Design Consideration for Immersion 193: Embedded Barrier Layer and Pattern Collapse Margin

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In immersion lithography the optical path between the lens element and the photoresist is currently water. Defects have been identified as a major roadblock for the introduction of immersion lithography to real device manufacturing. To address these immersion specific defect issues we have developed a novel additive approach for controlling the resist surface and its interaction with water. These additives have been designed to segregate within the resist film and migrate to the resist surface. Due to the high local concentration of these additives at the resist/water interface they create a very hydrophobic surface and allow control of surface properties. Data will be presented on this novel concept, illustrating the control of leaching and resist surface hydrophobicity. The use of this new technique allows control of leaching, resist surface contact angles and immersion specific defects.

As critical dimensions (CDs) in the semiconductor industry shrink, pattern collapse becomes a problem. We classify pattern collapse into three classes: adhesion failure, pattern strength failure and pattern film loss failure, and relate these to the capillary forces which drive failure. In this paper, the correlation of PCM with Ψ (carbon heteroatom ratio) of polymer composition, resist profile, PEB temperature and time are discussed. Finally, mechanism understanding of pattern failure and subsequent correlations gives us the tools to design advanced resist with excellent PCM.

Keywords: immersion lithography, top coat-free, EBL, pattern collapse

1. Introduction

Extending ArF lithography to the 45nm node and beyond challenges both tool makers and photoresist suppliers alike. As critical feature sizes become smaller, the physical and chemical forces resulting from the use of advanced lithography tools and processes fundamentally change photoresist design requirements. Immersion technology will be necessary to resolve sub 50nm features with acceptable process window. In immersion lithography increasing the refractive index of the medium between the photoresist and the projection lens enhances the resolution of the optical system.

The use of a liquid between the lens and the photoresist in immersion lithography requires precise control of the resist surface. Two key considerations are critical for immersion photoresist design: preventing leaching of resist components into the immersion liquid, and controlling the contact angles of the liquid with the resist surface. Contact angle control is considered to be the most viable route to minimizing immersion specific defects. In order to develop advanced materials for immersion applications it is necessary to develop a fundamental understanding of how resist design impacts the liquid-resist interactions.

To address immersion specific photoresist issues we have developed a novel additive approach for controlling the resist surface and its interaction with water. We took the novel approach of

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chemically designing an internal self-assembling barrier material that creates a thin embedded layer which functions as a topcoat. These additives have been designed to segregate within the resist film and migrate to the resist surface. Due to the high local concentration of these additives at the resist/water interface, they create a very hydrophobic surface and allow control of surface properties. In addition, this invention makes it very easy to convert existing dry 193 resist chemical approaches into an immersion 193 resist by simply adding the self-assembling barrier material as a trace additive to the original photoresist formulation. In this paper data will be presented on this novel self assembly concept, illustrating the control of leaching and immersion specific defects.

Another key aspect of photoresist design for resolving sub 50nm features is to adapt the photoresist to the greater capillary forces during the development process and subsequent drying process. Capillary forces are the primary cause of pattern collapse limitations observed by researchers in immersion lithography, Extreme Ultra Violet (EUV) and Electron Beam lithography. Developing a mechanistic understanding of why pattern collapse happens and what controls the pattern collapse phenomena is vital to improving resist performance in light of increasing capillary forces.

We have investigated the mechanisms of pattern collapse/failure and subsequently linked the mechanism to polymer and pattern profile differences. Understanding the fundamental reasons for pattern failure has allowed us to develop a platform of resist formulations, called EPICEB, which can withstand the capillary forces necessary to image 45nm and below, in addition to excellent lithographic performance and minimal line width roughness (LWR).

2. Experimental
2.1. Polymer Synthesis
Free radical polymerizations were carried out using standard techniques. Many reactions were run to optimize the choice of monomers and their ratios in order to maximize the desired properties.

