Methacrylonitrile Based Si-Containing Polymers for 157-nm Positive Resist

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Polymers based on silylated p-hydroxy-α-methylstyrene (SiMST) or methacrylic acid (SiMA) were prepared and their application to positive resists for 157 nm lithography was studied. These polymers were synthesized by silylation of the copolymer of methacrylonitrile (MAN) and p-hydroxy-α-methylstyrene (HMST) or copolymerization of corresponding monomers. Etch rate of the films using O₂ plasma was 12-45 nm/min which was lower than that for poly(vinylphenol) film (80 nm/min). The incorporation of Si atoms in the polymer side chain strongly enhanced the etch resistance. Changes in mass of the films during irradiation were studied by in-situ QCM method. Weight loss for SiMA units was larger than that for SiMST units. Dissolution properties of the films in 2.38 wt% TMAH aqueous solution were studied and resist pattern profile was simulated.

Keywords: 157 nm lithography, Si-containing polymer, silylation, methacrylonitrile, oxygen plasma, positive resist

1. Introduction

F₂ laser lithography has been one of the most prominent candidates as post ArF lithography for the fabrication of 100-70 nm pattern dimensions because of its theoretical capability in resolution and depth of focus.[1] Single-layer positive resists must be highly transparent at 157 nm, non-photocrosslinkable, non-photodegradable, non-outgassing, alkaline developable, and highly resistant to plasma etching. Most critical issue of those points to design resist materials is the low transmittance at 157 nm because a lot of organic polymers strongly absorb wavelengths of light at VUV regions.[2] Only fluoropolymers, polysilsesquioxane and polysiloxanes, in fact, provide highly transmittance at 157 nm. Polymers based on methacrylonitrile, tetrafluoroethylene, α-trifluoromethacrylates, norbornene and styrene derivatives bearing hexafluoroisopropanol groups have been designed as single-layer resists for 157 nm lithography.[3-6]

Dry etch resistance is also important for resists. To get high resistance to oxygen plasma etching, aromatic rings or bulky alicyclic hydrocarbons such as norbornene or adamantyl units have been introduced in polymer main-chain or side-chain. Aromatic rings decrease transparency at 157 nm and incorporation of fluorine atoms sometimes decreases etch resistance to oxygen plasma.

Si-containing polymers have been noted as resist materials because of good etch resistance to oxygen plasma. Polymers based on silylated p-hydroxy styrene or methacrylic acid showed high etch resistance to oxygen plasma and could be used as single-layer or bilayer resists for KrF lithography.[7-9] Furthermore, top surface imaging process using silylation of chemically amplified resists was reported to be bilayer resists for ArF lithography.[10,11] Recently, we reported that the copolymer of methacrylonitrile (MAN) and p-trimethylsiloxy-α-methylstyrene (TMSiMST) was able to be used as single-layer or bilayer positive resists for F₂ lithography, giving fine images.[12]

In this paper we report synthesis of MAN based Si-containing polymers and their
application to positive resist for F2 lithography. Copolymers of methacrylonitrile and silylated p-hydroxy-α-methyl styrene (SiMST) or silylated methacrylic acid (SiMA) were prepared by polymer reactions or copolymerization. Acid catalyzed deprotection reaction of silyl ethers or silyl esters, etch resistant to oxygen plasma, weight loss of polymer films during exposure and dissolution characteristics were examined. Resist pattern profiles were also estimated by the use of a lithography simulation program.

2. Experimental

2.1. Materials and reagents

Solvents for monomer syntheses were distilled before use. Silylation reagents such as 1,1,1,3,3,3-hexamethyldisilazane (from Wako Pure Chemical Industries), 1,3-diphenyl-l-1,1,3,3-tetramethyldisilazane (from Aldrich Chemical Company), tert-butyldimethylchlorosilane (from Shin-Etsu Chemical Co., Ltd.) and dimethylsilane (DMSDMA) (from Shin-Etsu Chemical Co., Ltd.) were used as received unless otherwise specified. Trimethylsilyl methacrylate (TMSiMA) was purchased from Aldrich and used after distillation.

