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PAG Incorporated Polymeric Resists for Sub-100 nm Patterning at 193 nm Exposure

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Chemically amplified systems, which provide improved sensitivity, are composed of a polymer matrix resin and small molecule photoacid generating units. The inherent discrepancies of the physical and chemical properties of these components can lead to phase separation thereby reducing resolution and sensitivity. Incorporating the PAG in the polymer main chain provides a unified system, which alleviates these problems. Furthermore, PAG incorporated in the chain may improve outgassing and acid diffusion control. Incorporated in the polymer chain was phenylmethacrylate dimethylsufonium triflate photoacid generator (Tf-PAG), which upon exposure generates triflic acid, providing the level of sensitivity required by chemical amplification (CA) systems. 2-Ethyl-2-adamantyl methacrylate (EAMA), and 3-hydroxy-1-adamantyl methacrylate (HAMA) served as the protecting group. γ-Butyrolactone methacrylate (GBLMA) provided necessary substrate adhesion. PAGs bound in the main chain resist were exposed with the ASML PAS 5500/950B 193 nm Step and Scan System, with 0.63 NA. Resolution of 110nm line space (220 pitch) and 80 nm isolated lines, with photospeed of 8.2 mJ/cm^2 and 2.5 mJ/cm^2 respectively, were obtained for these PAG in the chain resists.

***Keywords:* 193-nm lithography, photoacid generator (PAG), polymer resist.**

1. Introduction

Over the past four decades, feature size for electronic memory and logic devices has been reduced by a factor of two every eighteen months, providing incredible increases in computer power and reduction in computing cost [1].

$$\mathrm{Res} = (\kappa\lambda)/(\mathrm{NA}) \quad (\text{Eq. } 1)$$

The above Rayleigh lens equation (Eq. 1) relates resolution to processing conditions (k), the numerical aperture of the imaging system (NA), and the wavelength of exposure light (λ).

Utilizing the Rayleigh lens equation, the improvements in imaging tool processing parameters, facilitates this remarkable success in miniaturization [2]. To reduce feature size, these exposure tools utilize reduced wavelength light (193 nm or 13.5 nm EUV exposure). Furthermore, a fluid can be introduced between the last optical element and the photoresist, which allows increased NA and therefore higher resolution (193 nm immersion)[1].

However, if the industry is to continue its remarkable shrinking of feature size throughout the nanometer scale, changes in current resist materials must occur. At present, chemically amplified polymeric resists provide the sensitivity necessitated by the low power output of 193 nm

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exposure tools. These systems generally consist of two components: a polymeric resin and a photoacid generator (PAG), the molecule that produces the acid catalyst upon light exposure. These two components are often incompatible and form separate phases [3]. Phase separation of the polymer and the PAG results in non-uniform distribution of the PAG molecules and the photogenerated acid within the polymer matrix. The result of non-uniform distribution is unacceptable line edge roughness, and loss of resolution, especially for nanoscale features [4]. Furthermore, incompatibilities of the PAG and polymer can lead to decreased acid generation efficiency [5].

To avoid these incompatibility issues, the PAG could be directly incorporated into the polymer chain [6]. Also, binding the PAG to the polymer chain reduces outgassed photoproducts, which can damage optical equipment [7]. Furthermore, PAG attached to the polymer chain may also improve acid diffusion, which is the excess migration of acid from the exposed regions. This leads to blurring and decreased resolution [8]. According to the 2005 ITRS roadmap, acid diffusion may be the ultimate factor that limits resolution [9].

These polymer bound PAGs should contain onium salts which generate superacids upon exposure. Superacids, such as triflic acid, provide the level of strength required for chemical amplification. In fact, triflate PAG bound polymeric resists have been successfully imaged at EUV, which motivates continued research, specifically at 193 nm exposure [10]. The structures of vinyl functionalized PAGs (a, b) and blend PAG (c) are shown in Fig. 1. The design of the microstructure for 193 nm sensitive systems, with these PAGs bound to the polymer chain, is shown in Fig. 2. The methacrylate polymer backbone was used due to minimal 193 nm absorbance. γ-Butyrolactone methacrylate (GBLMA) was included in the microstructure to improve adhesion. 2-Ethyl-2-adamantyl methacrylate (EAMA), and 3-hydroxy-1-adamantyl methacrylate (HAMA), both acid-cleavable bulky alicyclic substituent groups, were included to improve resolution, sensitivity, and dry etch resistance [11]. Also, 0.2-wt % tetrabutyl-ammonium hydroxide (TBAH) base was added to control acid diffusion. Table 1 presents the entire list of all polymers synthesized and investigated.

