Novel EUV Resist Materials Design for 14 nm Half Pitch and below

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Polymers with a different Tg and activation energy were prepared to clarify influences of acid diffusion on resolution at 15 nm half-pitch (hp) and 14 nm hp using a EUV micro-field exposure tool (MET) at LBNL. Resolution on such a narrow pattern was limited by collapse and pinching. Clear relationship between pinching numbers and polymer Tg indicates that acid diffusion is one of major contributors on the pinching. In addition, polymers with a low thermal activation energy (Ea) on deprotection were effective for reducing pinching. This is probably originated from its high chemically amplification character even in low post-exposure bake (PEB) temperature to obtain both large chemical contrast and short acid diffusion. On the other hand, a good correlation between a cleanable outgassing amount and Ea indicates trade-off relationship between outgassing and resolution. Advantages of n-butyl acetate (nBA) developer have been investigated in viewpoint of dissolution uniformity. Surface roughness of a non-patterned resist film at half-exposed area, which was well correlated with LWR, was measured by AFM as indicator of uniformity in development process. To avoid any differences in resist chemistry other than development process, cross linking negative tone resist was applied for this study. The surface roughness obtained by nBA, which is conventional negative-tone imaging (NTI) developer, was 32 % lower than that obtained by 2.38 % TMAH solution. NTI resist system with a nBA developer and optimized resist reduced LWR from 4.8 nm to 3.0 nm in comparison with conventional positive tone resist with a 2.38 % TMAH developer. In addition, advantage on semi-dense trench patterning was well defined. New EUV sensitizer with 1.15 times higher EUV absorption resulted in 1.15 times higher acid yield by EUV exposure. Lithography performance of the new EUV sensitizer has been investigated by MET at SEMATECH Albany. Sensitivity was indeed improved from 20 mJ/cm² to 17 mJ/cm² according to the acid yield increase, but resolution was significantly degraded.

Keywords: EUV lithography, LWR, chemically amplified resist, negative tone imaging, EUV sensitizer

1. Introduction

EUV lithography is one of the most promising candidates for half-pitch (hp) 20 nm node device manufacturing and below[1]. The main issue for developing EUV resist is to satisfy the ITRS targets of resolution, line-width roughness (LWR), sensitivity, and outgassing simultaneously[1-4]. Although partial resolution of 14 nm hp has been recently demonstrated using chemically amplified resist (CAR) on micro-field exposure tool (MET) at LBNL and interference exposure tool at PSI, its sensitivity of 30 mJ/cm² is still far from target below 20 mJ/cm²[5-7]. In other words, current EUV resists and processes have serious limitations in high volume manufacturing because all of resists that showed
resolution below 14 nm hp had larger Esizes than 25 mJ/cm².

CAR serves as a key technology for photolithography of 20 nm hp patterning and below because of its high sensitivity and resolution. Many studies have revealed that most important process in CAR is post-exposure bake (PEB) process because deprotection reaction and diffusion kinetics of acid generated from photoacid generator (PAG) directly determines resolution and sensitivity[8-12]. Especially suppression of acid diffusion is dominant contributor to improve resolution and LWR in such a narrow pitch patterning. This approach, however, causes sensitivity loss, therefore, different material designs are strongly required based on a comprehensive understanding of reaction and diffusion processes during PEB process.

Noise mitigation through lithography process is important because photon-shot noise (PSN) dominantly determines resolution and LWR of current EUV resist[13, 14]. Our previous study revealed that sensitivity more than 30 mJ/cm² is necessary to obtain realistic pattern quality on 15 nm hp. We also revealed that one approach to mitigate PSN is applying special rinse process that affords smoothing of noise[13]. However, detailed understanding is still lacking how we can mitigate PSN using other lithography processes and materials.

The present study aims to clarify how we can improve lithography performance of 15 nm hp and 14 nm hp pattern. Accordingly, we prepared resist materials whose Tg and Ea are different with each other, to be exposed using a MET at LBNL. We also report herein a novel approach to improve resolution and LWR by using an EUV dedicated negative-tone imaging (NTI) system and a new EUV sensitizer.

2. Experimental
2.1 Materials
A series of protected co-polymers with different thermal activation energy were synthesized according to the conventional polymerization methods [15]. An organic solution containing each of polymer, PAG and organic amine as a quencher was prepared, and the resulting solution was filtered with 0.03 mm polyethylene filter for lithographic evaluation.