2.2. Preparation of monomers

tert-Butyldimethylsilyl methacrylate (tBDMSiMA)

According to the reported method [7], methacrylic acid (11.3 mL) was treated with tert-butyldimethylchlorosilane in the presence of imidazole (21.6 g) in DMF (50 mL) at 50 °C for 6 hr. To the reaction mixture was added 400 mL of aqueous NaCl solution (20 wt%) and the mixture was extracted twice with ethyl acetate. After drying the solution with anhydrous Na2SO4, ethyl acetate was evaporated under vacuum. tert-Butyldimethylsilyl methacrylate was purified by vacuum distillation. Yield: 7.8 g (25 %); b.p. 46 °C (4 mmHg). 1H-NMR (CDCl3; 270MHz): δ = 0.30 (6H, s, Si-CH3), 0.96 (9H, s, C(CH3)3), 1.93 (3H, s, CH3), 5.57-6.10 (2H, ss, CH2=).

2.3. Preparation of polymers

Poly(methacrylonitrile-co-trimethylsiloxy-α-methylstyrene) ((MAN(X)-TMSiMST(Y)) and poly(methacrylonitrile-co-dimethylsiloxyl-α-methylstyrene-co-p-hydroxy-α-methylstyrene) (MAN(57)-DMSiMST(39)-HMST(4)) were obtained by silylation of the copolymer of methacrylonitrile and p-hydroxy-α-methyl-styrene (MAN(X)-HMST(Y)) (X,Y; mol%) with silylation reagents. MAN(X)-HMST(Y) was obtained by thermolysis of poly(methacrylonitrile-co-t-butoxycarbonyl oxy-α-methylstyrene) ((MAN(X)-BOCMST(Y)). Synthesis of MAN(X)-BOCMST(Y) was previously reported.[3] Copolymer of methacrylonitrile(57) and t-butyldimethylsiloxyl-α-methylstyrene(43) (MAN(57)-tBDMSiMST(43)) was synthesized by the reaction of MAN(57)-HMST(43) with t-butyldimethylchlorosilane using imidazole as a catalyst in DMF at room temperature for 24 hr. Copolymer of methacrylonitrile(57) and dimethylsiloxyl-α-methylstyrene(43) (MAN(57)-DMSiMST(43)) was obtained by silylation of MAN(57)-HMST(43) with DMSDMA at 45 °C for 20 min, according to the reported method.[13]

MAN(58)-TMSiMA(42) and MAN(45)-tBDMSiMA(55) were prepared by radical copolymerization of MAN and TMSiMA or tBDMSiMA at 55°C using azobisisobutylonitrile as an initiator. Degree of silylation of phenolic OH groups was checked by disappearance of IR absorbance due to phenolic OH groups. Composition of MAN(57)-DMSiMST(39)-HMST(4) was determined by 1H-NMR using the ratio of the signals due to aliphatic (0.2-2.0 ppm), aromatic (6.0-7.5 ppm) and Si-CH3 (0 ppm) protons. Composition of MAN(58)-TMSiMA(42) and MAN(45)-tBDMSiMA(55) was determined by 1H-NMR using the ratio of the signals due to aliphatic (0.2-2.0 ppm) and Si-CH3 (0 ppm) protons. Structures and characteristics of polymers are summarized in Scheme 1 and Table 1, respectively.

2.4. Method

Sample films were spin-cast onto Si wafer and thickness after prebake at 90 °C for 1min was 0.2 µm. Casting solvent was propylene glycol methyl ether acetate (PGMEA). Thickness of the films was measured by interferometry (Nanometrics, NanoSpec/AFT M-3000). Triphenylsulfonium triflate (TPST) was used as photoacid generator and the content was 5 wt % in the polymer. The exposure was carried out with KrF excimer lump (146 nm, Ushio UER20H-146V) under N2 gas flow. Light intensity was 2 mW/cm² at the irradiated film surface. The distance between the film and the lamp was 6 cm. The films were developed with 2.38 wt% of tetramethylammonium hydroxide aqueous solution (TMAH) at 23 °C. Dissolution rate was measured with a resist
development analyzer RDA-790 (Litho Tech.
Japan). Resist pattern profiles were simulated
with a lithography simulation program SOLID-C (Sigma-C GmbH).

