Hybrid metal-organic clusters resist for next-generation high-NA EUV lithography

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Abstract

Extreme ultraviolet (EUV) lithography, predominantly with the advent of high-NA systems, imposes stringent requirements on photoresists to overcome the resolution-line-edge roughness (RLS) sensitivity trade-off for next-generation (NG) technology nodes. Hybrid metal-organic clusters (MOC) resists, particularly antimony (Sb)-based MOCs functionalised with a crosslinkable organic ligand, have emerged as promising NG, EUV (λ~13.5 nm) resists owing to higher EUV absorption and tunable molecular properties, with ligand-level control of solubility & cross-link architecture. We report the design, synthesis, and analysis of Sb-MOCs functionalised with organic ligands, using SCXRD and patterned via EBL as a prelude to EUV benchmarking. The minimum feature size of half-pitch (hp) line patterns was evaluated for an EBL-exposed negative-tone resist developed in different developer media. Development was carried out in Deionized (DI) water (pH = 7.2) for 30 s, and in *n*-heptane and iso-octane for 120 s. Minimum hp line widths of 25 nm, 45 nm, and 45 nm were achieved at exposure doses of 700 μC/cm², 800 μC/cm², and 4000 μC/cm² for DI water, *n*-heptane, and iso-octane developers, respectively. The corresponding line edge roughness (LER) values for the minimum achievable hp line patterns were 4.26 ± 0.12 nm for the 25 nm patterns developed in DI water. For the 45 nm patterns developed in *n*-heptane and iso-octane, the LER values were 3.87 ± 0.23 nm and 5.73 ± 0.48 nm, respectively. The high-resolution pattern fidelity underscores the Sb-MOC resists as a scalable platform for sub-25 nm patterning in high-NA, EUV lithography, offering a pathway to extend semiconductor scaling toward the sub-10 nm technology node.

Keywords: EUV photoresist; negative tone resist, antimony MOC, EBL pattering, high resolution (l/s) patterns, RLS trade-off.

1. Introduction

The transition to High-NA extreme ultraviolet lithography (EUVL) is redefining resist requirements for next-generation chip manufacturing as patterning approaches sub-16 nm half-pitch. EUVL has become a promising option for producing high-performance integrated circuits (IC) down to 10 nm nodes or below, following the celebrated Moore's Law [1]. At these nodes, stochastic variability driven by photon shot noise, limited absorption, and reduced resist thickness increasingly constrains pattern fidelity, demanding simultaneous optimization of resolution, LER, and sensitivity (RLS) [2]. Even though EUVL remains the industry standard for large-scale production, early-stage screening and prototyping are severely hampered by its high costs, and limited accessibility. Electron beam lithography (EBL), therefore, serves as a high-resolution proxy for evaluating resist contrast, cross-linking behaviour, and dissolution characteristics, enabling rapid formulation optimisation prior to EUV exposure studies [3-4]. Resist development is a key step in chip fabrication, directly influencing pattern fidelity and process compatibility with safe, sustainable manufacturing [5]. Aqueous development pathways are particularly attractive due to their compatibility with semiconductor processes, but they require precise control over swelling, collapse free behaviour, and dissolution contrast to support high-resolution patterning. Recent advances in water, processable resists further support green manufacturing by reducing toxicity, waste, and defect formation under tightly regulated process protocols [6]. Commercial aqueous developable CAR and nCAR resists, including sulfonium-based CAR/nCAR, HSQ, and MAPDST-co-ADSM, enable eco-friendly processing but face challenges with LER and sensitivity to high-resolution patterns. Despite water's cleaner dissolution, MAPDST-co-ADSM, nCARs patterned ~20-40 nm l/s lines but showed elevated LER due to molecular size

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constraints. Similarly, resolution, LER, and sensitivity trade-offs in aqueous systems were highlighted in $SnMSF_4$, nCAR, which resolved ~22 nm patterns at 2000-3000 µC/cm [7-9].