2.2. Contact angle measurements
Contact angle measurements were performed using a Kruss DSA100 with tilt table. Static contact angles were measured with a 2 microliter droplet, the reported number was an average of 3-5 test points. The advancing and receding contact angles are measured using a 50 micro-liter droplet. A video recording was made of the droplet as the table tilts down at a rate of 1mm/ second. The angles were measured at 1 frame prior to the droplet starting to move across the wafer. The tilt angle is recorded as the angle at which the droplet first starts to move across the wafer. The developer contact angles were measured using a 2 micro-liter droplet of CD-26 aqueous base developer.

2.3. Calculations
Collapse force calculations were performed on a theoretical resist with 35nm lines and 70nm pitch with a film thickness of 120nm, resulting in an aspect ratio of 3.43. Other required values are: theoretical resist water surface tension (72dyne/cm) and polymer modulus (3GPa).

2.4. Lithography
ArF photoresists were made of polymethacrylates, a sulfonium photoacid generator (PAG), a nitrogen based quencher and a surface leveling agent, all dissolved in a blend of PGMEA and cyclohexanone solvents.

Photoresists were coated to 1500Å film thickness (post-apply bake, PAB: 100°C/60sec) on top of an organic BARC (AR77®/ 840Å/205°C/60sec). The photoresists were exposed using a 193 phototool (ASML 1100/ 0.75NA using dipole illumination with 6% ATTPSM or alternatively the IMET/ 1.3NA). The resists were then given a post-exposure bake (PEB: 90-110°C/60sec) and developed with a surfactant-containing developer (MF26A), linewidth measurements were done using top-down SEM analysis on Hitachi 9300.

To minimize adhesion failures, AR77® was used through out this study. This organic BARC is well known to be particularly good for prevention of pattern collapse from a delamination type mechanism.

3. Immersion EBL
3.1. Immersion Polymer Design
In an immersion scanner, water is placed between the lens and the wafer. As the lens moves across the wafer two key contact angles, the advancing and receding, between the water and the resist film surface must be controlled. The leading edge forms the advancing contact angle and the back forms the receding contact angle. Optimization of each of these angles is important in order to minimize the defects that may be formed during the lithographic process.
If the advancing angle becomes too high, air can be trapped under the advancing water to form bubbles, which may adhere to the resist film surface. The bubbles can subsequently diffract the stepper’s light and deform the feature shape. This diffraction can lead to “bubble defects” in the lithography, shown in Fig. 1.

Figure 1: Bubble Formation

If the receding contact angle is too low, droplets of water can be pulled off of the receding water film edge, and be left on the wafer. These small droplets dry and can cause a circular defect shown in Fig. 2. The exact mechanism of why this droplet causes this type of defect is still a topic of debate.

Figure 2: Droplet formation

We have discovered a novel method to control the resist’s surface energy and thus contact angles which involves use of a polymeric additive that is designed to phase separate from the photoresist to form a barrier at the air/resist interface (first presented at the 25th IEEE Workshop on Lithography, August 3, 2006)\(^7\). This barrier layer, which we call “embedded barrier layer” (EBL) can be designed to have a wide variety of contact angles and has been shown to decrease photoacid generator (PAG) leaching below required specifications.

We expect that the EBL technique will become the key innovation in the practical application of 193 immersion photoresist technology. Full details are available in the 2006 published patent.\(^8\)

In designing the EBL, our copolymer design is as shown below. The surface modifying monomer is responsible for bringing the polymer additive to the air/resist interface. The surface modifying group (SMG) of this polymer is most commonly a perfluorinated alkane chain. The second monomer is a photo-generated acid labile group or leaving group (LG). Upon acid cleavage, the remaining carboxylic acid will allow the exposed areas to have a lower developer contact angle in addition to the EBL becoming aqueous base soluble.

![EBL Copolymer Design](image)

**EBL Copolymer Design**

To examine the effect of the surface modifying group on the resist’s surface properties we synthesized a series of polymers. Each polymer in the series contained an increasing amount the SMG monomer. Each of these polymers was formulated into a standard ArF photoresist. The EBL containing photoresists were then coated onto 200 mm Si wafer and baked at 110°C for 60 seconds, cooled for 30 seconds, and contact angles measured.

