Non-chemically amplified resists for 193-nm immersion lithography: influence of absorbance on performance

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ABSTRACT

The feasibility of three polymer systems for use as non chemically amplified resists for 193 nm lithography are discussed. The three systems are polycarbonates, polyphthalaldehydes and polysulfones. In general it was found that increased absorbance resulted in higher sensitivity to 193 nm light. However, the exception to this was the polycarbonates, which were found to undergo crosslinking due to an alkene group present in the polymer backbone. Although polyphthalaldehydes were very sensitive, their absorbance values were too high to be useful in a commercial environment. Absorbing polysulfones were found to be sensitive to 193 nm light and initial patterning results have been presented.

Keywords: Non-CAR, non-chemically amplified resists, EUV, polymer architecture, polycarbonates,

1. INTRODUCTION

In a bid to maintain growth and profitability, the semiconductor industry is continually seeking to improve the performance of integrated circuits. By and large this is achieved by reducing the size of components that are printed onto silicon wafers by the photolithographic process. Historically, the density of transistors on computer chips has doubled every 18-24 months (Moore's Law) and this benchmark remains the basis for the industries plans for manufacture into the future. The industry roadmap for achieving such continued improvements in performance has reached a junction where component manufacture using shorter wavelengths of light has become economically unviable. Specifically, the introduction of 157 nm VUV technology has been revealed to place unachievable requirements on certain critical components, such as the lens materials, the pellicle[1-5] and the resist itself.[6] Thus in the past several years the industry has concentrated on extending the life of 193 nm (ArF illumination) lithography through various means, most notably using immersion lithography (193*i*) in which the final lens element is a liquid, which at this time is water. It is expected that 193*i* lithography will allow the industry to move beyond the current 65 nm node to the 45 nm node. Initially, it was thought that the use of higher refractive index fluids[7] and photoresists [8-15], 193*i*+ lithography would be able to tackle the 32 nm node. However, the significant challenges associated with this technology have meant that it has been surpassed by double patterning technologies

For these reasons alternative strategies are being actively explored by the semiconductor industry. Several companies are devoting considerable effort to explore the possibly of EUV lithography to achieve the 32 and 22 nm nodes. At this time EUV faces formidable obstacles to being implemented commercially, for example the high cost and questionable reliability of the plasma source and contamination of the projection mirrors due to outgassing. Thus the industry is searching for innovative technologies to extend 193 (and 193*i*) lithography to the 32 and 22 nm nodes.

Current resists utilize chemical amplification to achieve the desired sensitivity, hence their name, chemically amplified resists (CAR). The concept of *chemical amplification* was proposed by Ito, Willson, and Fréchet in 1982.[16] In the chemical amplification scheme, a single photochemical event induces a cascade of subsequent chemical transformations in a resist film; irradiation produces active species that catalyze numerous chemical reactions. Although the active species could be either ionic or radical in principle, use of photochemical acid generators (PAGs), which was proposed in the original chemical amplification concept has become the primary and almost exclusive foundation for an entire family of advanced resist systems. Diffusion of photoacid is believed to be a dominant cause of LER for CAR platforms. [17-19] However, a number of other factors are believed to contribute to LER and the major contributors include

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mask roughness,[20] aerial image contrast,[21] polymer-developer interactions[22, 23] and energy blur such as diffusion of secondary electrons in the case of EUV lithography[24-26].

Despite the large amount of work investigating issues that effect LER a global understanding of all the different components is yet to be achieved. Furthermore, LER values for patterning at 32 or 22 nm nodes is yet to reach the goals set by the ITRS for immersion double patterning or EUV. Given that LER can have a significant effect on device performance,[27] it still remains important to develop polymers and processes that attempt to minimise LER and gain further understanding of the processes involved.