2.2 Lithographic evaluation using an EUV light

The resist solution was filtered and spin-coated on silicon wafer that was treated with an UL, and pre-baked at 100 °C for 60 sec to give a film thickness of 30 nm. The wafer was exposed with EUV light using an Exitech MET with 0.30 NA and Pseudo-PSM (PPSM) illumination at LBNL to give a patterned wafer. After the exposure, the wafer was baked (PEB) at each temperature for 90 sec, and developed with a 2.38 % TMAH aqueous solution or FN-DP001 (nBA) at 23 °C for 30 sec. The exposed wafer was analyzed by CD-SEM tool (S-4800, Hitachi). EUV exposure at Sematech Albany and EIDEC were performed in a similar manner to that of LBNL using an Exitech MET with 0.30 NA and quadrupole illumination (0.93/0.36) at Albany, and small-field exposure tool (SFET) at EIDEC with 0.30 NA and annular illumination (0.7/0.3), respectively. In this paper, pinching was defined as line break shorter than pattern pitch. Wafers similar to those described above were prepared for verification of NTI resist performance using an ASML NXE:3100 scanner with 0.25 NA and dipole 60X illumination (σouter / σinner = 0.83 / 0.41) for 1:1 L/S patterning and conventional illumination (σ = 0.51) for trench patterning with 30 nm line on 120 nm pitch mask..

2.3 Cleanable outgassing evaluation
Outgassing measurement was performed using a ROX at CNSE. Detailed about measurement method are described in another paper[16].

2.4 Thermal activation energy (Ea) of deprotection determination
To determine Ea of each polymer, PEB temperature dependency of E0 was measured using a Hitachi HL 750D (Vacc = 50 kev) E-beam tool. The resist coated wafers were prepared for the analyses using the similar manner to that described in section 2.2. Log slope of E0 and PEB temperature plots was used as Ea in this paper.

2.5 Surface Roughness analysis
The surface roughness of non-patterned resist film was measured with Veeco Nanoscope IIIa AFM system at various film formation conditions. A resist-coating sample was prepared using the similar procedure as described in section 2.2. A 1 cm x 1cm area of resist film was fully exposed by E-beam using Hitachi HL 750D (Vacc = 50 kev) without any patterning for the AFM measurement. The PEB and development procedures were also similar to that described in section 2.2.
3. Results and discussion

3.1. High Tg and Low Ea polymer for resolution

We have previously reported that major contributors on pinching are photon-shot noise and acid diffusion[13]. Design of PAG to suppress acid diffusion, for example PAG anion bound polymer, had been reported as a good candidate for short acid diffusion resist. Our previous study also indicates that low Ea polymer and low PEB temperature are necessary simultaneously to satisfy sensitivity and resolution targets probably due to its high chemical contrast and small diffusivity characters[13]. However, detail understanding about polymer Ea and Tg are still lacking.

Figure 1 shows $E_0$ obtained with EB exposure against normalized Ea at wide range of PEB temperature: i) standard, ii) standard - 40 °C, iii) standard - 60 °C for various resists which have different Ea. Low Ea resist showed higher sensitivity at the whole PEB temperature range as shown in Figure 1. This tendency is consistent with previous reports about the deprotection reaction of typical chemically amplified resist[13,17]. It is noteworthy that resist with lower Ea than 0.5 kept high sensitivity around 10 $\mu$C/cm$^2$ even at lowest PEB temperature of “standard - 60 °C”.

In order to investigate effect of Ea and Tg, we exposed various kind of resists at each optimum PEB temperature using a MET at LBNL with PPSM illumination to get 14 nm hp to 18 nm hp patterns. Polymers were systematically prepared to get different Ea and Tg. Table 1 summarizes EUV lithography performances of resists A ~ E which contain the different polymers with each other.

Figure 2a illustrates the relationship between number of pinching and Ea for resists, A, C, D, and E, in which polymers have Tg higher that PEB temperature. It clearly shows that normalized Ea less than 1.0 decreases the number of pinching. As a consequence, lowest Ea resist (resist A) showed best resolution among all of the resists (Table 1). Figure 2b illustrates the relationship between number of pinching and Ea for resists, A, C, D, and E, in which polymers have Tg higher that PEB temperature. It clearly shows that normalized Ea less than 1.0 decreases the number of pinching. As a consequence, lowest Ea resist (resist A) showed best resolution among all of the resists (Table 1).
Figure 2. Dependency of pinching on (a) normalized Ea and (b) $\Delta (T_g - \text{PEB temperature})$. The pinching was defined as line break shorter than pitch of pattern.

number of pinching and $\Delta (T_g - \text{PEB temperature})$ except for resist E containing high Ea polymer. As clearly seen in this Figure, $T_g$ at least 40 °C higher than PEB temperature resulted in less pinching. In summary, combination of “low Ea and high $T_g$ polymer” and “low PEB temperature” should open up a promising way simultaneously to satisfy E-size and pinching (LWR) requirements at least on 15 nm hp. Unfortunately, all resists didn’t resolve 14 nm hp because of severe pinching and collapse. A sudden increase of pinching in 14 nm hp may be explained by a sudden degradation of aerial image with this illumination conditions. Pattern collapse is, of course, key limiting factor on such an ultra narrow pitch because of large capillary force associated with a high surface tension of water, so a hydrophobic resist and a surfactant rinse material to give a low surface tension may be also necessary to resolve 14 nm hp L/S pattern.

