

ORIGINAL ARTICLE

Sensitivity of a chemically amplified three-component resist containing a dissolution inhibitor for extreme ultraviolet lithography

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A chemically amplified three-component resist was prepared by adding a dissolution inhibitor as the third component in an X-ray resist, and its sensitivity and resolution were evaluated for the application in extreme ultraviolet (EUV) lithography. The chemically amplified three-component resist, which consists of a base resin, a photo-acid generator (PAG) and a dissolution inhibitor, achieved high sensitivity with an exposure dose below 10 mJ cm^{-2} . Furthermore, the chemically amplified three-component resist exhibited higher resolution and sensitivity than a resist without a dissolution inhibitor. The dissolution inhibitor was converted to a dissolution promoter containing carboxylic groups by exposure to EUV light, and it acted as a plasticizer in the resist, resulting in facile diffusion of the protons generated from the PAG. Thus, novel EUV resists with high sensitivity and resolution could be designed utilizing chemically amplified three-component resist containing a dissolution inhibitor.

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INTRODUCTION

Process miniaturization is the current trend in lithography for manufacturing semiconductor devices. The development of next-generation dynamic random access memory requires the 32-nm half-pitch node or below. Recently, this issue has been resolved through the double patterning method utilizing an ArF excimer laser in immersion lithography ($\lambda = 193 \text{ nm}$).

Extreme ultraviolet (EUV; $\lambda = 13.5 \text{ nm}$) lithography is also regarded as the most promising next-generation lithography technology at 32-nm half-pitch and below. However, the current resist material does not respond to EUV exposure; thus, novel EUV resist materials are needed. Tremendous effort has been devoted to reducing polymer absorption during the development of KrF, ArF and F₂ excimer lithography. Under ionizing radiation, acid generators in the resist are not primarily sensitized by the incident radiation but by the secondary electrons.^{1,2} Most acid generators can react with electrons with energy as low as the thermal energy.^{3,4} The EUV resist requires high sensitivity because of the low intensity of the EUV exposure equipment. Therefore, the development of an EUV resist has been investigated using a chemical amplification mechanism. Chemically amplified EUV resists are generally composed of two components, a base resin and a photo-acid generator (PAG). The mechanisms by which PAG is sensitized by secondary electrons have

been investigated.^{5–8} Increasing the PAG concentration, the dielectric constant of the polymer, the thermalization distance of the secondary electrons, and the effective reaction radius of the PAG can increase the acid generation efficiency.^{5,6}

The performance of an EUV resist is characterized by parameters such as the line-edge roughness (LER), sensitivity and resolution, but the parameters are a relation of trade-off. Although some excellent EUV resists have been reported, the requirements for next-generation resist materials are so stringent that, despite great effort, the technical solutions for many parameters remain uncertain.^{9–15} Increasing the catalytic chain length to enhance the sensitivity leads to resolution blur. Reducing the feature size increases the degree of LER. Although LER is somewhat suppressed, as the exposure dose or base quencher concentration is increased, both can degrade the sensitivity. This trade-off has been investigated in detail.¹⁶

We have demonstrated the properties of a chemically amplified three-component EUV resist that consists of a dissolution inhibitor in a chemically amplified two-component resist.^{17,18} To achieve higher resolution in the chemically amplified EUV resist, we considered reducing the dissolution rate in an unexposed area using a dissolution inhibitor. We have previously reported the properties of various dissolution inhibitors as the third component in electron-beam (EB) lithography.^{17,19} Thus, isophthalic acid that is chemically modified

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with *tert*-butyl group was found to possess excellent properties as a dissolution inhibitor. In its reaction mechanisms, the EUV resist bears more similarity to EB and X-ray resists than photo resists.^{20,21} Therefore, bis(*tert*-butyl) isophthalate (B-IP) was adopted as the third component in a chemically amplified EUV resist, and the properties of this three-component resist were investigated for application in EUV lithography.

