Development of High-Performance Negative-tone Resists for 193-nm Lithography

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We have been developing negative-tone resist systems utilizing an acid-catalyzed intramolecular esterification of γ - and δ -hydroxy acid for ArF phase-shifting lithography. In this paper, α -acryloyloxy- β , β -dimethyl- γ -butyrolactone (DBLA), adamantane lactone acrylate (AdLA), and norbornane lactone acrylate (NLA) were examined as a precursor of hydroxy acid. It was found that AdLA and NLA are not hydrolyzed into hydroxy acid under an alkali hydrolysis condition. DBLA was found to produce γ -hydroxy acid, which is stable in the resist solution. The γ -hydroxy acid derived from DBLA becomes γ -lactone relatively easily by an acid-catalyzed reaction and can be used to make resists insoluble. Since the variation and the flexibility of the copolymer composition of the base polymer can be increased, the resist properties are controllable and the pattern quality can be improved by utilizing γ -hydroxy acid derived from DBLA.

Key words: ArF phase-shifting lithography, negative-tone resist, lactone, hydroxy acid, intramolecular esterification

1. Introduction

It has been reported that negative-tone resists have advantages over positive-tone resists in several aspects, such as smaller proximity effects (dark field mask) [1], a large process window [2], compatibility with dual damascene/low- k processes [3] for CMOS LSIs, and with alternating-type PSMs [4,5] for memory LSIs. However, so far most development of ArF resists has been focusing on positive-tone materials.

In the early stage of our research on negative-tone ArF single-layer resists [6–16] based on chemical amplification, we examined the use of acid-catalyzed insolubilization reactions between aliphatic carbinols and alicyclic polymers with carboxylic moiety [6]. Although the negative-tone resist exhibited high insolubilization reactivity, the obtained patterns showed swelling distortion in the sub-micrometer range. This distortion is due to the penetration of the aqueous-base developer into remaining carboxylic groups in the exposed area. It is thus necessary to reduce the number of carboxylic groups in the exposed region in order to avoid micro-swelling distortion of fine patterns.

The authors previously reported on ArF

negative-tone resists based on acid-catalyzed intramolecular esterification [13,14]. We developed alicyclic polymers with a γ -hydroxy acid structure (Scheme 1). It was found that because the acid-catalyzed intramolecular esterification of γ -hydroxy acid into γ -lactone proceeds in the exposed region, the aqueous-base developer cannot penetrate there. However, the transparency and shelf life of the polymer is limited because of the complex addition cyclization process of the polymer preparation and the low stability of the γ -hydroxy acid structure in the resist solution.

The authors next developed a negative-tone resist system based on acid-catalyzed intramolecular esterification of δ -hydroxy acid [15,16]. To improve the transparency and shelf life of the resist, an acrylate polymer with a pendant structure consisting of an androsterone derivative with δ -hydroxy acid (Scheme 2) was developed. It is well known that a steroid structure such as androsterone shows high transparency and high dry-etching durability [17–19]. And the δ -hydroxy acid structure is known to be more stable than the γ -hydroxy acid structure [20]. Since the number

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of carboxyl groups decreases drastically in the exposed area because of the acid-catalyzed intramolecular esterification reaction of δ -hydroxy acid to δ -lactone in the resist system, 100-nm line-and-space patterns were obtained without micro-swelling distortion.

Although the androsterone moiety has excellent dry-etching resistance, a drawback of androsterone is cost, because it is not widely used in industry. The current study investigated other structures and found that γ -hydroxy acid derived from a copolymer of α -acryloyloxy- β,β -dimethyl- γ -butyrolactone can be used. The present study focuses on the reactivity of the γ - and δ -hydroxy acids with different structures and recent progress of our ArF negative-tone resists utilizing the acid-catalyzed esterification reaction of hydroxy acid to lactone.

