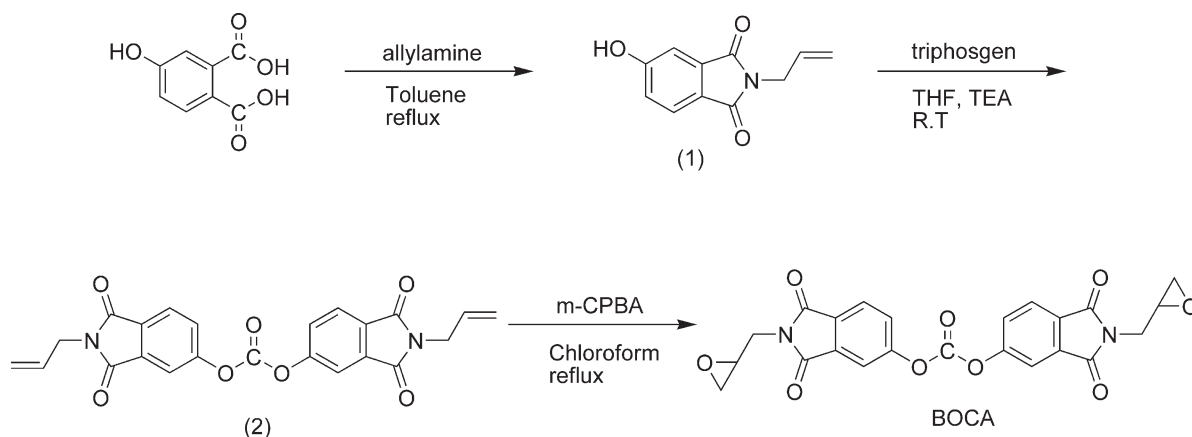


Figure 2 Synthetic route of BOCA.

and then dried over anhydrous magnesium sulfate. The solvent was evaporated to give a white powder in 86.7% yield.

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  (ppm) 2.84 (m, 4H, oxirane), 3.25 (m, 2H, oxirane), 3.95 (m, 4H,  $-\text{CH}_2-$ isoindolone), 7.67 (s, 2H, phenyl), 7.84 (s, 2H, phenyl), 7.98 (d, 2H, phenyl).

#### Synthesis of poly(AMMA-co-PTBS-co-MAA)

9-anthracenylmethylmethacrylate (AMMA) was prepared by a reaction between methacrylic acid (MAA) and 9-anthracenemethanol, according to a previous procedure.<sup>14</sup>  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  (ppm) 1.87 (s, 3H,  $\text{CH}_3\text{C}=\text{C}$ ), 5.41 (s, 1H,  $\text{C}=\text{CH}$ ), 6.00 (s, 1H,  $\text{C}=\text{CH}$ ), 6.12 (s, 2H,  $\text{CH}_2$ -anthracene), 7.38 (d, 2H, anthracene), 7.48 (d, 2H, anthracene), 7.89 (d, 2H, anthracene), 8.29 (d, 2H, anthracene), 8.32 (s, 1H, anthracene).  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  (ppm) 18.2 ( $\text{CH}_3^-$ ), 58.9 ( $\text{CH}_2^-$ anthracene), 123.8 ( $\text{CH}=\text{C}$ ), 124.9 (anthracene), 125.9 (anthracene), 126.2 (anthracene), 126.4 (anthracene), 128.9 (anthracene), 129.0 (anthracene), 130.8 (anthracene), 131.2 (anthracene), 135.9 ( $\text{C}=\text{CH}$ ), 167.4 ( $\text{C}=\text{O}$ ). A total of 2.20 g (7.97 mmol) of AMMA, 1.76 g (10 mmol) of *p*-tert-butoxy styrene (PTBS), 1.88 g (21.9 mmol) of methacrylic acid (MAA) and 0.17 g of azobis(isobutyronitrile) (AIBN) was dissolved in 24 mL of propylene glycol methyl ether acetate (PGMEA). The mixture was stirred for 5 h at 60°C under a dry nitrogen atmosphere. The reaction mixture was poured into *n*-hexane. The precipitate was further purified by several washes using *n*-hexane, and then dried in a vacuum oven. 1.92 g of the poly(AMMA-co-PTBS-co-MAA); (AMMA/PTBS/MAA = 20/25/55) was obtained as a yellowish white powder. Gel permeation chromatography (GPC) using polystyrene as the standard revealed a weight average molecular weight ( $M_w$ ) of 9000 g/mol and the number average molecular weight ( $M_n$ ) of 5000 g/mol.  $^1\text{H-NMR}$  ( $\text{DMSO}-d_6$ ):  $\delta$  (ppm) 0.82

