Higher Sensitive Extreme Ultraviolet (EUV) Resist Materials Derived From $p$-$t$-Butylcalix[n]arenes ($n = 4$ and 8)

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We examined the synthesis, physical properties, and resist properties of the various polymers and an oligomer containing fixed hole derived from calixarenes. By the condensation reaction of $p$-$t$-butylcalix[n]arene ($n = 4$ and 8) with 1,4-dichloro-2-oxabutane (DCB) and 2,5-dibromoacetyloxy-2,5-dimethylhexane (DBH), the soluble polymers poly(BCA[8]-co-DCB), poly(BCA[8]-co-DBH), and poly(BCA[4]-co-DCB), and an oligomer BCA[4]-DBH were obtained. They have good physical properties (solubility, film-forming ability, high thermal stability), excellent thickness loss property, and good acid-deprotection reactivity upon ultra-violet (UV) irradiation. The resist-sensitivity in an extreme ultraviolet (EUV) exposure tool indicated that poly(BCA[8]-co-DBH) and BCA[4]-DBH were good candidate to offer higher resolution resist pattern, i.e., $E_0 = 5.0 \text{ mJ/cm}^2$ [poly(BCA[8]-co-DBH) and 0.8 mJ/cm² (BCA[4]-DBH).

**Keywords:** Molecular resist, Calixarene, Botryosin, Extreme ultraviolet

1. Introduction

Extreme ultraviolet (EUV) lithography system has been just to be on the practical realization stage. However, the ideal EUV resist pattern cannot be offered, due to that the resist material has a serious problem, which is well known as a trade-off among three resist properties such as sensitivity of exposure tool, resolution and roughness of resist pattern [1]. Therefore, the development of a new EUV resist material has been required strongly.

Many researchers have considered the chemically amplified resist materials based on polymers. While the molecular resist materials such as phenol resins, calixarenes, and fullerene have been considered to overcome the trade-off issue, due to their uniform smaller sizes in the comparison of those of polymers [2]. Our research group has also reported on the synthesis and resist properties based on ladder cyclic oligomer noria (“noria” = water wheel in Latin) [3-11] hyperbranched polyacetal [12-14], nanoscale-gel-shaped compound botryosin (“botryosin” = botrys in Greek) [15,16], tellurium-containing molecules [17,18], and tannic acid [19]. We have also examined the resist sensitivities of various cyclic oligomers using EUV exposure tool [20]. As a result, the resist sensitivity tended to increase with larger holes. When the fixed hole size of the cyclic oligomer increases, its density decreases. That is, the resist sensitivity might increase with decrease of the density of the resist materials.

In this time, we examined the condensation
reaction of \( p-t \)-butylcalix[n]arene (BCA[n]; \( n = 4 \) and 8) and dichloro compounds containing acetal or \( t \)-butyl ester units, anticipating that soluble polymers have botryosin-like structures containing fixed holes and acid labile groups in the main chain, to offer higher resolution resist pattern.

2. Experimental
2.1. Materials
1-Methyl-2-pyrrolidone (NMP) was dried with calcium hydride (CaH\(_2\)) and purified by distillation under reduced pressure. Carbon tetrachloride (CCl\(_4\)), 2,5-dimethylhexane-2,5-diol, pyridine, \( t \)-butylcalix[4]arene (BCA[4]), \( t \)-butylcalix[8]arene (BCA[8]), potassium carbonate (K\(_2\)CO\(_3\)), tetraethylammonium bromide (TBAB), 1,4-dichloro-2-oxabutane (DCB), propylene glycol monomethyl ether acetate (PGMEA), propylene glycol monomethyl ether (PGME), triphenylsulfonium nonaflate (TPS-Nf) and tetramethylammonium hydroxide (TMAH) were used as commercially available.

