Preparation and Characterization of Molecular Photoresists: Crosslinkable Positive and Water Developable Negative Tones

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ABSTRACT: The development of a water-developable negative photoresist from β-CD using an acid-catalyzed chemical amplification method is investigated here. Tertiary butoxyl protected β-cyclodextrin (*t*-BOC-CD) is also synthesized and used to prepare a positive photoresist. Glutaraldehyde is added as a crosslinking agent for both positive and negative photoresists. Deprotection of *t*-BOC-CD is accelerated by a photo-induced acid. In the presence of glutaraldehyde and acid, both the deprotected *t*-BOC-CD and β-cyclodextrin are crosslinked. The introduction of a *t*-butoxyl group into the β-CD molecule and the addition of glutaraldehyde into the β-CD molecules are both found to decrease the crystallinity of the molecules, improving the resist film properties. The etching resistance of both positive and negative photoresist films is improved by the crosslinking method. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 109: 3849–3858, 2008

Key words: lithography; synthesis; photoresist; crosslinking

INTRODUCTION

Photoresist masks for patterning layers for electrical circuit fabrication are well documented.[1–3] The development of new photolithography systems is thus required to reduce the feature size of integrated circuits.[4,5] Moore's Law requires advanced lithographic images with new, shorter wavelength lasers, such as 193 nm, 157 nm, and electron beams (EBs).[6–8] For these technologies, new photolithography resist materials are required to have high transparency at the exposure wavelength and high stability under dry etching conditions. The chemical amplification resist system is a potential future photolithographic technology using a new irradiation method.[9–12] In this system, a generating acid is produced in a photo-acid generator when light is present, which promotes deprotection of the protecting groups, such as *t*-butyl ester, acetal, trimethylsilyl, and tetrahydropyranyl groups to achieve higher resolution resist materials.[13–16] Recently, many new chemical amplification photoresist materials have been reported using various polymers containing fluorine atoms and alicyclic groups, such as adamantane and norbornane.[17,18] From these studies, it seems that material molecular size is one of the important factors to achieve ultra-high resolution.

Recently, some studies have reported that novel low molecular weight organic compounds, which form stable amorphous glasses above room temperature, work well as EB resists.[19–22] These materials, called ''molecular resists,'' have attracted considerable attention. Meanwhile, cyclodextrins (CDs) are cyclic oligosaccharides containing many primary and secondary hydroxyl groups in small-size molecules, as well as calixarenes. We can consider introducing photo-reactive groups into the CDs by chemically reacting the hydroxyl groups. Furthermore, it is expected that CDs have good thermal stability, film-forming properties, and high transparency in the visible region because of their inherent structures.

There have been many reported studies regarding the synthesis of positive photoresists using novel materials.[23–26] Positive CD-derived photoresists have physical properties that are worse than other polymeric positive photoresists, due to the low molecular weight of the CD molecule. To maintain water developability and overcome the aforementioned molecular weight deficit, a water-developable negative-tone photoresist with CD functionality has been designed for an acid-catalyzed chemical amplification procedure. Tertiary butoxyl protected cyclodextrin (*t*-BOC-CD) is synthesized and used as a photoresist. Deprotection of *t*-BOC group is accelerated by a photo-induced acid. In the presence glutaralde-

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hyde and acid, both protected and unprotected CD may be cured for positive and negative photoresists. The results of decreasing molecular crystallinity and improving film properties are accomplished. Furthermore, the etching resistance in both positive and negative-tone photoresist films is improved by this crosslinking method.

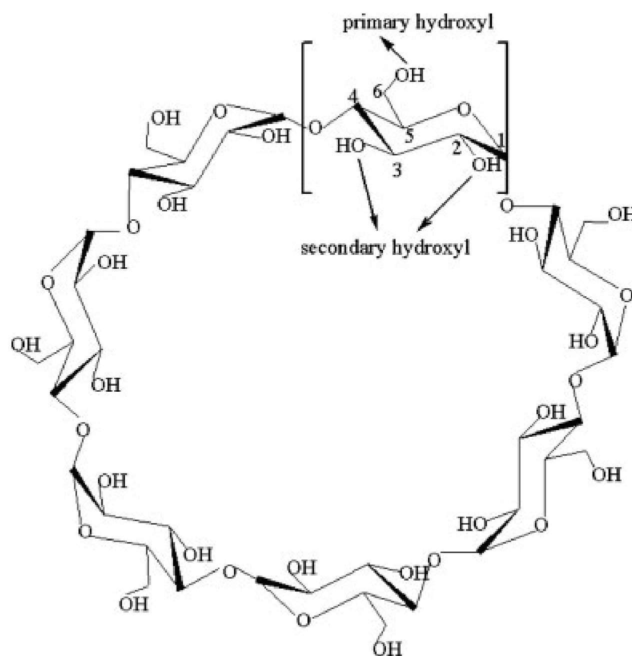
EXPERIMENTAL

Materials

Hexamethyldisilazane (HMDS; 99.0%), hydrogen peroxide (H_2O_2; 30% in water), and photo-acid generator triarylsulfonium hexafluoroantimonate salts (PAG; 50% in propylene carbonate) were purchased from Sigma-Aldrich Chemical. β-Cyclodextrin hydrate (β-CD; 99.0%; $[\alpha]_D = +161^\circ$, $c = 1$ in H_2O), di-*tert*-butyl dicarbonate (97.0%), glutaraldehyde (50% in water), and tetramethylammonium hydroxide (TMAH, 25% in water) were purchased from Acros Chemical. All organic solvents were purchased from Tedia. Tetrahydrofuran was distilled over calcium hydride under argon immediately before use, and other solvents were purified by standard methods. Polystyrene (PS; $M_w = 9.1 \times 10^3$) and poly(methyl methacrylate) (PMMA; $M_w = 7.5 \times 10^4$) were purchased from Tosoh and Fluka, respectively. Wafer substrates were cleaned in a mixture of H_2O_2/H_2SO_4 (3/7) solution at 90°C for 1 h, rinsed with deionized water and acetone, and then dried at 100°C for 1 h. HMDS was spin coated on the surface of the wafer and then dried at 110°C for 1 h.