2. Experiments

2.1 Materials

α -Acryloyloxy- β,β -dimethyl- γ -butyrolactone (DBLA) (Figure 1) was obtained commercially and

Scheme 1. Acid-catalyzed reaction of alicyclic polymer with γ -hydroxy acid

Scheme 2. Acid-catalyzed reaction of acrylate polymer based on androsterone with δ -hydroxy acid.

purified by distillation before use. Adamantane lactone acrylate [21] (AdLA) (Figure 1) was obtained from Daicel Chemical Industries, hydroxy adamantane acrylate was obtained from Idemitsu Petrochemical Co.; both monomers were used without any purification. 3- β -Acryloyloxy-androsterone was synthesized by one-step esterification using *trans*-androsterone with acryloyl chloride in the presence of pyridine [15]. The ketone structure of 3- β -acryloyloxy-androsterone was converted to δ -lactone by Baeyer-Villiger oxidation [22,23]. Norbornane lactone acrylate [11] (NLA) (Figure 1) was synthesized according to the literature [24,25].

Commercially available dimethyl-2,2'-azobis-isobutylate (V-601) was used as the initiator for free radical polymerization. Triphenylsulfonium triflate (TPSOTf) (Midori Kagaku Co.) was used as a photo-acid generator. Aqueous tetramethylammonium hydroxide (TMAH) solution with two different concentrations (2.38% and 0.05%) was used as developer (Tokyo Ohka Kogyo Company).

Figure 1. Alicyclic monomer with lactone structure

2.2 Polymer preparation

Polymerization using 3 mol% of V-601 was carried out in tetrahydrofuran (THF) at 70°C for three hours under a nitrogen atmosphere. After polymerization, hydrolysis of the lactone moiety in the obtained polymers was done in THF with excess aqueous sodium hydroxide solution (0.20 N) at room temperature for six hours. The conversion ratio from lactone to hydroxy acid was estimated from the ^1H and ^{13}C NMR spectra. The weight-average molecular weight of the polymers ranged from 2,000 to 3,000.

2.3 Characterization

The structures of the prepared polymers were determined by ^1H and ^{13}C NMR spectroscopy (Varian Mercury 200 MHz) in THF-d_8 . The molecular weight and the molecular-weight distribution of the prepared polymers were then measured by gel-permeation chromatography (GPC) on a Hitachi L-6000 liquid-chromatography system calibrated with polystyrene standards in THF. The system was equipped with Gelpack A150, 140, and 120 GPC columns (Hitachi Chemical Co.).

2.4 Lithographic evaluation

Resist films were formed by spin-coating onto silicon substrates from a solution in propylene glycol methyl ether (PGME). The resist films were then baked on a hot plate at 100°C . The thickness of the films was measured with an Alphastep 200 (Tencor Instruments) profilometer. The dissolution rate of the resist films was measured by an in-house-made interferometric dissolution rate monitor (DRM). Deep-UV exposure was made by using a Xe-Hg lamp through an interference filter with a maximum transmittance at 250 nm. The lithographic performance was by an ArF excimer-laser stepper (ISI Microstep, 0.60 NA, $\sigma = 0.3$) with a Levenson-type phase-shifting mask. For lithographic evaluations, a bottom anti-reflecting coating was formed on the silicon substrates.

Dry-etching of BARC and SiO_2 was performed by a UHF ECR plasma-etching system (Hitachi U612 and U622, etching conditions for SiO_2 : $\text{C}_4\text{F}_8/\text{O}_2/\text{Ar}$ gas; plasma power of 800W; and wafer bias power of 1400W, etching conditions for BARC: CF_4 gas; plasma power of 500W; and wafer bias power of 50W).

2.5 MOPAC calculation

The heat of formation of the lactone and the corresponding hydroxy acid was calculated by using a MOPAC simulation program, where PM3 was used as a parameter.

3. Results and Discussion

3.1 Performance of the ArF negative resists based on the androsterone structure with δ -lactone

Figure 2 shows the lithographic performance of

the ArF negative resist based on the androsterone structure with δ -lactone in combination with 1.5-wt% TPSOTf. The diluted developer 0.05% TMAH solution was used. Exposure dose was 25 mJ/cm^2 . By using an ArF excimer-laser stepper with a phase-shifting mask, 100-nm line-and-space patterns could be obtained.

Figure 2. Lithographic performance of the resist utilizing the androsterone with δ -lactone (polymer 2).