The contact angle measurement results are summarized in Table 1. Polymer A did not change the surface angles because it only contained LG monomer without any surface modifying monomer. These angles matched our standard ArF resist without any additive. As the percent SMG increased, from polymer B to F, the static contact angle increased. These contact angle trends correspond with an increase in hydrophobicity of the surface, consistent with additional fluorine in the polymer.

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<td>F</td>
<td>100.0</td>
<td>16.2</td>
<td>99.9</td>
<td>85.6</td>
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Table 1: Summary of EBL variations and changing Static angle, Tilt angle, Advancing angle (adv), and Receding angle (rec)
The static angles can also be adjusted by changing the amount of EBL added to the photoresist. Shown in Figs. 3 and 4, the hydrophobicity of resist surface increases rapidly as the surface becomes saturated with the EBL material. After this saturation point there is only a slight increase in static contact angle with increased addition of the EBL additive.

Figure 3. Advancing contact angle versus concentration of SMG in EBL additive polymer.

Figure 4: Static contact angle versus EBL material loading

The relationship between receding, and tilting contact angles are not always linear, but are somehow related. Fig. 5 shows how, in one case, these angles are related. The non linear response to the increasing of SMG monomer is still under investigation. However from this data the selection of the correct monomers, ratios and loading of the EBL polymer can be used to tune the contact angles to the desired values to minimize film defectively in immersion lithography.

A structure property relationship was developed for our EBL copolymers. We found that the surface properties of an EBL can be modified by adjusting the fluorinated surface modifying monomer and the leaving group. The contact angles of a photoresist can be adjusted by using embedded barrier layers of different compositions, or by adjusting the loading of the EBL materials. These materials allow for practical topcoat free immersions resists.

3.2. Switching Contact Angles

Exposed regions

With the increased hydrophobicity of the resist films, one concern is the possibility that defects generated by materials in the developer or the water rinse puddle might adhere to the resist surface. In order to address these concerns we varied the nature and concentration of the leaving group monomer within the EBL, to change surface hydrophobicity, and thus cause changes in developer contact angles between the initial film and after exposure and PEB. Prior to exposure a resist loaded with optimized EBL copolymer has contact angles of 89.0º with water and 88.7º with 0.26N developer. Upon exposure without a PEB, the contact angles have only a slight change in contact angle to 87.6º water and 87.5º in developer. The contact angles with 0.26N developer remains close to the water contact angles showing that there is no deprotection during exposure. (It should be noted that there was a half hour delay between the exposure and the measure of the contact angles.) After a 90ºC/60s PEB, the contact angles changes to 59º with water and 24.2º with developer. The deprotection of the leaving group on both the EBL and in the base polymer cause a drop in the contact angles as shown in Table 2. This deprotection also allows the EBL material to be soluble in the developer solution.

The large change in contact angle is believed to occur because the EBL deprotection occurs during the bake step and not during the exposure step. This change in EBL hydrophilicity on exposure is
helps provide smooth dissolution of exposed resist in standard alkaline developers.

<table>
<thead>
<tr>
<th>Table 2: Contact Angle of Resist + EBL</th>
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<tr>
<td></td>
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<tr>
<td>water</td>
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<tr>
<td>developer</td>
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Unexposed Regions:
A third monomer was added to the EBL polymer system to make an EBL terpolymer. This new polymer reacts with the developer to lower the contact angle of the resist in the unexposed regions during development. This is achieved by reacting a polymeric functional group with the developer to form acidic functionality at the interface. A model polymeric EBL compound was first synthesized using an already acidic monomer, to see the effect of the reacted material on resist contact angles. This chemical approach is similar to the chemical design of the switchable BARC materials we previously disclosed in 2006. The model acidic polymer when added to a 193 resist, gave a film surface with a static water contact angle of 80.2° and static developer contact angle of 72.1°. With the model compound confirming acidic functionality could provide lower contact angles, two different EBL terpolymers were formulated into a 193 photoresist. Contact angles with water, developer, developer after a sixty second developer puddle, and water after the 60s develop where measured as summarized in Table 3.

