193 nm Resists for Deep Sub-Wavelength Applications

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Among the chemistries/polymers reported for the 193 nm photoresist applications, methacrylate copolymers consisting of 2-methyl-2-adamantane methacrylate (2-MadMA) and mevalonic lactone methacrylate (MLMA) and cycloolefin polymers derived from derivatives of norbornene have shown promising results. We have studied the lithographic properties of these two different promising chemistries. Both systems offer linear resolutions down to 130 nm dense lines using conventional 193 nm illumination and high sensitivity at standard developer conditions. The etch rates of the methacrylate and cycloolefin based resists for polysilicon were found to be 1.4 and 1.3 times higher than that of novolak resist. Calculation of the normalized image log-slope (NILS) for both resist types shows that they can still resolve aerial images with a NILS of about 1.0, which is also confirmed by their 248 nm performance. The potential of 193 nm lithography is estimated at 110 nm dense line resolution if resists can be improved to match the performance of dense line resolution if resists can be improved to match the performance of the best current 248 nm resists which can resolve NILS values of about 0.7

Keywords: 193 nm resists, methacrylate copolymers, 2-methyl-2-adamantane methacrylate, mevalonic lactone methacrylate, polysilicon, novolak resist

1. Introduction

The introduction of 193 nm lithography into manufacturing is seen to occur at the earliest for the 130 nm technology node. This makes 193 nm lithography the first lithographic technology to be introduced at CD design rules that are substantially smaller than the wavelength of the imaging light. At the introduction of i-line lithography, the CD's printed were about 1.5 times larger than the 365 nm wavelength, while at the time of transition to mainstream 248 nm DUV lithography, the ratio had already shrunk to unity. If 193 nm lithography is introduced for the 130 nm technology node, the ratio will be as low as 67% of the imaging wavelength. Such deep sub-wavelength lithography will put special demands on photoresists, and it is presently not clear if 193 nm resist development

can be pushed to a performance that will allow the industry to avoid the widespread use of optical enhancements.

The task facing the resist chemists is not made easier by the constraints placed upon them by the nature of the problem. For any optical lithography beyond i-line, the design of resist systems is primarily determined by the polymer absorbance. For 248 nm lithography, the high absorbance of novolaks necessitated a switch to polyhydroxystyrene as a base polymer; since PHS does not show the same dissolution inhibition phenomena as novolak, functional group deprotection and chemical amplification had to be introduced. Similarly, for 193 nm lithography, the high absorbance of double

bonds has required the development of new wholly aliphatic polymers using carboxylic acid groups as a substitute for the acidity of the phenolic group. For 157 nm lithography, a technology which has recently seen a large boost in interest as a means of further extending optical lithography, even carboxylic acid groups are too absorbing, and current development concepts focus on fluoroalcohols or siloxanes as possible base resins.

The design of polymers for 193 nm photoresist applications requires the delicate act of balancing the hydrophilic/hydrophobic nature of the polymer with the introduction of etch resistant and adhesion promoting units. Pioneering work at IBM and Fujitsu showed the feasibility of imaging with aliphatic polymers but also the need to embody highly annelated carbon rings to provide dry etch resistance. The annelated structures in these polymers can be introduced either in the main chain or in the side chains. For instance, researchers at NEC¹ and Fujitsu²⁻³ have introduced tricyclododecyl and adamantane groups in the side chains of methacrylate type polymers, respectively. More recently, systems have been reported in which the annelated carbon rings are introduced in the main chain⁴⁻⁶ through cycloolefin monomers. From a synthesis point of view, both systems have several drawbacks. In the case of the methacrylate systems, making the polymer is easy but some of the annelated monomers are difficult to purify and are expensive unless produced in large quantities. The cycloolefin type monomers (usually norbornene and derivatives) are easily prepared, usually in quantitative yields, by the Diels-Alder reaction using cyclopentadiene and acrylate monomers. Free radical polymerization is, however, only possible by alternating copolymerization with electron deficient monomers such as maleic anhydride, a reaction for which good yields and reproducibility are often critical. Direct vinyl

addition polymerization can be effected by means of metal catalysis. All of the systems that are presently being developed for commercial use fall into one of these three classes, or are hybrids thereof.