While inorganic metal oxide-based resists, such as HfO_2 and ZrO_2, exhibit excellent resistance to plasma etching, they often suffer from weak photon absorption, limited process compatibility, and insufficient sensitivity to EUV exposure [10]. In contrast, purely organic polymer resists are generally easier to process but typically exhibit low EUV absorption and poor etch robustness. These trade-offs have motivated the development of hybrid metal-organic platforms that combine inorganic absorption advantages with ligand-enabled control over solubility and network formation. As reported, indium-based metal-organic clusters, including In-MAA and In-mTA, achieved half-pitch line patterns of ~35 nm and below, under electron-beam exposure; however, pattern development relied on isopropyl alcohol, a solvent generally considered incompatible with fab-friendly processing [11-12]. Antimony-based alternatives, such as $SbCl_5$-derived hybrids, exhibit enhanced water solubility through structural transformations (e.g., from tetramers to discrete units), enabling milder aqueous developers [13].

Therefore, a negative tone antimony-based resist (Sb-mTA) has been designed & developed to improve the development process for fab-friendly ecosystem. It uses the ideal pKa (~4.4) of meta-toluic acid for precise pattern solubility switching and the high atomic number of Sb for excellent EUV absorption. Various developers, including DI-water, n-heptane, and iso-octane, have been used to produce patterns ranging from ~25 to 90 nm and result the aqueous developer sensitivity at ~700 µC/cm². Aqueous developer outperformed organic solvents such as n-heptane (800 µC/cm²) and iso-octane (~4000 µC/cm²), which showed residue issues, for the Sb-based nCARs, while aqueous developer allowing for clean dissolution and high-resolution patterns down to ~25 nm (l/s) without swelling. While iso-octane balanced LER at 40-90 nm, N-heptane provided sharper edges for thicker films, but higher doses reduced throughput.

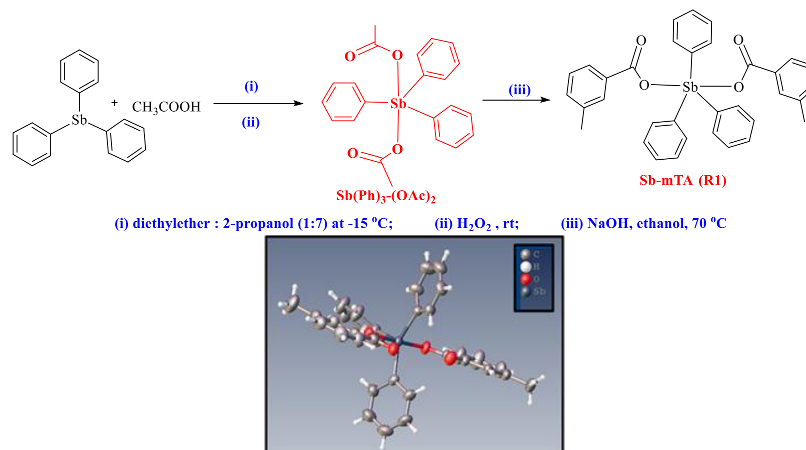
2. Experimental Section

2.1 Materials

Triphenyl antimony (III), *m*-toluic acid, and hydrogen peroxide were obtained from Loba Chemicals. Acetic acid and n-heptane were purchased from Thermo Fisher Scientific, while diethyl ether and sodium hydroxide were sourced from Merck. Chloroform, hexane, iso-octane, and ethanol were procured from SD Fine Chemicals (SDFCL), and ethyl lactate was acquired from TCI Chemicals. Solvents were of analytical or HPLC grade as appropriate.

2.2 Synthesis of metal organic cluster (MOC) resist

The acetate ligand on triphenyl antimony was replaced following a reported procedure with necessary modifications [14]. To synthesize the meta-toluic acid-functionalized antimony complex (Sb-mTA), meta-toluic acid was first dissolved in ethanol, followed by the addition of an equimolar amount of sodium hydroxide under continuous stirring to yield a homogeneous solution. In a separate flask, antimony (III) acetate was dissolved in ethanol to obtain a clear solution (Solution A).