Oxygen plasma etching was carried out at
room temperature using laboratory-constructed
apparatus where the oxygen plasma was
generated using two parallel electrodes and RF
power supplies. The typical etching conditions
were as follows: 30 W power (13.56 MHz),
power density of 1.0 W/cm², 80 mTorr, and
oxygen flow of 1.0 sccm.

According to the reported method [14], a
laboratory-constructed piezoelectric apparatus
was used to measure the changes in a mass of
polymer films during exposure. An AT-cut
quartz crystal with gold electrodes, showing a
resonance frequency of 10.000 MHz, was used.
With this crystal, a frequency shift of 1 Hz
corresponds to a mass change of about 1 ng.
Polymers were deposited onto the quartz crystal
(0.54 cm diameter) by casting from chloroform
solution of polymers. The quartz crystal was
placed in a chamber (7 cm from the lamp) and
irradiated under N₂ gas flow. The frequency
changes during irradiation were monitored by a
Yokogawa FC-864 frequency counter. The
frequency change, AF (Hz), is linearly related to
the mass on the quartz crystal plate according to
the equation. [15,16]

\[ AF = -2.66 \times 10^6 \frac{F^2 \Delta m}{A} \]

where \( F \) (MHz) is the initial frequency of the
quartz crystal used, \( \Delta m \) (g) is the mass on the
quartz crystal plate and \( A \) (cm²) is the area of the
crystal surface occupied by the polymer films.

3. Results and Discussion

3.1. Properties of Si-containing polymers

Copolymers based on silylated
p-hydroxy-α-methylstyrene (SiMST) were
easily prepared by silylation of MAN-HMST copolymers, except

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<table>
<thead>
<tr>
<th>Polymer</th>
<th>R</th>
<th>x</th>
<th>y</th>
<th>z</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAN(57)-DMSiMST(43)</td>
<td>SiH(CH₃)₂</td>
<td>57</td>
<td>43</td>
<td>0</td>
</tr>
<tr>
<td>MAN(75)-TMSiMST(25)</td>
<td>Si(CH₃)₃</td>
<td>75</td>
<td>25</td>
<td>0</td>
</tr>
<tr>
<td>MAN(66)-TMSiMST(34)</td>
<td>Si(CH₃)₃</td>
<td>66</td>
<td>34</td>
<td>0</td>
</tr>
<tr>
<td>MAN(61)-TMSiMST(39)</td>
<td>Si(CH₃)₃</td>
<td>61</td>
<td>39</td>
<td>0</td>
</tr>
<tr>
<td>MAN(57)-DMPSiMST(39)-HMST(4)</td>
<td>Si(CH₃)₂(CH₃)₂</td>
<td>57</td>
<td>39</td>
<td>4</td>
</tr>
<tr>
<td>MAN(57)-tBDMSiMST(43)</td>
<td>Si(CH₃)₂C(CH₃)₂</td>
<td>57</td>
<td>43</td>
<td>0</td>
</tr>
</tbody>
</table>

(x,y,z: mole%)

Table 1. Characteristics of polymers

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Mn (X10⁻⁴)</th>
<th>Mw/Mn</th>
<th>Tg (°C)</th>
<th>Td (°C)</th>
<th>T(%) at 157 nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAN(75)-TMSiMST(25)</td>
<td>1.9</td>
<td>2.6</td>
<td>121</td>
<td>232</td>
<td>- a)</td>
</tr>
<tr>
<td>MAN(66)-TMSiMST(34)</td>
<td>1.8</td>
<td>1.9</td>
<td>116</td>
<td>256</td>
<td>- a)</td>
</tr>
<tr>
<td>MAN(61)-TMSiMST(39)</td>
<td>1.5</td>
<td>1.7</td>
<td>106</td>
<td>220</td>
<td>19</td>
</tr>
<tr>
<td>MAN(57)-DMPSiMST(39)-HMST(4)</td>
<td>- a)</td>
<td>- a)</td>
<td>139</td>
<td>309</td>
<td>- a)</td>
</tr>
<tr>
<td>MAN(57)-tBDMSiMST(43)</td>
<td>2.1</td>
<td>2.2</td>
<td>96</td>
<td>323</td>
<td>- a)</td>
</tr>
<tr>
<td>MAN(58)-TMSiMA(42)</td>
<td>- a)</td>
<td>- a)</td>
<td>121</td>
<td>196</td>
<td>- a)</td>
</tr>
<tr>
<td>MAN(45)-tBDMSiMA(55)</td>
<td>5.6</td>
<td>1.9</td>
<td>110</td>
<td>277</td>
<td>21</td>
</tr>
</tbody>
</table>