Measurements

Fourier transform infrared spectroscopy (FTIR) spectra were recorded using a KBr disk on a Jasco VALOR III (Tokyo, Japan) FTIR spectrophotometer. ^1H NMR (400 MHz) spectra were obtained using a Bruker AMX-400 (Darmstadt, Germany) high-resolution NMR spectrometer in DMSO-d_6 and $CDCl_3$, and chemical shifts were reported in ppm with tetramethylsilane as an internal standard. Differential scanning calorimeter (DSC) was conducted using a Perkin-Elmer DSC 7 at a heating and cooling rate of 10 K min^{-1} under a nitrogen atmosphere. The crystallinity of resist films were investigated by an Olympus BH-2 polarized light microscope (POM). Thermal decomposition temperature data were recorded under a nitrogen atmosphere at a heating rate of 10 K min^{-1} with a thermogravimetric analyzer (TGA) Perkin-Elmer TGA 7. UV/Vis absorption spectra were measured with a Jasco V-550 spectrophotometer. Optical rotations were measured at 30°C in acetone using a Jasco DIP-370 polarimeter with the D-line of sodium ($\lambda = 589$ nm) at a precision of $\pm 0.001^\circ$. The measurements were performed using 1% solutions of substances in acetone. The X-ray diffraction data was recorded on a Rigaku RINT-2500 series instrument with Ni-filtered Cu Kα radiation. The sample in a quartz capillary was held in a temperature-controlled cell (Rigaku LC high-temperature controller). Scanning electron microscope (SEM) microphotographs were measured with a JEOL HR-FESEM JSM-6700F (Osaka, Japan) instrument. Film thickness and thickness variation of the fabricated patterns were estimated using Alpha step Tenco-200 instrument. Crosslinkable positive-tone *t*-BOC-CD photoresist was cured using UV irradiation at 0.65 mW cm^{-2} (254 nm; model UVG-54).



Scheme 1 Schematic illustrations of β-cyclodextrin.

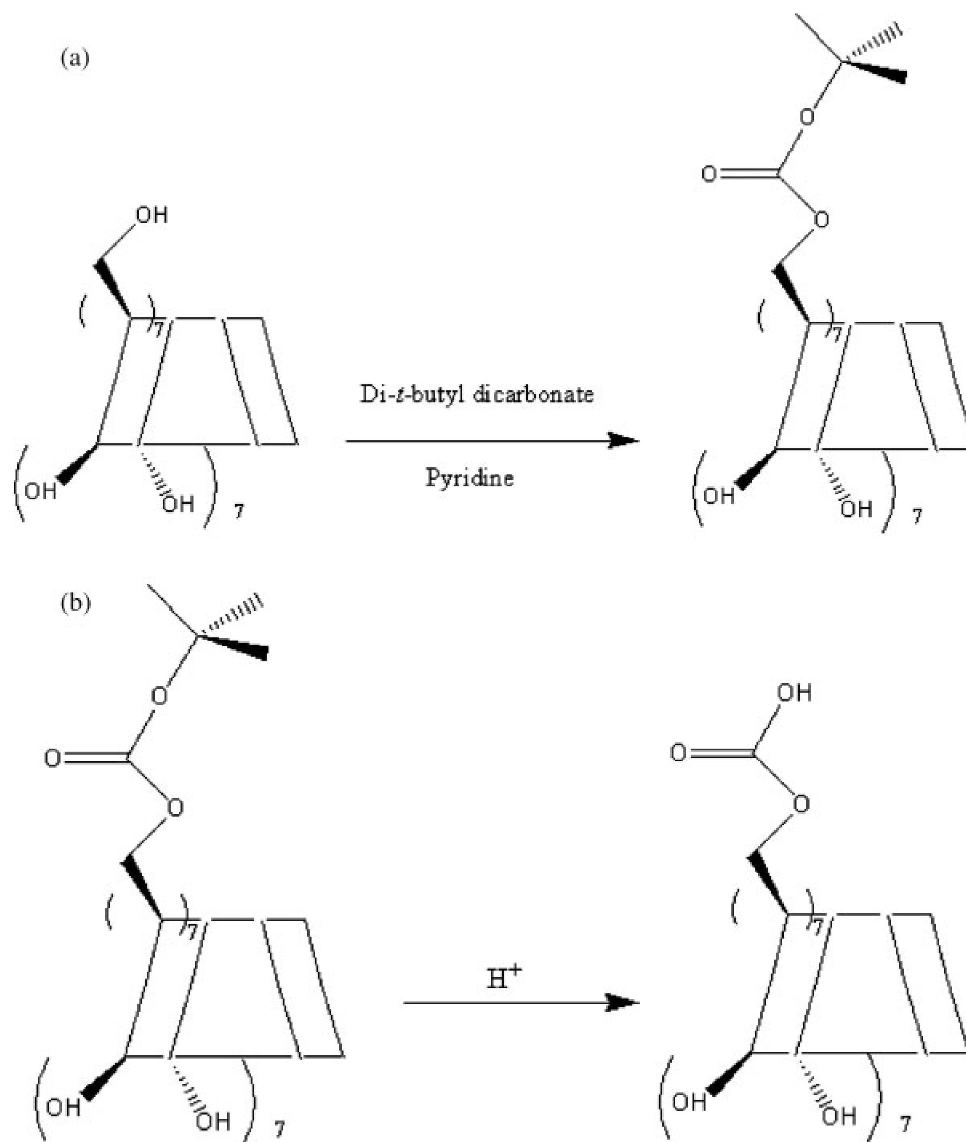
Synthesis of *t*-BOC-CD

First, 2.27 g (2 mmol) of β-cyclodextrin (β-CD) was dissolved in 20 mL pyridine at 0°C. Then, 5.97 g (27.4 mmol) of di-*t*-butyl dicarbonate was added dropwise to the solution. After completion of the adding process, the ice bath was removed and the mixture was stirred at room temperature for 12 h. The reacted mixture was then poured into 1000 mL of water. The white precipitate that formed was filtered and then dried at 70°C in vacuum for over 24 h.

