

Sub-50 nm feature sizes using positive tone molecular glass resists for EUV lithography

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Extreme ultra violet (EUV) lithography is one of the most promising next generation lithographic techniques for the production of sub-50 nm feature sizes with applications in the semiconductor industry. Coupling this technique with molecular glass resists is an effective strategy for high resolution lithographic patterning. In this study, a series of *tert*-butyloxycarbonyl (*t*-Boc) protected *C*-4-hydroxyphenyl-calix[4]resorcinarenes derivatives were synthesized and evaluated as positive tone molecular glass resists for EUV lithography. The amorphous nature of these molecules was confirmed using thermal analysis, FTIR and powder X-ray diffraction. Feature sizes as small as 30 nm with low line edge roughness (4.5 nm, 3σ) were obtained after patterning and development.

Introduction

The photolithographic fabrication of nanometre scale semiconductor devices requires increasingly high resolution techniques in order to maintain pace with Moore's Law.^{1,2} EUV lithography has emerged as one of the most promising candidates to meet the growing demands for ever-smaller feature sizes.^{3–5} Increasing attention has been given to amorphous low molecular weight compounds, known as molecular glasses, as new chemically amplified resists.^{6–11} The use of these monomolecular resists leads to lower line edge roughness (LER) in the fabricated pattern, less swelling or residual stress, and minimal chain entanglement compared to common photoresist polymers. Coupling small molecular size with short wavelength is expected to result in an effective process for the production of nanoscale lithographic features.

Small organic glass forming molecules are well known in the literature, and in particular the synthesis of non-planar structures with bulky side groups are thought to lead to the formation of stable glasses.^{12,13} Materials with higher glass transition temperatures ($T_g > 100$ °C) are generally more desirable for positive tone resists to maintain the integrity of the pattern during the post exposure bake (PEB) in the lithographic process.

To date, it has been demonstrated that several calixarene derivatives form molecular glasses with several desirable characteristics for a resist, including high T_g .^{14–17} Calixarene derivatives have been employed as both negative^{15,18,19} and positive²⁰ tone resists for E-beam lithography, and as positive and negative tone resists using 365 nm UV radiation.^{21,22} To date, no reports in the literature exist of a calixarene based resist for EUV lithography.

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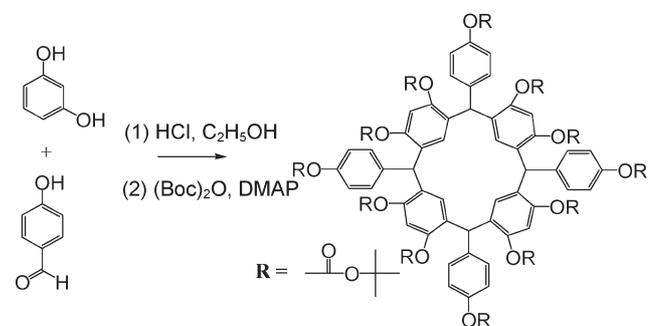
Here, we report the synthesis and characterization of a novel positive-tone *t*-Boc protected *C*-4-hydroxyphenyl-calix[4]resorcinarene (Scheme 1).

These materials are designed with bulky aromatic side groups to ensure high T_g , to inhibit crystallization, and ensure good etch resistance in lithographic processing. We then describe the patterning of these novel molecular glasses using EUV lithography, resulting in the production of sub-50 nm features.

Experimental

Materials and equipment

4-(Dimethylamino)pyridine (DMAP), di-*tert*-butyl dicarbonate, 4-hydroxybenzaldehyde, resorcinol, propylene glycol methyl ether acetate (PGMEA) and 1-methyl-2-pyrrolidinone (NMP) were purchased from Aldrich and used as received. All other solvents were from Fischer, and used as received unless otherwise stated. Fourier transform infrared spectra (FTIR) were measured using a Thermo Nicolet 870. Nuclear magnetic resonance (NMR) spectra were recorded on a Mercury Varian Inova-300 spectrometer. Thermogravimetric analysis (TGA)



Scheme 1 The synthesis of *t*-Boc protected *C*-4-hydroxyphenyl calix[4]resorcinarene.

was performed on a Thermogravimetric/Differential Thermal Analyzer TG/DTA 320 at a heating rate of $10\text{ }^{\circ}\text{C min}^{-1}$ under nitrogen. The T_g of C-4-hydroxyphenyl-calix[4]resorcinarene derivatives was measured on TA Instruments Q1000 Modulated differential scanning calorimeter (DSC) at a heating rate of $10\text{ }^{\circ}\text{C min}^{-1}$ under nitrogen. T_g was determined on the second heating cycle. Powder X-ray diffraction (XRD) was carried out on a Scintag, Inc. Theta-Theta Diffractometer at room temperature. EUV Lithography was conducted at the Advanced Light Source, Lawrence Berkeley National Lab, Berkeley, California. The patterned wafers were examined using a high resolution Hitach SEM. Line edge roughness (LER) was calculated using SuMMIT image analysis software, EUV technology, Martinez, CA.