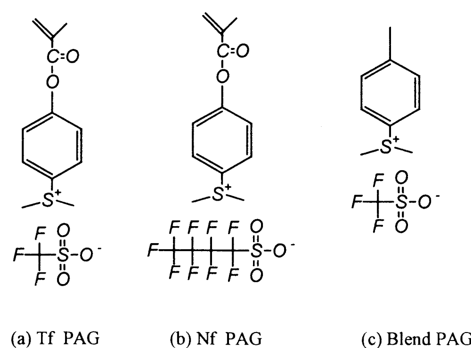

Figure 1: Chemical structures of PAGs: (a) phenylmethacrylate dimethylsufonium triflate photoacid generator (Tf PAG); (b) phenylmethacrylate dimethylsufonium nonaflate photoacid generator (Nf PAG); (c) 4-methylphenyl dimethylsufonium triflate (blend Tf PAG)

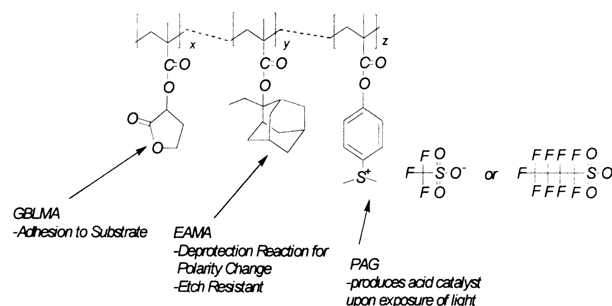

Figure 2: Microstructure design for 193-nm sensitive resist for sub 100-nm patterning

2. Experimental section

2.1. Materials

Materials were purchased from Sigma Aldrich and Acros unless otherwise noted. 2-Ethyl-2-adamantyl methacrylate (EAMA), 3-hydroxy-1-adamantyl methacrylate (HAMA), and γ-butyrolactone methacrylate (GBLMA) were obtained from AZ electronics or Idemitsu Kosan Co., Ltd. Phenylmethacrylate dimethylsufonium triflate photoacid generator (Tf-PAG), phenylmethacrylate dimethylsufonium nonaflate photoacid generator (Nf-PAG), and (4-methylphenyl) dimethylsufonium triflate (momeric Tf PAG) were synthesized according to the literature [6]. α,α'–Azobis(isobutyronitrile) (AIBN) was

purified twice by recrystallization from methanol. Hexamethyldisilazane (HMDS), cyclohexanone, and propylene glycol monomethyl ether acetate (PGMEA) were used as received. A 2.38-wt % aqueous solution of tetramethylammonium hydroxide (TMAH) was obtained from AZ Electronics. Six inch silicon wafers were obtained from Waferworld INC.

2.2. Characterization

The NMR spectra were obtained on a JEOL500 spectrometer with DMSO-d_6 and TMS as the solvent and internal standard, respectively. Gel permeation chromatography (GPC, Waters Breeze software) was performed with THF as the eluent. The molecular weights of polymers were calculated with respect to polystyrene as narrow M_w standards. Thermogravimetric analyses (TGA) were performed on a TA instrument, Hi-Res TGA 2950 Thermogravimetric Analyzer 7, at a rate of 10 °C/min, heating to 800 °C with N_2 gas flow of 25 cm^3/min. Differential scanning calorimetry (DSC) measurements were performed on a TA instrument (DSC 2920) at a heating rate of 10 °C/min. The resists were exposed using the 193 nm ASML 5500/950B optical lithography system, with a 0.63 NA aperture refractive lens (note: 0.85 is the state of the art NA). The light source of this system is a 10 watt 2000 Hz ArF laser. The test mask consisted of a series of 1:1 line space pairs and isolated lines with feature size ranging from 80 nm to 300 nm, and the exposed wafers were evaluated on a Raith 150 SEM. The thickness of the films was measured with a Woollam Variable Angle Spectroscope Ellipsometer. Atomic force microscopy (Digital Instruments Dimension 3100 AFM) in tapping mode provided surface characterization of the lithographic patterns. This experiment utilized standard TESP probes (Digital Instruments) with 125 μm long cantilever which has a resonant frequency range of 309 to 382 kHz. The spring constant of the tip was in the range of 20-00 N/m. The tip had a nominal radius between 5 and 10 nm, with cone half angle: 17° side, 25° front, and 10° back. Scans were taken at a speed of 1 Hz, with the fast scan axis perpendicular to the lines of the lithographic patterns.