Figure 3 shows the dry-etching durability of the developed resist material. It is clear that after BARC and SiO_2 etching the commercially available acrylate-type ArF positive resist shows serious resist-pattern deformation (Figure 3(b)). In contrast, the developed negative resist was durable under the dry-etching condition (Figure 3(a)). This durability is due to the high dry-etching resistance of the androsterone moiety. Although the negative resist based on the androsterone structure with δ -lactone shows the excellent properties, a drawback of androsterone is cost because it is not widely used in industry; another drawback is its incompatibility with the standard developer.

Figure 4 shows the relationship between δ -hydroxy acid content and dissolution rate in the 0.05% and 2.38% aqueous TMAH solutions. In 0.05% aqueous TMAH solution, the polymer films dissolved at a rate of 20 nm/s when δ -hydroxy acid content was more than 70%, but they did not dissolve when δ -hydroxy acid content was less than 70%.

In 2.38% developer, the polymer films dissolved at a rate of 100 nm/s when δ -hydroxy acid content was more than 80%. Interestingly, the polymer film with the δ -hydroxy acid content of 70% became insoluble in 2.38% aqueous TMAH solution. This seems to be caused by the so-called salting-out effect [26], which is attributed to the large hydrophobic androsterone molecules attached

a) Resist based on the androsterone with δ -lactone

b) ArF positive resist (alicyclic methacrylate type)

Figure 3. Dry-etching durability of the resist materials.

Figure 4. Relationship between δ -hydroxy content and dissolution rate.

to the small hydrophilic hydroxy acid group. Another commercially available structure, which has a smaller alicyclic group with hydroxy acid, was chosen for the study explained below.

3.2 Reactivity of lactones for alkali hydrolysis reaction

As a precursor of hydroxy acid, the three lactones shown in Figure 1 were examined. An acrylate which has a small alicyclic group with δ -lactone structure was tried to find at first, but such a monomer does not exist. NLA [11] and AdLA [21] are reported as the comonomer for the base polymer of ArF positive resists, and DBLA is commercially available. DBLA and NLA have a γ -lactone structure and AdLA has an ϵ -lactone structure.

At first, alkali hydrolysis of the three lactones was investigated. The condition for the alkali hydrolysis was the same that for preparing the androsterone derivative with δ -hydroxy acid. Figure 5 shows the ^{13}C NMR spectra of the carbonyl region of poly(DBLA-AdHA) before and after hydrolysis. Peak **f** at 172.6 ppm (assigned to the carbonyl carbon of the γ -lactone of DBLA) decreases after hydrolysis. And new peak **g** at 171.1 ppm (supposed to be assigned to the carbonyl carbon of the γ -hydroxy acid) appears. It can therefore be said that γ -lactone of DBLA can be easily hydrolyzed and produce γ -hydroxy acid.

Figure 6 shows the ^{13}C NMR spectra of the carbonyl region of poly(NLA-AdHA) before and after hydrolysis. Peak **h** at 179.4 ppm is assigned to the carbonyl carbon of the γ -lactone of NLA. After hydrolysis, peak **h** hardly changes and a new

peak corresponding to the carbonyl carbon of the γ -hydroxy acid does not appear. It is difficult to hydrolyze the γ -lactone of NLA under our alkali hydrolysis condition.

The ^{13}C NMR spectra of the carbonyl region of AdLA monomer are shown in Figure 7. Peak **i**, around 178.0 ppm, which is the carbonyl carbon of

the lactone of AdLA, does not disappear after the hydrolysis reaction. The lactone of AdLA monomer is therefore also stable against the alkali hydrolysis reaction. This difference in the reactivity of lactone comes from the steric structure of the lactone. It is discussed in section 3.7.

Figure 5. ^{13}C NMR spectra of poly(DBLA-AdHA) in THF- d_8 . (a) before hydrolysis and (b) after hydrolysis.

Figure 7. ^{13}C NMR spectra of AdLA monomer in THF- d_8 . (a) before hydrolysis and (b) after hydrolysis.

Figure 6. ^{13}C NMR spectra of poly(NLA-AdAH) in THF- d_8 . (a) before hydrolysis and (b) after hydrolysis.

Figure 8. Relationship between γ -hydroxy acid content and dissolution rate.