Yield: 76.0%. FTIR (KBr, ν_{max}/cm^{-1}): 1371, 1394 ($C(CH_3)_3$), 1741 (C=O), 2931, 2980 (CH), 3561 (OH). ^1H NMR (DMSO-d_6, δ in ppm): 1.2–1.4 (m, $C(CH_3)_3$). $[\alpha]_D = +105.8^\circ$ ($c = 0.02$ g/mL, acetone).

Crosslinkable positive *t*-BOC-CD photoresist

First, 0.75 g (0.41 mmol) of *t*-BOC-CD and 0.37 g (0.9 mmol, 25% aqueous solution) of glutaraldehyde

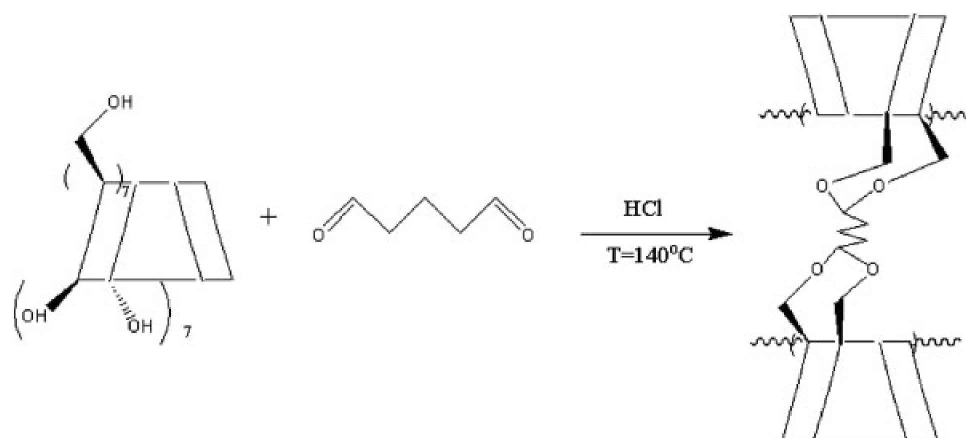


Scheme 2 (a) Synthesis and (b) acid-catalyzed deprotection of *t*-BOC-CD.

were dissolved in 5 mL propylene glycol methyl ether acetate (PGMEA) at 30°C. 0.04 g (50 wt % in propylene carbonate) of triarylsulfonium hexafluoroantimonate salts was added into the mixture as a photo-acid generator. The mixture was spin-coated onto a substrate and dried at 80°C under vacuum for 1 h. One half area of the fabricated photoresist was exposed to 254 nm UV light for 15 s, and then postbaked (PEB) at 90°C for 90 s. The exposed photoresist was developed with 2.38 wt % TMAH. The removed part as acted as a positive-tone photoresist. The residual area of the photoresist was then further exposed to 254 nm UV light for 15 s and PEB again at 140°C for 5 min. The resulting film was developed in TMAH and acetone separately to assess the film's resistance to the solvents. To estimate the resolution of the positive-tone photoresist, the film was exposed with a Karl Suss MJB-3 aligner.

Water-developable negative molecular photoresist

To start, 1.5 g (1.32 mmol) of β-CD and (0.75 g, 25% aqueous solution) of glutaraldehyde were dissolved in 10 mL *N*,*N*-dimethylformamide with stirring for 30 min. After that, 0.04 g (50% in propylene carbonate) of triarylsulfonium hexafluoroantimonate salts as the photo-acid generator was added into the mixture. The mixture was spin-coated onto a wafer, dried, and prebaked 90°C for 3 min. The photoresist was exposed with a Karl Suss MJB-3 aligner. After exposure, the resist was PEB at 140°C for 10 min,



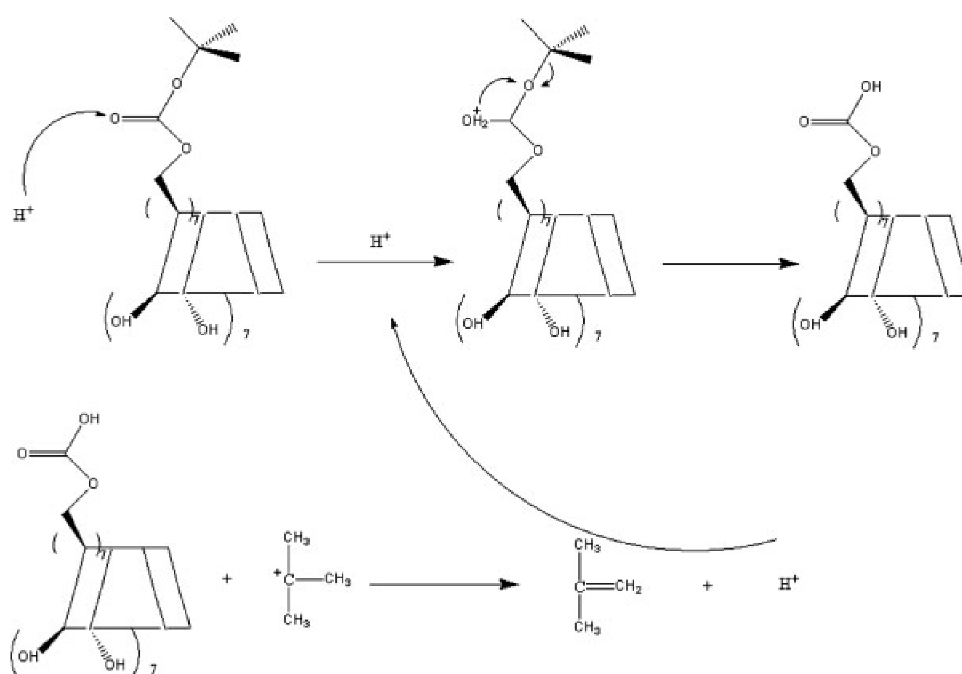
Scheme 3 Acid-catalyzed crosslinking of negative-tone photoresist.

and then developed by immersion in a water developer for 30 s.