<table>
<thead>
<tr>
<th>Table 3: Contact Angle Changes</th>
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<tbody>
<tr>
<td>poly</td>
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<tr>
<td>A</td>
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<td>B</td>
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Terpolymer EBL materials allow EBL materials to have lower contact angles with developer than with water. In the unexposed areas it is shown that a new developer reactive monomer allow for the developer contact angles to change significantly. Lowering these contact angles should allow for the developer to wet the surface better and provide smooth dissolution behavior. In addition, the lower water contact angle after the develop step should allow an improved post develop rinse. Optimizing all these EBL additive properties improve the defectivity of topcoat free 193nm immersion resists.

3.3. Defectivity
The EBL system described above was used in conjunction with a 193nm resist and tested for defects, on a ASML 1400i 193 nm immersion scanner with a scan speed of 500 mm/sec. Within the samples, the EBL polymer composition and its loading into 193 resist formulations was varied by design to yield a variety of contact angles. The results are summarized in Fig. 6, graphically relating the observed bubble defectivity to the advancing and receding angles.

![Figure 6. Contour plot showing observed bubble defectivity as a function of advancing and receding contact angles in a 193 resist containing various EBL additives](image)

Using this data to optimize the surface angles of an EBL-containing resist allows us to create low defect 193nm immersion resists. Defectivity results were verified using the optimized resist on a Nikon 193 nm immersion scanner with a 500 mm/sec scan speed, resulting in the defect type and count distribution shown in Fig. 7.

![Figure 7. Defect type and distribution for an optimized 193 immersion resist containing EBL](image)

3.4. Lithography with EBL Additives
193 immersion resist EPIC EB, containing EBL polymer, yielded high resolution 47nm
lithographic images, seen in Fig. 8. This resist showed a line edge roughness (LER, $3\sigma$) of 2.6 nm and a line width roughness (LWR, $3\sigma$) of 3.5 nm. The cross-sections showed good profiles with no scumming or footing as shown in Fig. 9.

Each of the 6 samples was coated onto 2 wafers and tested. Wafers 1 and 2 contain no EBL and the remaining samples increase in EBL loading. As the graph shows, even at the highest loading of EBL material there is no change in the bulk etch rate of the material.

4. Pattern Collapse
4.1. Profile Dependence on Pattern Collapse

One of the significant photoresist design challenges for next generation involves improving resistance to pattern collapse. Shrinking critical dimensions and limited etch budgets mean that aspect ratios must remain constant. As a result, the resist “footprint” for adhesion is decreasing, while capillary forces on the resists are increasing, a potent combination of factors which can induce pattern collapse. Two main factors affecting the capillary forces of water on the resist are: contact angle of water with the resist (hydrophilicity/phobicity of the resist) and the modulus of the resist (ability of the resist to bend under the force of the water). These two factors are different and will be addressed separately.

The capillary force ($F$) along the side of the resist covered with water can be described by the equation:

$$F = \frac{2sH}{R} \cos \theta \div w$$

Where $H$ is the resist thickness, $s$ the surface tension of the water-air interface, $R$ is the curvature of the water meniscus, $\theta$ the contact angle of the resist water interface and $w$ the spacewidth. Subsequently, the capillary force increases with the aspect ratio of the space and with the surface tension of the water.

The ramifications of these physical relationships become apparent when considering the capillary forces on resists with changing profiles. Shown in Fig. 11 is a series of resists with a range of side wall angle from tapered to reentrant. The lines in image $a$ will have a weaker capillary force acting on the lines because $w$ is bigger, alternatively the lines in image $c$ will have a greater capillary force acting on the lines because $w$ is smaller. These
trends are observed both theoretically and experimentally, as shown in Figs. 12 and 13. Fig. 12 shows the calculated increase in capillary force on the resist lines as the wall angle changes from 80° to 90°. Experimentally the same trend is observed; Fig. 13 is a graph of PCM (pattern collapse margin, the linewidth of the last line standing at over exposure) versus measured side wall angle. When the side wall angle is 81° the PCM is 47nm but as the profile approaches 90° the PCM drops to 60nm. The straighter the wall angle the more difficult it is to achieve good PCMs.

