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Directly patterned inorganic hardmask for EUV lithography

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ABSTRACT

This paper describes a metal oxide patternable hardmask designed for EUV lithography. The material has imaged 15-nm half-pitch by projection EUV exposure on the SEMATECH Berkeley MET, and 12-nm half-pitch by electron beam exposure. The platform is highly absorbing ($16 \mu\text{m}^{-1}$) and etch resistant ($>100:1$ for silicon). These properties enable resist film thickness to be reduced to 20nm, thereby reducing aspect ratio and susceptibility to pattern collapse. New materials and processes show a path to improved photospeed. This paper also presents data for on coating uniformity, metal-impurity content, outgassing, pattern transfer, and resist strip.

Keywords: Extreme Ultraviolet Lithography, EUVL, EUV resist, patternable hardmask, inorganic negative tone resist

1. INTRODUCTION

EUV lithography requires high-performance photoresists with 22-nm half-pitch resolution, and extendibility beyond 16 nm.¹ Researchers have enhanced the EUV imaging fidelity of polymer-based chemically amplified resists (CARs) through two very general strategies: a) reducing resist blur to improve resolution and linewidth roughness (LWR) while maintaining sensitivity; b) reducing pattern collapse by using very thin films with high surface adhesion and structural integrity. The effective blur of a CAR is primarily determined by acid diffusion and secondarily by the radius of gyration of the polymer, secondary electron scattering, and other mechanisms.^{2,3} Total resist blur is typically 12 nm or greater, but it has recently been reduced to ~ 10 nm in polymer-bound PAG systems.^{2,4} Photospeed improvements have focused on increased acid yield, although this gain can come at the expense of increased acid diffusion. Improvement in resolution requires a reduction in resist blur, while balancing the competing smoothing effect on LWR from blur. Pattern collapse can also be mitigated with thin resist films and small aspect-ratio features. For conventional materials, film thickness is bound by the constraints of etch resistance and EUV absorbance. Thin CARs dictate process window and complexity, necessitating the use of additional layers to support multistep pattern transfer. Absorbance determines the volume density of information content recorded in the film, a significant factor in the near-shot-noise-limited regime contemplated for EUVL at or below 20-nm half-pitch. Typical leading EUV resists have film thicknesses in the range of 40 to 45 nm.⁵

Looking ahead to the challenging future patterning requirements for semiconductor lithography, we consider here design principles for a sub-20-nm EUV imaging material. We highlight four key attributes of an integrated materials system:

- EUV absorption: efficient capture of incident EUV photons is central to maximizing the spatial information content extracted from the aerial image. Voxel (volume pixel) size scales inversely with the film's absorption coefficient. As shot noise becomes a factor in image fidelity, harnessing more photons directly, rather than through amplification processes becomes increasingly important. The volume density of direct exposure sites underpins the quality of sidewall formation during the transformation of a 2D aerial image into a 3D profile, especially when blur mechanisms are also minimized. Absorbance must be managed for appropriate penetration through the film and for producing a desired sidewall angle.
- Resist blur: effective total blur much smaller than the feature size is vital for reproducing the spatial fidelity of the aerial image. Specifically, the chemistry resulting in a change in solubility must happen within a short distance from the ionization point, i.e., where an incident EUV photon generates a photoelectron. This implies that the secondary electron mean free path should be short, and that any amplification mechanism should be restricted to a small volume. In addition, molecular building blocks much smaller than the critical dimension are beneficial for defining sharp transitions on the relevant distance scale, with appropriately low line roughness.

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- Quantum yield: to achieve a commercially viable photospeed, the captured EUV photons must be used efficiently. This efficiency comes from maximizing the secondary electron yield and within a controlled interaction volume, the impact of the electron-initiated chemical events that affect development.
- Etch resistance: resist films with high etch selectivity relax the aspect ratio required to perform pattern transfer within an acceptable process window. Since pattern collapse scales quadratically with feature aspect ratio, reducing aspect ratio is an important factor in mitigating pattern collapse.

Taken together, the ideal resist would have an appropriately high absorption with a sufficiently high etch resistance such that it could be made thin (aspect ratio <2) thereby avoiding pattern collapse. Such a resist would also have low blur and high quantum efficiency to achieve high RLS image fidelity. High EUV absorption, short secondary electron mean free path, and high etch resistance all imply the need for a dense film.

Inpria is developing inorganic photoresists for EUV lithography. In this paper, we describe Inpria's resist material in the context of the design principles put forth above. We present lithography results obtained with three different formulations, emphasizing process integration and initial data on coating uniformity, metal-impurity content, outgassing, pattern transfer, and resist strip.

2. METAL OXIDE RESIST PLATFORM

Inpria's resist materials are based on a fundamentally new approach that relies on the deposition of extremely high-quality amorphous oxide films from aqueous solution and efficient photon-induced network-forming reactions. These transition-metal amorphous oxide films are deposited with standard spin-coating equipment, yielding morphology typical of vacuum deposited films.^{6,7} Unlike standard polymer resists which can have surface roughness ~ 2 nm,⁸ Inpria's films have angstrom scale smoothness on the order of 0.2 nm RMS. For comparison, sol-gel deposition of metal oxides and inorganic films routinely produce films with roughness above 2 nm.^{9,10} Furthermore, metal oxides are an excellent etch mask, potentially eliminating one or more underlayers.