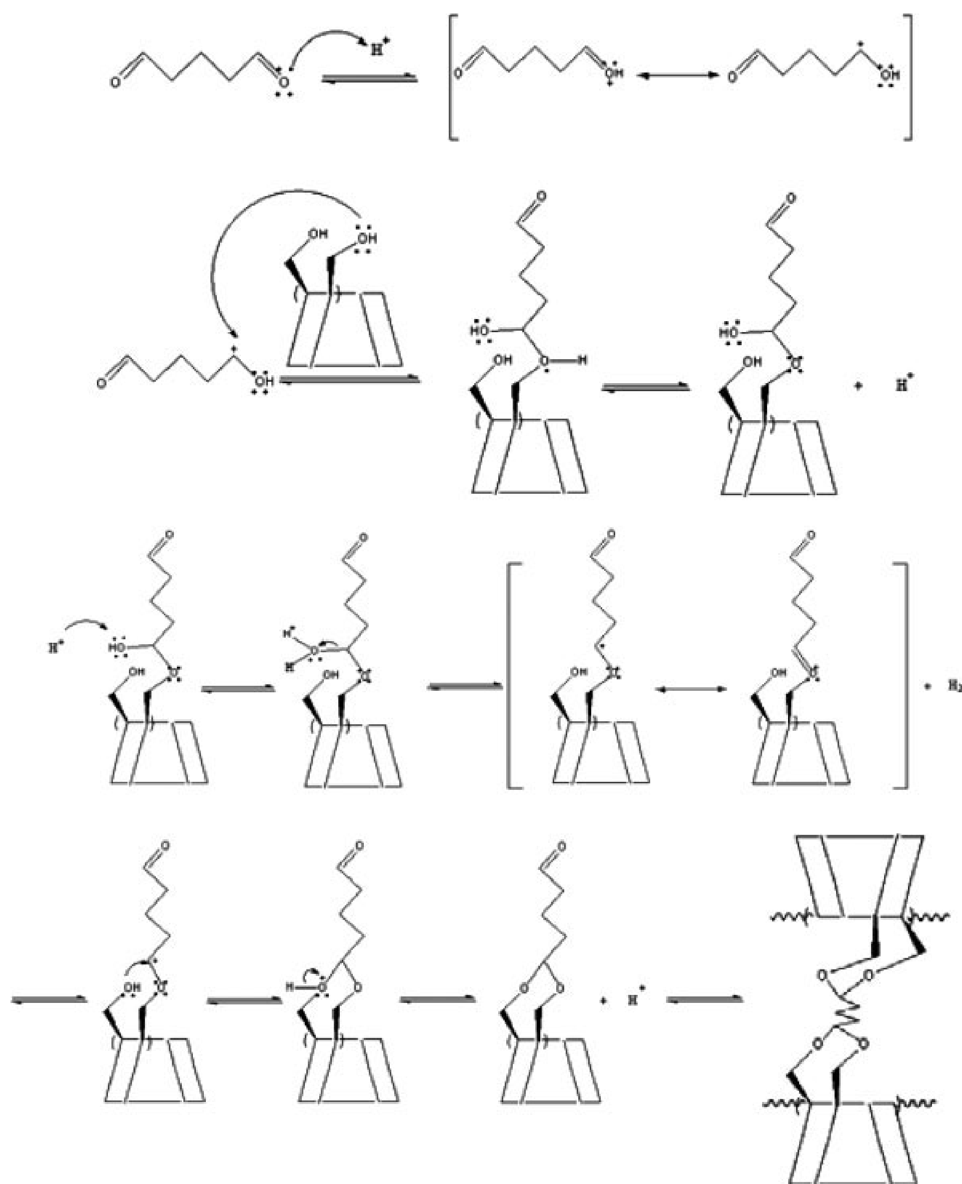
RESULTS AND DISCUSSION

Deprotection and crosslinking of *t*-BOC-CD

Scheme 1 depicts the molecular structure of β-CD, wherein the plural primary and secondary hydroxyl groups can be seen. Three kinds of cyclodexdrins (α, β, and γ) contain similar structures, but contain a different total number of alicyclic rings and primary and secondary hydroxyl groups. Considering the cost of the compounds, in this investigation, we used β-CD, as the least expensive option, to investigate the photoresist characteristics. Scheme 2(a,b) depict the introduction of *t*-butoxyl groups into β-CD and the deprotection of the *t*-butyl group from *t*-BOC-CD, respectively. The *t*-butoxyl groups usually react with the more reactive primary hydroxyl groups. After deprotection, the left carboxyl groups increase the solubility of the resulting compound, permitting it to be further crosslinked by glutaraldehyde in the presence of acid. Theoretically, the hydroxyl groups can also be crosslinked with the glutaraldehyde. Scheme 3 depicts the crosslinking of β-CD with glutaraldehyde in the presence of acid. In this case, the solubility of the crosslinked β-CD in developer is decreased. The variation in solubility can be used as a negative photoresist.



Scheme 4 Mechanism of acid-catalyzed deprotection of *t*-BOC-CD.



Scheme 5 Mechanism of acid-catalyzed crosslinking of β-CD.

The chemical amplification mechanism of the acid-catalyzed deprotection of the *t*-butyl group from *t*-BOC-CD and the crosslinking of β-CD molecules are depicted in Schemes 4 and 5, respectively. There are many functional groups in a β-CD molecule and consequently, crosslinking can occur at many points. Only one crosslinked point is shown in Scheme 5. Crosslinking points can also occur between different β-CD molecules. In the case of the positive photoresist, after removing the *t*-butyl group from *t*-BOC-CD, the hydrophilicity of the deprotected *t*-BOC-CD increases significantly. To investigate the acid-catalyzed deprotection, DSC thermograms of the *t*-BOC-CD with and without acid were carried out. The results are summarized in Figure 1. In the presence of acid, the deprotection was accelerated and showed an endothermic degradation from 40 to

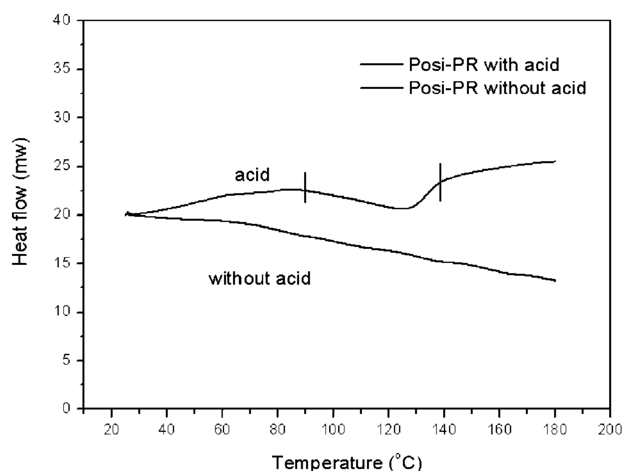


Figure 1 DSC thermogram of positive photoresist without UV exposure.

