Extreme Ultraviolet (EUV)-Resist Materials of Noria (Water Wheel-Like Cyclic Oligomer) Derivatives containing Acetal Moieties

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The synthesis and properties of noria derivatives (noria-CHVE's) with pendant acetal moieties were examined for application as extreme ultraviolet (EUV)-resist materials. The reaction of noria and cyclohexyl vinyl ether was carried out in the presence of pyridinium *p*-toluenesulfonate as a catalyst, to give the corresponding noria-CHVE's with various degrees of introduction (DI) of cyclohexyl acetal moieties by adjusting the feed ratio of reactant and reaction time. The physical properties (solubility, thermal stability, and film-forming ability) of noria-CHVE's were consistent with the differences in DI values. The patterning properties of noria-CHVE₅₀ (DI = 50) and noria-CHVE₅₉ (DI = 59) were investigated in an EUV-resist system, and it was found that noria-CHVE₅₀ gave higher resolution than noria-CHVE₅₉, providing a clear line and space pattern with a resolution of 37.9 nm and a line-edge roughness (LER) of 7.2 nm.

Keywords : noria, acetal, chemically amplification, extreme ultraviolet, resist

1. Introduction

New lithography systems employing electron beams (EB) and short-wavelength lasers, such as KrF ($\lambda = 248$ nm), ArF ($\lambda = 193$ nm), and extreme ultraviolet (EUV) ($\lambda = 13.5$ nm) are expected to produce the higher resolution semiconductor devices, and the development of new photo-resist materials has been strongly required. Among them, photo-functional polymers containing adamantane, norbornene, fluorine atoms, and certain protecting groups such as trimethylsilyl and tetrahydropyranyl groups showed higher photo-sensitivity, lower outgassing, and low line-edge roughness (LER).¹

Recently, many molecular glass resists based on dendritic oligomers,² calixarenes,³ low-molecular-weight polyphenols,⁴ and fullerenes⁵ could show high photo-sensitivity in EB and EUV lithography systems. We have also examined the synthesis and properties of the

calixarene derivatives containing photo-degradable groups, such as *t*-butyloxycarbonyl,⁶ *t*-butyl ester,⁷ and acetal groups.⁸ As the results, although these derivatives showed excellent photosensitivity, a clearly resolved pattern could not be achieved, due to that the line pattern was broken down during the etching process.⁹ This means that the mechanical property of calixarene derivatives is insufficient to construct the higher resolution patterns. Very recently, we have succeeded the synthesis of a ladder cyclic molecule "noria" (noria = water wheel in Latin) by the condensation reaction of resorcinol and 1,5-pentanedial under dynamic covalent chemistry (DCC) system.¹⁰ As shown in Scheme 1, the noria derivatives with pendant photo-degradable groups have been examined in the application as EB and EUV resist materials. The noria derivatives containing

t-butoxycarbonyl groups¹¹ and *t*-butyl ester groups¹² showed clear line and space patterns with resolutions of 50 ~ 70 nm in EB resist system. The noria derivative containing adamantate groups could show clear line and space patterns with resolutions of 26 nm in EUV resist system.¹³ Furthermore, the noria derivative with pendant oxetanyl groups was applicable as negative EUV resist material and showed clear line and space patterns with resolutions of 50 nm.¹⁴

Scheme 1

In this paper, we examined the synthesis of noria derivative containing cyclohexyl acetal moieties and its application as an EUV-resist material.

2. Experimental

2.1 Materials.

Tetrahydrofuran (THF) was dried over Na wire and purified by distillation before use. Other commercial organic solvents, pyridinium *p*-toluenesulfonate, trioctylamine, tetramethyl ammonium hydroxide (TMAH), triphenylsulfonium trifluoromethanesulfonate (TPS-TF), and triphenylsulfonium perfluorobutanesulfonate (TPS-PBF) were used without further purification. Cyclohexyl vinyl ether was donated from NIPPON CARBIDE INDUSTRIES Co., INC.