EXPERIMENTAL PROCEDURE

Materials and reagents

The chemically amplified three-component resist consisted of a base resin, a PAG and a dissolution inhibitor. UV-III (ROHM and HAAS Electronic materials, Philadelphia, PA, USA), which consists of poly(4-hydroxystyrene) copolymerized with poly(*tert*-butyl acrylate), was utilized as an X-ray-resist material. Triphenylsulfonium triflate, used as a PAG, was purchased from Aldrich, Tokyo, Japan. The dissolution inhibitor B-IP was prepared in a reaction between isophthalic chloride and *tert*-butyl alcohol in the presence of pyridine.¹⁷ The ethyl lactate solvent and the 1,1,1,3,3,3-hexamethyldisilazane adhesion promoter were purchased from Wako Pure Chemical Industry, Co., Ltd., Kyoto, Japan. A 2.38-wt% tetramethylammonium hydroxide solution was purchased from Tokyo Ohka Kogyo Co., Ltd., Tokyo, Japan and used as a developer.

Preparation of the three-component resist on a Si wafer

Figure 1 provides the chemical structures of the components in the EUV resists. UV-III was dissolved in ethyl lactate at a volume ratio of 1:1. The base resin content in the UV-III was approximately 28 and 5 wt% triphenylsulfonium triflate relative to the base resin was added to the solution. The two-component resist solution was then dissolved in a suitable quantity (10, 25, 50 and 75 wt%) of B-IP relative to the base resin (three-component resist).

A 6-inch silicon wafer was pretreated with 1,1,1,3,3,3-hexamethyldisilazane, and the prepared resist solutions were spin-coated onto the wafer at 2000 r.p.m. for 30 s. After spin-coating, the wafer was prebaked on a hotplate at 130 °C for 90 s. The thickness of the prebaked wafer was measured using a Dektak 6M surface profilometer (ULVAC, Chigasaki, Japan). The initial thickness of the two- or three-component resist was 213 or 260 nm, respectively. The wafer was exposed to EUV light (wavelength: 13.5 nm, intensity: 0.0189 mW cm⁻²) by an exposure system (EUV source: Electroless Z-Pinch, ENERGETIQ, Woburn, MA, USA). The post-exposure baking (PEB) of the exposed resist was performed on a hotplate at 110 °C for 60 s, and the resist was developed by immersion in 2.38 wt% tetramethylammonium hydroxide and rinsed with de-ionized water. The thickness of the developed resist was also measured using a Dektak 6M, and the sensitivity and discrimination curves were prepared from the ratio of the thickness before and after development under various conditions. The resolution and LER were difficult to evaluate because of the mask processing and analytical instruments. Therefore, the resolution of the resists was evaluated qualitatively using the dissolution rate modulus calculated from the discrimination curves.

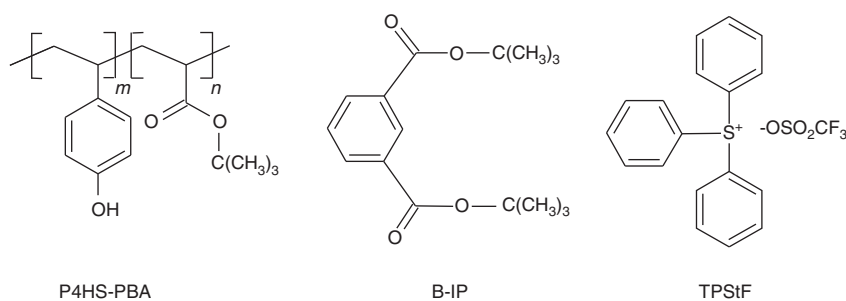


Figure 1 Chemical structure of the components in the extreme ultraviolet resists. From left to right: poly(4-hydroxystyrene) copolymerized with poly(*tert*-butyl acrylate) (P4HS-PBA) as a base resin, bis(*tert*-butyl) isophthalate (B-IP) as a dissolution inhibitor and triphenylsulfonium triflate as a photo-acid generator (PAG).

Evaluating the effect of the dissolution inhibitor via differential scanning calorimetry (DSC) and Fourier-transform infrared (FT-IR)

DSC was performed on a DSC-8230 (Rigaku, Tokyo, Japan) to evaluate the effect of the dissolution inhibitor and the PAG in the resist. The four-resist samples were prepared as follows: UV-III only, UV-III containing 5 wt% the PAG, UV-III containing 25 wt% dissolution inhibitor and UV-III containing binary mixtures. These sample solutions were spin-coated onto the silicon wafer using a speed program of 300 r.p.m. for 3 s to 1000 r.p.m. for 20 s and prebaked on a hot plate at 130 °C for 90 s. After prebaking, the samples were scraped off the silicon wafer, and 5 mg of the material was placed in an aluminum pan. The DSC measurements were performed with a heating rate of 5 °C min⁻¹ under atmospheric conditions.