2.2. Measurements
Infrared (IR) spectra were taken with a JASCO FT/IR4200. The \(^1\)H NMR spectra were recorded on JEOL ECS-400K (400 MHz for \(^1\)H NMR) instruments in DMSO-\( d_6 \) and CDCl\(_3\) with Me\(_4\)Si (TMS) as an internal standard for \(^1\)H NMR. The molecular weights (\( M_n \) and \( M_w \)) of the polymer samples were measured by gel permeation chromatography (GPC) at 40 °C using LiBr and phosphoric acid solution in DMF (20 mM) as a solvent, an eluent flow rate of 0.6 mL/min, and narrow polystyrene standards as reference samples. The measurements were performed using a TOSOH HLC-8320GPC system that was equipped with an EcoSEC GPC System detector using TSKgel SuperAW3000 columns (0.5 - 1000 kg/mol). Thermal analysis was performed on a Shimadzu thermogravimetric analyzer (TGA) TGA-50/50H at a heating rate of 10 °C/min under nitrogen.

2.3. Synthesis of 2,5-dibromoacetoxy-2,5-dimethylhexane (DBH)
2.3.1. Typical procedure
A solution of 2,5-dimethylhexane-2,5-diol (17 mmol, 2.49 g) and pyridine (40 mmol, 3.22 ml) in CCl\(_4\) (30 mL) was prepared at 0 °C. Then, bromoacetyl bromide (40 mmol, 3.22 mL) was slowly added to the solution and stirred at room temperature for 24 h. After the solution was filtered, diethyl ether was added and resulting solution was washed with 0.1 N hydrogen chloride solution, saturated NaHCO\(_3\) solution, and saturation NaCl solution thrice. Organic layer was dried over anhydrous MgSO\(_4\) and was concentrated by rotary evaporator to obtain yellow solid. Yield = 5.14 g (84%). IR (KBr, cm\(^{-1}\)): 2979 (\( \nu \) C-H of methyl), 1725 (\( \nu \) C=O of ester), 1205 (\( \nu \) C-O of ether). \(^1\)H NMR (400 MHz, CDCl\(_3\), TMS) \( \delta \) (ppm): 1.46 (s, -CH\(_3\)), 1.86 (s, -CH\(_2\)-), 3.77 (s, -O-CH\(_2\)-C(O)-).

2.4. Condensation polymerization of BCA[8] and 1,4-dichloro-2-oxabutane (DCB)
2.4.1. Typical procedure
A solution of BCA[8] (1.0 mmol, 1.30 g), tetrabutylammonium bromide (TBAB) (1.0 mmol, 0.32 g), and K\(_2\)CO\(_3\) (10 mmol, 1.38 g), in NMP (7.5 mL) was stirred at 80 °C for 2 h. Then, DCB (4.0 mmol, 0.516 g) was added, and the reaction mixture was stirred at 80 °C for 24 h. The resulting mixture was poured into a large amount of 0.1 N hydrogen chloride solution to precipitate a solid, which was collected by filtration on a membrane filter (Millipore LAWPO 4700 pore size 0.45 μm) and washed with water. The obtained product was dried \textit{in vacuo} at 60 °C for 24 h to afford a cream powder. The molecular weights were estimated by SEC and their structure was confirmed by IR spectroscopy and \(^1\)H NMR spectroscopy. The degree of reaction ratio (DR) of DCB was calculated by the integration ratios of aromatic protons and remained hydroxyl protons, to be DI = 73%. Yield = 1.18 g (83%). \( M_n = 1,730 \) and \( M_w/M_n = 1.40 \). IR (KBr, cm\(^{-1}\)): 3472 (\( \nu \) O-H), 2961 (\( \nu \) C-H of aromatic), 1603 (\( \nu \) C=C of aromatic), 1202 (\( \nu \) C-O of ether). \(^1\)H NMR (400 MHz, CDCl\(_3\), TMS) \( \delta \) (ppm): 0.62 ~ 1.42 (m, -CH\(_3\)), 3.45 ~ 5.71 (m, aromatic H), 8.12 (br, -OH), 5.99 ~ 7.25 (m, aromatic H), 8.43 (br, -OH).

