Extreme Ultraviolet (EUV)-Resist Material Based on Noria (Water Wheel-like Macrocycle) Derivatives with Pendant Alkoxy and Adamantyl Ester Groups

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1. Introduction

Photolithography system employing electron beam (EB) and shorter wavelength lasers, such as KrF ($\lambda = 248$ nm), ArF ($\lambda = 193$ nm), and extreme ultraviolet (EUV, $\lambda = 13.5$ nm) have been expected to offer higher resolution pattern. According to the developments of photolithography system, the development of new resist materials have been required. Very recently, extreme ultraviolet (EUV) laser has been expected to offer higher resolution pattern with less than about 20 nm resolution. However, there is a serious problem among the resolution of pattern, sensitivity of the photo-chemical reaction, and roughness of the pattern, i.e., these relationships are well known as “trade-off”. To overcome this “trade-off”, many molecular glasses such as calixarenes,¹⁻¹⁰ dendritic oligomers,¹¹⁻¹⁸ low-molecular-weight oligomers,^{19,20} and fullerenes²¹ have been reported and their resolutions showed about 50 nm regions. Recently, we could design a ladder type cyclic oligomer “noria” (noria = water wheel in Latin),²² and examined the synthesis, physical properties, and patterning properties of noria derivatives with pendant *t*-butyl ester groups,^{23,24} *t*-butoxy-carbonyl groups,²⁵ adamantyl ester groups,^{26, 27} cyclohexyl acetal moiety,²⁸ and oxetanyl groups²⁹

using electron beam (EB) or EUV exposure systems. Furthermore, noria derivative with pendant a protective group could achieve to offer clear 22 nm resolution pattern using EUV exposure system.³⁰

Meanwhile, noria analogues with pendant twelve alkoxy groups could be synthesized by the condensation reaction of 1,5-pentanediol with 3-methoxyphenol or 3-ethoxyphenol under dynamic covalent chemistry (DCC) system.³¹ In DCC system, noria derivatives with pendant twelve methoxy groups (noria_{MP}) and twelve ethoxy groups (noria_{EP}) could be obtained selectively and their solubility and film-forming ability are superior to those of noria. These results expected that noria_{MP} and noria_{EP} derivatives with pendant protective groups can have superior resist properties using EUV exposure system.

In this communication, we synthesized noria_{MP} derivatives (noria_{MP}-AD_n) with pendant adamantyl ester groups, and their physical properties and EUV patterning properties were examined.

2. Experimental

Materials.

3-Methoxyphenol (MP), tetrabutylammonium bromide (TBAB), and propylene glycol methyl ether acetate (PGMEA) were purchased from Tokyo Chemical Industry Co., Ltd (Tokyo, Japan). 1,5-Pentanedial (50% aqueous solution) and 1-methyl-2-pyrrolidone (NMP) were purchased from Kanto Chemical Co., Inc (Tokyo, Japan). Trifluoroacetic acid (TFA), trioctylamine (TOA), and potassium carbonate (K_2CO_3) were purchased from Wako Pure Chemical Industries, Ltd (Osaka, Japan). 2-Bromoacetyloxy-2-methyladamantane (BMA) was obtained from Idemitsu Kosan Co., Ltd (Tokyo, Japan). Anhydrous magnesium sulfate ($MgSO_4$) was purchased from Yoneyama Yakuhin Kogyo Co., Ltd (Osaka, Japan). Chloroform and methanol were purchased from Godo Co., Ltd (Tokyo, Japan). Triphenylsulfonium triflate (TPS-Tf) was obtained from JSR (Tokyo, Japan). Triphenylsulfonium nonaflate (TPS-Nf) was donated from Selete (Ibaraki, Japan). All chemicals were used as commercial grade without further purification.

Measurements.