The decomposition of the *tert*-butyl group in the resist samples was evaluated via FT-IR (Shimadzu, Kyoto, Japan, IR Prestage-21). The two- and three-component resists were prepared without prebaking, without exposure and with exposure at 10 mJ cm⁻² followed by baking at 110 °C for 60 s.

RESULTS AND DISCUSSION

To investigate suitable PEB temperature conditions, the chemically amplified two-component resists were prepared at various PEB temperatures and their sensitivities were evaluated. The sensitivity curves of the two-component resists prepared at various PEB temperatures are shown in Figure 2. The sensitivity and the thickness in the unexposed resist area decreased with increasing PEB temperature. The resist was believed to harden with treatment at high PEB temperature. The sensitivity of the EUV resist was expected to be approximately 10 mJ cm⁻², but lower for mass production.²² In the ITRS 2008 road map, the EUV lithography resist performance was targeted toward 16-nm resolution, 0.9-nm 3 σ LER and 10-mJ cm⁻² sensitivity. Each resist treated at a PEB temperature of 90–110 °C achieved high sensitivity at below 10 mJ cm⁻². The discrimination curves of the two-component resists prepared at various PEB temperatures are shown in Figure 3. The dissolution rate in the unexposed area was lower at higher PEB temperatures, with that in the exposed area exhibiting the opposite behavior. The resist was hardened via PEB at 110 °C and was hardly dissolved in alkaline solution. However, the dissolution rate in the exposed area was higher at elevated PEB temperatures because the diffusion of the protons generated from the PAG in the resist was enhanced by the PEB. The ratios of the dissolution rates in the exposed and unexposed areas increased with increasing PEB temperature with a maximum at 110 °C. The results indicated that high resolution could be obtained at a PEB temperature of 110 °C.

We previously reported the properties of various dissolution inhibitors as the third component in EB lithography; of these, isophthalic acid with chemically modified *tert*-butyl groups (B-IP)

was found most suitable.^{17,19} In the exposed area, B-IP having *tert*-butyl group was converted to carboxylic acid, and the dissolution rate of the resist toward an alkaline developer was increased. By contrast, the dissolution rate of B-IP in the unexposed area was slower than that of other dissolution inhibitors. Thus, the chemically amplified three-component resist for EUV lithography was prepared using B-IP as the third component. The sensitivity curves of the two- or three-component resists are shown in Figure 4, and the discrimination curves are shown in Figure 5. The sensitivity of the three-component resist exceeded that of the two-component resist, yielding a curve with a steeper slope. Generally, the resist having the curve with a steeper slope would be high resolution. The thickness in the unexposed area

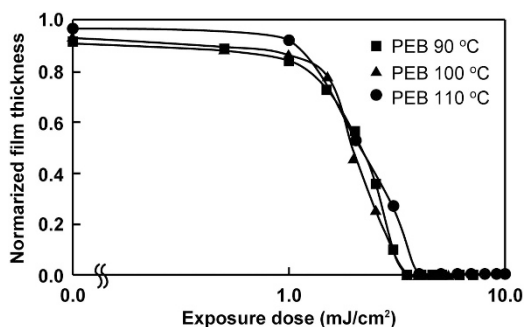


Figure 2 Sensitivity curves of the chemically amplified two-component extreme ultraviolet resists at various post-exposure baking (PEB) temperatures.

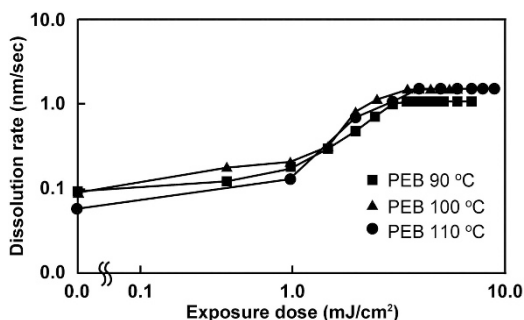


Figure 3 Discrimination curves of the chemically amplified two-component extreme ultraviolet resists at various post-exposure baking (PEB) temperatures.