Fig. 12: Calculated capillary force versus resist wall angle

Figure 13: PCM versus experimental resist wall angle

4.2. Hydrophilicity Dependence on Pattern Collapse

The resist-water interface is another critical factor in pattern collapse. A hydrophilic resist will have the strongest capillary force based on the equation above. Theoretical calculations of capillary force versus contact angle, shown in Fig. 14, suggest that resist should favor more hydrophobic materials. The challenge lies in creating a suitably hydrophilic resist sidewall surface: after PEB, development and the creation of a surface of partially aqueous base soluble material, the contact angle drops significantly.

4.3. Mechanisms

Analysis of pattern collapse becomes complex because as the resist design changes the failure mechanism can also change. Characterization of pattern collapse led to the identification of three main mechanisms of pattern collapse/failure: mottling, line breakage and adhesion failure.

"Line mottling" is defined as the uneven dissolution of the resist, an example is show in Fig. 15-a. This is normally characterized by the tops of the lines dissolving away unevenly, especially with overexposure. This eventually leads to the entire line dissolving away, as opposed to the line breaking or lifting off the substrate. Mottling seems to be attributed to the increasing hydrophilicity of the resist with increasing dose.

Line breaking or modulus failure is due to the line breaking mid-way down the line, as shown in figure 15-b. The amount of force (F) required to make a line move can be estimated by:

\[ d = \frac{3}{2} \left( \frac{F}{E} \right) \left( \frac{H}{w} \right)^{\frac{3}{5}} \]

where \( d \) is the amount of movement or sway and \( E \) is the Young’s modulus for the polymer. \( T^{10} \) From this equation, the importance of aspect ratio is evident due to the \((H/w)\) term being cubed. High
glass transition temperature of the polymer can be correlated with Young’s modulus.¹

Adhesion failure, shown in Fig. 15-c, is the delamination of the line from the substrate. This can be due to a number of factors: a poor matching of the surface energy between the substrate and the resist, poor interfacial bonding between resist, a pinched or undercut profile, or strong acidity of the substrate.¹¹, polymer stiffness or a high glass transition

4.4. Ψ Effect on Pattern Collapse Margin (PCM)

Polymer composition has been characterized by a new factor here defined as Ψ (Greek psi, carbon heteroatom ratio). By systematically changing the composition of a series of polymers it was possible to adjust the carbon to heteroatom (nitrogen, oxygen, sulfur or halogen) ratio. A smaller value of Ψ means that there are an increasing number of heteroatoms compared to carbon, alternatively a larger Ψ value represents a decreasing number of heteroatoms compared to carbon. Calculation of Ψ can be summarized by:

Ψ = (# Carbons/ # Heteroatoms)

In Fig. 16 the PCM versus Ψ is shown. A very clear trend is apparent, by increasing the Ψ the PCM worsens, in other words the profiles collapse at larger linewidths with fewer heteroatoms in the polymer. Alternatively, by increasing the number of heteroatoms, described by an increase in the Ψ, it is possible to improve the PCM. This trend was observed in two different polymer systems, shown in Figs. 16 and 17. The trend is consistent: decreasing the Ψ improves the PCM.

Figure 16: PCM versus Ψ for 193 resist polymer A

Figure 17: PCM versus Ψ for 193 resist polymer B

Considering two polymer systems, one could assume that the PCM is correlated with the contact angle, as described in the capillary force equation. However, when the contact angles were measured for these resists there was very little difference between the samples. Therefore it appears that the direct cause of the failure is something other than the changes in surface tension force. Fig. 18 shows the cross-section images of resists with a range of Ψ values at their collapse dose. There is an observable difference in the resist profile shape at collapse, for example the resist with a Ψ of 7.47 has very square lines and the collapse mechanism is line breaking. As the Ψ decreases, the side wall

Figure 18: □ and pattern collapse mechanism changes
angle of the resists becomes more and more tapered, the mechanism of collapse begins to change from line breaking to mottling. The resist with a low $\Psi$ is very mottled at collapse and most of the top of the line has dissolved away.