As a reference, E-beam patterning has been performed with a resist similar to C through 18 nm hp to 14 nm hp. Cross-section SEM data are listed in Figure 3. We can clearly conclude that pattern collapse and partial pinching are major limiting factors in EB patterning as well. Conclusion here is that we have to improve both collapse and pinching for 14 nm hp and beyond regardless of exposure system. 3.2. E-beam exposure results

3.2. Outgassing issue of low Ea polymer

In previous work, we revealed that lower sensitivity to obtain better resolution and pinching (LWR) caused worse outgassing[13]. Although outgassing has received a great deal of attention due to its impact on throughput and damages to optics, only several studies have been reported regarding chemistry of outgassing[19-23]. There have been only one report that deals with influence of Ea on outgassing[23]. Therefore, we have investigated, in particular, effects of Ea on outgassing using a wide variety of polymers with a different acid labile groups (ALG).

Figure 4 illustrates normalized Ea versus “cleanable outgas contamination thickness (CG)” over $E_0$. The CG was obtained from witness-based outgassing measurement that was taken with a ROX tool. A monotonic decrease of CG/$E_0$ with increase of normalized Ea was obtained. Some papers pointed out that low Ea polymer thermally reacts with light-generated acid at room temperature and generates volatile species during exposure step. This result in Figure 4 strongly

18nm hp 16nm hp 15nm hp 14nm hp

Figure 3. X-section images of resist C derivative printed by E-beam (50 keV). HP of line and space is varied from 18 nm line/space to 14 nm line/space.
suggests that low Ea polymer has a potential risk on outgassing due to its high thermal reactivity character even at room temperature. It was estimated from the Esize of NXE scanner and E0 of ROX that 30 mJ/cm² Esize for NXE scanner is equal to 11.5 mJ/cm² E0 for ROX tool[24]. CG/E0 limit of 0.26 nm/mJ/cm² was estimated from this plot and the correlation between Esize of NXE scanner and E0 of ROX, with the condition of 30 mJ/cm² Esize for 14 nm hp. Unfortunately, low Ea limit of 1.0 for 14 nm HP resolution (see section 1.6) can not meet the specification of CG/E0 0.26. Key breakthrough technology for simultaneous improvement on both outgassing and resolution is to apply topcoat as shown in our previous paper[13].

3.3. Noise mitigation approach (1): dissolution uniformity of nBA developer for LWR
Recent studies revealed that NTI system had a clear advantage on LWR due to low swelling character of nBA and high chemical contrast character of NTI specific resist[25, 26]. These investigations, however, do not distinguish individual influences of deprotection contrast, uniformity of development, and swelling in development process. To elucidate root cause of LWR advantage of NTI is important for any improvement of NTI system in future.

We selected here a cross-linking type negative tone resist for a comparison of nBA and 2.38% TMAH aqueous developers, because cross-linking system essentially show the essentially same de-protection contrast in PEB step regardless of developer. Figure 5a shows contrast curves of the resist processed with nBA or 2.38% TMAH developers. Figure 5b shows patterning data by E-beam. Similar patterning results on both developers indicate that nBA and TMAH have a same swelling character in this case (Figure 5b).

Exposure dose dependency of surface roughness (Ra) is shown in Figure 6 using the cross-linking type negative tone resist developed with nBA or 2.38% TMAH solution. The Ra increased with increasing exposure dose and reached a maximum value, and then decreased at higher dose. This tendency is quite similar with each other between nBA and 2.38% TMAH. On the other hand, AFM image of half-exposed area was significantly different with each other. It is interesting to note that resist surface after nBA development was very smooth compared with that after TMAH development (Ra 4.5 nm for nBA, 6.6 nm for TMAH). In this experiment, we consider that any influences of de-protection contrast and swelling in development process are excluded, therefore, we conclude that nBA developer has a potential advantage on uniformity in development process. This should be the reason for superior LWR in NTI process reported so far both in ArF immersion and EUV lithography[26-28].
3.4. Demonstration of lithography performances using a latest material dedicated for NTI process

Based on the results shown in section 3.3, we designed new NTI resist to demonstrate advantage of LWR compared with conventional positive tone EUV resist. Lithography performance of a conventional positive tone resist and the newly developed NTI resist were evaluated at 45 nm 1:1 L/S pattern using a SFET at EIDEC with an annular illumination (NA 0.30, σo/i = 0.7/0.3), and summarized in Table 2. As clearly seen in Table 2, LWR was drastically improved from 4.8 nm to 3.0 nm using a FN-DP001 (nBA) as developer with maintaining good sensitivity.