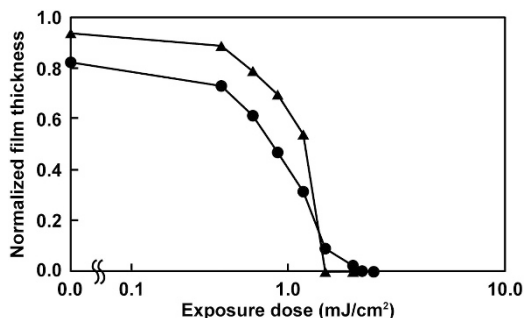


Figure 4 Sensitivity curves of the chemically amplified two- or three-component extreme ultraviolet resists. The filled triangle indicates the sensitivity curve of the three-component resist; the filled circle indicates that of the two-component resist.

of the three-component resist decreased less than that in the two-component resist because of the dissolution inhibitor. B-IP used as the dissolution inhibitor was insoluble in alkaline solutions. The sensitivity in three-component resist having B-IP would be lower than that of the two-component resist, because the decomposition of the *tert*-butyl groups requires more protons from the PAG. However, the sensitivity of the three-component resist exceeded that of the two-component resist, despite the trade-off between sensitivity and resolution. This unique behavior can be explained that the dissolution inhibitor is acted as a plasticizer in the resist and the proton generated from the PAG was easily diffused through the resist. The dissolution rate in the unexposed area of the three-component resist was below that of a two-component resist, and the dissolution rate in the exposed area of the three-component resist was higher than that of a two-component resist as indicated by Figure 5. In the exposed area, the *tert*-butyl groups of the B-IP were decomposed by the protons generated from the PAG, and the B-IP was converted to a small molecule containing carboxyl groups. After exposure, the B-IP acted as a dissolution promoter in the resist, and the dissolution rate of the three-component resist in the exposed area increased. From the result of the ratio of the dissolution rates in exposed and unexposed areas, it could estimate that the three-component resist has higher resolution than the two-component resist.

This effect was confirmed by adding a dissolution inhibitor to the resist along with DSC measurements. The DSC curves of the various EUV resist components are shown in Figure 6 and confirmed an endothermic peak for each sample. The peaks anticipate the melting

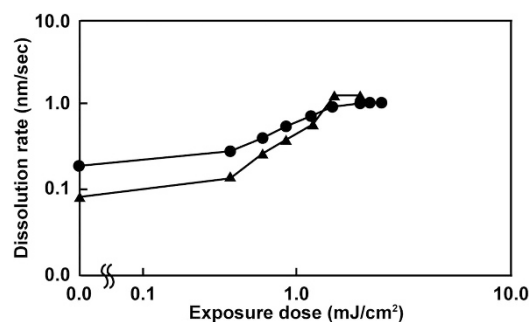


Figure 5 Discrimination curves of the chemically amplified two- or three-component extreme ultraviolet resists. The filled triangle indicates the sensitivity curve of the three-component resist; the filled circle indicates that of the two-component resist.

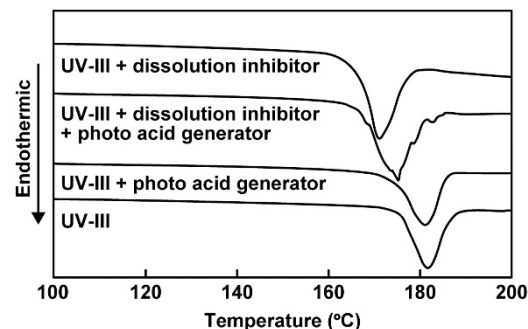


Figure 6 Differential scanning calorimetry curves of the chemically amplified extreme ultraviolet resists with various components. The measurements were carried out with 5 mg samples and a 5 °C min⁻¹ rate of temperature increase.

point of the base resin, UV-III. The individual peak for UV-III and the peak for the two-component resist (UV-III plus PAG) were both confirmed at 182 °C. By contrast, the peak for UV-III after the addition of a dissolution inhibitor shifted toward a lower temperature of 172 °C. The peak of the three-component resist also shifted toward a lower temperature of 175 °C. The melting point of UV-III decreased with the addition of a dissolution inhibitor. Thus, the resist could become plasticized with the addition of a dissolution inhibitor. Consequently, the sensitivity of the three-component resist was improved to diffuse easily the proton in the exposed area through the plasticizing of the resist polymer.