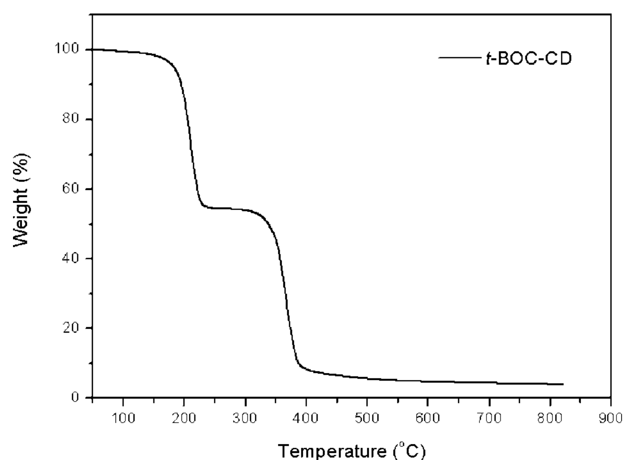


Figure 2 TGA thermogram of *t*-BOC-CD without acid.

80°C. An exothermic crosslinking from 90 to 140°C could also be observed. In contrast to these findings, molecular thermal deprotection and crosslinking can only occur gradually when acid is not present.

Figure 2 shows a TGA thermogram of *t*-BOC-CD without acid. Two stages of thermodegradations can be seen. A slight weight loss can be seen at around 100°C due to the evaporation of absorbed water inside the β-CD molecules. The first labile stage occurred at around 180°C, and is due to the degradation of *t*-butyl groups bonded at the primary hydroxyl groups. Theoretically, about 40 wt % *t*-butyl groups can be introduced into a primary hydroxyl group. Similarly, a 40 wt % degradation can be seen in Figure 2. The second stage of degradation begins at around 320°C due to the degradation of CD molecules.

To investigate the acid-catalyzed crosslinking of CD molecules, DSC thermograms of β-CD with glutaraldehyde were conducted. Figure 3 summarizes the results of DSC thermograms with and without the presence of acid. For both systems, an endothermic curve between 60 and 100°C can be observed, and is due to the evaporation of water absorbed inside the β-CD. An exothermic curve can be observed between 100 and 140°C for the acid-con-

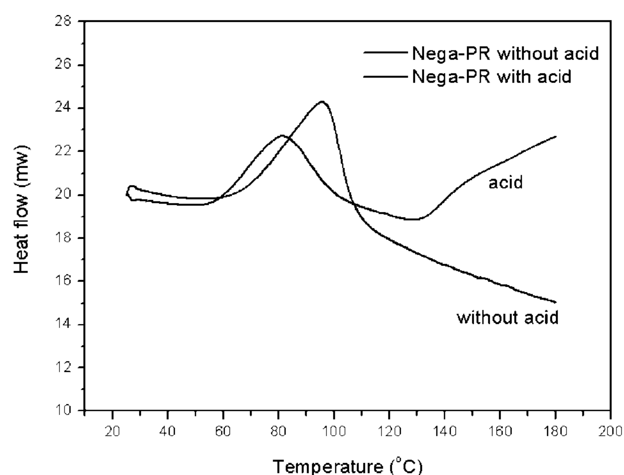
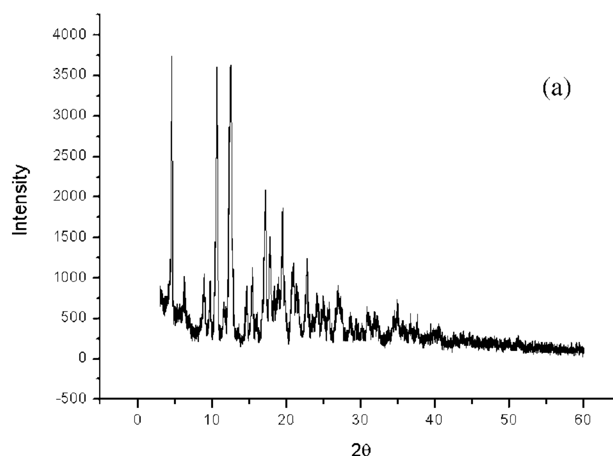
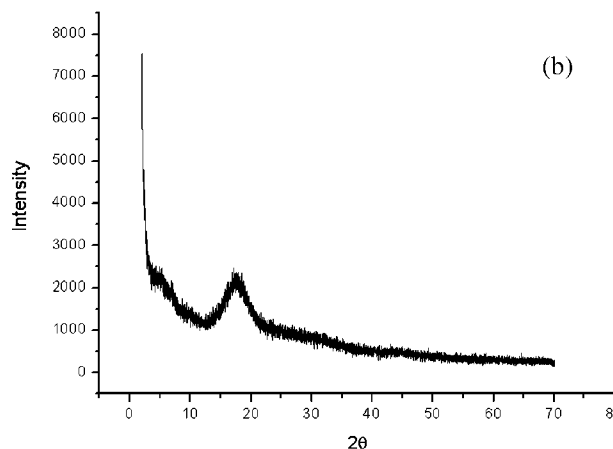


Figure 3 DSC thermogram of negative photoresist without UV exposure.





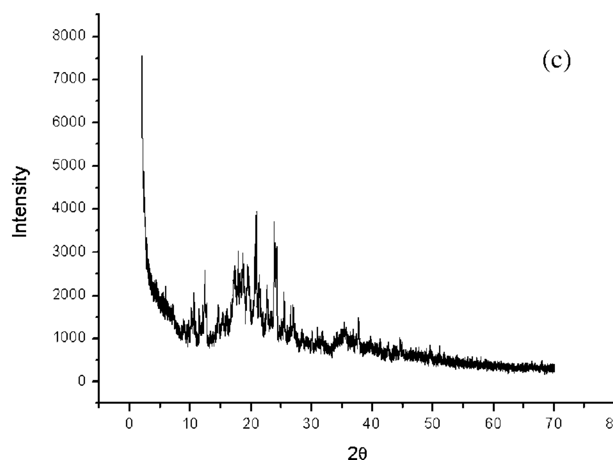


Figure 4 X-ray diffraction of (a) β-CD, (b) *t*-BOC-CD, and (c) a mixture of β-CD and glutaraldehyde.