Another key attention is to recognize overall advantage of NTI process, for example ultimate resolution, trench and contact hole patterning capability. Full-field EUV exposure have been performed using a NXE:3100 (NA 0.25) with (a) dipole 60X illumination (σouter / σinner = 0.81 / 0.43) for 1:1 L/S and (b) conventional illumination (σ= 0.51) for trench. As shown in Figure 7a, NTI resist have a potential resolution capability down to 20 nm hp 1:1 L/S. Ultimate resolution of NTI was rather worse than that of current positive tone
resist because of pattern collapse. However, optimization of process and material, such as underlayer, development recipe, nozzle, and rinse material, should open up a new route for 14 nm hp resolution with good LWR. Trench patterning capability was investigated using a mask of 30 nm line on pitch 120 nm with an over exposure as shown in Figure 7b. This demonstrates that NTI resist can resolve trench around 18 nm at reasonable dose of 20 mJ/cm², although the resist formulation has not been optimized yet. This indicates that NTI has a potential advantage on trench patterning similar to ArFi case[28-29].

Lithography performance of dense contact hole are now under evaluation using a different type of NTI resist because contact hole exposure in NTI generally results in much higher sensitivity below 10 mJ/cm² if we apply the same resist for 1:1 L/S patterning. This shift might be originated by flare in EUV scanner.

3.5. Noise mitigation approach (2): novel EUV sensitizer using a latest material dedicated for NTI process

Maximization of acid generation yield is important in EUV lithography because EUV needs high sensitivity to achieve high throughput due to the limitation on available light source power[1]. In addition, PSN gives big impact to 15nm HP patterning in the dose region at least below 30 mJ/cm² for 15 nm hp.14. However, it could be considered that chemical noise would be minimized by maximizing chemical contrast of generated acid concentration. Therefore, resist design that exhibits a higher acid yield than current EUV resist would be effective to achieve better pattern quality.

Sensitization mechanism of EUV resist has been well investigated by many researchers [30-40]. Absorption of high-energy EUV light (13.5 nm) mainly by polymer is the first step of EUV sensitization. Subsequent ionization of polymer and release of electron is next important step of the sensitization. This electron is then trapped by PAG through reductive electron transfer and activates PAG to generate acid. It has been reported that acid generation yield under EUV exposure is strongly affected by polymer structure[32-36], PAG structure[37-39], and its loadings[38-40], respectively.

We have revealed that reductive electron transfer to PAG and activation of PAG to generate acid are already maximized, so important process to increase acid yield should be EUV absorption
step. It is well known that EUV absorption is generally dependent on absorption cross section of atom[41]. Based on the absorption cross section and basic synthetic chemistry, we designed new EUV sensitizer which has EUV absorption coefficient of 5.7 that is 1.15 times higher than absorption coefficient 4.3 of model co-polymer of 4-hydroxy styrene and methyl methacrylate (PHS/MMA). Figure 8 shows acid yield of the new EUV sensitizer against PHS/MMA under EUV exposure. This clearly shows that the new EUV sensitizer showed 1.15 times higher acid yield than PHS/MMA in accordance with calculated EUV absorption coefficient.

Lithography performance of a resist containing the new EUV sensitizer was studied by MET at SEMATECH Albany. Unfortunately, resolution of this sensitizer was tremendously bad even on relaxed hp of 28 nm probably due to its low Tg and large swelling properties. However, remarkable conclusion here is sensitivity was indeed improved from 20 mJ/cm² to 17 mJ/cm² by using the new sensitizer according to the acid yield increase.

4. Conclusions

This study has shown the potential factor that determines 14 nm hp resolution using MET at LBNL. A Low Ea and high Tg polymer with low PEB temperature has a significant improvement on resolution of 15 nm hp and 14 nm hp. Cleanable outgas is major concern for using the low Ea polymer, and need breakthrough simultaneously to satisfy lithography performance targets and outgas specification. On the other hand, we again show that pattern collapse is still key limiting factor on such a narrow pitch. Quantitative experiment revealed that nBA has an advantage on smoothness of pattern, in other words, LWR. Newly developed NTI resist dedicated for EUV demonstrated advantage of LWR and trench patterning capability. Big progress in resolution down to 20 nm HP was achieved. New EUV sensitizer gave a 1.15 times higher acid yield than conventional polymer by EUV exposure. Lithography performance of the new sensitizer was not good, but keeping attention to the design of new EUV sensitizer should lead to revolutionary route for 14 nm hp device manufacturing with an acceptable throughput. These results indicate that our materials design now provides a novel method to breakthrough RLS trade-off relationship even in unfavorable situation of EUVL against PSN.

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