As part of this search for a solution, we are considering non-CAR resists for 193 nm and EUV lithography [28-31]. An issue with this class of resists has been the poor sensitivity [32]. The next generation of excimer lasers have a more powerful laser source which will provide the capability to deliver significantly larger doses to the resist at current scan speeds. Hence, for 193 nm Immersion lithography in particular it is likely that the industry will accept less sensitive resists. However despite this further effort is still required to increase the sensitivity of non-CAR resists

2. EXPERIMENTAL

2.1 Synthesis of Polyaldehydes

Typical polymerization: phthaldialdehyde (1.04 g, 0.782 M) and 10 mL of dichloromethane were added to an ampoule. The ampoule was deoxygenated by three freeze-thaw-pump cycles, sealed and placed in an ice bath (dried ice / acetone) at -78 ^{o}C. 4.56μL (3.6×10^{-5} mol) of boron trifluoride diethyl etherate (4.56μL, 3.6×10^{-5} mol) was added to initiate the reaction for 24h before terminated by addition of acetic acid (2mL)/ pyridine (1mL) mixture. The polymer was precipitated in methanol and the yield obtained was ~85-9%%.

All other experiments were carried out in the same way but with the addition of varying amounts of initiator to phthaladialdehyde, type of initiators and monomers.

2.2 Synthesis of Polycarbonates

A typical ring-opening polymerization was as follows: 2.2.1]heptane-2,5 -[1,3]dioxan]-2 –one (0.43g, 2.62×10^{-2} mol), 1,3-benzene-dimethanol (0.45mg, 4.16×10^{-6} mol), Tin(II) 2-ethylhexanoate (0.17mg, 4.17mg $\times 10^{-7}$ mol) were dissolved in 1 mL of toluene. The polymerization mixture were transferred to 5 mL glass tubes, deoxygenated by three successive freeze-evacuate-thaw cycles, flame-sealed under vacuum and polymerized at 110 °C. The polymer was then precipitated into methanol and dried under vacuum.

All other experiments were carried out in the same way but with different cyclic carbonate monomers.

2.3 Measurements

^{1}H NMR spectroscopy was carried out using a Bruker Avance DRX 500 spectrometer operating at 500.13 MHz for protons and equipped with a 5 mm triple resonance z-gradient probe. Deuterated chloroform ($CDCl_3$) was used to dissolve the organic samples. An internal standard, either tetramethylsilane (TMS) or the residual proton signal of the deuterated solvent was used. Thermogravimetric analysis (TGA) was performed at a heating rate of 10 °C/min in N_2 on a METTLER TOLEDO instrument STAR^e Thermogravimetric analyzer. Differential scanning calorimetry (DSC) was performed at a heating rate of 10 °C/min on a METTLER TOLEDO instrument STAR^e Differential Scanning Calorimeter. Molecular weights of polymers were measured using gel permeation chromatography. The chromatographic system consisted of a 1515 Isocratic pump (Waters), a 717 autosampler (Waters), Styragel HT 6E and Styragel HT 3 columns (Waters) run in series, a light scattering detector DAWN 8+ (Wyatt Technology Corp.) and a 2414 differential refractive index detector (Waters). Tetrahydrofuran (THF) was used as the mobile phase at a flow rate of 1mL/min. ASTRA (Wyatt Technology Corp.) and Empower 2 (Waters) were used for data collection and processing. For the determination of molar mass by conventional SEC, the columns were calibrated by polystyrene standards (Waters) covering the molar mass range of 1060–1,320,000 g/mol. Fourier Transform Infrared spectra of the thin films on silicon wafers were obtained using a Nicolet Nexus 5700 FTIR spectrometer (Thermo Electron Corp., Waltham, MA) equipped with a Harrick grazing angle attenuated total reflectance accessory (Harrick Scientific Products, Pleasantville, NY) fitted with a KRS-5 MIR polarizer (Harrick Scientific Products, Pleasantville, NY). P-Polarised illumination was used. Spectra were recorded at 4 cm^{-1} resolution for at least 128 scans with an optical path difference (OPD) velocity of 1.8988 cm s^{-1}. The thin film side of the Si wafer was pressed directly onto the germanium internal reflection element of the ATR accessory and a pressure of 56 lbs in^{-2} was applied. Spectra were manipulated using the OMNIC 7 software package (Thermo Electron Corp., Waltham, MA). Optical properties of the thin films, phi and delta, were measured using a J.A. Woollam Vacuum UV – Variable Angle Spectroscopic Ellipsometer (VUV-VASE). Using a model these parameters were used to calculate, film thickness, refractive index and absorbance.