Infrared ray (IR) spectra were taken by NICOLET 380 FT-IR (ThermoFisher Scientific Inc. USA). Hydrogen magnetic resonance (1H NMR) spectra were recorded on a JNM-ECA-600 (JEOL Ltd. Japan) by dissolving samples in deuterated $CHCl_3$ and dimethylsulfoxide (DMSO). Matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-TOF MS) analysis was carried with an AXIMA-CFR plus (Shimadzu Co. Japan) in positive mode. Thermogravimetric analysis (TGA) was performed on EXSTAR 6000 (Seiko Instruments Inc. Japan) with the sample in an inert nitrogen atmosphere. Prior to testing, the samples were dried at 100 °C for 20 min under nitrogen and then thermally ramped in the TGA to 600 °C at a heating rate of 10 °C/min. The glass transition temperature (T_g) was measured on a Seiko Instruments differential scanning calorimeter (DSC) model EXSTAR6000/DSC6200 at heating rate of 10 °C/min under nitrogen. Size exclusion chromatography (SEC) was performed by HLC-8220 (Tosoh Co. Japan) having a Shodex Asahipak GF-510 HQ (7.5 i.d. $\times$ 300 mm) and two GF-310 HQ columns (7.5 i.d. $\times$ 300 mm), and the DMF solution containing 20 mM anhydride LiBr and 20 mM H_3PO_4 was used as a mobile phase. Film thickness was measured by ellipsometer (DHA-OLX, Mizojiri Optical Co., Ltd.). The EUV lithography was performed by small field exposure

tool (SFET) with 0.3 numerical aperture connected to a coater/developer track system ACT-12 in a chemically controlled environment. The scanning electron microscope (SEM) images were observed by critical-dimension SEM (CD-SEM, S9380II and S9220, Hitachi). Powder X-ray diffraction (PXRD) analysis was performed by MultiFlex (Rigaku Corporation) on the room temperature.

Synthesis of *noria*_{MP}.

A mixture of 1,5-pentanedial (50% in water, 10 g, 50 mmol), 3-methoxyphenol (24.8 g, 200 mmol), and TFA (37.5 g, 330 mmol) as an acid catalyst in chloroform (50 mL) was stirred at reflux for 48 hours. The reaction solution was poured into methanol, and the precipitation was collected by filtration and then dried *in vacuo* at 60 °C for 24 hours. The yield was 12.2 g (79%). IR (KBr, cm^{-1}): 3411 (ν OH), 2998 (ν C-H aromatic), 2933 and 2858 (ν C-H aliphatic), 1616 and 1589 (ν C=C aromatic), 1294 and 1100 (ν C-O-C ester). 1H NMR (600 MHz, TMS, $DMSO-d_6$): δ (ppm) 0.44-3.02 (m, 36H, $-CH_2CH_2CH_2-$), 3.24-3.99 (m, 36H, $-OCH_3$), 4.04-4.86 (m, 12H, $>CH$), 5.53-7.52 (m, 24H, arH), 7.80-9.67 (m, 12H, OH). MALDI-TOF mass: calcd for [*noria*_{MP}+Na] $^+$: 1897.2, found: 1897.2.

Synthesis of *noria*_{MP} derivatives with pendant adamantyl ester groups.

Typical procedure for synthesis of *noria*_{MP}-AD₁₈: A mixture of *noria*_{MP} (8.0 g, 4.2 mmol), K_2CO_3 (8.2 g, 60 mmol), and TBAB (0.064 g, 0.2 mmol) in NMP (95 mL) was stirred at 60 °C for 3 h. After that, a solution of BMA (4.3 g, 15 mmol) in NMP (5 mL) was added slowly to resulting mixture, and was stirred at 60 °C for 48 h. The obtained solution was poured into chloroform, and washed with 0.1 M oxalic acid aqueous solution and water. The organic layer was dried over $MgSO_4$, and concentrated by rotary evaporator. The concentrated solution was added to methanol to obtain the white solid. The resulting precipitate was collected by vacuum filtration, and dried at 60 °C *in vacuo* for 12 h. The degree of introduction of adamantyl ester groups (DI) was calculated by 1H NMR integration ratios of the methyl protons of methoxy moieties, and methyl and methylene protons of *noria*_{MP} and adamantyl ester moieties. DI = 18%. The yield was 6.7 g (68%). IR (film, cm^{-1}): 3402 (ν OH), 2997 (ν C-H aromatic) 2927 and 2861 (ν C-H aliphatic), 1748 and 1724 (ν C=O ester), 1615 and 1588 (ν C=C aromatic), 1296 and 1054 (ν C-O-C ether), 1102 (ν O=C-O-C ester). 1H NMR (600 MHz, $CDCl_3$, TMS) δ (ppm) = 0.34 - 2.37 (m, $-CH_3$ and $-CH_2-$,