By combining the metal oxide precursors described above with a high-contrast solubility switch, the system is transformed into a directly patternable inorganic hardmask. We start with a metal oxide sulfate (MSOx) and introduce a peroxy complexing agent (see Figure 1). During spin coating and post-application bake, this agent hinders network formation by capping small inorganic clusters. During exposure, EUV photons lead to the generation of secondary electrons, which subsequently break peroxide bonds, thereby decomposing the polymerization inhibitor. Active metal sites are created that lead to crosslinking and condensation. Three-dimensional framework formation can therefore proceed, both immediately after the exposure and during a post-exposure bake. This large change in the size from small clusters to larger units results in well-defined differences in solubility and the resulting selective dissolution of unexposed regions in a base developer, e.g., TMAH. The resulting system functions as a negative-tone resist.

For the current generation of Inpria's EUV resists, we have selected hafnium as the metal because of its high EUV absorption cross section relative to many other metals. We have directly measured the electron density of these materials with X-Ray Reflectivity (XRR). This measurement is combined with compositional information from Electron Probe Micro Analysis (EPMA) to calculate a mass density of 4.7 g/cm^3 . This mass density and the compositional information are used to calculate EUV absorption from the Center for X-Ray Optics database,¹¹ which yields an absorption coefficient of $16 \text{ } \mu\text{m}^{-1}$. For comparison, conventional polyhydroxystyrene (PHS) CARs have an EUV absorption coefficient of approximately $4 \text{ } \mu\text{m}^{-1}$, so the MSOx materials are $\sim 4\text{X}$ more absorptive (see Table 1). This absorption enables the use of relatively thin films and efficient capture of a significant fraction of incident EUV photons.

Table 1. Comparison of absorption values

Resist	Density	Absorption (μm^{-1})
Typical PHS ¹²	1.19	4.1
Typical Inpria Resist	4.7	16

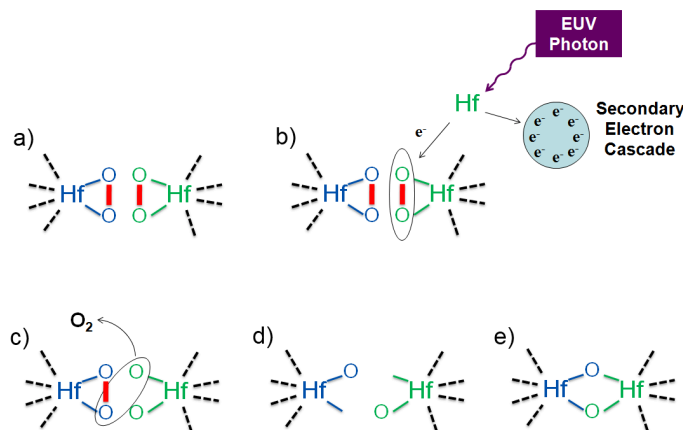


Figure 1. Diagram representing a simplified exposure mechanism in Inpria resist; a) During spin coating, polymerization inhibiting peroxo groups prevent further network formation. b) An EUV photon generates a photo electron which induces a secondary electron cascade. A secondary electron decomposes the peroxo group. c) An oxygen radical is generated, which when combined with another oxygen form a neighboring peroxo group, leaves the film as an oxygen molecule. d) Neighboring metal centers now have unsatisfied coordination sites. e) Three dimensional framework formation occurs as the coordination of the metal is satisfied.

Since the resist is entirely inorganic and dense, etch selectivity comparable to vapor-deposited hardmask materials can be realized. This selectivity provides a means for deep etching even with very thin films. We can therefore readily work with film thicknesses near 20 nm, thereby reducing stresses leading to pattern collapse by approximately 4X relative to a 40-nm film. Given the 4X higher absorption of the metal oxide, such a film would also still have approximately 2X the absorbance of a 40-nm PHS film.

The solubility switch for this resist system is not based on acid deprotection, so the system does not have the high diffusion blur associated with acid reactions in a conventional CAR. Instead, we believe the key residual diffusion is associated with secondary electron blur. Due to the higher density of the metal oxide we expect that the secondary electron blur is also somewhat reduced relative to an organic film. Low diffusion blur enables the system to have intrinsically very high resolution.

We note that with higher absorption, the incoming photons are collected 4X more efficiently by volume. This is aligned directly with the strategy for mitigating the impact of shot noise. Specifically, the resist should interact with as many photons as possible.

The MSOx resist is aqueous and contains no organic components (either as ligands, photoinitiators, or as casting solvent). The resist, therefore, does not outgas organic compounds, eliminating the issues of organic contamination under exposure. As described below, the resist does outgas water and oxygen, but at levels below the specifications for use in exposure tools.

3. LITHOGRAPHY RESULTS

Exposures were performed at the SEMATECH-Berkeley EUV MET (BMET) and also on a 30-keV electron beam tool. The resist was applied with standard spin-coat techniques on silicon substrates after a brief oxygen plasma treatment to render the surface hydrophilic. Film thicknesses were approximately 20 nm. Post-application and post-exposure bakes were in the range of 50 to 100 °C. Except where noted, development was performed in 25% w/w TMAH.