2.2 Measurements.

Infrared (IR) spectra were measured on a Thermo Electron Model NICOLET 380 spectrometer. The ¹H NMR spectra were recorded on JEOL Model JNM α-500 (500 MHz for ¹H NMR) instruments in DMSO-*d*₆ and CDCl₃ using Me₄Si (TMS) as an internal standard

reagent for ¹H NMR. The number-average molecular weight (*M_n*) and molecular weight distribution (*M_w*/*M_n*) of the polymers were estimated by size exclusion chromatography (SEC) with a Tosoh model HLC-8120 GPC equipped with refractive index and ultraviolet detectors using TSK gel columns (eluent THF, calibrated with narrow-molecular-weight polystyrene standards). Thermal analysis was performed on a Seiko Instruments thermogravimetric analyzer (TGA) Model EXSTAR6000/TG/DTA6200 at a heating rate of 10 °C/min under nitrogen. The rate of photochemical deprotection reaction of the acetal moieties was measured by means of real-time IR (RT-IR) spectroscopy (BIO RAD model Excalibur FTS3000MX spectrometers).

2.3 Synthesis of noria derivatives (noria-CHVE's).

A mixture of noria (34.1 g, 20 mmol) and cyclohexyl vinyl ether (CHVE) (102 mL, 720 mmol) in THF (150 mL) was stirred for room temperature. After 2 h, pyridinium *p*-toluenesulfonate (3.62 g, 14 mmol) was added, and resulting mixture was stirred at room temperature for 12 h. NEt₃ (5.6 mL, 40 mmol) and CHCl₃ (500 mL) was added and resulting solution was washed with saturated aqueous sodium hydrogen carbonate. The organic phase was dried over MgSO₄ and concentrated in a rotary evaporator. The residue was dissolved in CHCl₃, followed by precipitation from *n*-hexane to obtain a solid (noria-CHVE). The degree of introduction (DI) of cyclohexyl acetal moieties, calculated from the integration values of aromatic protons of the noria moiety at 5.85 ~ 8.07 ppm, as well as methine protons of noria moiety, methane protons of acetal moieties, and methine protons of the cyclohexyl moieties at 2.78 ~ 5.85 ppm, was found to be 50%. Yield 21.6 g (42 %). IR (film, cm⁻¹): 3407 (ν OH), 2987, 2982, and 2856 (ν CH₂), 1608 and 1582 (ν C=C), 1023 (ν C-O-C), 981 (ν O-C-O acetal moieties). ¹H NMR (500 MHz, CDCl₃, TMS) δ(ppm) = 0.21 ~ 2.45 (m, 220H, methylene protons of cyclohexyl moiety and -CH₂-CH₂-CH₂- of noria moiety, methyl protons of acetal moiety), 2.78 ~ 5.85 (m, 43H, methine protons of noria, acetal, and cyclohexyl moieties), 5.85 ~ 8.07 (m, 24 H, aromatic H). Other noria-CHVE's with various DI values of 59 and 91 % were similarly synthesized by modifying the feed ratio and reaction time, as shown in Table 1.

2.4 Photoinduced deprotection of noria-CHVE.

A solution of noria-CHVE₅₀ (64 mg, 0.02 mmol) and TPS-TF (4.9mg, 5 mol %) as a photo-acid generator (PAG) in THF (3 mL) was cast on a KBr plate and dried *in vacuo* for 0.5 h. The obtained film was irradiated with a 250 W high-pressure mercury lamp (Ushio Electric Co., 5 mW/cm²) without a filter under nitrogen for 15 min at room temperature. The rate of decrease of cyclohexyl moieties was monitored with an RT-IR spectrometer at 980 cm⁻¹ which was corresponding peaks of acetal moiety.