2.3. Polymerization

The free radical mechanism provides facile polymerization of resist materials. The following general polymerization scheme was used throughout. First, appropriate molar ratios of monomers were obtained through weighing. Approximately 2 grams of monomers and approximately 0.1 grams AIBN were directly added to enough THF for a 25 ml total volume. Vinyl functionalized PAG (a or b) was dissolved in approximately 5-10 ml acetonitrile before being added to the reaction mixture. This solution (placed in a 100 ml ampoule) was degassed by freeze and thawing under vacuum for at least 15 minutes to remove oxygen. Polymerization was carried out at 65 °C in an oil bath for 24 hours. Polymer was precipitated by dropwise addition to 500 ml of appropriate organic solvent and allowed to stir for 15 minutes. The appropriate organic solvent, usually petroleum ether, diethyl ether, or methanol was determined by solubility tests. The precipitated polymer was allowed to settle at the bottom of the beaker. The organic solvent was then decanted. This precipitate-decant process was repeated three times, and the polymer was finally isolated by gravity filtration and washed with additional organic solvent. This precipitated polymer was then dried under reduced pressure to remove any residual solvent.

The details of the synthesis and characterization of the polymeric resists included Table 1 are provided below in Tables 2-6.

2.4. Resist Processing

Resist solutions with PAG incorporated into the polymer chain were prepared by dissolving enough polymer into cyclohexanone or PGMEA to prepare a 6.6 wt % solution. Blended PAG resist solutions were similarly prepared, and blend PAG (c) was added (5 wt % of polymer). Resist solutions were allowed to dissolve overnight, then filtered through a 0.2 micron Teflon filter.

Table 1: Comprehensive list of synthesized polymeric resists*

Nomenclature	Structure
poly(GBLMA-co-EAMA-co-Tf)	
poly(GBLMA-co-EAMA-co-Nf)	
poly(GBLMA-co-HAMA-co-Tf)	
poly(GBLMA-co-EAMA-co-HAMA-co-Tf)	
poly(GBLMA-co-EAMA)	blended with

* Note: Details of the synthesis and characterization of the polymeric resists in table 1 are provided below in tables 2-6.

Table 2: Polymerization Results of poly (GBLMA-co-EAMA)

Polymer	Mole Feed Ratio		Copolymer Composition		Yield /%	Mw (PDI)
	GBLMA	EAMA	GBLMA	EAMA		
1	20	80	42.1	57.9	13.4	3600 (2.7)
2	30	70	58.5	41.5	18.7	5300 (1.9)
3	40	60	64.1	35.9	28.3	7400 (1.9)
4	50	50	71.7	28.3	21.9	5400 (1.9)
5	60	40	73	27	46.4	6500 (2.5)
6	70	30	77.0	23.0	53.4	8000 (2.2)
7	100	0	100	0	72.0	2800 (2.0)

Table 3: Polymerization Results of poly (GBLMA-co-EAMA-co-Tf-PAG)

Polymer	Mole Feed Ratio			Polymer Composition			Yield /%	Mw (PDI)
	GBLMA	EAMA	PAG	GBLMA	EAMA	PAG		
8	25	72.5	2.5	46.8	47.2	6	30.7	3700 (1.3)
9	25	70	5	43.5	40.8	15.7	13.7	1800 (1.7)
10	35	60	5	58.8	31	10.7	28.1	2000 (1.8)
11	35	62.5	2.5	56.5	37.4	6.1	27.6	4500 (1.5)
12	55	40	5	71.8	20.2	8	42.6	1800 (1.7)

Table 4: Polymerization results of poly (GBLMA-co-EAMA-co-Nf- PAG)

Polymer	Mole Feed Ratio			Polymer Composition			Yield /%	Mw (PDI)
	GBLMA	EAMA	PAG	GBLMA	EAMA	PAG		
13	25	71	4	47.8	46.8	5.4	31.1	1900 (2.6)
14	35	61	4	57.8	37.7	4.5	35.4	1400 (2.6)