3.1 Resolution

The resolution capabilities of the BMET were tested by using the Pseudo Phase Shift Mask technique^{4,13} and Inpria's XE15AB resist. XE15AB and the associated process were designed to maximize resolution, providing improvement over the original results presented in 2010.¹³ Line/space patterns were clearly resolved to 15-nm half-pitch with patterning performance demonstrated to 13 nm (Figure 2). These results match well with the aerial-image predictions. The 16-nm

half-pitch has a CD of 13 nm and LER of 2.0 nm. Figure 3 shows 12-nm half-pitch features resolved in XE15AB by electron-beam lithography. These results demonstrate that ultimate resolution of the resist is below 12 nm.

Although the EUV exposure dose for XE15AB was 70 mJ/cm^2 – considerably higher than commercially viable – this particular formulation provides a means to evaluate the ultimate resolution of an EUV lithography system. It also provides a basis for reducing dose while maintaining high resolution.

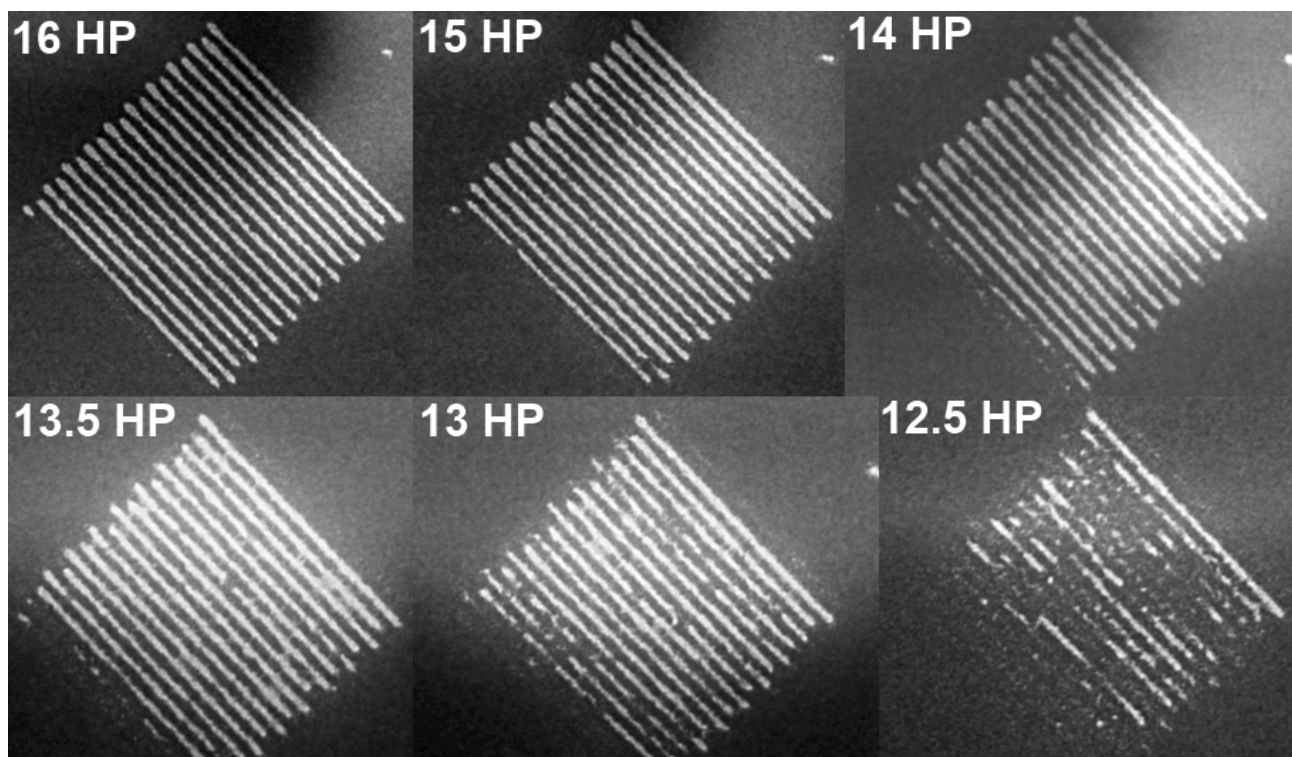


Figure 2. 16 nm HP – 12 nm HP lines produced by F2X mode on the SEMATECH-Berkeley MET with XE15AB resist at 70 mJ/cm^2 . The 16-nm HP lines printed at 13-nm CD, and they have a LER of 2.0 nm.

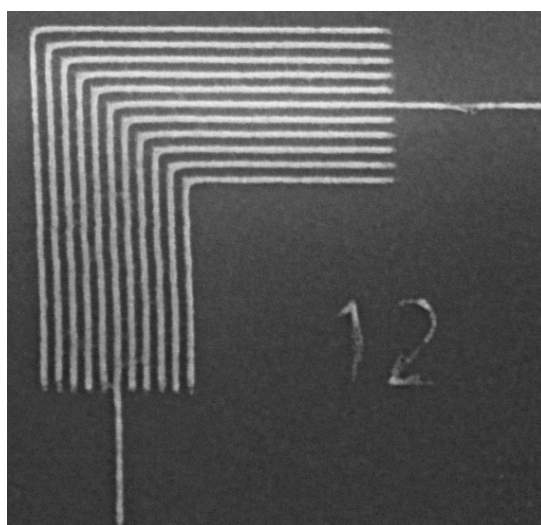


Figure 3. 12-nm half-pitch lines patterned with e-beam lithography, XE15AB resist at $930 \mu\text{C/cm}^2$ (30 keV).