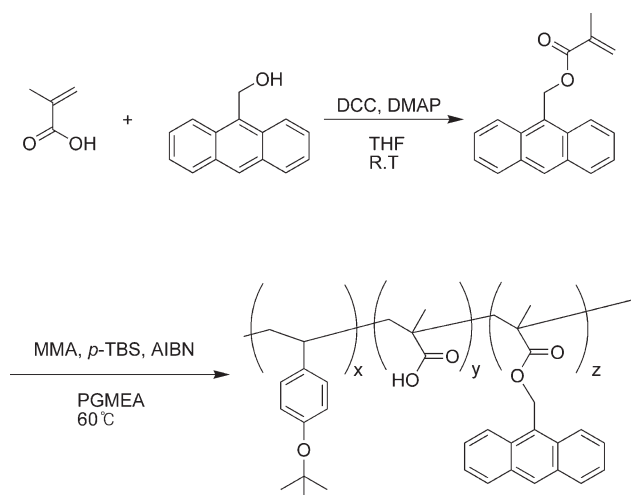
(m,  $\text{CH}_3\text{C}-$ ), 1.20 (m,  $\text{CH}_3\text{C}-$ ), 1.38 (m, polymer backbone), 1.57 (s,  $-\text{OC}(\text{CH}_3)_3$ ), 1.84 (m, polymer backbone), 1.99 (m, polymer backbone), 5.61 (s,  $\text{CH}_2$ -anthracene), 6.54–6.95 (m, phenyl), 7.54 (m, phenyl), 7.78 (d, anthracene), 8.01 (d, anthracene), 8.09 (d, anthracene), 12.08 (br, MAA).  $^{13}\text{C-NMR}$  ( $\text{DMSO}-d_6$ ):  $\delta$  (ppm) 17.9 ( $\text{CH}_3^-$ ), 20.0 ( $\text{CH}_3^-$ ), 28.55 ( $(\text{CH}_3)_3\text{CO}$ ), 44.4, 49.7, 51.4 (backbone), 58.3 ( $\text{CH}_2$ -anthracene), 123.1 (phenyl), 124.9 (anthracene), 125.9 (anthracene), 126.1 (anthracene), 126.8 (anthracene), 128.3 (phenyl), 128.7 (anthracene), 128.9 (phenyl), 129.2 (anthracene), 130.5 (anthracene), 130.9 (anthracene), 152.6 (phenyl), 165.2, 168.3 ( $\text{C}=\text{O}$ ).

#### Preparation of photo-patternable cross-linked epoxy film

1.84 g of poly(AMMA-co-PTBS-co-MAA), 0.144 g (9 mol % of MAA in copolymer) of BOCA and 0.021 g (10 mol % of BOCA) of PAG were dissolved in 8 g of PGMEA. The resulting solution was coated on a Si wafer using a spin coater at 5000 rpm for 30 s. The film was cured at 180°C on a hot plate for 1 min to make a cross-linked film. For a micron size pattern process, the cross-linked film was exposed to UV light at 500 mJ/cm<sup>2</sup> through a mask with a pattern of 50  $\mu\text{m}$  lines. The film was then developed in a 2.38% tetramethyl ammonium hydroxide (TMAH) aqueous solution for 30 s and rinsed in deionized water.

## RESULTS AND DISCUSSION

Bis(2-(oxiran-2-ylmethyl)-1,3-dioxisoindolin-5-yl) carbonate (BOCA) was synthesized by a three-step sequence reactions as shown in Figure 2. Initially, 2-allyl-5-hydroxyisoindolin-1,3-dione (1) was prepared by a reaction between 4-hydroxyphthalic acid and allylamine in toluene using azeotropic distillation. Bis(2-allyl-1,3-dioxisoindolin-5-yl) carbonate (2) was prepared from compound (1) and triphosgen