Figure 7 provides the FT-IR spectra of the two- and three-component resists with and without prebaking and PEB. In the FT-IR spectra, the unique peaks at 1370 and from 1385 to 1395 cm^{-1} indicate the stretching vibrations of the *tert*-butyl groups in the base polymer. The peaks for the *tert*-butyl groups are decreased in the FT-IR spectrum of the two-component resist after prebaking, which indicates the decomposition of the *tert*-butyl groups occurs only by prebaking, thus decreasing the film thickness in the unexposed area. In contrast, the FT-IR spectral peaks for the three-component resist exhibited no decrease after prebaking, and those after exposure and PEB decreased because of the decomposition of the *tert*-butyl groups. Thermo-treating the three-component resist by adding B-IP inhibited the decomposition of the *tert*-butyl groups, and the contrast between the dissolution in the exposed and unexposed areas improved significantly.

To evaluate the effect of the *tert*-butyl groups on the sensitivity and resolution of the resist, the ratio of the *tert*-butyl groups in the poly(4-hydroxystyrene) or the quantity of dissolution inhibitor were varied in the three-component resist. The ratio of the *tert*-butyl groups in the poly(4-hydroxystyrene) varied from 0 to 30, 50 and 100%, and the quantity of dissolution inhibitor varied from 10 to 30, 50 and 75 wt%

relative to the base resin. The decrease in film thickness in the unexposed area of the base resin functionalized by *tert*-butyl groups at various ratios is depicted in Figure 8a. A simple poly(4-hydroxystyrene) base resin was easily dissolved in tetramethylammonium hydroxide while developing. The residual film thickness increased with increasing ratios of *tert*-butyl groups, with no variation in the film thickness before or after developing with a base resin containing completely functionalized *tert*-butyl groups. The film thickness of the base resin containing *tert*-butyl groups functionalized at ratios of 30% and 50% decreased approximately 10% and 5%, respectively, relative to the initial thickness.

Figure 8b provides the relationship between the normalized residual film thickness and the quantity of dissolution inhibitor added. The normalized residual film thickness in the unexposed area increased slightly as the quantity of dissolution inhibitor increased. The sensitivity curves of the resists containing various quantities of dissolution inhibitor are shown in Figure 9a. The residual film thickness in the unexposed area increased with as the quantity of dissolution inhibitor increased from 10 to 25 and 50 wt%. The sensitivity of the resist containing 25 wt% dissolution inhibitor was approximately 2 mJ cm^{-2} , which exceeded those of the resists containing 10 or 50 wt%. Furthermore, the slope of the sensitivity curves exhibited identical behavior. These results indicated that the quantity of dissolution inhibitor providing the highest resist sensitivity was approximately 25 wt%.

The resolution of the resists containing various quantities of dissolution inhibitor was evaluated using the discrimination curves shown in Figure 9b. The dissolution rate in the unexposed area decreased as the quantity of dissolution inhibitor increased (10, 25 and 50 wt%). In the exposed area, the resist containing 25 wt% dissolution inhibitor exhibited a higher dissolution rate than the others. The dissolution contrasts calculated using the exposed and

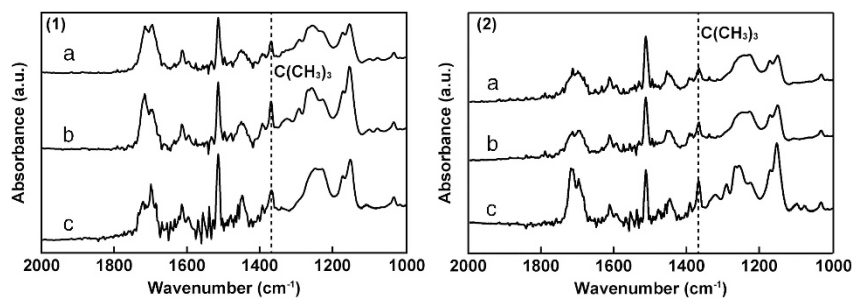


Figure 7 Fourier-transform infrared spectra of the chemically amplified three- (1) or two-component (2) extreme ultraviolet resists under the following thermo-treatments: (a) prebaking at 110 °C, exposure at 10 mJ cm^{-2} and post-exposure baking at 130 °C, (b) prebaking only at 110 °C and (c) untreated.