3.2 Process Characterization

We have established a baseline process on the SEMATECH-Berkeley MET as a starting point for studying a wide range of formulation and process modifications. Formulation XE15CB was designed to improve sensitivity while maintaining high resolution. Annular illumination ($0.35 < \sigma < 0.55$) was used to simultaneously characterize the resist with 2D patterns and under conditions with low NILS. Process windows for 26 and 20 nm HP are shown in Figures 4 and 5. Greater than 10% exposure latitude was obtained down to 20-nm half-pitch. We expect to observe a process window improvement with more advanced illumination conditions.

A simple resist model was built in PROLITH and effectively used to predict performance of the material over the process window. Through examination of exposure latitude over a range of feature geometries, an initial upper bound of 7 nm has been estimated for resist blur. This initial result suggests that the material indeed has a low intrinsic diffusion.

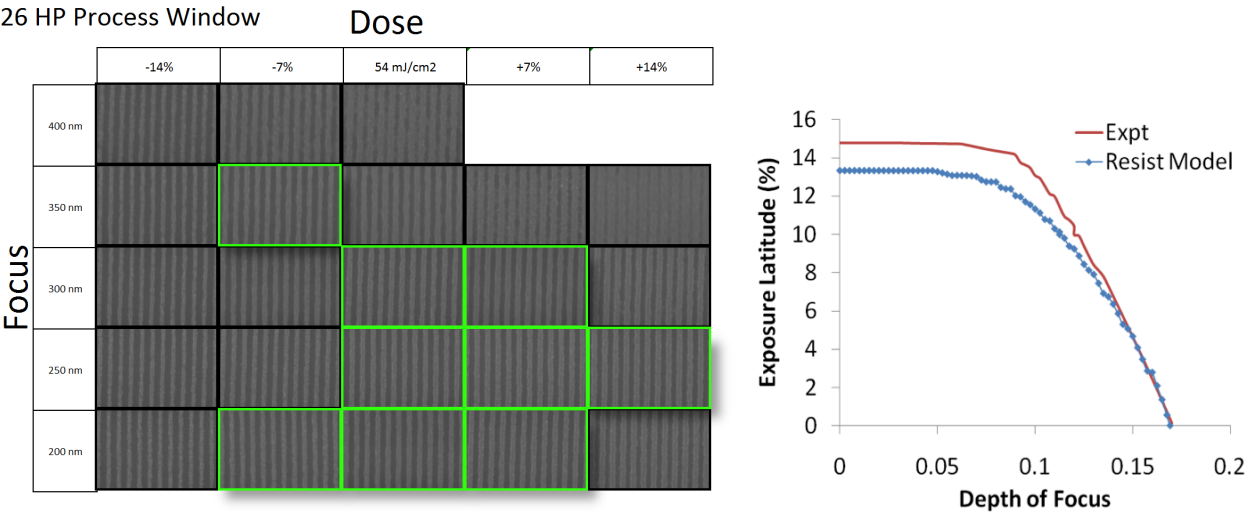


Figure 4. EL vs. DOF for 26 HP lines and resist model, annular illumination, SEMATECH-Berkeley MET

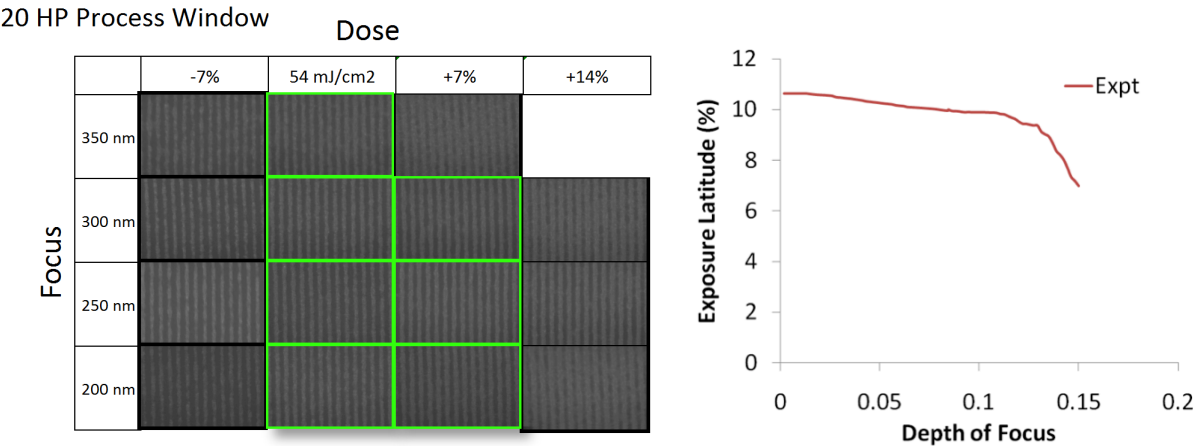


Figure 5. EL vs. DOF for 20 HP lines, annular illumination, SEMATECH-Berkeley MET

Systematic studies of process modifications have been initiated. An example of such a modification is shown in Figure 6. The baseline resist was developed with reduced concentration TMAH and a proprietary developer additive. This process modification resulted in a ~40% reduction in dose with a slight increase in LWR.