**Figure 3** Base polymer for the patternable antireflective coating system.

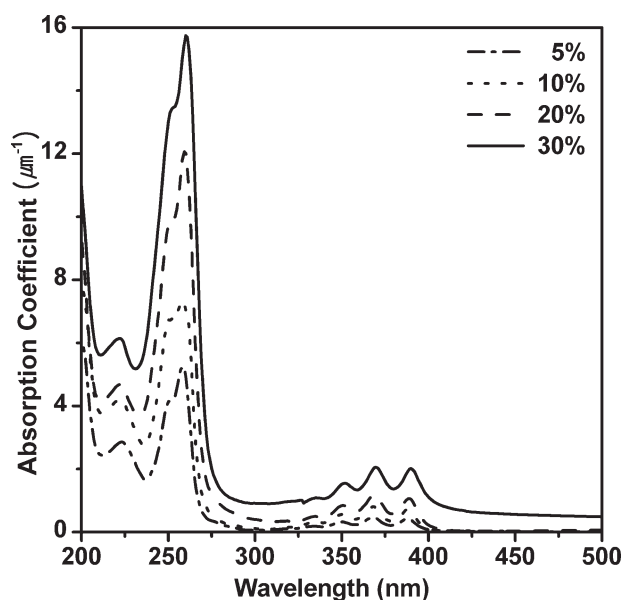
with triethylamine. Finally, compound (2) was oxidized using *m*-CPBA to produce a dioxirane compound, BOCA. The product of each reaction was confirmed by  $^1\text{H-NMR}$ .

Poly(AMMA-*co*-PTBS-*co*-MAA) was used as a base resin for the preparation of this photo-patternable cross-linked epoxy system. This polymer was synthesized through the conventional free radical polymerization and their structure is shown in Figure 3. AMMA was introduced to the polymer as a chromophore to absorb 248 nm and the content was determined by measuring the absorption coefficient and reflective index at this wavelength. The absorption coefficient of  $8.97 \mu\text{m}^{-1}$  and the reflective index of 1.59 for the polymer film containing 20 mol % AMMA were observed (Fig. 4). Because these values have been known to impart effective antireflective properties to the polymer materials, 20 mol % of AMMA was used in this study.<sup>15</sup>

MAA was introduced into the polymer because it can impart dual functions such as cross-linking with the oxirane groups of BOCA to generate a stable cross-linked epoxy film and increasing the solubility of the decomposed film (third structure in Fig. 1) in the basic developer, tetramethyl ammonium hydroxide (TMAH) 2.38% aqueous solution. The third component, *p*-ter-*t*-butoxy styrene (PTBS), is also essential for the photo-patternable cross-linked epoxy system.<sup>16</sup> Initially, the hydrophobic properties of the PTBS group can impart improved solubility of the polymer in PGMEA widely used in semiconductor industry for photoresist or coating application because of its high boiling point and low toxicity.<sup>17</sup> Because PGMEA was used as a solvent for both polymerization and coating processes, good solubility in this solvent is essential for the development of the photo-patternable cross-linked epoxy system.

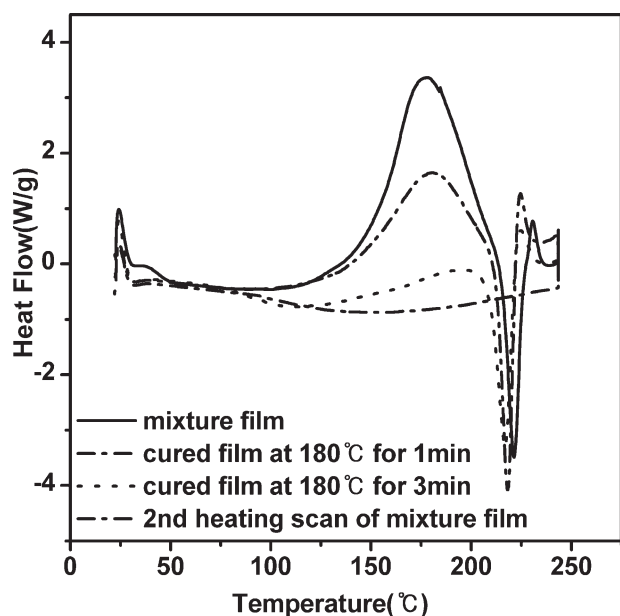
When the polymers composed of only AMMA and MAA groups were prepared without a PTBS unit, they were not soluble in PGMEA. The PTBS group in the cross-linked epoxy system can also improve the solubility of the decomposed film to the basic developer, TMAH, during the decomposition process because a hydrophilic hydroxy functional group can be generated by PAG through UV irradiation. The contents of MAA and PTBS were determined from development tests shown later in this article.