2.5. Condensation reaction of BCA[8] and 2,5-dibromoacetoxy-2,5-dimethylhexane (DBH)
The reaction of BCA[8] and DBH was carried out in the same way for the reaction of BCA[8] and DCB. DR = 99%. Yield = 1.04 g (73%). \( M_n = 2,110 \) and \( M_w/M_n = 1.63 \). IR (KBr, cm\(^{-1}\)): 3416 (\( \nu \) O-H), 2957 (\( \nu \) C-H of aromatic), 1752 (\( \nu \) C=C of aromatic), 1020 (\( \nu \) C-O of ether). \(^1\)H NMR (400 MHz, CDCl\(_3\), TMS) \( \delta \) (ppm): 0.37 ~ 2.24 (br, -CH\(_3\)), 2.78 ~ 5.36 (br m, -CH\(_2\)- and >CH-), 5.99 ~ 7.25 (m, aromatic H), 8.12 (br, -OH).

2.6. Condensation reaction of BCA[4] and DCB
The reaction of BCA[4] (0.5 mmol, 0.33 g) and
DCB (1.0 mmol, 0.10 mL) was carried out in the same way for the reaction of BCA[8] and DCB. DR = 80%. Yield = 0.61 g (67%). $M_n = 1740$ and 520. $M_w/M_n = 1.06$ and 1.05. IR (KBr, cm$^{-1}$): 3392 (µO-H), 2959 (µC-H of aromatic), 1482 (µC=O of ester), 1192 (µC-O of ether). $^1$H NMR (400 MHz, DMSO-$d_6$, TMS) $\delta$ (ppm): 0.65 ~ 1.49 (m, -CH$_3$), 3.45 ~ 4.60 (br m, -CH$_2$- and >CH-), 5.37 (s, O-CH$_2$-O) 6.62 ~ 7.37 (m, aromatic H), 9.12 (s, -OH).

2.7. Condensation reaction of BCA[4] and DBH

The reaction of BCA[4] (0.5 mmol, 0.65 g) and DBH (1.0 mmol, 0.39 g) was carried out in the same way for the reaction of BCA[8] and DCB. DR = 50%. Yield = 0.34 g (60%). $M_n = 560$ and $M_w/M_n = 1.17$. IR (KBr, cm$^{-1}$): 3426 (µO-H), 2960 (µC-H of aromatic), 1737 (µC=O of ester), 1482 (µC=O of aromatic), 1201 (µC-O of ether). $^1$H NMR (400 MHz, CDCl$_3$, TMS) $\delta$ (ppm): 0.96 (s, -CH$_3$), 1.20 (s, -CH$_3$), 1.49 (s, -CH$_2$-), 3.83 (dd, >CH-), 4.86 (d, -O-CH$_2$-C(O)-), 6.83 (d, aromatic H), 8.06 (s, -OH).

2.8. Thickness loss property on the silicon wafer after soaking in 2.38 wt% tetramethylammonium hydroxide (TMAH) aq.

The solutions of the synthesized polymers and oligomer in PGMEA were spin-coated on the silicon wafer to prepare corresponding thin films with about 50 nm thickness. The value of thickness of the thin films was determined by ellipsometry before and after soaking in 2.38 wt% TMAH aq. for 30 sec.

2.9. Photo-deprotection reaction rate

The solutions of the synthesized polymers and oligomer (0.5 g) and TPS-Nf (10 wt%) as a photoacid generator in CHCl$_3$ (2 mL) was cast on a KBr plate and dried in vacuo for 2 h. The obtained film was irradiated with a UV lamp (614 nm, 50% transmission), TMS) and after soaking in 2.38 wt% TMAH aq. for 30 sec. The film thickness was measured with an ellipsometry to obtain sensitivity curves.