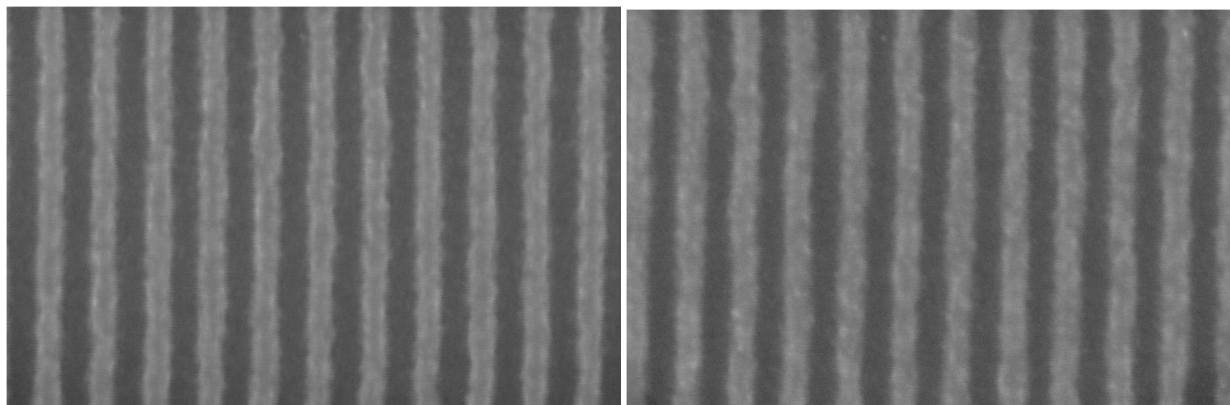


Figure 6. 26nm half-pitch l/s; (left) XE15CB, LWR 3.7 nm, 54 mJ/cm². This image was selected from the process window presented in Figure 3. (right) XE15CB using developer modifications, LWR 4.6 nm, 33 mJ/cm².

We have performed initial characterization of contact hole patterning using quadrupole illumination. Since this is a negative resist, a bright-field mask was used. This exposure results in a substantially reduced dose, but it also projects a significantly lower NILS. Patterning results for this challenging illumination condition are shown in Figure 7. It is worth noting that the BMET delivers roughly the same aerial image contrast for 20-nm hp line/space with annular illumination and for 30-nm hp contacts from a bright field mask with quadrupole illumination. In both cases, the resist captures the resolution limit of the tool under these illumination conditions.

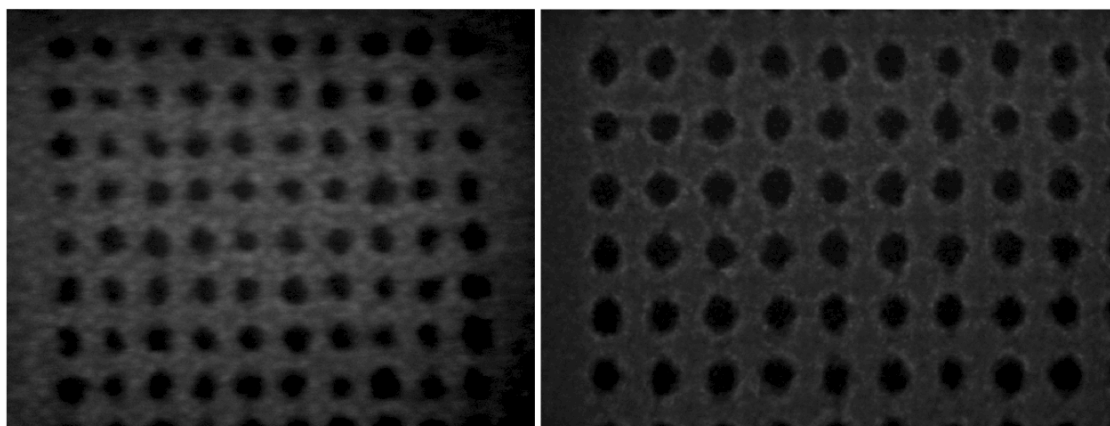


Figure 7. Dense contact hole arrays patterned with bright field mask, quadrupole illumination, a) 30nm hp, 21 mJ/cm² (left); b) 40nm hp, 26 mJ/cm² (right)

3.3 Improved Materials

New resist compositions and synthesis approaches have resulted in an improved formulation – Inpria XF20JD. The primary expected advantage is increased photospeed with retention of high resolution and small LWR. The 22-nm half-pitch features shown in Figure 8 were patterned by e-beam at 249 $\mu\text{C}/\text{cm}^2$ (30 keV). This dose has been separately well characterized to correspond to an EUV dose of 25 mJ/cm². Secondary benefits of this formulation are access to a larger design space for compositions, process conditions, and development chemistries. A substantial boost to shelf life at room temperature is also realized.