a) Not measured. b) Tg: Determined by DSC. Td: Determined by TGA. c) Thickness: 100 nm.
for MAN(53)-DMPSiMST(39)-HMST(4). 1,3-
diphenyl-1,1,3,3-tetramethyldisilazane seems to 
be more bulky than other silylation reagents, so 
MAN(53)-DMPSiMST(39)-HMST(4) left 
phenolic OH groups. Copolymers based on 
silylated methacrylic acid (SiMA) were not 
obtained by silylation of the copolymer of MAN 
and methacrylic acid. Therefore, MAN based 
SiMA copolymers synthesized by conventional 
radical copolymerization of corresponding 
monomers.

Thermal decomposition temperatures (Td) of 
copolymers based on SiMST (220-323 °C) and 
SiMA (196-277 °C) were higher compared to 
MAN(51)-BOCMST(49) (165 °C). The glass 
transition temperatures (Tg) of SiMST based and 
SiMA based copolymers were 96-121 and 
110-121 °C, respectively.

Transmittances at 157 nm for 
MAN(61)-TMSiMST(39) and MAN(45) 
tBDMSiMA(55) films at 100 nm thick were 19 
and 21 %, respectively, which were lower than 
that for MAN based copolymer such as MAN(51)-BOCMST(49) (35%), 
 methacrylonitrile(51)-4-t-butoxy-α-methylstyrene 
(49) (37%) and methacrylonitrile(54) 
-4-hexafluoro-2-t-butoxybenzoxoysopropyl-α-
 methylstyrene(46) (44%).[12] Silyl ether and 
silyl ester units significantly reduced the 
transmittance at 157 nm.

3.2. Etch resistance

Etch resistance of sample films was checked 
with O2 plasma. Etch rate and content of Si 
atoms in the films are shown in Table 2. Etch 
rate of the films containing Si atoms was low 
(12-25 nm/min) compared with the rate for 
poly(vinylphenol) films (80 nm/min), except for 
MAN(75)-TMSiMST(25) (45 nm/min) because 
content of Si atoms in the MAN(75) 
-TMSiMST(25) (6.9 %) was lower than that in 
other Si-containing polymers (7.4-10.5 %). 
Although etch conditions were not the same, etch 
resistance of MAN based Si-containing polymers 
was the same or relatively lower than that 
reported silylated KrF or ArF polymers.[8,17] 
The difference of etch resistance between SiMST 
and SiMA based copolymers was not observed 
because content of Si atoms in SiMA based 
polymer was the same as that in SiMST based 
polymer (Table 2). Although contents of Si 
atoms in the polymers was low at 6.9-10.5 %, 
introduction of Si atoms to polymer side chains 
enhanced the etch resistance

3.3. Weight loss of polymer films

One of the most critical problems for silicon 
containing resists is outgassing from polymer 
films during exposure, which can contaminate the 
exposure system. Therefore, investigation on 
change in a mass of films during irradiation is 
very important. Fig.1 shows the weight loss of 
the polymer films during exposure. The 
decrease of mass of SiMST based copolymer was 
lower than that of SiMA based copolymer. 
Thickness loss of poly(methacrylates) was 
reported to be fast because of decomposition of 
ester units on irradiation at 146 nm.[18] 
Therefore, it seems that weight loss of SiMA 
based polymer films results from degradation of 
ester groups on irradiation, as will be discussed 
below. SiMST based copolymers seem better 
than SiMA based copolymers for F2 lithography.