As was shown in Fig. 12, the capillary forces on a line with a 90° wall angle are significantly higher than on a line with 80° wall angle. Fig. 18 shows the more square profile has a worse PCM and the more tapered profile has a better PCM. Alternatively, the more hydrophilic the resist the greater the capillary force that acts upon the lines. The PCM for the resist with a $\Psi = 7.47$ is much worse than a resist with a $\Psi = 3.57$, going from a collapse linewidth of 57.7nm to 32nm respectively.

Most likely the PCM differences are due to the change in profile/wall angle with $\Psi$ as opposed to contact angle changes and subsequent capillary force changes. The profile changes can be attributed to the $\Psi$ variations.

4.5. Lithographic Processing Conditions

Thermal processing conditions are important factors for pattern collapse. Post exposure bake (PEB) temperature and time are important factors affecting the interfacial bonding of the resist with the substrate and the amount of residual leaving group in the film. There are limitations around the range of PEBs that can be used, which are determined for the individual resists, but there is a range of appropriate PEB temperatures and times for each resist. This work analyzes the relationship between PEB temperature and time with pattern collapse. For this study, seven polymers were tested in the same formulation and the PEB conditions varied, the results are summarized in Table 4. Polymer 1 was tested at a PEB of 105°C with two different PEB times, by increasing the PEB time from 60 to 90 seconds the PCM was improved 4nm. Polymers 2 to 7 were compared at 5°C variations in PEB temperature. In every case, increasing the PEB 5°C resulted in improved PCM. At higher PEB temperatures, the 5°C difference had more impact on PCM shown with polymer 4 and 5 with an 8nm improvement in PCM. At lower PEB temperatures, the 5°C had less impact on PCM as seen with polymer 7, when baked at 90/95°C the PCM only improved 1nm. Similar trends have been seen regarding PAB (post-apply bake) studies and the film strength with various bake temperatures and times.12 PEB conditions can not be changed infinitely but this trend does offer some opportunity to improve PCM with very small process condition changes. Another important factor in PEB conditions is the effect on pattern collapse mechanism, which could be one of the factors controlling the PCM. From a resist design perspective, a higher PEB process will allow greater control of the PCM with a smaller change in temperature. The influence of PEB times and temperatures allows fundamental understanding of the importance of resist substrate interaction and how to improve PCM.

Table 4: Thermal Conditions and PCM

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<th>Polymers</th>
<th>PEB (°C/sec)</th>
<th>PCM (nm)</th>
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4.6. Improved PCM Platform

By developing fundamental understanding of pattern collapse and the mechanisms behind pattern collapse, a new polymer platform has been developed. This new platform is capable of maintaining high aspect ratios, without mottling or collapse, at very small linewidths. This performance is necessary for hyper-NA applications where resolution down to 40nm 1:1 features is required. Shown in Fig. 19 are images of dense lines through dose, where lines remain standing down to 33nm at 100nm pitch. This is one example of significant improvement in pattern collapse margin.

5. Conclusions

Next generation photoresist design relies on developing mechanistic understanding of the challenges and improving the resist based on the physical factors that control the performance. For top-coat free immersion lithography self assembling EBL are introduced and a systematic study of EBL and subsequent surface properties the key role of EBL loading and composition in controlling water contact angles. This work led to the development of new polymeric EBLs which have switchable contact angles and lower lithographic defects. Pattern collapse is another challenge for next generation lithography due to increasing capillary forces as the resist line dimensions decrease. Investigations into the mechanisms behind resist failure and the development of a new polymer characterization factor (Ψ) led to the creation of a new family of resists with improved pattern collapse margins.

References

1. For licensing inquiries regarding Rohm and Haas' EBL technology, contact R. Hemond at rhemond@rohmhass.com