2.5 Patterning ability.

The patterning properties of noria-CHVE were examined as follows. A photo-resist was prepared from noria-CHVE, TPS-PBF as a photoacid generator (10 wt %), trioctylamine as a quencher (1.25 wt %), and PGMEA as the solvent. The photo-resist solution was filtered through a 0.5 μm filter prior to evaluation. The resist sample was coated at 51 nm film thickness on a silicon wafer. The photo-resist film was baked (SB: Soft Bake), exposed, re-baked (PEB: Post Exposure Bake) and then developed with TMAH (2.38 wt %) aqueous solution for approximately 30 seconds. All imaging exposures were made using the 0.3 numerical aperture (NA) small field exposure tool (SFET)¹⁵ connected to a coater/developer track system ACT-12 in a chemically controlled environment. Patterns were observed using a critical-dimension scanning electron microscope (CD-SEM, S9380II, Hitachi).

3. Results and discussion

3.1 Synthesis of noria-cyclohexyl vinyl ether (noria-CHVE).

The noria derivatives (noria-CHVE's) with pendant cyclohexyl acetal moieties were synthesized by addition reaction of noria and cyclohexyl vinyl ether in the presence of pyridinium *p*-toluenesulfonate as a catalyst in THF. By the control of the feed ratio and

reaction time, noria-CHVE's with various degrees of introduction of cyclohexyl acetal moieties (DI) = 50 ~ 91 % were synthesized (Scheme 2). These conditions and results are summarized in Table 1.

The structure of noria-CHVE's was confirmed by IR and ¹H NMR spectroscopy. Figure 1 depicts the ¹H NMR spectrum of noria-CHVE₅₀ (run 1 in Table 1), along with that of noria. The spectrum shows new peaks assignable to methylene protons and methine protons of the cyclohexyl acetal moiety at 0.21 – 2.45 ppm and 2.78 – 5.85 ppm, respectively, and indicated that the reaction of noria and cyclohexyl vinyl ether proceeded to give acetal moieties, i.e., the noria derivative with pendant cyclohexyl moiety was obtained. The degree of introduction of cyclo-

Scheme 2

hexyl moiety (DI) was calculated from the integration ratios of aromatic protons of noria moiety at 5.85 ~ 8.07 ppm, and the methine protons of the noria moiety, acetal moiety, and cyclohexyl moiety at 2.78 – 5.85 ppm in the ¹H NMR spectrum, and was 50%. We could synthesize noria-CHVE's with DI's = 50 ~ 91 %

Table 1. The synthesis of noria-CHVE's^a

Run	feed ratio	reaction time (h)	noria derivative	D.I. ^b	Yield ^c (%)	M _n ^d	M _w /M _n ^d	T _{di} ^e
1	noria / CHVE = 1/1.5	12	noria-CHVE ₅₀	50	42	2360	1.04	112
2	noria / CHVE = 1/2	12	noria-CHVE ₅₉	59	42	2540	1.03	121
3	noria / CHVE = 1/2	6	noria-CHVE ₉₁	91	41	2650	1.03	135

^aThe reaction of noria and CHVE was carried out in the presence of pyridinium *p*-toluenesulfonate in THF at room temperature. ^bCalculated by ¹H NMR. ^cMethanol insoluble part. ^dEstimated by SEC (eluent; THF). ^eT_{di} = The initial thermal decomposition temperature determined by TGA.

by appropriately adjusting the feed ratio and reaction time (Table 1). Number-average molecular weight (M_n), weight-average molecular weight (M_w), and polydispersity ratio (M_w/M_n) were also determined by means of SEC. Although the values of M_n and M_w of noria-CHVE's (DI = 50, 59, and 91%) increased with increasing value of DI. However, the values of M_w/M_n were all the same (1.03 ~ 1.04), i.e., the reaction can be controlled very precisely.

Figure 1. ^1H NMR spectra of noria and noria-CHVE₅₀. (A) noria (500 MHz, $\text{DMSO}-d_6$). (B) noria-AD₅₀ (DI = 50%) (500 MHz, CDCl_3).

3.2 Physical properties (solubility, film-forming ability, and thermal stability) of noria-CHVE's.

The physical properties of noria-CHVE's were examined relevant to application as EUV-resist materials.