Synthesis of C-4-hydroxyphenyl-calix[4]resorcinarene. Resorcinol (16.5 g, 150 mmol) and conc. HCl (25 mL) were dissolved in ethanol (150 mL) and was stirred at $0\text{ }^{\circ}\text{C}$ for 10 min using ice water bath. *P*-Hydroxybenzaldehyde (18.3 g, 150 mmol) was added to the solution and the mixture was refluxed for 6 h. After cooling to room temperature, the precipitated solids were collected by vacuum filtration. The crude solids were washed with ethanol (100 ml) two times and acetone (150 ml) three times, followed by drying at $50\text{ }^{\circ}\text{C}$ in vacuo during overnight.

Yield: 75%. Characterization data was identical to that reported by Tunstad *et al.*²³

Synthesis of dodecyl-*O*-*tert*-butyl carbonated C-4-hydroxyphenyl-calix[4]resorcinarene (100% *t*-Boc protected C-4-hydroxyphenyl-calix[4]resorcinarene). To a solution of C-4-hydroxyphenyl-calix[4]resorcinarene (1 g, 1.17 mmol) and DMAP (0.057 g, 0.47 mmol) in NMP (15 mL) di-*tert*-butyl dicarbonate (3.44 g, 15.8 mmol) was added slowly using a dropping funnel. The evolution of CO_2 gas occurred immediately. This mixture was stirred for 24 h at room temperature, then precipitated into a four-fold excess of distilled water, and the resulting solids were collected by vacuum filtration. The crude product was dissolved in ethyl acetate (30 mL) washed with water ($3 \times 50\text{ mL}$) and brine ($1 \times 50\text{ mL}$). The collected organic layer was dried over Mg_2SO_4 , filtered and solvent was evaporated under reduced pressure, resulting in the collection of a fine powder which was dried at $50\text{ }^{\circ}\text{C}$ under reduced pressure overnight. Yield: 1.7 g (70%). The partially protected compounds were obtained using a similar procedure, with varying amounts of di-*tert*-butyl dicarbonate.

Compound A (100% *t*-Boc protected C-4-hydroxyphenyl-calix[4]resorcinarene. Yield: 70%. FTIR (film, cm^{-1}): 2983, 2939 (ν CH), 1765 (ν C=O), 1508, 1486, 1471 (ν C=C aromatic), 1157 (ν C–O–C). $^1\text{H-NMR}$ (300 MHz, CDCl_3 , TMS) δ (ppm) 1.2–1.6 (m, 108H, CH_3), 5.7–6.5 (m, 4H, CH), 6.7–7.1 (m, 24H, aromatic H). **Compound B** (90% protected) Yield: 62%. FTIR (film, cm^{-1}): 3531 (ν OH), 2983, 2966 (ν CH), 1764 (ν C=O), 1508, 1486, 1469 (ν C=C aromatic), 1151 (ν C–O–C). $^1\text{H-NMR}$ (300 MHz, CDCl_3 , TMS) δ (ppm) 1.2–1.6 (m, 97.2H, CH_3), 5.7–6.5 (m, 4H, CH), 6.6–7.1 (m, 24H, aromatic H). **Compound C** (80% protected) Yield: 65%. FTIR (film, cm^{-1}): 3517 (ν OH), 2984, 2939 (ν CH), 1766 (ν C=O), 1508,