Table 5: Polymerization of poly (GBLMA-co-HAMA-co-Tf-PAG)

polymer	Mole Feed Ratio			Polymer Composition			Yield /%	Mw (PDI)
	GBLMA	HAMA	PAG	GBLMA	HAMA	PAG		
15	56	40	4	53.8	42.9	3.3	71.4	4800 (3.4)
16	52	40	8	49.4	43.4	7.1	64.5	2400 (3.4)

Table 6: Polymerization Results of poly (GBLMA-co-EAMA-co-HAMA-co-Tf-PAG)

Polymer	Mole Feed Ratio				Polymer Composition				Yield /%	Mw (PDI)
	GBLMA	EAMA	HAMA	PAG	GBLMA	EAMA	HAMA	PAG		
17	27.5	62.25	7.5	2.75	38.7	46.0	10.7	4.6	46.4	2500 (2.4)
18	27.5	61.75	7.5	3.25	40.1	46.3	8.7	5.0	42.3	1800 (2.5)
19	27.5	53.75	15	4.25	32.2	45.5	17.0	5.3	54.6	2300 (2.5)
20	27.5	60.5	7.5	4.5	37.8	44.9	10.5	6.8	41.4	1400 (2.6)

For resist formulation, Six inch wafers were primed with 20 % HMDS/ 80 % PGMEA solution, or bottom antireflective coating (BARC) by spin coating at 2500 rpm for 60 seconds. The primed wafers were then baked at 115 °C for 90 seconds. Wafers were placed on chill plates and allowed to return to near room temperature after each baking step. Resist solutions were spin coated onto the wafers at 2500 rpm for 60 seconds. Approximately

2 ml of resist solution were required to coat the entire 6 inch wafer. The post apply bake was carried out at 100° C for 90 seconds. The exposed wafers were baked at 100 °C for 90 s. The development was conducted in conventional 2.38-wt % TMAH for 5-25 s followed by deionized water rinse. The thickness of the resists was 150-180 nm.

3. Results and Discussion

3.1. Polymerization and charcterization

^{1}H NMR (DMSO-d_6, ppm) analysis was used to determine polymer composition. Poly(GBLMA-co-EAMA-co-Tf-PAG) provided the following signals: 7.4-8.2 (phenyl); 3.3 (CH_3S-); 5.6 (lactone: CHCOO); 4.3 (lactone: $COOCH_2$); 0.7-2.5 (alkyl). Incorporation of HAMA provided an additional signal at 4.7 (-OH).

Throughout the polymerization, EAMA (the protecting group) avoided incorporation in the polymer chain (Tables 2 and 3). The molar composition of EAMA was always considerably less than the original feed ratio. Also, homo-EAMA was not obtained on several attempts, while GBLMA readily homo-polymerized (Table 2), the resistance of EAMA polymerization is attributed to the steric hindrance of the bulky adamantyl pendant group. Considering intermolecular interactions, this non-polar adamantyl group is poorly compatible with the polar lactone of GBLMA and the polar THF solvent as well.

Considering the decreased yield of resist polymers containing a large proportion of EAMA, another protecting group, HAMA was considered. HAMA contains a polar hydroxyl group, which increases compatibility with the ionic PAG monomers and the polar GBLMA. Utilizing HAMA instead of EAMA greatly increased yields (Table 5), however, these samples showed a considerably lower sensitivity to exposure. Therefore, HAMA was used in addition to EAMA in order to increase yield and preserve exposure sensitivity (Table 6).

Inclusion of the protecting group EAMA showed decreased temperature of decomposed (***Tdec***). Poly (GBLMA) exhibited ***Tdec*** of 234 °C, while poly (GBLMA-co-EAMA) exhibited ***Tdec*** of 147-186°C. The cleavage of the COOR bond (which also serves as the deprotection reaction) is readily facilitated at elevated temperatures.

The glass transition temperatures (***Tg***) and ***Tdec*** for all the polymers are provided in Table7.