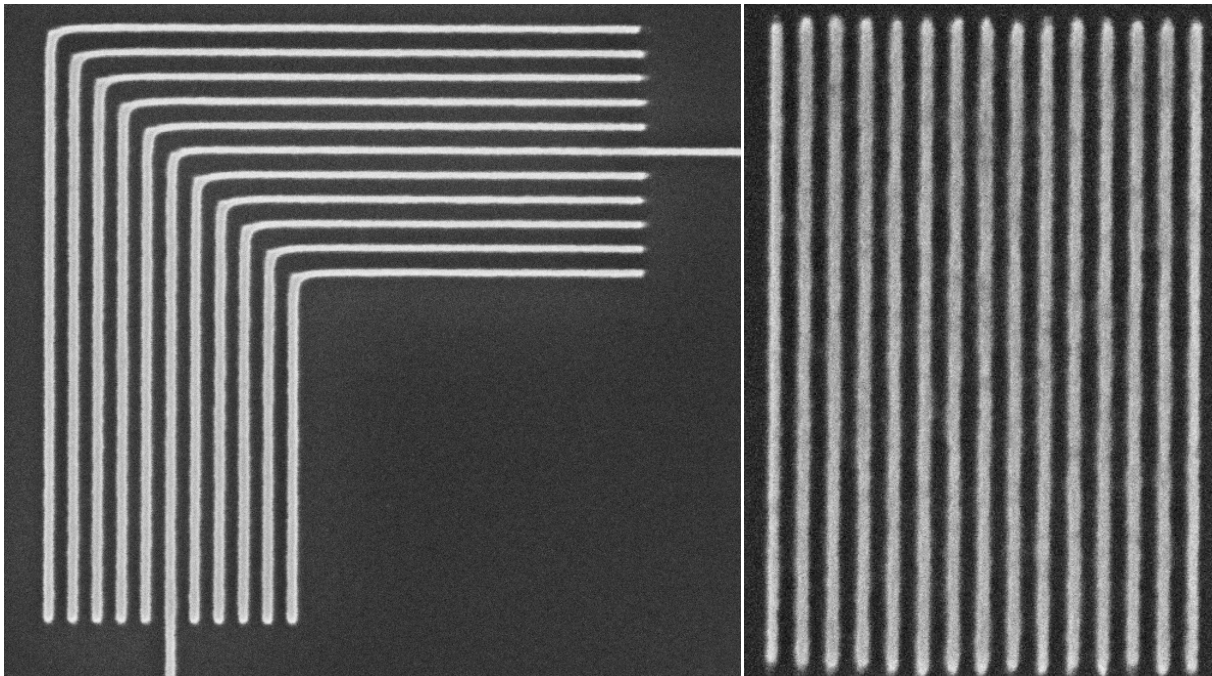


Figure 8. Resist XF20JD patterned by e-beam lithography (30 keV); (left) 26 hp elbow structure, V-lines; CD 25.0 nm, LWR 2.2 nm, $276 \mu\text{C}/\text{cm}^2$, EUV Equivalent dose $28 \text{ mJ}/\text{cm}^2$. H-V bias is due to tool specific write bias. (right) 22 nm HP lines, CD 20.0 nm, LWR 3.0 nm, $249 \mu\text{C}/\text{cm}^2$, EUV equivalent dose $25 \text{ mJ}/\text{cm}^2$

4. PROCESS INTEGRATION

An initial survey of process integration issues was performed to assess risks associated with bringing the MSOx resist materials into a standard semiconductor lithography environment. Experiments were performed to address: coating uniformity and defectivity, the level of critical impurities in the resist, outgassing during EUV exposure, pattern transfer, and wet and dry strip.

4.1 Uniformity and Defectivity

To analyze both the coating uniformity and the level of defectivity of the resist films, the resist was coated on 300-mm wafers by using a Tokyo Electron ACT 12 automated wafer track. All wafers were pre-rinsed with DI water for 10 s and spun dry. The resist was then deposited by manual dispense with a syringe and $0.2\text{-}\mu\text{m}$ PPE filter in a puddle in the middle of the stationary wafer. The wafer was spun at 2500 rpm for 30 s with an acceleration of 500 rpm/s. After coating the wafer was baked at 80°C for 180 s.

To demonstrate the reproducible nature and quality of the resist coating, film thickness was measured at 49 points across the wafer for five coated wafers by ellipsometry using a KLA-Tencor Aleris tool. Seven points, representing variation across the entirety of each wafer, are plotted in Figure 9. Thickness ranges for each wafer were under 1.5 nm, indicating a reproducible, smooth and otherwise high quality film across each 300 mm wafer. The thickness uniformity had a range of 1.5 nm ($\sim 6\%$ film thickness) and can be improved upon by automated dispense and spin optimization.

Defectivity of coated wafers was analyzed by using a KLA-Tencor SP2 surface scan instrument. The mode was adjusted to scan at an oblique angle at 10% laser power to obtain a signal-to-noise ratio of 30 for the smallest defects analyzed. Defects were measured above the filtration level of $0.2 \mu\text{m}$. Measurements were conducted on three separate coated wafers to obtain an average defectivity. Defectivity of the resist was then compared to the defectivity of a wafer coated only with DI water filtered through an identical $0.2\text{-}\mu\text{m}$ filter. The defectivity of the water-coated and resist-coated wafers were 0.13 cm^{-2} and 0.075 cm^{-2} , respectively, indicating that the major source of defects at this level is not the resist. Significant improvements can likely be made through use of advanced filtration and by employing automated dispense and spin optimization.

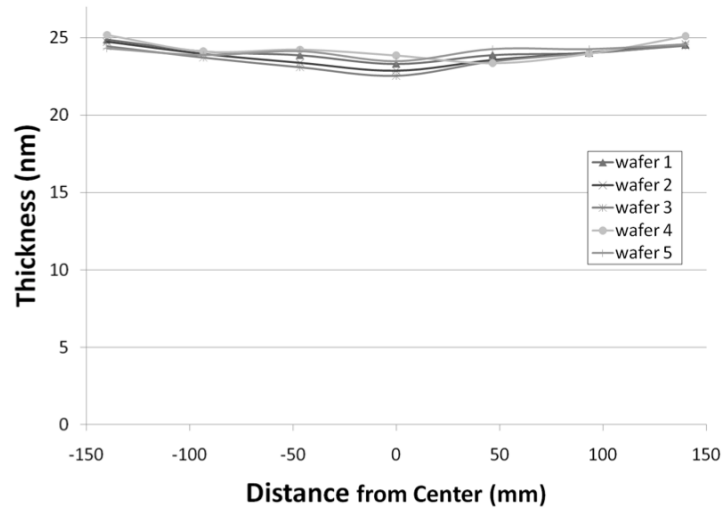


Figure 9. Plot of film thickness across the diameter of five different 300 mm wafers, as measured by ellipsometry.