1481, 1471 (ν C=C aromatic), 1168 (ν C–O–C). $^1\text{H-NMR}$ (300 MHz, CDCl_3 , TMS) δ (ppm) 1.2–1.6 (m, 86.4H, CH_3), 5.7–6.5 (m, 4H, CH), 6.7–7.2 (m, 24H, aromatic H). **Compound D** (70% protected). Yield: 67%. FTIR (film, cm^{-1}): 3502 (ν OH), 2983, 2939 (ν CH), 1764 (ν C=O), 1509, 1486, 1469 (ν C=C aromatic), 1155 (ν C–O–C). $^1\text{H-NMR}$ (300 MHz, CDCl_3 , TMS) δ (ppm) 1.2–1.6 (m, 75.6H, CH_3), 5.5–6.3 (m, 4H, CH), 6.4–7.1 (m, 24H, aromatic H). **Compound E** (60% protected) Yield: 63%. FTIR (film, cm^{-1}): 3490 (ν OH), 2983, 2937 (ν CH), 1762 (ν C=O), 1511, 1486, 1473 (ν C=C aromatic), 1153 (ν C–O–C). $^1\text{H-NMR}$ (300 MHz, CDCl_3 , TMS) δ (ppm) 1.2–1.6 (m, 64.8H, CH_3), 5.6–6.3 (m, 4H, CH), 6.4–7.1 (m, 24H, aromatic H).

Synthesis of triphenyl sulfonium 2-(phenoxy)tetrafluoroethane-1-sulfonate [$\text{C}_6\text{H}_5\text{OCF}_2\text{CF}_2\text{SO}_3^- \text{S}^+(\text{C}_6\text{H}_5)_3$] photoacid generator. The photacid generator, triphenylsulfonium 2-(phenoxy)tetrafluoroethane-1-sulfonate (Fig. 1) was synthesized by combining a protocol published independently by Fiering and Wonchoba²⁴ and Crivello and Lam.²⁵ Detailed synthesis and characterization of the novel PAG will be published elsewhere.

Briefly, potassium phenoxide was reacted with 1,2-dibromotetrafluoroethane, resulting in 1-bromo-2-(phenoxy)tetrafluoroethane. This was transformed to 2-(phenoxy)tetrafluoroethane-1-sulfinate on reaction with sodium dithionite and sodium bicarbonate in aqueous acetonitrile. The sulfinate was converted to 2-(phenoxy)tetrafluoroethane-1-sulfonate by reaction with elemental chlorine in water to sulfonyl chloride, then oxidation with lithium hydroxide in aqueous THF. Subsequently an ion exchange reaction of sulfonate with diphenyliodonium chloride in aqueous acetonitrile gave diphenyliodonium 2-(phenoxy)tetrafluoroethane-1-sulfonate, which by reaction with diphenyl sulfide and copper(II) acetate was converted to triphenylsulfonium 2-(phenoxy)tetrafluoroethane-1-sulfonate. This was purified by recrystallization from dichloromethane–ether mixture. Yield: 70%. $^1\text{H-NMR}$ (300 MHz, acetone- d_6) δ (ppm) 7.37–7.30 (m, 3H, aromatic protons, anion), 7.37–7.46 (m, 2H, aromatic protons, anion), 7.80–8.0 (m, 15H, aromatic protons, cation). ^{19}F NMR (282 MHz, acetone- d_6) δ (ppm) –82.40 (s, OCF_2), –118.01 (s, CF_2SO_3^-). Elemental analysis, C% calc = 58.2, C% found = 57.5, ESI MS (m/e) = 273.18 ($\text{C}_6\text{H}_5\text{OCF}_2\text{CF}_2\text{SO}_3^-$), 263.38 ($(\text{C}_6\text{H}_5)_3\text{S}^+$)

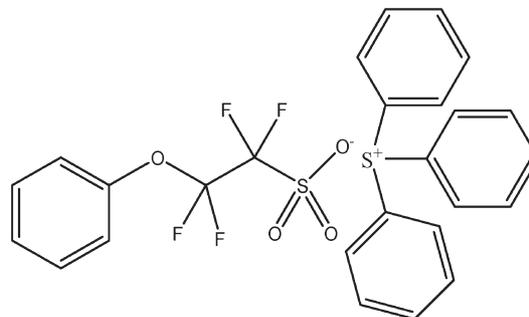


Fig. 1 Triphenyl sulfonium 2-(phenoxy)tetrafluoroethane-1-sulfonate.

Lithographic evaluation

Typically, the resist compounds were dissolved in propylene glycol methyl ether acetate (PGMEA) making a 5% wt solution. A novel PAG, triphenylsulfonium 2-(phenoxy)tetrafluoroethane-1-sulfonate, (5% wrt resist), trioctylamine (0.12 wt% wrt resist) was added and the solution was filtered through a 0.2 μm membrane filter three times. The solutions were then spin coated onto a 4 inch HMDS primed silicon wafer (2000 rpm, 30 s) leading to a film approximately 100 nm thick. This was subjected to a post application bake (PAB) at 100 $^{\circ}\text{C}$ for 120 s, then exposed using EUV radiation. After exposure, the wafer was baked at 80 $^{\circ}\text{C}$ for 30 s, before development in an aqueous solution of 0.026 N tetramethylammoniumhydroxide (TMAH), rinsed with water then dried with a stream of nitrogen.