Table 7: Glass transition temperature(***Tg***) and thermal stability(***Tdec***)

Sample	*Tg*	*Tdec*
poly(GBLMA)	118	234
Poly (GBLMA-co-EAMA)	107-134	147-186
Poly (GBLMA-co-EAMA-co-Nf)	102-107	109-115
Poly (GBLMA-co-EAMA-co-Tf)	103-113	110-119
Poly (GBLMA-co-HAMA-co-Tf)	120-124	140-142
Poly (GBLMA-co-EAMA-co-HAMA-co-Tf)	95-104	108-115

Inclusion of PAG decreases the thermal stability. Poly (GBLMA-co-EAMA-co-Tf) and poly (GBLMA-co-HAMA-co-Tf) exhibited a ***Tdec*** of 110-119 °C and 140-142 °C respectively. Poly (GBLMA-co-EAMA-co-HAMA-co-Tf) provided ***Tdec*** of 108-115 °C. PAG, which decomposes upon exposure to light, also readily decomposes at elevated temperatures.

Inclusion of the protecting group EAMA generally increased the glass transition temperature. Poly (GBLMA) exhibited a *Tg* of 118 °C, while poly (GBLMA-co-EAMA) exhibited *Tg* of 107-134 °C. It is proposed, the bulky adamantyl pendant groups restrict motion, which increases *Tg*.

Inclusion of PAG generally decreases glass transition temperatures. Poly (GBLMA-co-EAMA-co-Tf) and poly (GBLMA-co-HAMA-co-Tf) provides *Tg* of 103-113 °C and 120-124 °C respectively. Poly (GBLMA-co-EAMA-co-HAMA-co-Tf) provided *Tg* of 95-104 °C.

3.2. Lithographic properties

Fig.3-7 present images of lithographic patterns, which were obtained using a Raith scanning electron microscope (SEM). 110 nm line space features was observed for these systems exposed at 193 nm wavelength and NA =0.63. This resolution was observed for GBLMA-EAMA-Tf-PAG and GBLMA-EAMA-HAMA-Tf-PAG resists with approximately 40 mole % EAMA and 5-7 mole % Tf-PAG (Fig 3-5). The associated exposure dose was 8-9 mJ/cm^2.

Extrapolation of these results with the Rayleigh Lens Equation (Eq. 1) provides resolution potential with state of the art exposure tools (which use improved NA of 0.85), Plugging in wavelength λ = 193 nm, NA=0.63, and resolution=110 nm provides k equal to 0.36. Plugging in this k, with wavelength=193 nm, and improved NA=0.85,

provides 82 nm line space resolution for exposure on current state of the art tools. Line space feature size of 82 nm compares well to the current ITRS requirement of 80 nm line space features produced with these tools [9].

Conversely, triflate PAG blended resists provided only 130 nm line space features (Fig. 3 and Fig. 7 respectively). Potentially higher acid diffusion control associated with PAG bound samples could account for improved resolution. Also, incompatibilities of blended monomeric PAG and polymer could result in decreased resolution.

Base (0.2 wt %) was added to triflate PAG incorporated samples to control acid diffusion. Added base improved fidelity of 110 nm lines space features (Fig 4). However, exposure dose increased 0.45 mJ/cm^2 in comparison with similar systems without base. More base should be included on subsequent exposures to further improve resolution. Triflate PAG incorporated resists provided 80 nm isolated lines (Fig 3). Interestingly, only samples with low amounts of EAMA (approximately 20%) provided high resolution isolated features (80 nm), while only samples with large amounts of EAMA (approximately 40%) provided high resolution dense line space features (110 nm). The increased diffraction associated with dense line space patterns accounts for the resolution discrepancy between isolated features (80 nm), and line space features (110 nm) [12].

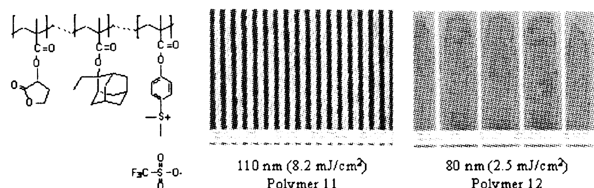

Figure 3: SEM images of poly(GBLMA-co-EAMA-co-Tf)

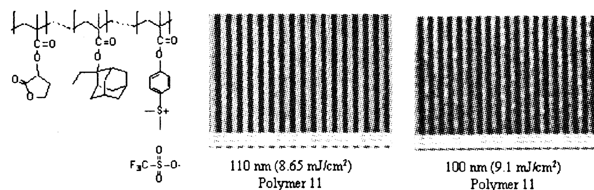

Figure 4: SEM images of poly(GBLMA-co-EAMA-co-Tf) with 0.2 wt% TBAH base added

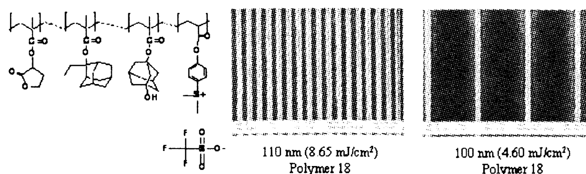

Figure 5: SEM images of poly(GBLMA-co-EAMA-co-HAMA-co-Tf)