2.4 Resist Evaluation

Samples were prepared by dissolving the polymers in cyclohexanone. The resist solution were then spin coated at a thickness of approximately 20~30 nm and subjected to a 120 °C post applied bake (PAB). The wafers were then exposed with varying dose by 193 nm laser. Afterwards, the wafers were applied a post exposure bake and then developed in organic solvents. The contrast curves were normalized by comparison to the initial film thickness and remaining film thickness.

3. RESULTS & DISCUSSION

3.1 Polycarbonates

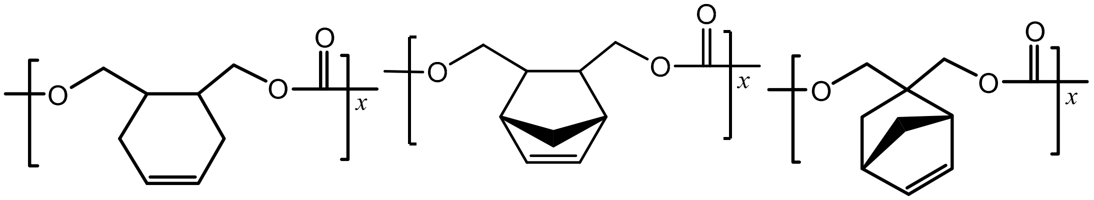
Previously, we have demonstrated that polycarbonates are good candidates as non-CAR resists for EUV lithography [30, 31]. Hence, we decided to investigate the utility of these materials as non-CARs for 193 nm lithography. Initially, we tested the polymers described in Whittaker et al. [31], but these polymers had a low absorbance at 193 nm and hence had E_0 values in excess of 1J cm^{-2}, (data not shown). For this reason we set out to synthesize more absorbing variants.

Three systems have been prepared and Table 1 details the thermal and properties of polycarbonates prepared via ring opening polymerization (ROP) of cyclic carbonates PC1- PC3. The glass transition temperature (T_g) of polycarbonates reported are ≥100 °C and refractive indices (n_{193nm}) are ≥1.7. Both PC1 (~4.93 μm^{-1}) and PC3 (~4.93 μm^{-1}) are within the desirable range. The decomposition temperatures for the polymers were greater than 207 °C, so these materials should withstand typical resist processing temperatures. PC2 was unable to be analyzed due to insolubility in all coating solvent available.

Table 1. Properties of polycarbonates prepared via ROP of cyclic carbonates.

S/N	Molecular Weight$^{\alpha}$	T_g (°C)	T_d $^{\delta}$ (°C)	n_{193nm}	k_{193nm}	Abs_{193nm} (μm^{-1})	*Thk* (nm)
(PC1)	*Mn:* 24855 *M*w: 49271 *PDI*: *1.98*	*~100*	*322*	1.71	0.15	4.93	26.42
(PC2)	*Insoluble in all available coating solvents*						
(PC3)	*Mn: 20399* *M*w: *42045* *PDI*: 2.06	*(108)^x*	*(207)^x*	*1.71*	*0.09*	3.19	*16.73*

α Molecular weights are reported relative to polystyrene standards. β Conversion was measured gravimetrically. δ T_d is measured at the onset of decomposition. x Thermal properties (T_g & T_d) *based on literature*



(PC1) **(PC2)** (PC3)

Figure 1 Structures of polycarbonates used in this study.