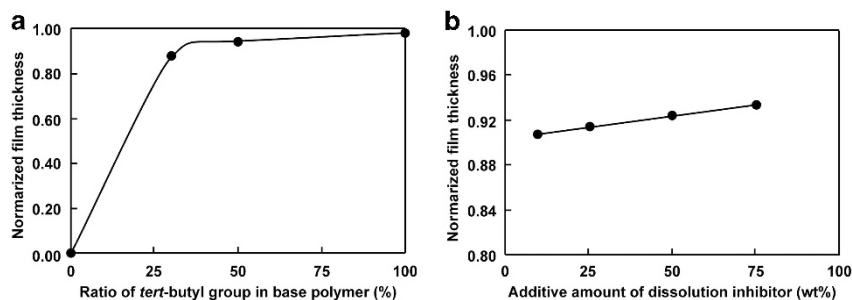


Figure 8 Effect of the *tert*-butyl groups on the resist solubility. (a) Relationship between the ratios of *tert*-butyl groups in the base polymer and the normalized film thickness. (b) Relationship between the quantity of dissolution inhibitor added and the normalized film thickness.

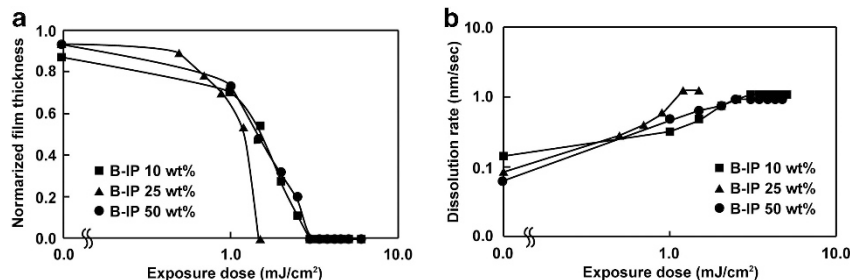


Figure 9 (a) Sensitivity and (b) discrimination curves for the chemically amplified three-component resist with various quantities of dissolution inhibitor. B-IP, bis(*tert*-butyl) isophthalate.

unexposed areas of each resist containing 10, 25 and 50 wt% dissolution inhibitor were 7.5, 18.6 and 15.4, respectively. Consequently, the resist containing 25 wt% dissolution inhibitor was found to have the best sensitivity and resolution for EUV applications.

CONCLUSIONS

A chemically amplified three-component resist was prepared by adding a dissolution inhibitor as the third component in an X-ray resist for applications in EUV exposure, and its sensitivity and resolution were evaluated. The optimal PEB temperature in the chemically amplified resist was determined at 110 °C, resulting from sensitivity and discrimination curves of the two-component resist. The resists achieved higher sensitivity at below 10 mJ cm⁻². The three-component resist, which consists of a base resin (poly(4-hydroxystyrene) copolymerized with poly(*tert*-butyl acrylate)), PAG (triphenylsulfonium triflate) and a dissolution inhibitor (B-IP), exhibited improved resolution and sensitivity over the two-component resist, although the relationship between the sensitivity and resolution was a trade-off. The dissolution inhibitor was converted into a dissolution promoter containing carboxylic groups under exposure to EUV light and acted as a plasticizer in the unexposed area, resulting in the facile diffusion of the proton generated from the PAG. Consequently, the design of novel EUV resists with high sensitivity and resolution was accomplished using an X-ray resist containing a dissolution inhibitor. However, the resolution and LER could not be precisely evaluated because of the mask processing and analytical instrumentation. In the near future, we wish to further evaluate the properties of the chemically amplified three-component resist including its resolution and LER toward application as an industrial EUV.

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