3. Results and discussion

3.1. Reaction of $p$-tert-butylcalix[8]arene (BCA[n]) (n = 4 and 8) with 1,4-dichloro-2-oxabutane (DCB) and 2,5-dibromoacetoxy-2,5-dimethylhexane (DBH) (Scheme 1)

Reaction of BCA[8] and DCB was examined using potassium carbonate as a base in NMP at 80 °C for 24 h in the presence of TBAB as a catalyst, yielding the soluble polymer poly(BCA[8]-co-DCB) with $M_n = 1,730$ and $M_w/M_n = 1.40$ in 83% yield (Scheme 1 [A]). Its structure was confirmed by $^1$H NMR and IR spectroscopy. In $^1$H NMR spectroscopy, the peaks at 6.31 ~ 7.59 and around 8.43 ppm were seen, which were assignable to aromatic protons and hydroxyl groups, respectively. This means that condensation reaction of BCA[8] and DCB proceeded to give corresponding a soluble polymer containing both units of BCA[8] and DCB. In our previous report, we examined the reaction of BCA[8] with hexamethylene disiocyanate (HDI), and the only soluble polymer poly(BCA[8]-co-HDI) could be obtained in high yield. Analysis by $^1$H NMR, IR, MALDI-TOF mass spectroscopy, and AFM indicated that poly(BCA[8]-co-HDI) consists of nanoscale-gel-shaped structures resembling a bunch of grapes, presumably because the hydroxyl groups of BCA[8] are located at the lower rim and the polymerization of BCA[8] and HDI proceeded homogeneously not to three dimensional cross-linked polymers. We named the soluble polymer botryosin, after the Greek word botrys, meaning a bunch of grapes. By the consideration based on our mentioned previous report, the synthesized poly(BCA[8]-co-DCB) should have botryosin-type structure as shown in Scheme 1. These conditions and results are also summarized in Table 1.

In the same way for the reaction of BCA[8] and DCB, the reaction of BCA[8] and DBH was...
performed to give corresponding soluble polymer poly(BCA[8]-co-DBH) with $M_n = 2,110$ and $M_w/M_n = 1.63$ in 73% yield. The synthesized poly(BCA[8]-co-DCB) and poly(BCA[8]-co-DBH) have acetal and tertiary ester units in the main chain, which are applicable to a main-chain scission type positive resist material.

Furthermore, the condensation reaction of BCA[4] with DCB and DBH was examined in the same way as mentioned above. As the results, no cross-linking product could be obtained, and only soluble compounds were obtained. In the case of DCB, the mixture compounds containing a polymer with $M_n = 1740$ and $M_w/M_n = 1.06$ and an oligomer with $M_n = 650$ and $M_w/M_n = 1.05$ were obtained in 67% yield, which were estimated by SEC (Fig. 1). The polymer part is expected to be a botryosin-type polymer and the oligomer one is expected to be an intramolecular cyclization reaction product. However, in the case of DBH, only a low molecular compound was obtained. Figure 1 depicts the SEC profiles of the synthesized polymers and oligomer. Although poly(BCA[8]-co-DCB), poly(BCA[8]-co-DBH), poly(BCA[4]-co-DCB) showed multimodal peaks, the BCA[4]-DBH showed an unimodal peak and its $M_n$ and $M_w/M_n$ were 560 and 1.17, indicating that a single compound could be obtained.

The $^1$H NMR spectroscopy of BCA[4]-DBH showed that it was consistent with both unit of BCA[4] and DBH with composition ratio of BCA[4]/DBH = 1/1. The peaks at around 8.1 ppm were also seen assignable to hydroxyl groups of BCA[4], and degree of reaction ratio (DR) was calculated to be almost 50%. This result means that the condensation reaction BCA[4] and DBH proceeded to give a soluble compound containing intramolecular cross-linked DBH unit, i.e., BCA[4]-DBH might have cup-shaped structure, tertiary ester groups and two hydroxy groups in the main chain as shown in Scheme 1[B]. These results are also summarized in Table 1.