The exothermic transition observed from the DSC heating scans have been used to identify the reaction temperatures of oxirane and carboxylic acid groups by others.<sup>18</sup> Therefore, DSC experiments were performed to determine the reaction temperature between the oxirane groups in BOCA and the carboxylic acid group in the polymer (Fig. 5). The first DSC heating curve of an equivalent mixture film of BOCA and poly(AMMA-*co*-PTBS-*co*-MAA) shows a large exothermic peak at  $177^\circ\text{C}$  that is caused by a cross-linking reaction, and the endothermic peak at around  $220^\circ\text{C}$  was assigned to decomposition of the carbonate group in BOCA.<sup>19</sup> In the second heating scan, both the endothermic and exothermic peaks disappeared because all the cross-linking and decomposition reactions were completed during the first heating scan. When the mixture was cured at  $180^\circ\text{C}$ , the intensity of the exothermic peak decreased because a cross-linking reaction occurs at this temperature. Obviously, the peak intensity (145 J/g) of this exothermic transition for a longer reaction time (3 min) is less than that (285 J/g) for the shorter reaction time (1 min) (Fig. 5), whereas the



**Figure 4** UV spectrum of base polymer as a function of AMMA contents.





**Figure 5** DSC curves of the BOCA and base polymer mixture film.

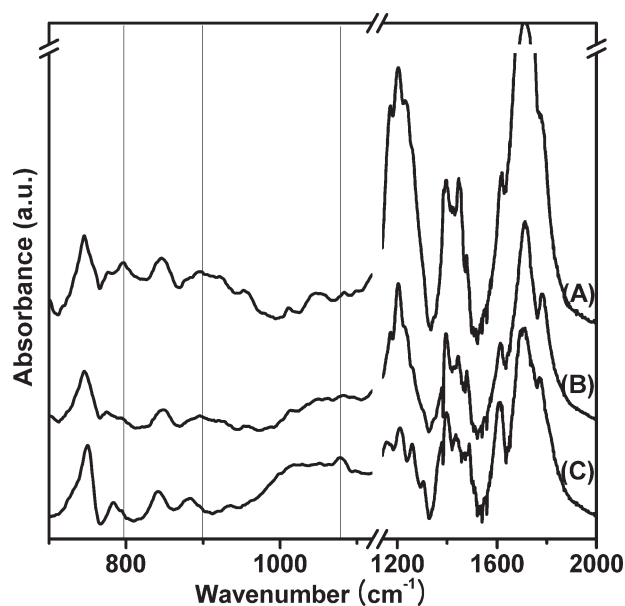
intensity of the endothermic peak (41 J/g) at approximately 220°C was almost identical at different curing times because the decomposition of carbonate does not occur at 180°C.

This cross-linking reaction was also monitored by FT-IR spectroscopy, as shown in Figure 6. Although the carbonyl stretching peaks in the range of 1600–1800  $\text{cm}^{-1}$  gives large absorptions and a change in peak intensity was observed, they cannot be used easily to monitor the reaction because of the overlap of several carbonyl peaks from the different groups of BOCA (imide and carbonate groups), the base polymer (carboxylic acid and ester), and the cross-linked product (ester generated from the reaction between oxirane and carboxylic acid), which makes the spectra in this region quite complicated. Therefore, the oxirane peaks at 893  $\text{cm}^{-1}$  (C–O–C ring deformation) and 793  $\text{cm}^{-1}$  (C–O–C ring stretching of oxirane) and the secondary alcohol peaks (generated through cross-linking reaction) at 1079  $\text{cm}^{-1}$  were used to monitor the cross-linking reaction. There was a decrease in the intensities of the oxirane peaks at 893 and 793  $\text{cm}^{-1}$  when the mixture was heated to 180°C for 1 min, and these peaks disappeared completely when heated at 220°C. On the other hand, secondary alcohol peaks appeared at 1079  $\text{cm}^{-1}$  when the mixture was heated. Therefore, the cross-linking reaction occurs at 180°C for 1 min and is complete at 220°C for 1 min when an equivalent mixture film of BOCA and poly(AMMA-*co*-PTBS-*co*-MAA) is heated.