Scheme 1: Schematic representation of synthesis and SCXRD structure of Sb-mTA.

The sodium salt of meta-toluic acid was then added dropwise to Solution A at 70 °C under constant stirring. The reaction mixture was maintained at this temperature overnight to ensure complete substitution. Upon completion, the product was isolated by washing sequentially with diethyl ether and hexane to remove impurities. The solid was dried in an oven at 50 °C for 12 hours. Final purification was achieved by recrystallization from a methanol-hexane solvent system. The structure of the final product, Sb-mTA, was confirmed by single-crystal XRD.

2.3 Resist formulation and thin film preparation

To prepare the resist formulation, 2.5 wt.% of the synthesised Sb-mTA material (R1) was dissolved in ethyl lactate and thoroughly mixed using a vortex mixer to ensure complete dispersion. The resulting solution was deposited onto a silicon wafer (pre-cleaned using the RCA method) via spin coating at ~2500 rpm for 45 seconds, producing a uniform thin film. The coated substrates were then subjected to a soft bake at 90 °C for 1 minute to eliminate residual solvent. A post-exposure bake was performed under identical thermal conditions (90 °C for 60 seconds) to promote network formation and enhance the film's suitability for subsequent development. Film thickness measurements indicated an average thickness of approximately~20 nm.

2.4. Deep Ultraviolet (DUV) Exposure and Pattern Development

Sb-mTA resist thin films were subjected to deep ultraviolet (DUV) irradiation at λ ~ 254 nm, using a conventional proximity-based lithography stepper (EVG-610) with an exposure energy range of ~100 mJ/cm^2. Following exposure, the films underwent a post-exposure bake (PEB) at 90 °C for 60 seconds to promote chemical transformations essential for pattern development. The development of the exposed resist samples was analysed under three different solvent systems: (DI) water (for 30 seconds), n-heptane (for 120 seconds), and iso-octane (for 120 seconds), deionised to evaluate developer compatibility and pattern resolution. In addition, to better understand the resist patterning mechanism, unmasked Sb-mTA films were exposed to uniform DUV flood irradiation. This was done as a control experiment to examine the change in the resist's chemical kinetics under DUV irradiation.

2.5 Electron beam lithography (EBL) and pattern development:

Electron beam lithographic (EBL) patterning was performed using the e-Line PLUS EBL system (Raith GmbH, Dortmund, Germany) with a 17 keV focused electron beam. A resist film with an average thickness of ~29 nm was exposed under carefully controlled parameters. An aperture of ~10 µm was selected, and the beam current was maintained at ~17.4 pA. Various electron doses were systematically applied to optimise pattern resolution and exposure efficiency. To achieve well-resolved nano-patterning, freshly prepared Sb-mTA MOC resists, spin-coated to a thickness of ~29 nm, were exposed under e-beam over a broad range of exposure doses. Following e-beam exposure, the resist samples underwent pattern development in a solution of afore-mentioned developer solutions, which are iso-octane, n-heptane, and DI water.

2.6 Characterisation Techniques

To evaluate the nanoscale patterning performance of the resist, high-resolution imaging was conducted using Field Emission Scanning Electron Microscopy (FESEM; Zeiss Gemini SEM 500, Germany), enabling detailed analysis of pattern dimensions and structural uniformity. The presence and nature of functional groups, and the structural elucidation of the Sb-mTA complex, were determined by single-crystal X-ray diffraction, recorded at 296 K with an Agilent SuperNova diffractometer equipped with a CCD detector and using graphite-monochromated Cu Kα radiation (λ = 1.54184 Å). Film thickness across the coated silicon substrates was measured using Atomic Force Microscopy (AFM; Bruker Icon) to ensure accurate topographical profiling. Additionally, critical metrics, such as line-edge roughness (LER) and line-width roughness (LWR), of the exposed patterns were quantitatively evaluated using SuMMIT® metrology software, which is commonly used to assess lithographic precision and nanoscale surface quality.