Results and discussion

The synthesis of *C*-4-hydroxyphenyl-calix[4]resorcinarene has been previously reported.²⁶ We modified the synthetic procedure to include ethanol as a solvent, and obtained the unprotected products as fine pink powders. Protection of the phenol groups using di-*tert*-butyl dicarbonate lead in the desired structure, depicted in Scheme 1. The ^1H NMR spectrum of the protected species was recorded in CDCl_3 , and showed multiplet *t*-Boc peaks around 1.2–1.6 ppm, revealing the existence of a variety of different structural configurations of the target molecule,²³ a factor which helps to inhibit crystallization. By controlling the amount of di-*tert*-butyl dicarbonate added during the protection step, we were able to obtain partially protected compounds in order to probe the effects of resist structure on lithographic performance. In total, five compounds with average *t*-Boc protection ranging from 100% to 60% protected were synthesized. All protected compounds showed good solubility in common lithographic solvents, such as propylene glycol methyl ether acetate (PGMEA).

The thermal characteristics of the compounds were examined by thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC). TGA showed that the observed percentage mass loss of *t*-Boc upon thermal deprotection was in good agreement with those calculated for the average degree of protection for each compound (Table 1).

TGA showed that thermal deprotection occurred at temperatures higher than 150 $^{\circ}\text{C}$ in all cases, and the observed weight loss upon deprotection was found to be in good

Table 1 Thermal characterization of fully and partially *t*-Boc protected *C*-4-hydroxyphenyl-calix[4]resorcinarene

Compound	A	B	C	D	E
<i>t</i> -Boc (mol%) ^a	100	90	80	70	60
% Weight loss (calc) ^b	58.9	56.4	53.4	50.0	46.2
% Weight loss (found) ^c	58.7	56.9	52.8	50.0	47.6
T_g / $^{\circ}\text{C}$ ^d	117	118	116	118	127

^a Theoretical mol% protection by *t*-Boc. ^b Theoretical % weight loss upon thermal deprotection. ^c Actual % weight loss upon thermal deprotection (measured by TGA). ^d Measured by DSC, heating/cooling rate 10 $^{\circ}\text{C}$ min⁻¹.

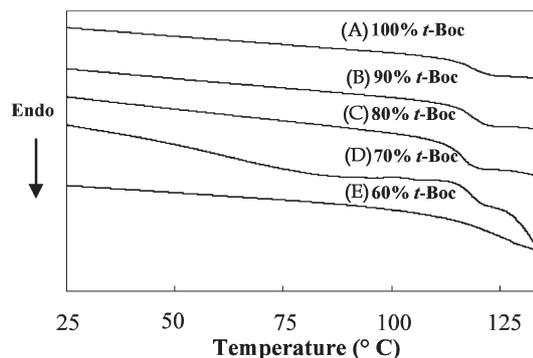


Fig. 2 DSC analysis of *C*-4-hydroxyphenyl-calix[4]resorcinarene with various *t*-Boc protecting compositions.

agreement with the theoretical *t*-Boc coverage in each case. DSC analysis revealed that all of the compounds exhibited a T_g around 120 $^{\circ}\text{C}$, with compound E (60% protection) having the highest T_g of 127 $^{\circ}\text{C}$ (Fig. 2).

FTIR was used to investigate hydrogen bonding in the partially protected compound. The intensity of free hydroxyl group peak at around 3500 cm^{-1} increased gradually with decreasing *t*-Boc protecting ratios, as expected. Compound E showed the broadest peak around 3500 cm^{-1} due to multiple hydrogen bonding interactions. The frequency of the free hydroxyl group peak shifts to lower wavenumbers with decreasing *t*-Boc coverage, also indicating an increase in hydrogen bonding (*i.e.*, compound B, 90% protected, $\nu_{\text{OH}} = 3517 \text{ cm}^{-1}$ compared to compound E, 60% protected $\nu_{\text{OH}} = 3490 \text{ cm}^{-1}$). The spectra can be seen in Fig. 3.