72.7H), 3.51 - 3.83 (m, $-\text{OCH}_3$, 36H), 4.22 - 4.53 (broad m, $-\text{O}-\text{CH}_2-\text{C}=\text{O}$, $>\text{CH}$, 16.3H), 6.20 - 6.30(m, ArH, 24H). 7.31 (broad, $-\text{OH}$, 9.8H). $M_n = 2172$, $M_w/M_n = 1.09$.

Noria_{MP}-AD₁₁;

IR (film, cm^{-1}); 3416 (ν OH), 2997 (ν C-H aromatic), 2932 and 2860 (ν C-H aliphatic), 1737 and 1726 (ν C=O ester), 1619 and 1588 (ν C=C aromatic), 1294 and 1053 (ν C-O-C), 1102(ν O=C-O-C). ^1H NMR (600 MHz, CDCl_3 , TMS) $\delta(\text{ppm}) = 0.32 - 2.39$ (m, $-\text{CH}_3$ and $-\text{CH}_2-$, 58.4H), 3.50 - 3.85 (m, $-\text{OCH}_3$, 36H), 4.23 - 4.54 (broad m, $-\text{O}-\text{CH}_2-\text{C}=\text{O}$, $>\text{CH}$, 14.6H), 6.21 - 6.31(m, ArH, 24H). 7.33 (broad, $-\text{OH}$, 10.7H). DI = 11 %. Yield; 60%. $M_n = 1990$, $M_w/M_n = 1.09$.

Noria_{MP}-AD₄₅;

IR (film, cm^{-1}); 3436 (ν OH), 2997 (ν C-H aromatic), 2920 and 2860 (ν C-H aliphatic), 1749 and 1722 (ν C=O ester), 1610 and 1586 (ν C=C aromatic), 1297 and 1058 (ν C-O-C ether), 1102 (ν O=C-O-C ester). ^1H NMR (600 MHz, CDCl_3 , TMS) $\delta(\text{ppm}) = 0.33-2.41$ (m, $-\text{CH}_3$ and $-\text{CH}_2-$, 127.8H), 3.51 - 3.85 (m, $-\text{OCH}_3$, 36H), 4.20 - 4.55 (broad m, $-\text{O}-\text{CH}_2-\text{C}=\text{O}$, $>\text{CH}$, 22.8H), 6.21 - 6.31(m, ArH, 24H). 7.31 (broad, $-\text{OH}$, 6.6H). DI = 45%. Yield= 78%. $M_n = 2483$, $M_w/M_n = 1.08$.

Noria_{MP}-AD₅₃;

IR (film, cm^{-1}); 3423 (ν OH), 2998 (ν C-H aromatic), 2919 and 2860 (ν C-H aliphatic), 1750 and 1721 (ν C=O ester), 1610 and 1586 (ν C=C aromatic), 1298 and 1060 (ν C-O-C ether), 1102 (ν O=C-O-C ester). ^1H NMR (600 MHz, CDCl_3 , TMS) $\delta(\text{ppm}) = 0.26-2.40$ (m, $-\text{CH}_3$ and $-\text{CH}_2-$, 146.2H), 3.53 - 3.85 (m, $-\text{OCH}_3$, 36H), 4.24 - 4.56 (broad m, $-\text{O}-\text{CH}_2-\text{C}=\text{O}$, $>\text{CH}$, 24.7H), 6.20 - 6.33(m, ArH, 24H). 7.34 (broad, $-\text{OH}$, 5.6H). DI = 53%. Yield = 78%. $M_n = 2457$, $M_w/M_n = 1.02$.