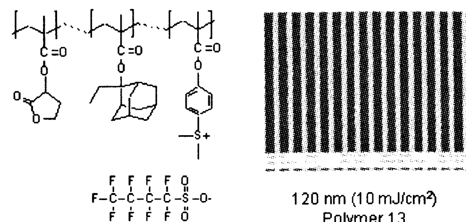

Figure 6: SEM images of poly(GBLMA-co-EAMA-co-Nf)

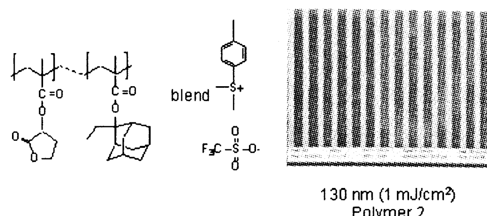

Figure 7: SEM images of poly(GBLMA-co-EAMA) with blended PAG

AFM images of 80 nm isolated lines are essentially identical to SEM images and provide additional height analysis as well (Fig. 8).

Blend ionic PAG resists were more sensitive than bound ionic PAG resists, this was indicated by the result: Tf-PAG blended resist (polymer 2), and Tf-PAG bound resists (polymer 11) were 1 mJ/cm^2, 8.2 mJ/cm^2 for 130 nm line space, respectively. These results are consistent with acid generation investigations by Lee at el [13]. In these investigations, FT-IR spectroscopy studies determined the level of deprotection in blend samples to be higher than bound PAG samples under similar exposure and bake conditions. From these results, it was determined that the acid generation rate constant (also known as Dill C) of blend PAG samples were higher than bound PAG

samples, further indicating that blended PAG resists are more sensitive than bound PAG resists.

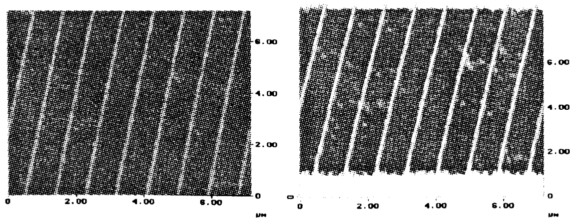

Figure 8: AFM image showing a 3D rendering of 80 nm isolated lines. Feature height and width is 70 nm and 84 nm respectively. Distance between lines is 799 nm.

As previously mentioned, the low yield of EAMA necessitated the use of HAMA which increased yield considerably. Yet, HAMA provides very poor sensitivity. Therefore polymers were synthesized with a combination of EAMA and HAMA which provided good yield and appropriate sensitivity.

The poor sensitivity of HAMA compared to EAMA is explained by the intermediates and products of the deprotection reaction. The deprotection of HAMA forms an unstable carbocation. The sterics of the ring prevent the formation of the preferred planar conformation. The non planar alkene product is also unstable for the same reasons. Although the carbocation from EAMA is on the ring, it is also connected to a sterically unhindered ethyl group. This ethyl group can maneuver to provide a stable planar carbocation intermediate and stable planar alkene product as well.

4. Conclusion

In conclusion, novel resist materials with triflate (Tf) or nonaflate (Nf) PAGs attached to the polymer backbone were synthesized and characterized. The lithographic capabilities of these resists at 193 nm exposure with 0.63 NA were determined. These triflate PAG bound resist provided 110 nm 1:1 line space and 80 nm isolated lines, with exposure doses of 8.65 mJ/cm^2, and 2.5 mJ/cm^2, respectively.

According to the ITRS roadmap, acid diffusion may be the ultimate factor which limits resolution[9]. Tf-PAG bound resists present a potential solution to improve acid diffusion control and continue the progression of the lithography industry for obtaining nanofeatures down to the 32 nm node. The acid diffusion of Tf-PAG bound resists is ongoing. Furthermore, PAG bound resists with the acid component directly attached to the polymer chain, are also undergoing investigation.

Acknowledgments

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