However, the carbonyl band at 1765 cm^{-1} was largely unchanged regardless of the degree of protection. These results indicate that hydroxyl–hydroxyl hydrogen bonding interactions predominate over hydroxyl–carboxylic interactions between adjacent molecules, possibly because intermolecular hydrogen bonding between hydroxyl groups and carbonyl groups are hindered by the presence of the bulky tertiary butyl

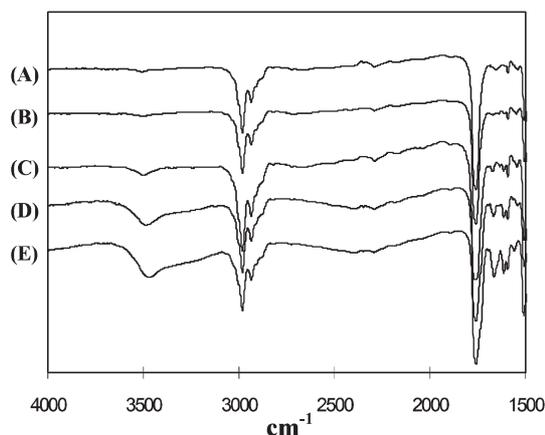


Fig. 3 FTIR of fully and partially protected *C*-4-hydroxyphenyl-calix[4]resorcinarene recorded as thin films. Note the increase in hydrogen bonding indicated by the broadening of the peak at 3500 cm^{-1} as the degree of protection decreases (A), 100%, to (E) 60%.

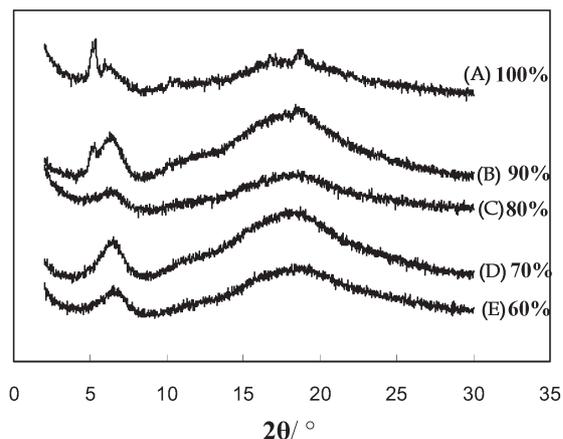


Fig. 4 XRD analysis of fully or partially *t*-Boc protected C-4-hydroxyphenyl-calix[4]resorcinarene, recorded at room temperature.

functionalities. We believe that the increase in hydrogen bonding is responsible for the increase in T_g in compound E. In order to confirm the amorphous properties of the compounds, the powder XRD traces were recorded at room temperature (Fig. 4).

All compounds show two halo peaks indicative of amorphous materials ($2\theta =$ approximately 6.3 and 18.3°). The d -spacing of the two amorphous halo peaks at narrow and wide angles are around 1.4 and 0.5 nm respectively. Interestingly, compound (A) has a sharp peak at $2\theta = 5.3^\circ$ and a less intense peak at $2\theta = 10.5^\circ$. These two sharp peaks suggest the existence of a lamellar structure. This result may indicate the existence of a layered mesophase structure in (A), and to a lesser extent in (B). Previous studies on calix[4]resorcinarene derivatives have observed similar phenomena.²³ As the degree of *t*-Boc coverage decreases (compounds C-E), the sharp peaks gradually disappear indicating amorphous compounds, and suggesting that an increase in the number of structural conformers in the material results in a complete absence of short range ordering.¹³

Preliminary solubility tests during spin coating revealed that compound C, 70% protected, was the optimal resist material when spin coated onto a silicon wafer primed with HMDS. The compounds with higher protection ratios (A and B) showed poor adhesion to the silicon wafer, while compounds with lower protection ratios (D and E) were slightly soluble in aqueous base even prior to exposure, leading to a reduction in image integrity upon development. Full details about the preparation of the resists for lithographic evaluation can be found in the experimental section. No crystallization of the spun coated films was observed, even after storage for several months, indicating stability of the amorphous resists.

SEM images demonstrate that 30 nm resolution was achieved using EUV lithography, with a resist formulation consisting of 5% by weight of the novel photoacid generator triphenylsulfonium 2-(phenoxy)tetrafluoroethane-1-sulfonate and an exposure dose of 22 mJ cm^{-2} (Fig 5A and B).