Plot (a) in Figure 2 shows chemical contrast curves of PC3 with and without post exposure bake (PEB). No visible differences were observed between the two curves. This indicates that thermal depolymerization of the polymer is not occurring during the PEB. PC3 was unable to clear up to an exposure dose of 2000 mJ cm^{-2} after development with IPA. Swelling of PC3 was visible at $\leq$200mJ, implying that cross-linking may be occurring in the system before degradation dominates. This is likely due to the alkene functional group in the pendent ring. Alkenes are known to undergo thermal and photocrosslinking in the presence of free radical initiators in more rubbery systems. We are currently investigating alternative absorbing groups for these polymers.

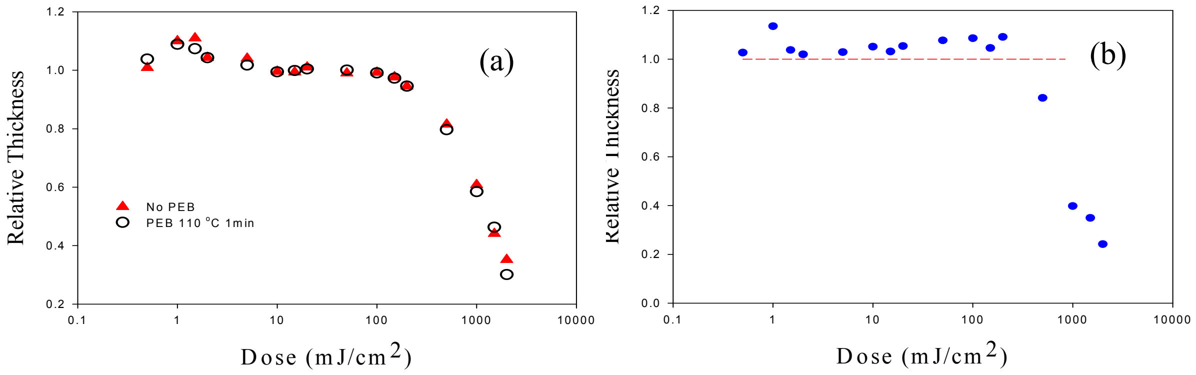


Figure 2. (a) Chemical contrast curve and (b) contrast curve of PC3.

3.2 Polyphthalaldehydes

Self-developing resists are a class of materials which volatilizes during exposure to radiation, eliminating the need for subsequent development steps. The advantage of avoiding the use of the solvents, is that issues such as resist swelling can be avoided. In chemical terms these polymers have a low ceiling temperature and depolymerise to volatile materials. Polyphthalaldehyde [33] is one such material. The T_c for the conventional polyphthalaldehyde is around -43 °C. Willson and Ito have reported the use of a KrF excimer laser, to irradiate polyphthalaldehyde without addition of photoacid generators, to yield clean, spontaneous "self-development" to the substrate [34]. We are seeking to investigate the properties of this polymer when exposed to 193 nm light.

Cationic polymerization of phthalaldehyde is a spontaneous reaction, which normally leads to high weight average molar mass and product yields (~80%). Lower weight average molar mass can be achieved by controlling the rate of polymerization, which is dependent on the polymerization time and amount of initiator. For instance, EXPT IV in Table 2 demonstrates that by decreasing the mole% of initiator from 0.83 to 0.42, lower weight average molar can be achieved (M_w decreased from 56k to 23k). Optical properties, such as n & k, of polyphthalaldehyde at 193nm were analyzed using a VUV-Vase ellipsometer. The refractive index (n) was ~1.7 and the k constant is ~0.9. From this k value the absorbance value of polyphthalaldehyde was calculated to be ~30 μm^{-1} at 193nm.