3.3 Alkali solubility of the polymer with γ -hydroxy acid

The affinity of a polymer containing γ -hydroxy acid, poly(DBHA-AdHA) derived from poly(DBLA-AdHA), to aqueous developer was investigated. Figure 8 plots the dissolution rate of the polymer films as a function of γ -hydroxy acid content.

The poly(DBHA-AdHA) films dissolved at a rate of 50–60 nm/s in 0.05% TMAH developer, even if the γ -hydroxy acid content was reduced to 10 mol%. Moreover, the dissolution rate of poly(DBHA-AdHA) films was not affected by the DBHA content and stayed around 50–60 nm/s. Even in the 2.38% TMAH developer, the films dissolve in a similar manner at a rate of around 220 nm/s. Note that as the alicyclic part of poly(DBHA-AdHA) is smaller, the salting-out effect was not observed.

3.4 Stability of the polymers with hydroxy acid

The stability of the polymers in resist solutions was examined. The polymers were stored in PGME solutions at 20°C. The dissolution rate of the polymer films as a function of the number of days stored is shown in Figure 9. And the dissolution rates of alicyclic polymer with γ -hydroxy acid (polymer 1) and δ -hydroxy acid (polymer 2) previously reported [13,15] are also plotted. The rate for polymer 1 decreases rapidly when it is stored at 20°C, but the rate for polymer 2 decreases little by little at 20°C. This decrease seems to be due to an intramolecular esterification

reaction of hydroxy acid to lactone in solution. Since the δ -hydroxy acid structure is known to be generally more stable than the γ -hydroxy acid structure [20], the difference is reasonable. However, surprisingly, the dissolution rate of poly(DBHA-AdHA) was hardly changed. Although poly(DBHA-AdHA) contains γ -hydroxy acid, it is found to be more stable than polymer 2 which contains δ -hydroxy acid. One of the reasons for this inversion of the order of stability may be the structure of the hydroxy acid, especially the existence of an adjacent alicyclic structure.

3.5 Acid-catalyzed reaction of the polymer having γ -hydroxy acid

Using poly(DBHA-AdHA) and 1.5-wt% TPSOTf, we prepared a two-component resist system. The resist films were exposed by 250-nm deep-UV light and developed in a 0.05% aqueous TMAH developer. The exposure characteristic curves for the resists are shown in Figure 10. Poly(DBHA20-AdHA80) and poly(DBHA30-AdHA70) functioned as negative resists with moderate sensitivity and high contrast in combination with the photo-acid generator. They show lower sensitivity than the resist composed of polymer 2. Although the γ -hydroxy acid structure in these polymers was stable in the PGME solution itself, it exhibits moderate sensitivity to the acid-catalyzed insolubilization reaction. Moreover, poly(DBHA50-AdHA50) does not work as a negative resist. Although the reason for the results is not fully understood, it is thought that too much

Figure 9. Stability of the polymers in PGME solution at 20°C.

hydrophilic γ -hydroxy acid prevents the insolubilization of the resist.

As the lactone of AdLA is stable under the alkali-hydrolysis condition, as mentioned in section 3.2, it is possible to synthesize poly(DBHA-AdLA) and poly(DBAH-AdHA-AdLA) by the selective hydrolysis of the DBLA moiety. As shown in Figure 11, these polymers also function well as negative resists in combination with the photo-acid generator. As every polymer shown in Figure 11 contains 20 mol% of γ -hydroxy acid moiety, their sensitivities to the exposure are almost the same.

Figure 12 shows the ^{13}C NMR spectra of the carbonyl region of poly(DBHA-AdHA) before and after acid-catalyzed reaction in the THF- d_8 solution by adding a small amount of methansulfonic acid. Peak g, at 171.0 ppm, assigned to the carbonyl carbon of the γ -hydroxy of DBHA, disappeared after the acid-catalyzed reaction. And a new peak f, at 172.7 ppm, assigned to the carbonyl carbon of the γ -lactone, appeared. The spectrum after the acid-catalyzed reaction is clearly identical to that

before hydrolysis (Figure 5). It is therefore concluded that the γ -hydroxy acid derived from DBLA can easily produce the γ -lactone by the acid-catalyzed reaction.