4.2 Impurity Contamination

Since the resist is wholly inorganic, with hafnium as a major constituent, a significant concern is the potential contamination risk through undesired impurities in the material. Resist samples were submitted to three independent labs for analysis by a combination of ICP-MS, ICP-OES, DCP-OES, and TXRF. Results are summarized in Table 1.

Table 2. Measurements of critical impurities concentrations for Inpria XE15AB resist.

Element	ICP-MS	ICP-OES	DCP-OES	TXRF
Ag	<50ppb	<20ppb		
Au		<5ppb	<1ppm	
Ca				<1ppm
Co	<50ppb	<5ppb		4ppm**
Cu	<50ppb	<20ppb		ND
Cr				ND
K		<20ppb	<1ppm	
Fe		<5ppb	<1ppm	ND
Ir			<5ppm	
Li		<20ppb	<1ppm	
Mn	<50ppb	1130ppb*		ND
Ni	<50ppb	<5ppb		ND
Pd	<50ppb	<50ppb		
Pt		<5ppb	<1ppm	
Ru	<50ppb			
Zn	<50ppb	<5ppb		ND

*suspected interference

**known interference with Hf

< indicates lower detection limit

Other than the following exceptions, all critical metal impurities were measured at 50ppb or better at all three labs. This fits with generally accepted levels for unintended metal contaminants. Reported manganese levels varied among the labs, with one lab reporting a higher level (~1ppm) than the other two (<50ppb); this may be the result of spectral overlap caused by the relatively high concentration of hafnium. Also, due to the difficulty in measuring iridium in solution, a lower detection limit of only 5ppm has been achieved thus far. We are pursuing alternative analysis techniques in order to address these issues and provide an acceptable detection limit.

4.3 Resist Outgassing

Resist outgassing has been measured using standard EUV resist qualification procedures at IMEC and ASML. Given the inorganic nature of the Inpria resist platform, it follows that no hydrocarbons were found to outgas from the resist. The sole components in the outgassing RGA spectrum were water and oxygen (Figure 10). No carbon contamination has been observed from Inpria resist, even by the new and more stringent E-gun based contamination test at ASML. In addition, no increase in the non-cleanable contamination component was observed. Inpria materials have consequently been cleared for use on the ASML ADT at IMEC and provisionally on the ASML NXE-3100.

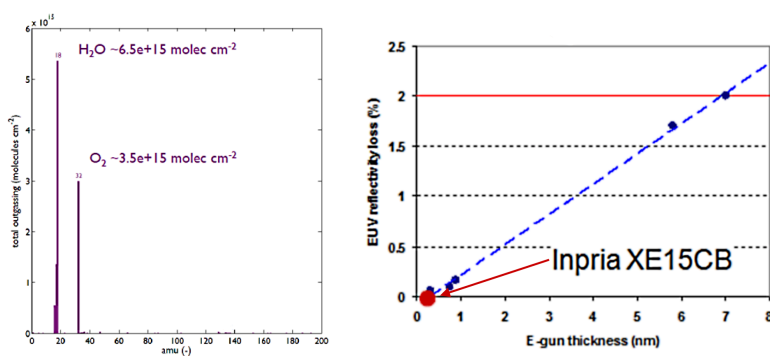


Figure 10. RGA outgassing measurement data and E-gun induced contamination testing results of Inpria resist XE15CB. These results show only water and oxygen outgassing and no contamination growth. Data courtesy of IMEC and ASML.

4.4 Pattern Transfer

A unique property of Inpria resist materials is a very high etch resistance in some etch gasses. Etch selectivities are similar to typical hardmask materials and other inorganic resists such as Dow Corning Fox-12 (HSQ). A process, also serving as a proxy for etch studies, is being developed at the Center for X-ray Optics at Lawrence Berkeley National Labs for the production of high quality X-ray optics. This process utilizes the etch selectivity of Inpria resist over silicon materials to create high aspect-ratio features. The etch selectivity of XE15CB to HSQ was evaluated for several baseline etch chemistries. After hardbake, HSQ is considered a low density SiO₂ like material. This etch survey was conducted in an Oxford Plasmalab 1500 dual chamber ICP/RIE at CXRO. Etching was conducted for 5 minutes for each etch gas; film thickness was subsequently measured by reflectometry. As shown in Figure 11, no etching was observed in Cl₂+O₂ or SF₆, while the film was completely stripped in BCl₃. Though not evaluated here, no etching is expected in an O₂ plasma since the resist is a metal oxide. The overall low etch rates and resilience to SF₆ demonstrate the capability of performing pattern transfer with very thin resist films. High selectivity to all materials containing Si can be obtained in SF₆, even if the Si-material etch rate is very slow, e.g., silicon nitride. The high etch rates in BCl₃ provide a path to a dry-strip process.