The chemical contrast curve of polyphthaladehyde is shown in Figure 3, where two different weight average molar mass (M_w:56k & 273k) are used to look at the effect of molar mass under 193nm irradiation. Plot (a) shows that lower weight average molar mass (M_w:56k) had a faster rate of thickness loss than the higher molar mass sample. For examples, ~25% thickness lost (M_w:56k) compare to 5% thickness lost at 5mJ (M_w: 273k). Both plots reached a plateau at ~50mJ, implying a possible completion of depolymerisation. As reported in the literature, the depolymerisation of the polyphthaladehyde will result in residues which include the starting monomer and other possible side products. To verify the degree of unzipping characteristic of polyphthaladehyde, the exposed coated wafer (M_w:56k) was developed using commercial base developer (TMAH, 2.38%). A contrast curve (b) was plotted which demonstrates that the dose to clear (E_0) is less than 10mJ with ~20% thickness retained. The E_0 is closer to ideal commercial resists (7-8mJ cm^{-2}). However, the high absorbance of the polyphthalaldehyde, of ~30 μm^{-1}, makes this material unsuitable in its current form to be used as a viable resist polymer. We are currently investigating lower absorbance variants of this class of polymers.

Table 2 Experimental results from the cationic polymerization of phthalaldehyde in the presence of boron trifluoride etherate ($BF_3 \cdot O(Et)_2$) at 60 °C.

EXPT	**Molecular Weight**$^{\alpha}$	**Initiator (mol%)**	**Conversion (%)** $^{\beta}$	**Time (h)**	T_d $^{\delta}$ **(°C)**	n_{193nm}	k_{193nm}	***Abs*** (μm^{-1})
I	M_n=156k M_w=273k PDI=1.5	5.15	79 (0.82g)	48h	155	1.78	0.93	30.44
II	M_n =190k M_w =345k PDI=1.5	1.42	77 (0.77g)	24h	155	1.72	0.91	29.61
III	M_n =289k M_w =56k PDI=1.5	0.83	81 (0.8g)	4h	168	1.71	0.86	27.98
IV	M_n =8.3k M_w =23.7k PDI=1.5	0.42	78 (0.78g)	4h	186	t.b.a	t.b.a	t.b.a

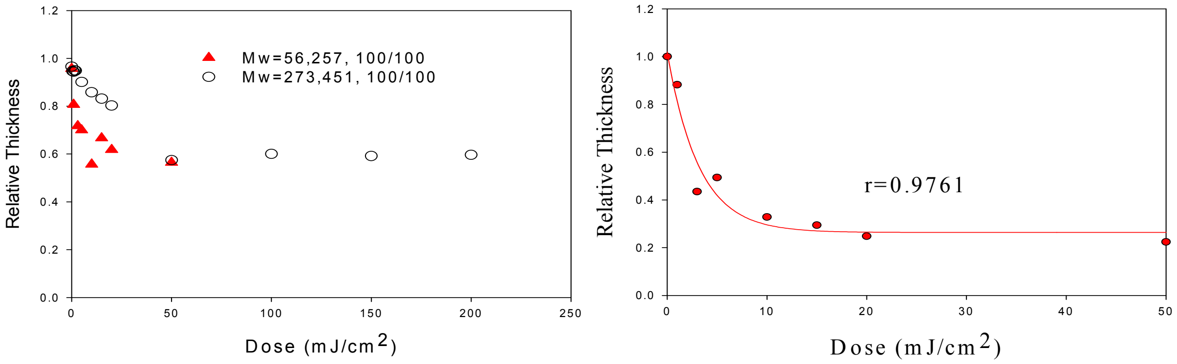
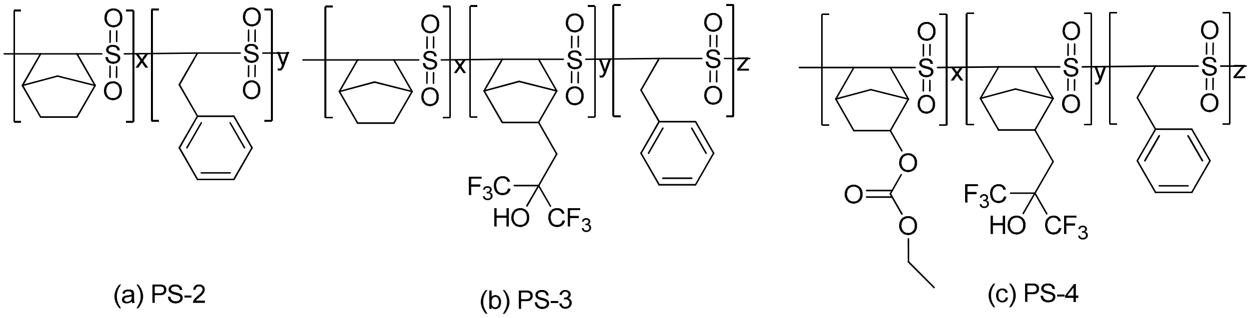