It has been pointed out by several groups that the performance of 193 nm polymers is yet inferior to that of 248 nm resist materials. A direct performance comparison is easily carried out since the 193 nm polymers are also transparent at 248 nm, and because the formulations commonly contain the same onium salt sensitizers used for 248 nm resists. In this paper, we use the normalized image log-slope (NILS) as a measure to determine the imaging capability of our 193 nm systems, and to compare them to that of state-of-the-art 248 nm systems.

In particular, we have studied the synthesis and lithographic properties of resist formulations based on a copolymer of 2-methyladamantylmethacrylate (2-MAdMA) with mevalonic lactone methacrylate (MLMA), and of a number of polymers derived from norbornene carboxylates and maleic anhydride.

2. Experimental

2.1 Synthesis of 2-MAdMA, MLMA and their copolymers

As shown in Scheme 1, 2-MAdMA was synthesized by the reaction of methacryloyl chloride and 2-methyladamantanol in presence of triethylamine base at -40 °C. It was preferentially purified by distillation under reduced pressure (105 °C/ <1 mm Hg). MLMA was prepared by reacting methacryloyl chloride and mevalonic lactone. This monomer can also be purified either by distillation under reduced pressure, however, great care is necessary during purification of this monomer as it is prone to elimination reaction under the reaction

conditions. Polymerizations were carried out in THF using AIBN initiator at 70 °C for over 5 hours under nitrogen atmosphere. Typical polymerization yields were above 65 %.

2.2 Polymer characterization

Polymer structures were confirmed by both IR and NMR. Molecular weights were determined on gel-permeation chromatography (GPC) using polystyrene standard. Thermal studies were conducted by thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) or modulated temperature differential scanning calorimetry (MT/DSC) as described in the literature.⁷

2.2.1 Resist formulations, exposures and etch rate measurements

The AZ[®]EXP AXTM1000P resist is based on a close to 1:1 MAdMA/MLMA copolymer. This formulation was chosen for commercial sampling activities and is available in gallon quantities. Resist formulations were made in ethyl lactate solution of the respective polymer, onium type photoacid generator (about 2 w/w% solids), and a small amount of base additive was included. Exposures were carried out on an ISI

0.60 NA mini stepper at International SEMATECH, Austin/Texas. Resists were coated either on bare silicon wafers or on wafers coated with 39 nm of AZ EXP ArF-1 193 nm bottom coat (baked at 175°C for 60 seconds) on a FSI Polaris coating track. The wafers were soft baked at 115°C for 60 seconds on a hot plate. Resist thickness was determined to be 0.451 μm on a Prometrix film thickness measurement tool. After exposure, the wafers were post exposure baked at 110°C for 60 seconds and developed in a 60 sec spray/puddle process with LD26W (2.38 wt% aqueous TMAH) developer at 23°C using a short pre-wet cycle. The cross-sections of the developed patterns were observed by scanning electron microscopy. The blanket polysilicon etch rate measurements were done on a LAM 9400 machine at 300W/150W/10mT/50sccm Cl_2 /150 sccm HBr/65 °C.