Noria_{MP}-AD₇₅ ;

IR (film, cm^{-1}); 3423 (ν OH), 2999 (ν C-H aromatic), 2916 and 2860 (ν C-H aliphatic), 1749 and 1720 (ν C=O ester), 1609 and 1585 (ν C=C aromatic), 1298 and 1062 (ν C-O-C ether), 1102 (ν O=C-O-C ester). ^1H NMR (600 MHz, CDCl_3 , TMS) $\delta(\text{ppm}) = 0.30 - 2.38$ (m, $-\text{CH}_3$ and $-\text{CH}_2-$, 189H), 3.51 - 3.83 (m, $-\text{OCH}_3$, 36H), 4.22 - 4.53 (broad m, $-\text{O}-\text{CH}_2-\text{C}=\text{O}$, $>\text{CH}$, 30H), 6.20 - 6.30(m, ArH, 24H). 7.33 (broad, $-\text{OH}$, 3H). DI = 75%. Yield = 85%. $M_n = 2705$, $M_w/M_n = 1.06$.

Patterning property of noria_{MP}-AD by EUV lithography system.

A solution of PGMEA was prepared from noria_{MP}-AD₁₈ (90wt%), TPS-Nf as a photo-acid generator (9wt%), and TOA as a quencher (1wt%). The resulting solution was filtered through a 0.45 μm membrane filter and spin-coated on silicon

wafer to form thin film with 60 nm thickness. The film was baked at 130 °C for 60 sec to remove residual solvent, exposed by small field exposure tool (SFET) with 0.3 numerical aperture (NA), and re-baked (PEB; post exposure bake) at 130 °C for 60 sec. The exposed film was developed in 2.38wt% TMAH aq. for 30 sec. The resulting patterns were observed using a CD-SEM (S9220, Hitachi).

3. Results and discussion

Synthesis and characteristics of noria_{MP}-AD.

Noria_{MP} derivatives with pendant adamantyl ester groups (noria_{MP}-AD_n) were synthesized by the substitution reaction of noria_{MP} and BMA in the presence of K_2CO_3 and TBAB in NMP (Scheme 1). By the control of feed ratio, noria_{MP}-AD_n with various degrees of introduction of adamantyl ester moieties were synthesized. These conditions and results are summarized in Table 1.

Scheme 1

The structure of noria_{MP}-AD_n was confirmed by ^1H NMR and IR spectroscopy. Figure 1 depicts the ^1H NMR spectrum of noria_{MP} derivative (run 4 in Table 1), along with that of noria_{MP}. The spectrum shows new peaks assignable to methyl and methylene protons of the AD moieties at 0.26 - 2.40 ppm, and indicated that the noria_{MP} derivative with pendant AD could be obtained. The degree of introduction of AD (DI) was calculated from the integration ratios of methyl protons of methoxy moiety of noria_{MP} at 3.53 ~ 3.85ppm, and the methyl and methylene protons of the noria_{MP} and noria_{MP}-AD moiety at 0.26 ~ 2.40 ppm in the ^1H NMR spectra, and was 53 %. Furthermore, FT-IR spectra showed two characteristic absorptions at 1750 and 1102 cm^{-1} due to C=O and C-O-C

stretching vibrations of adamantyl ester groups, respectively. As the results, $\text{noria}_{\text{MP}}\text{-AD}_n$ with $\text{DI} = 11 \sim 75\%$ could be obtained by the control of the feed ratio (Table 1).