Figure 3 (a) Chemical contrast curve and (b) contrast curve of polyphthaladehyde

3.3 Polysulfone-Based Polymers

Polysulfones have long been known for their high sensitivity to degradation resulting from interaction with high-energy photons.[35-39] In other projects at the University of Queensland we have developed methodologies and apparatus for the free radical alternating polymerization of sulfur dioxide with olefins. Although polysulfones, such as poly-1-butene sulfone (PBS), have been shown to be sensitive to certain wavelengths of light, such as EUV, PBS lacks of etch resistance. In this current project we propose developing copolymers of SO_2 and cyclic olefins, in an attempt to overcome these drawbacks.

Previously, we had reported the investigation of the possibility of poly (bicyclo[2.2.1]hept-2-ene)sulfone as a potential non-CARs for 193 nm immersion lithography.[28] This polymer was found to have desired properties for a good photoresist: (a) glass transition temperature (T_g) at ~120°C, (b) decomposition temperature (T_d) at ~200°C and (c) refractive index (at 193 nm) at ~1.74. However, PS-1 had a low absorbance value (0.17 μm^{-1}) at 193nm which is ~20 times below the target range of 3-5μm^{-1}. This greatly affected the sensitivity of PS-1, which was reported to be approximately > 1J.[40] In order to improve the sensitivity, we have recently reported the synthesis and polysulfones containing aromatic groups such as allybenzene to raise the absorbance value at 193nm.[41] This was found to enhance chain scission and decrease the E_0 values. Values as low as 50 mJ cm-2 were obtained. The structures of some of these polysulfones is detailed below



(a) PS-2 (b) PS-3 (c) PS-4

Figure 4 . Bicyclo[2.2.1]hept-2-ene based polysulfones with aromatic and adhesion precursors incorporated.

Figure 5 shows the top-down scanning electron micrographs of 120~130 nm half pitch line space patterns for PS-2 which were prepared by dry 193 nm interference lithography at an exposure dose of 61mJ.cm^{-2}. Figure 6 shows patterning results for PS-4 (a) at 60 nm hp and (b) 130 nm hp. The images exhibit unexpected line edge roughness, which we believe is a function of the polymers polydispersity index (PS-4) as well as unfavorable polymer-developer interactions (PS-2 & 4). We are working towards developing lower PDI polymers and gaining a better understanding of the effects of resist-developer interactions on LER.