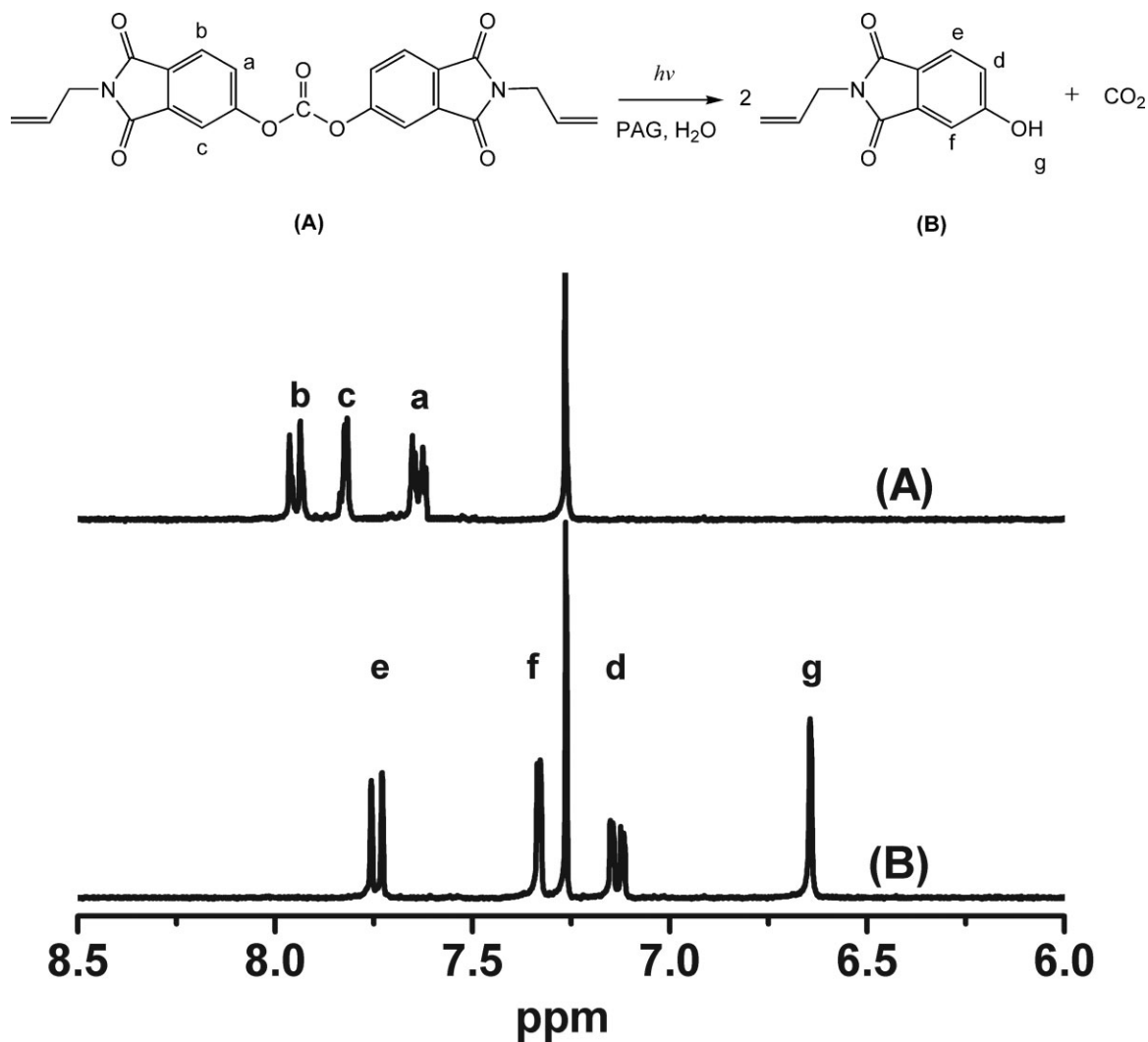
Photo acid generator (PAG), radiation-sensitive acid generator, is converted to a strong acid upon