3. Performance of 193 nm Resists

3.1 Lithographic Properties of AZ[®]EXP AX-1000P Resist

On the SEMATECH ISI 193 nm ministepper system (NA=0.60, $\sigma = 0.70$), the AZ[®]EXP AXTM1000P resist shows high resolution up to

Scheme 1. Synthesis of 2-MAdMA, MLMA and poly(2-MAdMA-co-MLMA)

Figure 1 (right): Dense line performance of AZ®EXP AX™1000P (binary mask, standard illumination; 0.451 μm film thickness (E_{max}), 17 mJ/cm^2)

Figure 3. Dense line performance of AZ®EXP AX™1000P using a strong phase shift mask ($NA = 0.60$, $\sigma = 0.30$)

0.13 μm (Figure 1) for dense lines with a conventional chrome-on-glass mask. The depth of focus for 130 nm dense lines is about 0.40 μm , that for 150 nm dense lines about 0.50 μm (Figure 2). The sensitivity of the resist (dose to print) under these conditions is 13 mJ/cm^2 on a bottom A.R.C. Using a strong phase shift mask and $\sigma = 0.30$, one obtains dense line resolution

Figure 2: Depth of focus for 0.15 μm L/S in AZ®EXP AX™1000P resist (binary mask, standard illumination).

Figure 4. Depth of focus for 0.15 μm L/S of AZ®EXP AX™1000P using phase shift mask

down to 0.10 μm (Fig. 3), which was the smallest dense feature on the mask, and a depth of focus of 1.0 μm for the 0.15 μm dense lines (Fig. 4). One of the resist's characteristic features is a low iso-dense line bias, which is believed to be reduced below the intrinsic bias of the aerial image by a near-optimal diffusivity of the catalytic acid. The resist also shows good performance for contact hole patterns (Fig. 5), and has high thermal stability of up to 135 $^{\circ}\text{C}$ (Fig 6). The polysilicon etch rate using Cl_2/HBr etchant was determined to be 1.4 times to that of novolak. One issue that is still being addressed with this type of formulation is the adhesion at high aspect ratios on silicon substrates which currently limits resolution to 0.15 to 0.14 μm in about half-micron thick films.

When investigating the thermal flow stability of developed AZ®EXP AX™1000P resist structures, no flow of the resist features was observed for heating temperatures as high as

Figure 5: Dense contact hole performance of AZ®EXP AX-1000P (binary mask, 26 mJ/cm²)

Figure 6: Thermal flow stability of AZ®EXP AX™1000P

135 °C (Fig. 6). The observed thermal flow temperature was thus higher than the T_g of 105 °C previously quoted in the literature.⁷ It was therefore concluded that due to the difficulty of the experiment, the earlier measurement must have been in error, and that the T_g of the photoresist must substantially exceed that temperature.

The determination of glass transition temperatures of stiff polymers such as those used in today's 193 nm resist systems is very difficult with standard DSC methods. Acrylic backbones with large pendant side groups exhibit only small heat capacity changes during the glass transition, which moreover may stretch out over a larger than normal temperature range. Very often, the glass transition temperatures of these polymers cannot be determined due to a lack of sensitivity of standard Differential Scanning

Calorimetry (DSC), or, in some cases, because the transition is convoluted with chemical changes in the polymer or photoresist. We have recently reported⁷ on the use of Modulated Temperature Differential Scanning Calorimetry (MT-DSC) and other methods to overcome these difficulties. As a result of these studies, it was found that the glass transition temperature of the 2-MadMA/MLMA 1:1 copolymer was about 155 °C, and that the glass transition temperature of AZ®EXP AX™1000P is as high as 150 °C (Figs. 7 and 8).

While these results are in full confirmation of the thermal flow experiment of Fig. 6, they also indicate that contrary to what had been previously thought, the AZ®EXP AX™1000P

Fig. 7: Glass transition and decomposition temperatures for 2-MadMA/MLMA copolymers.⁷

Fig. 8: Glass transition and decomposition temperatures of AZ®EXP AX™1000P resist.⁷

resist is not annealed under the present softbake conditions (115 °C, 1 min). As a result of this, one would expect to find sensitivity of the resist to atmospheric contamination. Delay time studies show that this is indeed the case: after 10 min in a filtered environment (ca. 3 - 5 ppb base contamination level), the resist begins to show signs of T-topping, which has become severe after 30 min (Fig. 9, A); after 1 hour, the resist fails completely.