3.6 Lithographic performance

The resists mentioned in the previous section were patterned by an ArF excimer-laser stepper (0.6 NA, 0.3 σ) with a phase-shifting mask (Figure 13). Resist films were spun to a 0.25- μm thickness on a silicon substrate (process conditions: prebake at 90°C for 2 min; PEB at 100°C for 2 min; and development in 0.05% aqueous TMAH for 15 sec). As shown in Figure 13(b), 0.12- μm line-and-space patterns were obtained with a dose of 20 mJ/cm². The pattern quality of these new resists is not so good since the formulation of the resists is not optimized. Accordingly, utilizing the γ -hydroxy acid derived from DBLA, the variation and flexibility of the copolymer composition increased. Since the resist properties are controllable, the pattern quality should be thus improved.

Figure 10. Exposure characteristic curves for the resist utilizing γ - and δ -hydroxy acid.

Figure 11. Exposure characteristic curves for the resist utilizing γ -hydroxy acid.

Figure 12. ^{13}C NMR spectra of poly(DBHA-AdHA) in THF- d_8 . (a) before adding methansulfonic acid and (b) after adding methansulfonic acid.

Table 1. Difference between heats of formation (lactone [with H_2O]– hydroxy acid).

	Lactone	Alkali hydrolysis	Hydroxy acid	Acid catalyzed intramolecular esterification	Lactone	$\Delta H(\text{kcal/mol})$ Lactone (with H_2O) – Hydroxy acid
AdLA	ϵ			—	—	-2.29
NLA	γ			—	—	-0.80
DBLA	γ					2.52
Polymer 1	γ					7.83
Polymer 2	δ					13.47

Figure 13. SEM photographs of patterns produced by ArF eximer-laser stepper (0.60 NA, phase-shifting mask) with resist containing (a) poly(DBHA20-AdHA80), (b) poly(DBHA20-AdHA40-AdLA40), and (c) poly(DBHA20-AdLA80).

3.7 Estimation of the difference between the heats of formation of lactone and that of hydroxy acid

The difference of the heat of formation ΔH (lactone group with one molecule of H_2O minus hydroxy acid group) was estimated in the case of several simplified monomeric structures (Table 1). It is clear from the table that ΔH become negative in the case of AdLA and NLA, which means the lactone structure is more stable than the hydroxy acid structure. This explains the result that neither AdLA nor NLA was hydrolyzed as mentioned in section 3.2. It is considered that the strain of the alicyclic moiety plays a major role in these results.

In contrast, ΔH is positive in the case of DBLA. This result suggests that the hydroxy acid is more stable than the corresponding lactone. And ΔH is positive in the case of the γ -hydroxy acid of polymer 1 and δ -hydroxy acid of polymer 2, which also shows that the hydroxy acid is more stable than the corresponding lactone. Therefore, the estimated ΔH s agree with the experimental results.

In conclusion, the difference in the heat of formation is zero to 15 kcal/mol is suitable for the hydroxy acid used in negative resists. The order of ΔH is polymer 2 (13.47), polymer 1 (7.83), DBLA (2.52); however, the order of the stability in the resist solution is DBLA, polymer 2, polymer 1. The actual stability and reactivity of the hydroxy acid therefore does not correspond to the difference

in the estimated heats of formation. It is thus concluded that the reactivity and stability of the hydroxy acid are affected by the steric effect and the polarity of the comonomer.

4. Summary

As ArF negative-tone resist materials utilizing intramolecular esterification reaction, several kinds of lactone structures were examined. It was found that AdLA and NLA are not hydrolyzed into hydroxy acid under an alkali hydrolysis condition. This result was supported by the estimation of the difference between the heat of formation of lactone and that of hydroxy acid. DBLA was found to produce γ -hydroxy acid, which is stable in the resist solution. The γ -hydroxy acid derived from DBLA becomes γ -lactone relatively easily by an acid-catalyzed reaction and can be used to make resists insoluble. Since the variation and the flexibility of the copolymer composition of the base polymer can be increased, the resist properties are controllable and the pattern quality can be improved by utilizing γ -hydroxy acid derived from DBLA.

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