Table 1 Synthesis and thermal properties of $\text{noria}_{\text{MP}}\text{-AD}_n$.^{a)}

run	Feed ratios (noria_{MP} $\times 12$ /BMA)	DI ^{b)} (%)	Yield ^{c)} (%)	M_n (M_w/M_n) ^{d)}	$T_d^{5\%e)}$ °C
1	1/0.15	11	60	1990(1.09)	189
2	1/0.30	18	68	2170(1.09)	179
3	1/0.65	45	78	2480(1.08)	183
4	1/0.80	54	69	2520(1.06)	191
5	1/1	75	85	2710(1.06)	196

^{a)}The reactions of BMA and noria_{MP} was carried out at various feed ratios in NMP at 60 °C for 48 hours. ^{b)}DI = degree of introduction of adamantyl ester groups into noria was calculated from ^1H NMR data (see Text). ^{c)}Methanol-insoluble part. ^{d)}Estimated by SEC(DMF). ^{e)}The temperature of 5% weight loss ($T_d^{5\%}$) was measured by means of TGA.

Fig. 1. ^1H NMR spectroscopy of noria_{MP} ([A]) and $\text{noria}_{\text{MP}}\text{-AD}_{53}$ ([B]) (run 4 in Table 1).

Physical properties (solubility, film-forming ability, and thermal stability) of $\text{noria}_{\text{MP}}\text{-AD}_n$.

The physical properties of synthesized

$\text{noria}_{\text{MP}}\text{-AD}_n$ were examined to relevant to application as EUV-resist materials.

Solubility.

Table 2 summarize the solubility characteristics of noria , noria_{MP} , and $\text{noria}_{\text{MP}}\text{-AD}$. Although, noria was soluble in aprotic highly polar solvents, such as DMSO and DMF, noria_{MP} was soluble in common organic solvents, such as THF, acetone, and chloroform. This means that hydroxyl groups of noria had replaced with methoxy groups in noria_{MP} , and inter-hydrogen bond effect between hydroxyl groups of the synthesized noria_{MP} could be reduced. Furthermore, noria_{MP} derivative with pendant AD moiety $\text{noria}_{\text{MP}}\text{-AD}_{11}$ ($\text{DI} = 11\%$) was slightly soluble in common organic solvents and soluble in 2.38wt% TMAH aqueous solution. On the other hand, other $\text{noria}_{\text{MP}}\text{-AD}_n$ ($n = 18, 45, 53$, and 75) were soluble in common organic solvents and insoluble in 2.38wt% TMAH aqueous solution. These mean that $\text{noria}_{\text{MP}}\text{-AD}_n$ with the DI value of more than 18% could be applicable as positive-type photo-resist material.

Table 2. Solubility of noria , noria_{MP} , and $\text{noria}_{\text{MP}}\text{-AD}_n$.

Solvent	noria	noria_{MP}	$\text{noria}_{\text{MP}}\text{-AD}_{11}$ (DI = 11%)	$\text{noria}_{\text{MP}}\text{-AD}_n$ (DI=18-75%)
Water	-	-	-	-
DMSO	++	++	++	++
DMF	+-	++	++	++
Methanol	-	-	-	-
THF	-	++	++	++
Acetone	-	++	++	++
Ethyl lactate	-	+-	+-	++
Ethyl acetate	-	+-	+-	++
Ethyl ether	-	-	-	-
Chloroform	-	++	++	++
PGMEA ^{a)}	+-	++	++	++
<i>n</i> -Hexane	-	-	-	-
Diglyme ^{b)}	-	++	++	++
PGME ^{c)}	+-	+-	+-	++
2.38wt%	++	++	+	-
TMAH				

^{a)}PGMEA = propylene glycol monomethyl ether acetate,

^{b)}Diglyme = diethyleneglycol dimethyl ether, ^{c)}PGME = propylene glycol monomethyl ether, ++ ; Soluble at room temperature, + ; Soluble at heating, +- ; Partially soluble, - ; Insoluble

Film-forming ability.

The film-forming ability was examined by spin coating a solution of noria_{MP} and $\text{noria}_{\text{MP}}\text{-AD}_n$ 50 mg in PEGMEA 1.0 ml in silicon wafer, and drying the wafer *in vacuo* at room temperature. As the results, all $\text{noria}_{\text{MP}}\text{-AD}_n$ had good film forming ability. However, $\text{noria}_{\text{MP}}\text{-AD}_{11}$ is not candidate for resist material, because it is soluble in TMAH aqueous solution.