As one of the approaches we are pursuing to improve the delay time stability of this type of resist, we have investigated the admixture of an additive based on a 193 nm-adapted version of

Fig. 9 (right): Delay time stability of 2-MAdMA/MLMA-based resist with 193 nm version of photobase additive for 140 nm dense lines. A) Additive level 0X (12 mJ/cm²) = AZ®EXP AX™1000P (contains non-photobase additive), B) additive level 1X (14 mJ/cm²), C) additive level 2X (28 mJ/cm²). Filtered environment with 3-5 ppb airborne base. Except for the SEM in the lower left corner, all experiments were carried out on the same day.

AZ's patented photobase technology. Three levels of additive were studied: at the 1X level, the photospeed was approximately equal to unmodified AZ®EXP AX™1000P (14 mJ/cm²), at the 2X level, it had approximately doubled (28 mJ/cm²), and at the 3X level, the photospeed had become too low for a practically useful resist (>45 mJ/cm² as extrapolated from the dose-to-clear of 18 mJ/cm²). Delay time studies for the 1X and 2X levels showed that there was no T-top formation for a period of up to 1 hour in filtered atmosphere (Fig. 9: B, C). The formulation at the 1X level was used as the basis for the new, delay time stable AZ®EXP AX™1010P resist system.

Fig. 10: Dependence of linewidth on PEB temperature for AZ®EXP AX™1000P. The reduced PEB dependence seen is a result of the lowered activation energy of deprotection.

As a result of the presence of beta substituents in 2-methyl-2-adamantanol and of the conjugation of the double bond and carbonyl group in the anhydromevalolactone cleavage product (cf. Scheme 1), the MAdMA/ MLMA resist system exhibits a lowered activation energy of the deprotection reaction. Fig. 10 shows the dependence of the linewidth of AZ®EXP AX™1000P resist on PEB temperature for 140 to 160 nm features. Independent of the feature size, a slope of about 5 nm/°C is observed, which is about half of what is seen for t-butyl leaving groups.

3.2 Lithographic Properties of Cycloolefin Resist Systems

Resist systems based on cycloolefin polymers are attractive because of the easy accessibility and potentially low cost of raw materials and

because of their high etch resistance, particularly in fluorine plasma.⁴⁻⁶ Systems based on cycloolefin/maleic anhydride alternating copolymers are particularly attractive because they can be synthesized by standard free radical polymerization. One such system which has demonstrated high resolution and good adhesion on silicon is based on a tetrapolymer of butyl norbornecarboxylate (BNC), hydroxyethyl norbornecarboxylate (HNC), norbornene-carboxylic acid (NC), and maleic anhydride.⁴

Resists based on the above polymer have been reported to resolve dense line features down to 130 nm with standard illumination.⁴ Figure 10 shows the performance for dense line features in a non-optimized resist formulation containing BNC/HNC/NC/MA tetrapolymer. The resist was processed on B.A.R.C coated substrate using a film thickness of 451 nm, a soft bake of 115°C/60sec, then exposed on the ISI 0.6 NA ministepper at $\sigma = 0.7$, baked at 110°C/60sec and developed using 2.38 wt% aq. TMAH. In the present non-optimized form, the resist resolved 0.15 μm dense lines with reasonable wall angle and film retention. The poly-silicon etch rate using Cl_2/HBr etch chemistry of this resist is 1.35 times that of novolak resist as measured on a LAM 9400 etcher, and the resistance to fluorine etch chemistries has been reported as 1.00 for CHF_3 and 1.07 for SF_6 based etches.⁴ Further optimization work of this type of resist formulation is on-going, and the results will be reported elsewhere.