3.3 Solubility.

Table 3 summarizes the characteristics solubility of noria-CHVE's (DI = 50, 59, and 91%) and noria. Noria was soluble in TMAH aqueous solution, whereas all noria-CHVE's were insoluble. This means that noria-CHVE's can be applicable to alkali-developable resist material. In the case of polyethylene glycol monomethyl ether (PGME), noria-CHVE₅₀ and noria-CHVE₅₉ were soluble, while noria-AD₉₁ was soluble in part. These results indicate that noria-CHVE₅₀ and noria-CHVE₅₉ might be suitable as EUV-resist materials.

3.4 Film-forming ability.

The film-forming ability of noria-CHVE's (DI = 50, 59, and 91%) was examined by spin coating a solution of noria or noria-CHVE's 50 mg in poly(ethylene glycol) methylether acrylate (PEGMEA) 1.0 ml on a silicon wafer, and drying the wafer *in vacuo* at room temperature. All noria-CHVE's had good film forming property with high transparency.

3.5 Thermal stability.

The thermal decomposition of noria-CHVE's was measured by means of TGA. Decomposition started in the range of 112 ~ 137 °C, as summarized in Table 1. It was observed that the thermal stability increased with increasing the values of DI of synthesized noria-CHVE's. This might be because acidic phenol groups had been replaced with acetal groups, i.e., acidic phenol groups might promote the thermal decomposition reaction of noria-CHVE's. However, we expected that the remaining hydroxyl groups of noria-CHVE result in higher hydrophilicity and thus provide good adhesion to the substrate.

As the result, we considered that noria-CHVE₅₀, and noria-CHVE₅₉ are good candidates for EUV-resist materials.

3.6 Photoinduced deprotection reaction of noria-CHVE.

The photoinduced deprotection of films prepared with 5 mol % of TPS-TF as a photoacid generator was carried out under UV irradiation with a 250 W high-pressure mercury lamp. Figure 2 depicts the RT-IR spectra during the exposed with UV of noria-CHVE₅₀ (run 1 in Table 1). Before the deprotection reaction, a peak at around 980 cm^{-1} was seen, assignable to the stretching vibration of O-C-O of acetal moiety (Figure 2 [a]). After heating for 1 min, a

Table 2. The solubility of noria-CHVE

Solvent	noria	noria-CHVE ₅₀	noria-CHVE ₅₉	noria-CHVE ₉₁
water	-	-	-	-
DMSO	++	-	+-	-
DMAc	++	++	+-	+-
DMF	+-	+-	+-	+-
Acetonitrile	-	-	-	-
Methanol	-	-	-	-
2-Propanol	-	+-	+-	+-
1,4-Dioxane	-	++	++	++
THF	-	++	++	++
Acetone	-	++	++	+
Methyl ethyl ketone	-	++	+-	++
<i>o</i> -Dichlorobenzene	-	++	++	++
Ethyl acetate	-	++	++	++
Ethyl ether	-	++	++	++
Chloroform	-	++	++	++
Anisol	-	++	++	++
Chlorobenzene	++	++	++	++
Toluene	-	++	++	++
Cyclohexanone	-	++	++	++
<i>n</i> -Hexane	-	-	+-	++
PGMEA ^a	-	++	++	++
PEGME ^b	-	++	++	+-
TMAH ^c (2.38 wt%)	++	-	-	-
a.q.				
Film-forming ability	×	○	○	○

^a Poly(ethylene glycol) methylether acrylate (PEGMEA).^b Polyethylene glycol monomethyl ether (PEGME).^c Tetramethyl ammonium hydroxide (TMAH).

new broad peak appeared at around 3300 cm⁻¹, which was assignable to the stretching vibration of hydroxy groups (Figure 2 [B]). This result shows that the deprotection reaction of acetal moieties proceeded, to produce noria. The peak at 980 cm⁻¹ due to O-C-O of acetal moieties decreased during the exposure. From the change of this peak, it is possible to calculate the conversion of the photoinduced deprotection reaction, and after 15 min, the conversion rate was calculated to be 94% (Figure 2 [E]).