Furthermore, thermal stability of the synthesized poly(BCA[8]-co-DCB), poly(BCA[8]-co-DBH), poly(BCA[4]-co-DCB), and BCA[4]-DBH was determined by thermo gravimetric analysis (TGA), and their initial decomposition temperatures ($T_d$) were in the range between 185 and 385 °C. These compounds also showed good solubility for common organic solvents such as DMSO, DMF, THF, CHCl₃, propylene glycol monomethyl ether acetate (PGMEA), and propylene glycol monomethyl ether (PGME), and good film forming ability. These results indicate that the synthesized polymers and oligomer are applicable to photoresist materials.

### 3.3. Thickness loss property on the silicon wafer after soaking in 2.38 wt% TMAH aq. solution

Next, the value of film thickness loss of the thin films prepared from the synthesized poly(BCA[8]-co-DCB), poly(BCA[8]-co-DBH), poly(BCA[4]-co-DCB), and BCA[4]-DBH was determined anticipating for the application of positive-type resist materials. As a result, they showed good
substrate adhesion. These results are summarized in Table 2. The decrease of their thicknesses was hardly observed, i.e., the ratios of decrease were <1%. This means that they can be used as positive resist materials with 2.38 wt% TMAH aq. as an alkaline developer.

3.4. Photo-induced deprotection reaction rate

Photo-induced deprotection reaction was examined in the film state in the presence of photoacid generator (PAG) under UV irradiation. The films were prepared with 10 mol% of TPS-Nf as a PAG and were performed under UV irradiation (614 μW/cm²) using a filter (Extract only 254 nm light) under atmosphere for 30 min, followed by heating at 110°C. The rate of decrease of acetal or tertiary-alcohol ester groups was monitored with FT-IR spectrometer around 980 and 950 cm⁻¹, respectively. Figure 2 depicts the IR spectra before and after the photoinduced deprotection reactions of BCA[4]-DBH. Before the deprotection reaction, a peak at around 950 cm⁻¹ was seen, assignable to the stretching vibration of tertiary ester group (Fig. 2 [A]). After heating for 5 min, a new broad peak about 3350 cm⁻¹, which was assignable to the stretching vibration of carboxylic acid groups (Fig. 2 [B]). This result shows that the deprotection reaction of tertiary ester groups proceeded, generating carboxylic acid groups with release of a 2,5-dimethylhexa-2,4-diene as shown in Scheme 2.

From the changing of these peaks, it is possible to calculate the conversion of the photoinduced deprotection reaction, and after 30 min, the conversion rate was calculated to be 71% (Fig. 2 [C]). In the same way, the conversion ratio of poly(BCA[8]-co-DCB), poly(BCA[8]-co-DBH), and poly(BCA[4]-co-DCB) were calculated to reach 25, 40 and 48%, respectively. Figure 3 shows the time courses of conversion in these photo-induced deprotection reactions. Thus, the order of photo-induced deprotection reactivity is BCA[4]-DBH > poly(BCA[4]-co-DCB) > poly(BCA[8]-co-DBH) > poly(BCA[8]-co-DCB), i.e., the reactivity appears to be consistent with the structures of the resist materials.