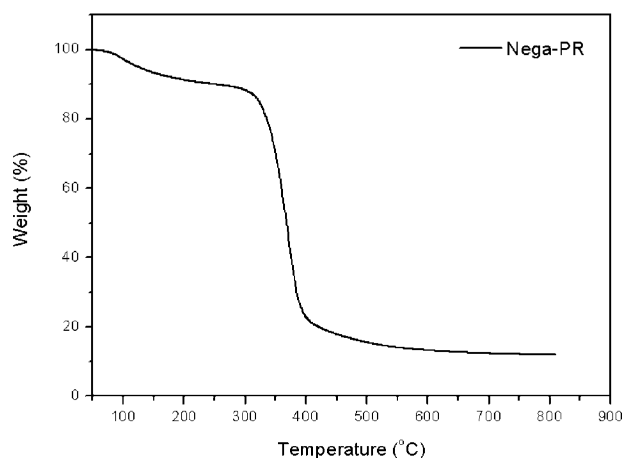


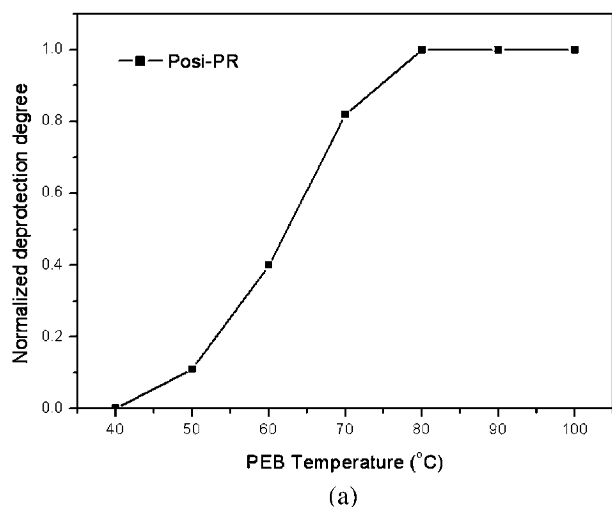
Figure 5 TGA thermogram of negative photoresist after UV exposure and postbaking.

taining system. This result suggests that acid-catalyzed crosslinking occurred in this range. Beyond 140°C, acid-catalyzed thermal degradation may occur gradually. Based on the DSC thermograms shown in Figure 3, 140°C was used as the postbaking temperature for the acid-catalyzed crosslinking. In the case of the system without acid, thermal crosslinking occurred slowly, leading to the appearance of the exothermic curve.

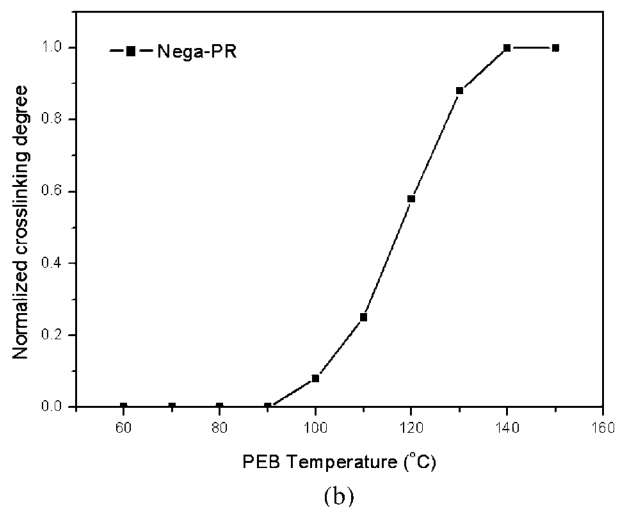
Noncrystalline materials are required for high performance behavior of a photoresist. Figure 4(a–c) show the results of the X-ray diffraction patterns of β-CD, *t*-BOC-CD, and β-CD with glutaraldehyde, respectively. As seen in Figure 4(a), the sharp peaks reveal high crystallinity of the β-CD molecules. The broad band in Figure 4(b) demonstrates that the introduction of *t*-butoxyl groups into β-CD decreases the crystallinity of the synthesized *t*-BOC-CD. As shown in Figure 4(c), the addition of glutaraldehyde can also decrease the crystallinity of the β-CD molecules. The added glutaraldehyde may disturb the intermolecular forces, and subsequently, the arrangement of β-CD, leading to a decrease in the crystallinity. From the physical properties of spin-coated films, a decrease of the crystallinity increases the homogeneity of films. The results in Figure 4 show that both positive-tone photoresist with *t*-BOC-CD and negative-tone photoresist with glutaraldehyde are expected to reveal better film properties. The crystallinity of resist films were confirmed using a POM. Anisotropic crystals can be distinguished as colorful areas under a crossed POM. The prepared films of *t*-BOC-CD and the mixture of β-CD and glutaraldehyde revealed dark appearance under POM. The results of POM are consistent with those observed in Figure 4.

Estimation of PEB temperature and developing time

As seen in Figure 1, the deprotection of *t*-butyl group and the crosslinking of β-CD molecules occurred at a certain temperature. To investigate the thermal stability of the crosslinked photoresists, the negative-tone photoresist was exposed to UV and then PEB. The resultant film was rinsed in water for 60 s to ensure the crosslinking of β-CD molecules. Figure 5 shows the results of the TGA thermogram and the crosslinked negative film. The first 10 wt % decrease is due to the evaporation of water absorbed inside the β-CD molecules. Second stage degradation occurred around 320°C. When compared with the degradation temperature shown in Figure 2 and the decomposition temperature of the β-CD molecule, this temperature point should reveal the decomposition of the alicyclic ring. These results suggest that



(a)



(b)

Figure 6 Effect of PEB temperature on normalized (a) deprotection and (b) crosslinking of positive and negative photoresist after UV exposure.