Thermal stability.

The thermal properties of $\text{noria}_{\text{MP}}\text{-AD}_n$ were examined by TGA and DSC. The 5 % weight loss temperatures were measured by means of TGA, and were in the range between 179 and 196 °C, as summarized in Table 1. It was shown a tendency of that the thermal stability increased with increasing DI values of $\text{noria}_{\text{MP}}\text{-AD}_n$. This might be because of that acidic phenol groups had been replaced with adamantate groups, i.e., acidic phenol groups promote the thermal decomposition reaction of $\text{noria}_{\text{MP}}\text{-AD}_n$.

$\text{noria}_{\text{MP}}\text{-AD}_{53}$ in the EUV exposure system. Figure 2 depicts the SEM images of the patterns with resolution in the range between 25 and 50 nm, which were obtained using trioctylamine as a quencher at 130 °C soft bake (SB) temperature and 130 °C post exposure bake (PEB), and 2.38 wt% TMAH aqueous solution as a developer. In the case of $\text{noria}_{\text{MP}}\text{-AD}_{18}$, a clear 50 nm resolution pattern was obtained with lower exposure dose (8.5 mJ/cm²) and lower LWR (6.1 nm) compared to those of $\text{noria}_{\text{MP}}\text{-AD}_{53}$ (Figure 2[A]). This means that noria_{MP} derivatives with lower DI of AD groups would have higher potential to offer higher resolution pattern. Furthermore, 25 ~ 40 nm resolution patterns were examined with slightly increased exposure dose. These results also depict Figure 2 [B] ~ [F]. Clear resolved patterns at 32 nm (LWR = 10.5 nm) with dose = 9.0 mJ/cm² and 40 nm (LWR = 8.9 nm) with dose = 10.0 mJ/cm² could be obtained with $\text{noria}_{\text{MP}}\text{-AD}_{18}$ and $\text{noria}_{\text{MP}}\text{-AD}_{53}$, respectively.

	(A)	(B)	(C)	(D)	(E)	(F)
Mask size L/S (nm)	50	40	32	30	28	25
$\text{Noria}_{\text{MP}}\text{-AD}_{18}$
Dose (mJ/cm ²)	8.5	9.0	9.0	9.0	9.0	9.0
LER (nm)	6.1	7.7	10.5	- a)	- a)	- a)
$\text{Noria}_{\text{MP}}\text{-AD}_{53}$
Dose (mJ/cm ²)	10	10	10.8	10.8	10.8	10.8
LER (nm)	6.8	8.9	- a)	- a)	- a)	- a)

Fig. 2. Scanning electron microscopy images of resolution patterns of $\text{noria}_{\text{MP}}\text{-AD}_{18}$ and $\text{noria}_{\text{MP}}\text{-AD}_{53}$.

Patterning properties of $\text{noria}_{\text{MP}}\text{-AD}$.

Patterning properties were examined using thin-film prepared from $\text{noria}_{\text{MP}}\text{-AD}_{18}$ and

Furthermore, the possibility to offer 25 nm resolution were also found in Figure 2[F]. It is noteworthy that these high resolution patterns

could be obtained with less than 10 mJ/cm^2 , i.e., the sensitivity of resist materials based of noria_{MP}-AD is superior compared to our previous reports of noria derivatives noria-AD₂₃²⁷ and noria-CHVE₅₀²⁸. Noria-AD₂₃ and noria-CHVE₅₀ provided the resist patterns of 26 nm resolution with exposure dose = 14.5 mJ/cm^2 and 35 nm resolution with exposure dose = 14.5 mJ/cm^2 in an EUV system, respectively. These results mean that noria_{MP}-AD might have high potential to offer higher resolution resist pattern as next-generation EUV-resist materials. Furthermore, the examination resist patterning property of noria_{MP} and noria_{EP} derivatives by the means of interference EUV-exposure system is now under investigation.

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