### Table 1. Condensation reaction of BCA[n] (n = 4 and 8) with CPP and DBH.

| Run | Feed Ratios | Composition Ratios | Yield (%) | $M_n (M_w/M_n)$ | DR (%) | $T_{d1} (°C)$ |
|-----|-------------|--------------------|-----------|-----------------|--------|--------------|
| 1   | BCA[8]/DCB = 1/4 | BCA[8]/DCB = 1/4 | 83        | 1,730 (1.40)    | 73     | 300          |
| 2   | BCA[8]/DBH = 1/4  | BCA[8]/DBH = 1/4  | 73        | 2,110 (1.63)    | 99     | 204          |
| 3   | BCA[4]/DCB = 1/2  | BCA[4]/DCB = 1/2  | 67        | 1740 (1.06), 650 (1.05) | 77     | 266          |
| 4   | BCA[4]/DBH = 1/2  | BCA[4]/DBH = 1/2  | 60        | 520 (1.17)      | 50     | 185          |

*a Condition; K$_2$CO$_3$ in NMP at 80 °C. bEstimated by SEC based on polystyrene standards; LiBr and phosphoric acid solution in DMF (20 mM). cDR; degree of reaction ratio of hydroxyl groups calculated by $^1$H NMR spectroscopy. d$T_{d1}$ = Initial thermal decomposition temperature determined by TGA.

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### Table 2. The ratio of thickness lost after soaking in TMAH aq.

| Compounds | poly(BCA[8]-co-DCB) | poly(BCA[8]-co-DBH) | poly(BCA[4]-co-DCB) | BCA[4]-DBH |
|-----------|----------------------|----------------------|----------------------|------------|
| Thickness Lost | <1% | <1% | <1% | <1% |
3.5. Resist sensitivity

The resist sensitivity of poly(BCA[8]-co-DCB), poly(BCA[8]-co-DBH), poly(BCA[4]-co-DCB), and BCA[4]-DBH was examined using EUV exposure tool. Solutions of these compounds in the presence of 10 wt% of TPS-Nf as a PAG were spin-coated on silicon wafers to prepare thin films of about 40 nm thickness. The postexposure baking (PEB) temperature and time were 110 °C and 30 s, respectively. Next, the silicon wafer allows development by dipping in 2.38 wt% TMAH aq. at room temperature for 30 sec. afterward rinsed in deionized water before drying. The value of the thickness of remained thin film was measured by means of ellipsometer after the lithography process. These results are illustrated in Fig. 4.

Although, thin films of poly(BCA[8]-co-DCB) and poly(BCA[4]-co-DCB) remained mostly after the lithography process with 10 mJ/cm² EUV-exposure dose, the thin films of poly(BCA[8]-co-DBH) and BCA[4]-DBH were fully soluble in aqueous alkaline solution after 5.0 mJ/cm² and 0.8 mJ/cm², i.e., $E_0$ (value of resist sensitivity) = 5.0 mJ/cm² and 0.8 mJ/cm², respectively. Furthermore, the photoresist contrast values $\gamma$ could be extracted from the sensitivity curves of poly(BCA[8]-co-DBH) and BCA[4]-DBH to be 2.5 and 8.0, respectively, i.e., BCA[4]-DBH has higher value of $\gamma$ and is expected to offer higher contrast resist pattern.

4. Conclusion

In summary, we examined the condensation reaction of $p$-tert-butylcalix[n]arene (BCA[n]) (n = 4 and 8) with 1,4-dichloro-2-oxabutane (DCB) and 2,5-dibromoacetyloxy-2,5-dimethylhexane (DBH), yielding soluble polymers poly(BCA[8]-co-DCB), poly(BCA[8]-co-DBH), and poly(BCA[4]-co-DCB), and a oligomer BCA[4]-DBH. The synthesized poly(BCA[8]-co-DCB), poly(BCA[8]-co-DBH), poly(BCA[4]-co-DCB), and BCA[4]-DBH showed good physical properties (solubility, film-forming ability, high thermal stability), excellent thickness loss property, and good acid-deprotection reactivity upon UV irradiation.

These mean that they are applicable to positive-working photoresist materials. Furthermore, their resist sensitivity was examined in the film state using EUV exposure tool, and it was observed that BCA[4]-DBH has higher sensitivity: $E_0 = 0.8$ mJ/cm², indicating that it is good candidate EUV resist material to offer higher resolution resist pattern.

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