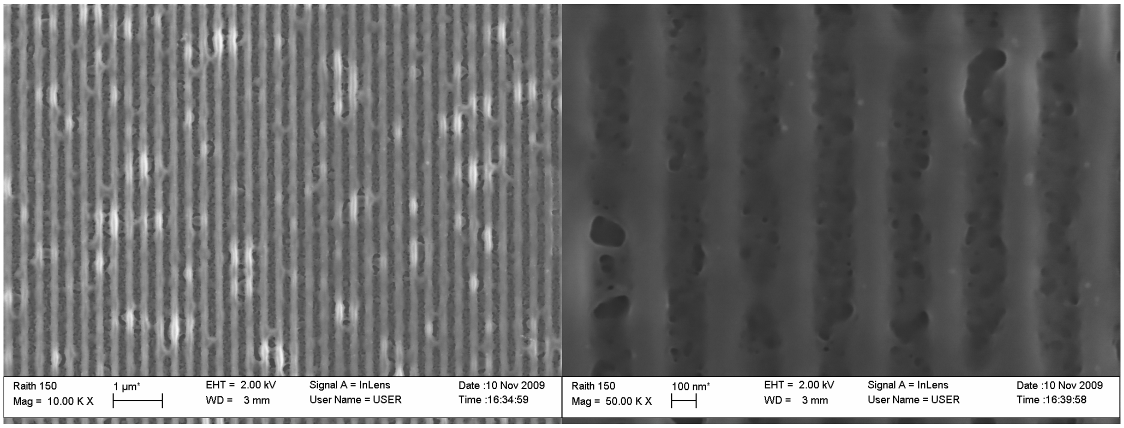


Figure 5 Patterning results for PS-2. Images shows lines patterned at 120~130 half pitch.

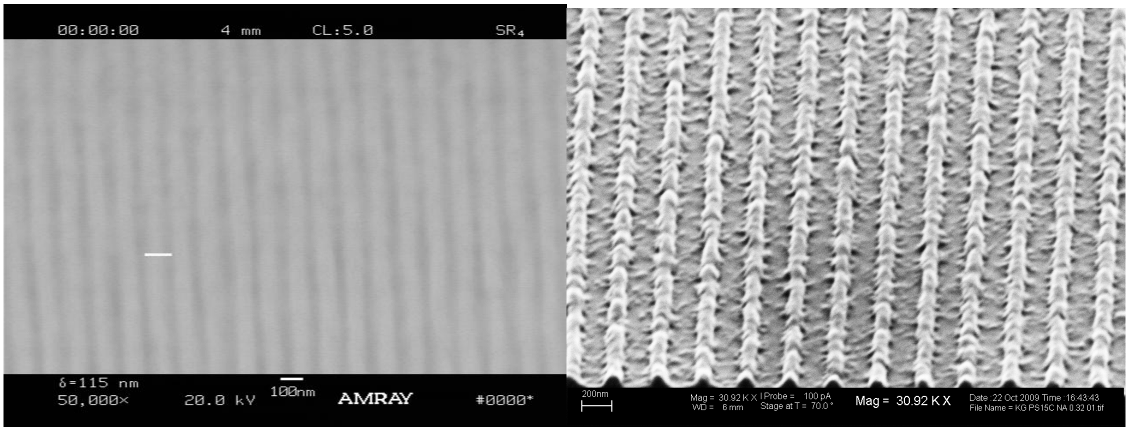


Figure 6 Patterning results for PS-3. Images show (a) patterning at 60 nm hp at 0.82 NA and (b) 130 nm hp at 0.32 NA.

4. CONCLUSIONS

Three polymer systems have been discussed for use as non-CARs in 193 nm lithography. These are polycarbonates, polyphthalaldehyes and polysulfones. Non-CARs with low absorbance values have previously been shown to result in high E0 values. Absorbing polycarbonates were synthesized, however the alkene group that gives the polymers the desired absorbance value resulted in crosslinking of the polymer. The polyphthalaldehydes were found to be highly sensitive, but the absorbance values were too high to be able to be used in as a commercial resist. Finally, polysulfones were able to be prepared with appropriate absorbance values, which resulted in E0 values as low as 50 mJ cm-2. Initial imaging results have been presented, where 60 and 120 nm 1:1 line spaces have been demonstrated. However, the LER values were unexpectedly high. It was hypothesized that these high values were due to the high PDI of the polymers as well as unfavorable developer-resist interactions. We are actively working toward generation of low PDI variants and optimizing the developer systems to better suit the novel polymers that have been synthesized.

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