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Etch rate (nm/min)</th>
<th>Si (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVP</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>MAN(57)-DMSiMST(43)</td>
<td>20</td>
<td>10.0</td>
</tr>
<tr>
<td>MAN(75)-TMSiMST(25)</td>
<td>45</td>
<td>6.9</td>
</tr>
<tr>
<td>MAN(66)-TMSiMST(34)</td>
<td>24</td>
<td>8.3</td>
</tr>
<tr>
<td>MAN(61)-TMSiMST(39)</td>
<td>12</td>
<td>9.0</td>
</tr>
<tr>
<td>MAN(57)-DMSiMST(39)-HMST(4)</td>
<td>25</td>
<td>7.4</td>
</tr>
<tr>
<td>MAN(57)-tBDMSiMST(43)</td>
<td>16</td>
<td>8.3</td>
</tr>
<tr>
<td>MAN(58)-TMSiMA(42)</td>
<td>19</td>
<td>10.5</td>
</tr>
<tr>
<td>MAN(45)-tBDMSiMA(55)</td>
<td>16</td>
<td>10.4</td>
</tr>
</tbody>
</table>

Table 2. Etch rate of the polymers

Fig.1 Decrease of film weight upon irradiation. Polymer:
(○) MAN(61)-TMSiMST(39),
(△) MAN(57)-DMSiMST(39)-HMST(4),
(□) MAN(57)-tBDMSiMST(43),
(◇) MAN(58)-TMSiMA(42),
(◇) MAN(45)-tBDMSiMA(55).
Weight: 1139-2088 ng.
3.4. Deprotection of silyl groups

Fig. 2 shows IR spectral changes of irradiated MAN(61)-TMSiMST(39) film on heating at 120 °C. After irradiation at 146 nm, the absorbance due to Si-O-C (930 cm⁻¹) decreased and the absorbance due to phenolic OH groups (3400 cm⁻¹) appeared. It suggests that the acid catalyzed deprotection reaction resulted from acid by TPST, followed by hydrolysis reaction of silyl ether units (Scheme 2). Fraction of decomposed silyl ether units was calculated by IR absorbance changes due to Si-O-C bonds. Decomposed fraction of silyl ether units decreased in the order MAN(57)-DMSiMST(43) > MAN(61)-TMSiMST(39) > MAN(57)-DMPsSiMST(39)-HMST(4) > MAN(57)-tBDMSiMST(43) as shown in Fig. 3. The highest rate of hydrolysis was observed for dimethylsilyl group. Acid catalyzed hydrolysis of silylated phenol derivatives was studied and

![IR spectral changes of MAN(61)-TMSiMST(39) film containing TPST(5 wt%) on heating at 120 °C. Irradiation: 146 nm for 5 sec. (---); before irradiation, (---); after irradiation, (---); after bake for 1 min.](image1)

![Scheme 2](image2)

![Decomposed fraction of silyl ether unit of polymer films with 5 wt% of TPST on heating at 120 °C after irradiation at 146 nm with 10 mJ/cm²](image3)
the rate of hydrolysis was observed to be governed by the steric hindrance of the substituted silyl group. The hydrolysis rate decreased in the order dimethylsiloxybezene > trimethylsiloxybenzene > phenyldimethylsiloxybenzene > t-butyldimethylsiloxybenzene. The order of hydrolysis rate of the polymers was agreement with those of model compounds as phenol derivatives. Dimethylphenylsilyl group was the most suitable for the protecting group.