absorption of a photon with the extent of reaction being governed by exposure energy.<sup>20</sup> So thermally cross-linked film contained BOCA and PAG was decomposed by UV irradiation and then dissolved in developer solution. The decomposition of the carbonate group in BOCA was confirmed from a model reaction study. Because only a small amount of BOCA (MAA content in the copolymer is 55 and 9 mol % of BOCA was added to the sample) is used, and there are several other carbonyl groups in the film, it was impossible to detect the change in carbonate content before and after the decomposition reaction of the cross-linked epoxy films by IR and NMR. Therefore, as a simple model test to observe the decomposition carbonate group, a chloroform solution of (2) (60 mmol) and PAG (IRGACURE PAG 203) was exposed to the UV lamp for 1 h. IRGACURE PAG 203 is one of the most popular PAG for 248 nm photoresist because of high sensitivity at 248 nm though low absorption at 248 nm. Additionally, it has good solubility in PGMEA, thermal stability and low volatility. The  $^1\text{H-NMR}$  spectra of the model reaction reactant and product in Figure 7 clearly show that compound (2) decomposed into 2-allyl-5-hydroxyisoindoline-1,3-dione (1) after UV irradiation.

After confirming the cross-linking reaction of BOCA with poly(AMMA-*co*-PTBS-*co*-MAA) and the decomposition reaction of the carbonate groups in BOCA from the experimental results, an attempt was made to determine the composition of poly(AMMA-*co*-PTBS-*co*-MAA), the cross-linking temperature, and the content of BOCA in the mixture to



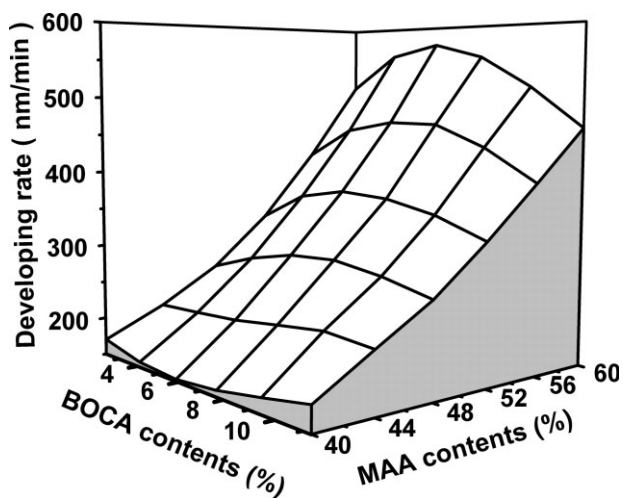
**Figure 6** IR spectra of the BOCA and base polymer mixture film. (A) simple mixture, (B) heated at 180°C for 1 min, (C) heated at 220°C for 1 min.



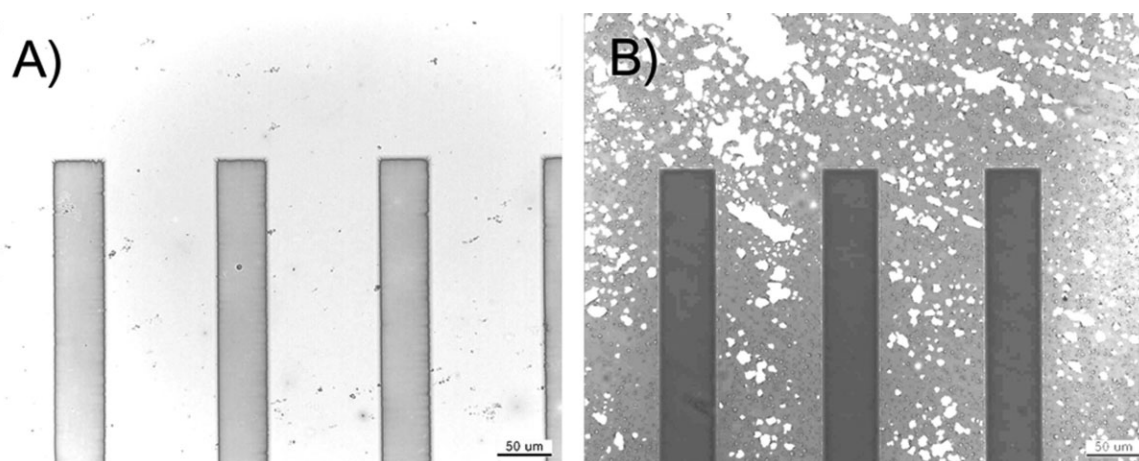
**Figure 7** Photo acid catalytic decomposition scheme and  $^1\text{H-NMR}$  spectra : (A) Bis(2-allyl-1,3-dioxoisindolin-5-yl) carbonate (2), (B) decomposed product.

obtain an optimized epoxy coating system for the UV patterning process.