Scheme 2: Synthesis of BNC/HNC/NC/MA tetrapolymer

Figure 11: Dense line performance of a non-optimized cycloolefin type resist based on BNC/HNC/NC/MA tetrapolymer

4. Performance Characterization of 193 nm Photoresists

Photoresists for 193 nm lithography have come a long way in a relatively short time, and today's materials are capable of meeting the requirements for early production. However, it has already been shown by other studies⁸ that 193 nm resists still lag other chemically amplified systems in imaging performance. Our investigations confirm that this is the case: when imaged at 248 nm, even the best current 193 nm

resists, which resolve 130 nm dense features with $NA=0.60$ and $\sigma=0.70$, fall short of the performance of state-of-the-art 248 nm resists.

For the 248 nm experiments, the AZ[®]EXP AX[™]1000P formulation shown in Figs. 1-6 was modified by increasing the amount of PAG in order to arrive at a reasonable photospeed at 248 nm (Fig. 12a, 40 mJ/cm²). This modification was necessary since the PAG absorption at 248 nm is much lower than that at 193 nm. The modified formulation was imaged at 248 nm

Fig. 12a: Performance of a modified 193 nm resist in 248 nm exposure ($NA=0.55$, $\sigma=0.55$).

Fig. 12b: Performance of AZ[®]EXP DX2549 DUV resist with limiting resolution of 150 nm dense lines ($NA=0.55$, $\sigma=0.8$)

Fig. 13: Definition of the Normalized Image Log-Slope (NILS).

with a stepper set at $NA=0.55$ and $\sigma=0.55$. The resulting images showed resolution down to about 190 nm dense lines (Fig. 12a). Under the same stepper settings, the most advanced 248 nm resists will show resolutions of down to 160-170 nm dense lines under the same stepper settings. With higher NA and/or sigma values, dense line resolution of 150 or better are possible (Fig. 12b).

In order to be able to compare the resolving power of resists under different settings of the optical exposure tools, we have employed the normalized image log-slope (NILS) as a measure of the quality of the aerial image that the resist can still resolve. For line features, the NILS is defined as the product of the feature size times the derivative of the natural logarithm of the light intensity in the direction perpendicular to the line, evaluated at the mask edge (see Fig. 13).

The NILS is generally recognized as a good measure of the aerial image quality.⁹ In order to use it for resist performance evaluations, one has to determine what NILS value of the aerial image a resist can still resolve. The lower the NILS value the better the resist. A poor resist system might require a NILS of 3 or 4 to resolve the aerial image, while state-of-the-art resists can work with much lower NILS, as will be shown below.

Fig. 12b shows the performance of the AZ[®]EXP DX 2549 resist system on a stepper with $NA=0.55$ and $\sigma=0.8$. Under these conditions, this resist shows a limiting resolution of 150 nm for dense line features. The 150 nm lines are only resolved in the center part of the 5-line dark field structure, i.e., the part that most closely corresponds to an infinite dense line array, which also is the area where aberrations such as coma will have the least impact. The calculation of the corresponding NILS value with the Prolith/2 simulator (Fig. 14) shows that for this sigma value, the resolution of 150 nm dense lines shows that the resist is capable of resolving aerial images with NILS values as low as about 0.7.

In contrast, the performance of the modified AZ[®]EXP AX[™]1000P formulation at 248 nm corresponds to a NILS value of about 1.1 (Fig. 15). For purposes of comparison, it shall be

Fig. 14: Contour diagrams of NILS values as a function of focus and partial coherence for 200, 180, and 150 nm dense lines ($NA=0.55$, 248 nm exposure wavelength).

Fig. 15: Contour diagrams of NILS values as a function of focus and partial coherence for 130, 120, and 110 nm dense lines (NA=0.60, 193 nm exposure wavelength).

Fig. 16: Contour diagrams of NILS values as a function of focus and partial coherence for 130, 120, and 110 nm dense lines (NA=0.70, 193 nm exposure wavelength).

pointed out that the performance of i-line resists, which have reached quarter-micron resolution after many decades of development, corresponds only to a NILS value of about 1.3.