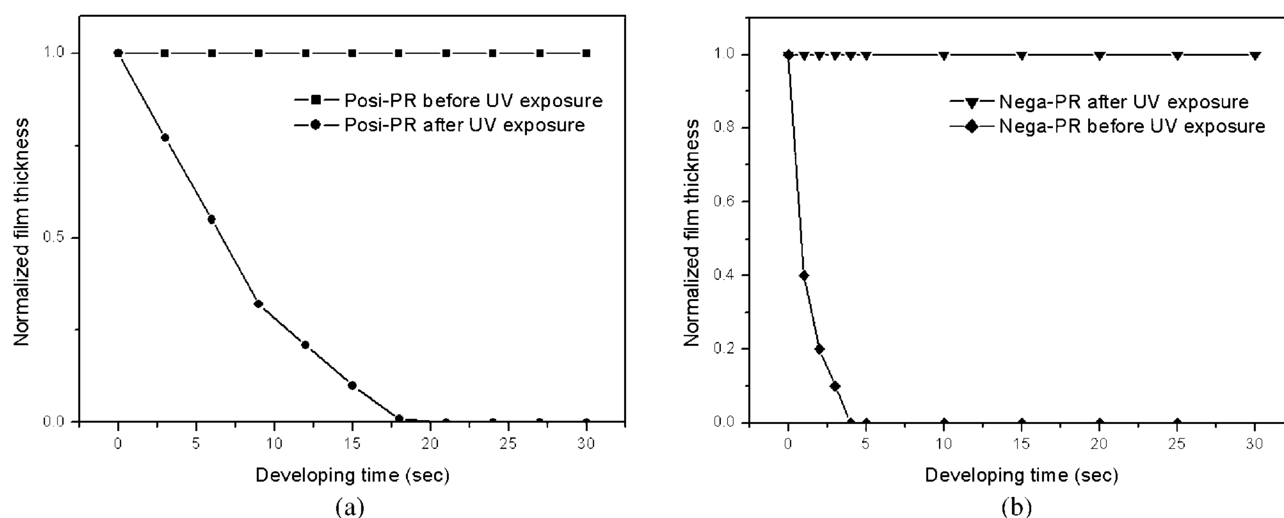


Figure 7 Dependence of normalized film thickness on developing time after UV exposure and PEB for the (a) positive and (b) negative photoresists.

the crosslinking of the β-CD molecule decreases the solubility of films, but does not increase its thermal stability. The degradation temperature was determined by the thermal stability of the alicyclic ring, which forms the β-CD molecule.

Figure 6 shows the effect of PEB temperature on normalized deprotection and crosslinking of the positive and negative photoresist after UV exposure. The PEB temperature for the positive-tone and the negative-tone photoresist in this investigation were estimated as 80°C and 140°C, respectively. Figure 7 shows the dependence of normalized film thickness of both positive- and negative-tone photoresist on developing time. For the positive-tone photoresist, after UV exposure and PEB at 80°C, the film was developed in 2.38 wt % TMAH. Without UV exposure, the positive photoresist did not dissolve in the developer. In the case of the UV exposed positive film, the deprotected *t*-BOC-CD dissolved in the developer and led to a contrasting appearance. As seen in Figure 7(a), 18 s is the minimum requirement for the developing time. At 18 s, a maximum value of contrast can be obtained. For negative photoresist, after UV exposure and PEB at 140°C, the film was developed in water. Figure 7(b) shows the result of the development and the minimum required development time of 4 s. The result suggests that without UV exposure, CD can not be crosslinked and was dissolved in water in 4 s. Acid-catalyzed crosslinked β-CD molecules were not dissolved in water.

Characteristic curves

To investigate both the positive and negative functions of the β-CD and *t*-BOC-CD molecules, photoresist films were prepared using the conditions summarized in Table I. Water was used as a developer for the negative photoresist. Figure 8(a) shows the exposure characteristic curve of the positive photoresist. The photoresist consisted of *t*-BOC-CD molecules and triarylsulfonium hexafluoroantimonate salts, which was added as a photo-acid generator. When compared with the polymeric photoresist, *t*-BOC-CD contains only a unitary molecular weight,

TABLE I
Conditions for the Preparation and Development of the Photoresist[a]

Process	Positive	Negative
Spin speed	Step 1 = 500 rpm × 10 s Step 2 = 1000 rpm × 20 s	Step 1 = 500 rpm × 10 s Step 2 =1000 rpm × 20 s
Composition	t-BOC-CD + GA 30 wt % PGMEA solution	β-CD + GA 30 wt % DMF solution
PB	100°C, 2 min	100°C, 3 min
UV exposure	30 mJ/cm^2, 254 nm	30 mJ/cm^2, 254 nm
PEB	Step 1 = 90°C, 120 s Step 2 = 140°C, 300 s	140°C, 5 min
Developer	2.38 wt % TMAH	H_2O

[a] In the presence of 10 wt % PAG (triphenylsulfonium hexafluoroantimonate).