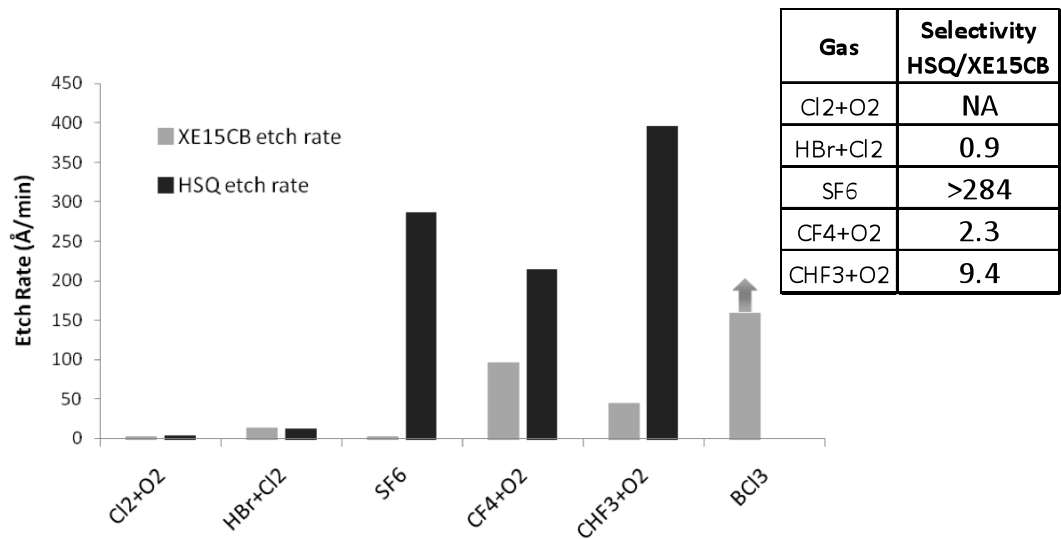


Figure 11. Etch rates for XE15CB and HSQ and selectivity of HSQ/XE15CB. The etch rate of XE15CB in SF₆ is zero and in BCl₃ is greater than 157 Å/min as the film was completely stripped in this etch.

4.5 Resist Strip

A traditional process for stripping organic resists is not suitable for rework or wet strip of Inpria's MSOx resist. After a hard bake, MSOx materials are chemically similar to inert hafnia. At IMEC, high-k gate oxide is stripped off using a dedicated dilute HF bath. In collaboration with IMEC, we have implemented and evaluated a process for cleaning wafers coated with a typical MSOx resist. Fresh wafers were measured for defectivity on the KLA-Tencor SP2 surface scan instrument described above. The wafers were then passed through a track, coated with resist, baked, and stripped. A control wafer was passed through the same process, but it did not receive a resist coating. A diagram of this process is shown in Figure 12. Defectivity of these wafers was measured to the 0.1-μm level. All wafers showed defectivity that was equivalent to or below the fresh-wafer defectivity level. We conclude from this initial experiment that a dilute HF bath (standard for high-k gate oxide processes) efficiently strips Inpria's MSOx resist.

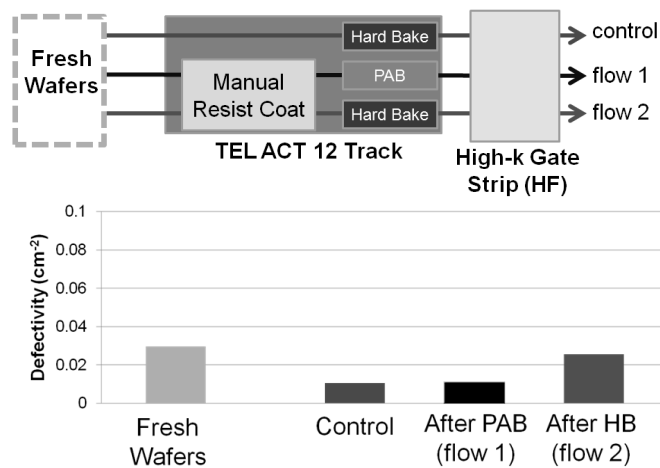


Figure 12. Process diagram for the evaluation of resist strip

5. CONCLUSION

We describe a novel metal oxide patternable hardmask for EUV lithography. For one formulation, we demonstrated a resolution of 15-nm half-pitch on the SEMATECH Berkeley MET and 12-nm half-pitch by electron beam exposure. The material was capable of a process window at 20-nm half-pitch with 10% exposure latitude and 100-nm depth of focus using annular illumination on the BMET. Improvement to sensitivity enabled contact hole patterning at 21 mJ/cm² for 30-nm half-pitch, and line/space patterning at 33 mJ/cm² for 26-nm half-pitch. Continued improvement in sensitivity remains an important area for future development. As an entirely inorganic metal oxide thin film, the resist has very high etch selectivity for many materials, including >100:1 for silicon. Initial data confirmed a strip process and coatability on 300-mm wafers with defectivity consistent with hand dispense. Since the resist has an absorbance of 16 μm^{-1} and high etch resistance, it is well suited for use at a film thickness of ~ 20 nm, thereby mitigating pattern collapse while reducing the potential impact of shot noise. The MSOx patternable hardmask described herein provides a new route to the high-resolution, high-absorbance, high-etch resistance materials required for sub-20-nm EUV lithography.

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