Furthermore, the resulting product was soluble in 2.38 wt% TMAH aqueous solution. The conversions of noria-CHVE's (DI = 59 and 91) were similarly calculated to reach 94 and 93 %, respectively. This means that the noria-CHVE's are good candidates for positive-type working resist materials.

3.7 Patterning properties of noria-CHVE.

In our previous report, we examined the synthesis and patterning property in EUV system of noria derivatives noria-AD with pendant adamantate groups. As the result, noria-AD₂₃ with DI = 23 % provided a more clear line and space pattern with a resolution of 26 nm compared to that of noria-AD₄₄ with DI = 44 %. This indicated that noria-AD with lower DI of adamantate groups would achieve higher resolution. In this time, we examined the patterning properties of the films prepared from noria-CHVE₅₉ and noria-CHVE₅₀.

Figure 3 (A) depicts the clear patterns in the case of the mask size at 45 nm obtained with noria-CHVE₅₉ and noria-CHVE₅₀, using trioctylamine as a quencher at 90 °C soft bake (SB) temperature and 90 °C

post exposure bake (PEB) temperature. In the case of noria-CHVE₅₉, a 45.3 nm resolution pattern with LER = 8.0 nm could be obtained using a exposure dose = 17.0 mJ/cm². Noria-CHVE₅₀ showed that a 45.6 nm resolution pattern with LER = 6.9 nm could be obtained using a exposure dose = 14.5 mJ/cm² (Figure 3 [A]).

This means that noria-CHVE₅₀ had higher potential to offer higher resolution patterns, due to that a pattern at 45 nm mask could be achieved

with lower exposure dose and lower LER compared to those of noria-CHVE₅₉. We next examined 40, 35, and 32 nm sizes of mask in EUV resist system. These results are depicted in Figure 3 (B), (C), and (D). Clearly resolved patterns at 37.9 nm with LER = 7.2 nm could be obtained with noria-CHVE₅₀. This indicates that noria derivatives with lower DI of cyclohexyl moieties would achieve higher resolution. i.e., the critical value of DI in noria-CHVE might be lower than 50%. It seems that critical point is dramatically solubility change point upon photodeprotection, offering both higher photosensitivity and higher pattern resolution.

Figure 2. RT-IR spectra of noria-CHVE₅₀ before and after photoirradiation. (A) noria-CHVE₅₀ (B) After 1 min. (C) After 5 min. (D) After 10 min. (E) After 15 min.

	(A)	(B)	(C)	(D)
Mask size L/S (nm)	45	40	35	32
Noria-CHVE ₅₉ Dose = 17.0 mJ/cm ²
Critical Dimension (LER) (nm)	45.3 (8.0)	- a)	- a)	- a)
Noria-CHVE ₅₀ Dose = 14.5 mJ/cm ²
Critical Dimension (LER) (nm)	45.6 (6.9)	41.8 (6.5)	37.9 (7.2)	- a)

Figure 3. EUV-resist patterns obtained with noria-CHVE₅₀ and noria-CHVE₅₉ at various exposure doses at 32 ~ 45 nm mask sizes. (A) Mask size = 45 nm. (B) Mask size = 40 nm. (C) Mask size = 35 nm. (D) Mask size = 32 nm. ^{a)} Not determined.

Conclusion

The synthesis, physical properties, and EUV-resist properties of noria derivatives (noria-CHVE's) with pendant cyclohexyl acetal moieties were examined. The noria-CHVE's was synthesized by the reaction of noria and cyclohexyl vinyl ether and their values of degree of introduction (DI) of acetal moieties were controlled by the adjustment of feed ratios and reaction time. i.e. DI = 50, 59, and 91%. It was showed that all noria-CHVE's had good physical properties for application as resist materials. Noria-CHVE₅₀ and Noria-CHVE₅₉ provided clear line and space patterns of 37.9 nm resolution pattern with LER = 7.2 nm and 45.3nm resolution pattern with LER = 8.0 nm, respectively. These results showed that noria-CHVE's had high potential as next-generation EUV-resist materials, and noria-CHVE's with lower DI would achieve higher resolution.

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