Low line edge roughness (LER) is purported to be a major advantage of using molecular glass resists.^{6,20} This quantity is usually defined as a deviation in the edge of a feature from an

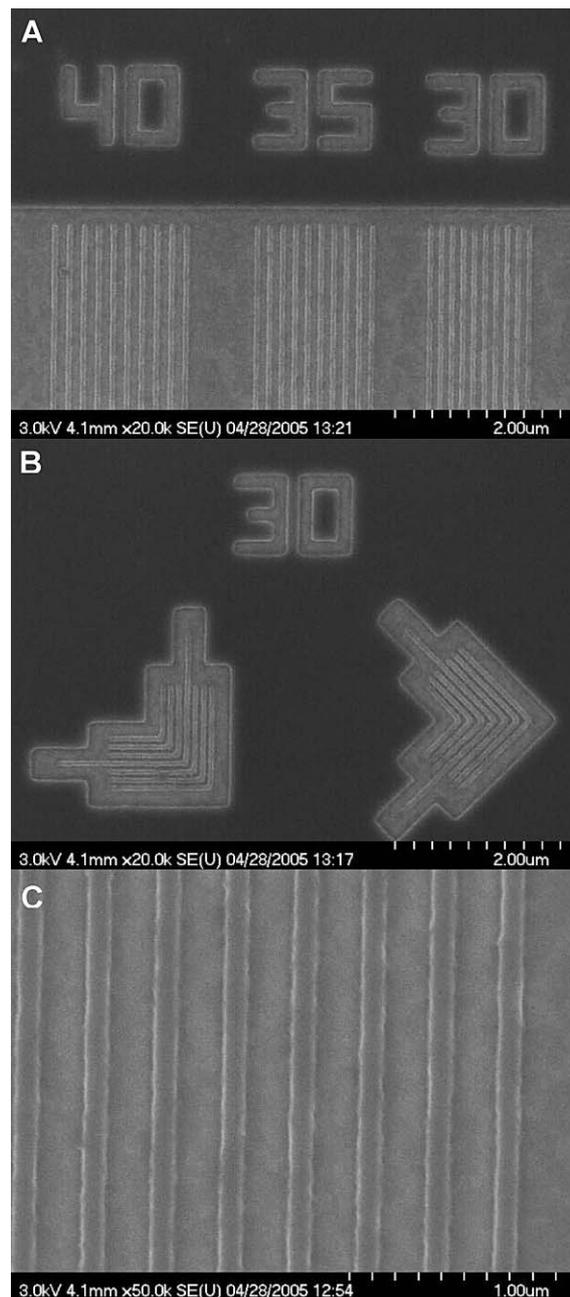


Fig. 5 (A) and (B) SEM images of 30 nm patterns obtained using compound C, 70% protected C-4-hydroxyphenyl-calix[4]resorcinarene with EUV lithography, 5% PAG, dose 22 mJ cm^{-2} . (C) 100 nm lines demonstrating low LER (4.6 nm, 3σ , from 50 nm dense lines). Full details regarding lithographic evaluation can be found in the experimental section.

ideal fitted line averaged across several features. The LER of the features obtained *via* EUV lithography were calculated from these images using SuMMIT image analysis software. LER (3σ) was calculated from 50 nm dense lines to be 4.6 nm. With conventional polymeric resists, LER of 12–14 nm have been encountered.²⁷

The exact reason for the low LER often encountered when molecular glass resists are employed is still under debate. The

conventional explanation is that the smaller size of these resists compared to conventional polymeric resists results in higher resolution features through smaller pixel sizes. Several groups^{3,28} have examined the LER obtained using polymeric resists, and showed that even a 12 fold increase in M_w (from 2.9–33.5 kg mol⁻¹) does not affect LER, which suggests molecular size alone is not the complete explanation. One significant difference between molecular glass resists and polymeric resists is polydispersity. Molecular glass resists are monodisperse, compared to polymer resists which always consist of a range of molecular weights. Lower molecular weight polymers have faster dissolution rates compared to higher molecular weight samples.³ This implies that the dissolution behavior of a polymeric resist upon development is a composite value depending on its molecular weight distribution and degree of deprotection, which leads to higher values of LER. We hypothesise that monodisperse MG resists, in which dissolution rate should be comparatively constant across the exposed regions of the sample, develop in a more homogenous manner and hence return lower values of LER.

Conclusions

In summary, *t*-Boc protected C-4-hydroxyphenyl-calix[4]resorcinarenes were synthesized and studied as positive-tone resists for advanced lithography. The compounds were found to be amorphous materials with high T_g 's. Feature sizes as small as 30 nm with excellent low LER were produced after patterning with EUV wavelength radiation. Future research goals will focus on the synthesis of new calixarene analogues as target materials for EUV resists.

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