3.5. Dissolution properties.

Dissolution properties of polymer films were studied using 2.38 wt% of TMAH. Sample films were cast from PGMEA, except for MAN(58)-TMSiMA(42) and MAN(57)-DMSiMA(43). MAN(58)-TMSiMA(42) films were cast from chloform and MAN(57)-DMSiMA(43) films were prepared by direct silylation of MAN(57)-HMST(43) film. Pre-bake treatment was carried out at 90 °C for 1 min. Unirradiated MAN(57)-DMSiMST(43) and MAN(58)-TMSiMA(42) films were soluble in the TMAH (2.38 wt%) solution. When irradiated and then followed by PEB at 120 °C, MAN(57)-BDMSiMA(43) was not dissolve in TMAH (2.38 wt%) solution. Relationship between exposure dose and remaining thickness of polymer films is shown in Figs. 4 and 5. Thickness changes were not observed during irradiation. After PEB treatment, thickness loss was small for Si-containing polymers (5-16 %). Although MAN(61)-TMSiMST(39) provided high sensitivity, contrast was low as shown in Fig.4(a). Higher contrast was obtained for MAN(57)-DMPSiMST(39)-HMST(4) as shown in Fig.4(b) because dimethylphenylsilyl group had relatively high hydrolysis rate and good stability in the absence of acid. MAN(45)-tBDMSiMA(55) needed exposure dose higher than MAN(61)-TMSiMST(39) and MAN(57)-DMPSiMST(39)-HMST(4) to dissolve in TMAH (Fig.4(c)). Fig.5 shows effect of MAN content on dissolution properties of the copolymer of MAN and SiMST. It has been reported that poly(methacrylonitrile) was highly transmittant at 157 nm, so it can be seen that higher MAN content in polymer, thicker films we use. MAN(75)-TMSiMST(25) did not dissolve in TMAH solution completely because the content of MAN units was too high (Fig. 5).

Relationship between exposure energy and
The dissolution rate is shown in Fig. 6. After irradiation, the dissolution rate of the copolymer films rapidly increased because exposed films were soluble in TMAH solution. Table 3 shows $R_{\text{max}}$, which is the maximum value of dissolution rate of the films. $R_{\text{max}}$ of PVP (120 nm/sec) was very high but MAN(61)-HMST(39) (7.5 nm/sec) and other Si-containing polymers (1.1-1.8 nm/sec) were low because the MAN unit is not so hydrophilic. Therefore, MAN based Si-containing polymers require long development time.

### 3.6 Patterning simulation

Patterned resist profiles were estimated by the use of a simulation program. The simulations were performed using values of dissolution rate measured by RDA-790. In the case of MAN(57)-DMPSiMST(39)-HMST(4) (thickness: 0.2 µm), 0.2 µm L/S patterns were estimated to be obtained, when irradiated with 12.5 mJ/cm² by F2 laser at 157 nm, baked at 120 °C for 1 min, and followed by development with 2.38 wt% of TMAH for 300 sec (Fig. 7).

### Table 3. Dissolution rate of the polymer films

<table>
<thead>
<tr>
<th>Polymer</th>
<th>$R_{\text{max}}$ (nm/sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVP</td>
<td>121</td>
</tr>
<tr>
<td>MAN(61)-HMST(39)</td>
<td>7.5</td>
</tr>
<tr>
<td>MAN(61)-TMSiMST(39)</td>
<td>1.8</td>
</tr>
<tr>
<td>MAN(57)-DMPSiMST(39)-HMST(4)</td>
<td>1.1</td>
</tr>
<tr>
<td>MAN(45)-TMSiMA(55)</td>
<td>1.2</td>
</tr>
</tbody>
</table>

$R_{\text{max}}$: Maximum value of dissolution rate.

Fig. 6 Relationship between exposure dose and dissolution rate of polymer films irradiated at 146 nm. Development: TMAH (2.38%) for 200 sec. (Δ) MAN(61)-TMSiMST(39), (□) MAN(57)-DMSiMST(39)-HMST(4), (◇) MAN(45)-BDMSiMA(55).

Fig. 7 Resist pattern simulation of MAN(57)-DMPSiMST(39)-HMST(4) film for 0.2 µm lines and spaces. Thickness: 0.2 µm. Development time: 300 sec. Exposure dose: 12.5 mJ/cm². NA: 0.54.

### 4. Conclusion

MAN based silylated p-hydroxy-α-methylstyrene polymers were prepared by silylation of MAN-HMST pre-polymer. MAN based silylated methacrylic acid copolymers were synthesized by the radical copolymerization of corresponding monomers. Incorporation of Si atoms in the polymer side chains enhanced the etch resistance to O₂ plasma. The mass decrease of SiMA based copolymers was larger than that of SiMST based copolymers on irradiation at 146 nm. The rate of hydrolysis of copolymers depended on the silyl group structure. 0.20 µm L/S patterns were suggested to be obtained by the use of lithography simulation program. Thus, MAN based Si-containing polymers seem to be useful for 157 nm single-layer and bilayer resists, which show high etch resistance.

### References