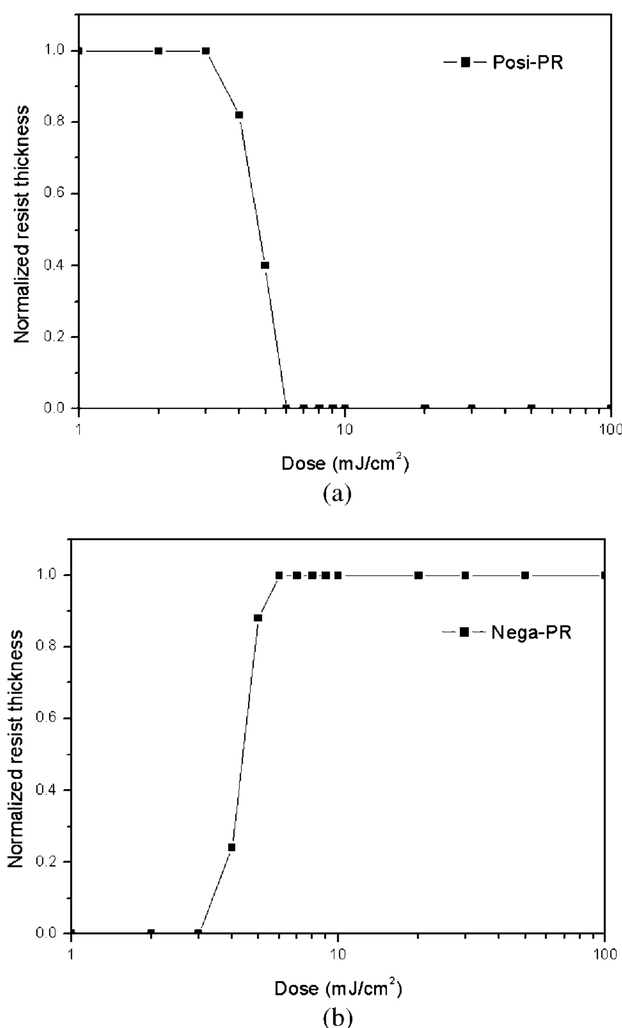


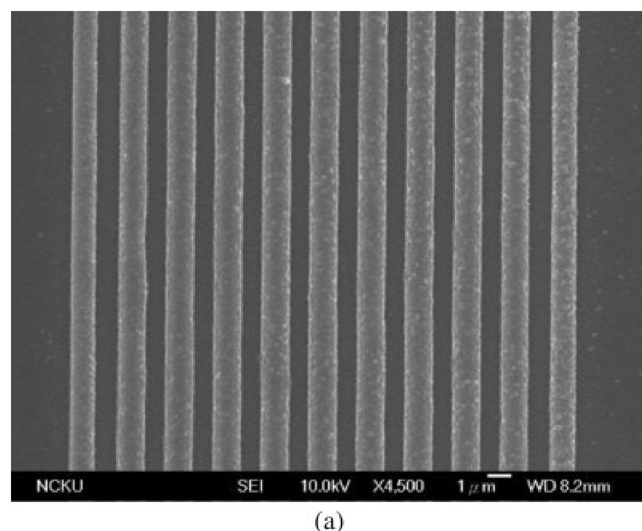
Figure 8 Exposure characteristic curves of the (a) positive and (b) negative photoresists.

and can theoretically provide superior photoresist contrast due to the unitary molecular weight. The sensitivity of the positive-tone photoresist calculated from Figure 8(a) is 5 mJ/cm^2. The contrast was evaluated as 6.45 using following equation:

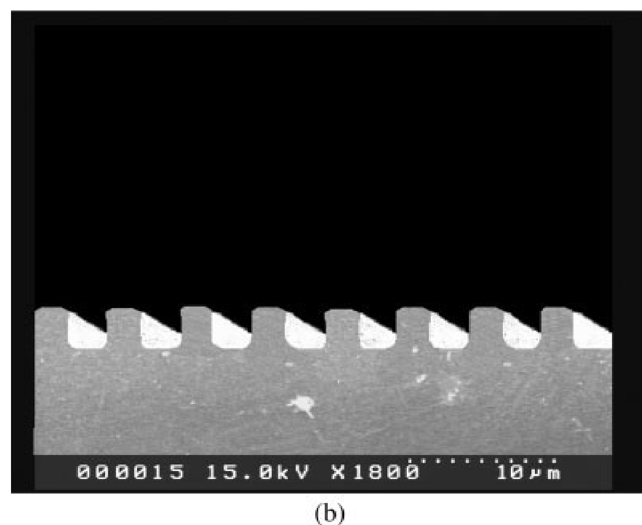
$$\gamma_P = 1/(\log D_s - \log D_t), \quad (1)$$

where, γ_P is the contrast of positive-tone photoresist, D_s is the sensitivity of photoresist, and D_t is the minimum requirement of UV exposure energy. The negative-tone photoresist consists of β-CD and glutaraldehyde molecules. The low molecular weight negative system is expected to reveal excellent exposure characteristics. The sensitivity of the negative-tone photoresist calculated from Figure 8(b) is lower than 5 mJ/cm^2. The contrast was evaluated to be 5.16 using eq. (1). Figure 9 shows the resolution of the photoresist. As seen in the Figure 9(a,b), a resolution of 1 μm is achieved for the systems.

The etching resistance of *t*-BOC-CD positive- and negative-tone photoresists was investigated and compared with PS and PMMA. The results are summarized in Table II. The etching rates of the crosslinked positive and negative films are estimated to be 20 and 18.3 Å sec^{-1}, respectively. The difference in etching resistance between both positive and negative systems may be due to the difference in the crosslinked molecular structures. As seen in Figure 2 and 3, both carboxyl groups in the deprotected *t*-BOC-CD and hydroxyl group in the β-CD can be crosslinked through glutaraldehyde. Contrary to the thermal stability described in the previous section, both positive and negative photoresist films revealed superior etching resistance compared with PMMA (M_w = 7.5 × 10^4), which is most likely due to the efforts of the thermal stable alicyclic rings. Furthermore, due to the existence of *t*-butoxyl groups,



(a)



(b)

Figure 9 Real image recording of the crosslinked (a) positive molecular photoresist, and (b) negative photoresist.

TABLE II
Etching Resistance of Resists[a]

Resist	PS[b]	*t*-BOC-CD	Posi-PR	Nega-PR	PMMA[b]
Etching rate (Å/s)	16.7	38.3	20.0	18.3	23.3

[a] CF_4:O_2 = 50 : 5 standard cubic centimeters per minute gas flow, 60 mTorr, 150 W, 300 s treatment.
[b] Molecular weight of comparison polymer: Polystyrene = 9000, Poly(methyl methacrylate)= 75,000.

t-BOC-CD shows the worst etching resistance, which is even lower than that of PMMA.

CONCLUSIONS

t-BOC-CD was synthesized and used to prepare a positive photoresist. A crosslinkable positive photoresist was designed by combining *t*-BOC-CD, glutaraldehyde, and a photo-acid generator. A water-developable negative photoresist was developed successfully using β-CD, glutaraldehyde, and a photo-acid generator. The introduction of the *t*-butoxyl group into the β-CD molecule and the addition of glutaraldehyde into the β-CD molecules were both found to decrease the crystallinity of molecules, leading to improved resist film properties. The etching resistance of both the positive and negative resist films was improved by the crosslinking method.

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