The above study of the NILS values¹⁰ thus leads to the conclusion that 248 nm DUV resists currently are the most advanced and most highly resolving resists available, with 193 nm resists still trailing in performance but already occupying a middle position between i-line and 248 nm systems. The use of chemical amplification has enabled the resist chemists to leapfrog the long period of development of i-line resists in only a few years. The history of optical lithography has been one of life cycle extensions of existing technologies, pushing feature sizes into the deep sub-wavelength region. As it is to be anticipated that 193 nm

lithography will suffer a similar fate at the hands of 248 nm lithography, it is important to know where the performance limits of 193 nm lithography will lie. The performance of 193 nm resists is one of the most important factors that will determine for which technology nodes 193 nm lithography can still be employed.

One way to approach this problem is to ask what performance one would expect at a wavelength of 193 nm if the resist performance were equal to the highest demonstrated one. In other words, what resolution performance would we see in 193 nm lithography if 193 nm resists would be as good as the best current 248 nm resists? While it is not clear whether such performance can be achieved within the constraints of 193 nm resist design (e.g., the need to work with carboxylic acid groups in

Fig. 17: Contour diagrams of NILS values as a function of focus and partial coherence for 110, 90, and 80 nm isolated lines (NA=0.60 and 0.70, 193 nm exposure wavelength).

standard strength developers), the historical precedent of 248 nm resist development would lend confidence to such an expectation.

Figs. 15 and 16 show calculated contour plots of the NILS for dense lines as a function of focus and partial coherence for a number of NA values and feature sizes (binary mask, no optical enhancements). From these diagrams, one can conclude what lithographic improvements could be realized if 193 nm resists were to become able to use a NILS value of 0.7. For a stepper with NA = 0.60 and $\sigma = 0.70$, the resolution capability would be improved by only 10 nm; however, the DOF for the 130 nm features would be increase to more than 0.7 μm . A partial coherence increase to 0.8 would make it possible to barely open the 100 nm lines. If one assumes that scanners with NA=0.70 become available, the NILS=0.7 cutoff would move to the 100 nm dense line range (Fig. 16).

For isolated lines, the current 193 nm resist resolution is about 110 nm at NA=0.60 and $\sigma=0.7$, which again corroborates a NILS limit of 1.1 (Fig. 17). If the required NILS were 0.7, the isolated line resolution at this stepper setting would increase to 90 nm, and to 80 nm if a stepper with NA=0.7 was used. It should be noted that the optimum partial coherence for the isolated lines ($\sigma=0.5$ to 0.6) is lower than for the dense lines.

5. Conclusions

Current 193 nm resists are capable of 130 nm resolution with binary masks and standard illumination on the first generation of exposure tools (NA=0.60). The 2-methyl-2-adamantane methacrylate/ mevalonic lactone methacrylate-based AZ[®] EXP AX[™]1000P resist shows good isolated and dense line performance, i.e., linear resolution down to 0.13 μm with a binary and

less than 0.10 μm with a strong phase-shift mask. Contact hole performance is 150 nm with a binary mask. The resist is not annealed under the present processing conditions and consequently shows delay time issues. With the addition of a new additive related to AZ's 248 nm photobase technology, resists can be formulated for which the delay time in filtered environments exceeds one hour.

Calculation of the NILS values shows that if resist performance can be improved to the level currently exhibited by 248 nm resists (i.e., a required minimum NILS of 0.7), 120 nm dense and 90 nm isolated lines can be opened. If for future stepper systems, the NA is additionally increased to 0.7, the resolution limit of 193 nm lithography without optical enhancements is predicted to lie at 100 nm dense lines and 80 nm isolated lines. However, a robust production process requires higher latitude than can be obtained at the final resolution of a resist. The 100 nm dense line technology node of the year 2004/2005 will therefore have to use optical enhancements for 193 nm lithography, unless the performance of 193 nm resists can be pushed beyond that of today's 248 nm materials.

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