To determine the appropriate content of MAA, a development test was performed using coated films composed of poly(AMMA-*co*-PTBS-*co*-MAA), PAG, and BOCA on a Si wafer (Fig. 8). In this formulation, the AMMA content was fixed at 20 mol % because this was found to impart good antireflective property to the polymer as mentioned above. After heating the coated film to  $180^\circ\text{C}$  for a given time followed UV irradiation ( $500 \text{ mJ}/\text{cm}^2$ ), the sample was dipped into a developer, tetramethyl ammonium hydroxide (TMAH) 2.38% aqueous solution, and the developing rate (dissolution speed of the film) was checked as a function of the BOCA contents and MAA content of the polymer. Figure 8 shows that the developing rate was not affected significantly by the BOCA content, whereas it increases as the content of MAA increase. As expected, the



**Figure 8** Developing rate of patternable antireflective coating materials as a function of the MAA and cross-linker contents.



**Figure 9** Optical image of antireflective coating material pattern made with different BOCA contents: (A) 9.0, (B) 18.0 mol % of MMA contents of base polymer.

UV irradiated areas were washed out in the developer around at 30 s, whereas the unexposed areas were maintained throughout this development process. A rapid developing rate is normally desired to dissolve the film clearly. However, if dissolution is too fast, such as less than half the developing process time, it can cause injure of pattern, which is known as a “overhang profile”.<sup>21,22</sup> Therefore, the MAA content in the polymer was determined to be 55 mol % to give a proper developing rate. At this developing rate, 85 nm film thickness was perfectly dissolved around half of the developing process time (30 s). As a result, the optimum composition of the polymer was determined to be AMMA/PTBS/MAA = 20/25/55 (mol ratio).

From the DSC and FT-IR results 180°C were determined as an optimum curing temperature to prepare the photo-patternable cross-linked epoxy film. When a lower temperature, e.g., 130°C, was used, the UV unexposed area was also peeled by the developer because good cross-linking network was not formed. At higher temperature, e.g., 230°C, even the UV exposed area was not fully washed out by the developer possibly because the cross-linking density is too high. The desired pattern could be obtained when 180°C was used for the curing process.

The optimum BOCA content in the epoxy system were determined through a micron size pattern process by UV irradiation. When BOCA content in the epoxy system was less than 4.5 mol % to MMA content, the UV unexposed area was also peeled by the developer. When the BOCA content was between 4.5 and 18 mol %, a good rectangular pattern was obtained [Fig. 9(A)]. When the BOCA content was larger than 18 mol %, even the UV exposed area was not fully washed out [Fig. 9(B)]. This residue was not removed by additional development or washing processes. Therefore, the optimum amount

of cross-linker, BOCA, was determined to be 9 mol % to MAA content of the polymer.

Because proper UV lithography equipment for 248 nm is not available, we could not obtain good cross-sectional micrographs of the line and grating patterns using this epoxy system as a patterenable antireflective coating system with any photoresist materials suitable for the 248 nm process. However, advanced results from the 248-nm UV lithographic process are expected because this epoxy system has both patterning and antireflective properties. New patterenable antireflective coating systems could be developed if the chromophore is changed from 9-anthracenylmethacrylate to other monomers that can absorb other UV light such as 193 or 157 nm. Recently, this cross-linked epoxy system was granted a KR, JR and US patent<sup>23-25</sup> and further studies on the practical applications of the UV lithography process as a patterenable bottom antireflective coating material are under progress.

## CONCLUSIONS

A new photo-patternable cross-linked epoxy system was developed through a cross-linking reaction of oxirane groups in bis(2-(oxiran-2-ylmethyl)-1,3-dioxoisindolin-5-yl) carbonate with the carboxylic acid groups in a polymer containing 9-anthracenylmethacrylate (AMMA), *p*-*tert*-butoxy styrene (PTBS), and methacrylic acid (MAA) monomeric units. Dissolution or patterning of the cross-linked system was achieved by the addition of a photo acid generator through the UV irradiation. Because this epoxy system can be patterenable and has antireflective properties, it is expected that it can be applied as a patterenable bottom antireflective coating material for the UV lithography processes and such works are under progress.

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