3. Results and Discussion

The lithographic performance of the Sb-mTA, MOC resists in different developer solutions, such as iso-octane, n-heptane, and DI water have been evaluated using EBL as a preliminary assessment for EUV benchmarking.

3.1 Lithographic patterning of resist (Sb-mTA) formulation

3.1.1 Lithographic Performance of Sb-mTA Resist (developed with DI water) (pH = 7.2)

The lithographic performance of $Sb(Ph)_3(OOC\text{-}C_7H_7)$, i.e., Sb-mTA, was evaluated using electron beam lithography (EBL). The resulting patterns obtained after developing the Sb-mTA resist in DI water (pH 7.2) are shown in Figure 1(a–d). Half-pitch (*HP*) line patterns with features of~ 25, 45, 65, and 90 nm line-width were successfully patterned with a development time of 30 seconds at an e-beam exposure dose of ~ 700 $\mu C/cm^2$. The measured LER values were 4.79 ± 0.20 nm for 90 nm half-pitch lines, 6.63 ± 0.28 nm for 65 nm half-pitch lines, and 4.26 ± 0.12 nm for 25 nm half-pitch lines. The FESEM micrographs clearly demonstrate successful *HP* patterning down to ~25 nm using DI water as the developer. The ability to develop Sb-mTA resist in DI water, a fab-friendly and environmentally benign ecosystem, represents a significant practical advantage. Aqueous based development eliminates the need for toxic, corrosive, or alcohol-based developer solutions, thereby simplifying process integration and improving safety in high volume semiconductor process environments. From a manufacturing perspective, this attribute of Sb-mTA is particularly attractive for device fabrication lines, as it reduces chemical handling, lowers environmental impact, and enhances compatibility with existing cleanroom infrastructure.

Developer: DI water for 40 s @ 700 $\mu C/cm^2$

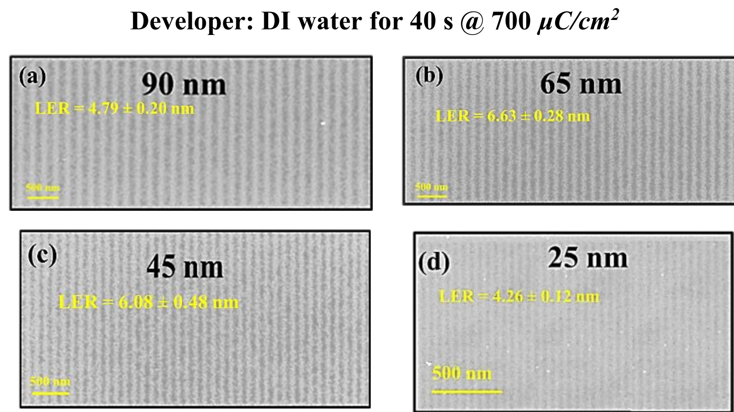


Figure 1. FESEM micrographs of Sb-mTA resist patterns generated using EBL, showing (a) 90 nm half-pitch (HP), (b) 65 nm HP, and (c) 45 nm (d) 25 nm HP line patterns, using DI water (pH = 7.2) as a developer.

To further examine pattern fidelity, the EBL-exposed Sb-mTA resist thin films were also developed using two other compatible developer solutions. The outcomes are discussed in the following sections.

3.1.2 Lithographic Performance of Sb-mTA Resist (developed with n-heptane)

When the Sb-mTA resist formulation developed in *n*-heptane, the Sb-mTA resist demonstrated the capability to pattern *HP* line features of 45, 65, and 90 nm at an e-beam exposure dose of ~ 800 $\mu C/cm^2$, as shown in Figure 2(a–c). However, 25 nm *HP* patterns could not be resolved using *n*-heptane as the developer. The resulting *HP* patterns exhibited reduced LER values as: 3.69 ± 0.32 nm for 90 nm lines, 3.53 ± 0.15 nm for 65 nm lines, and 3.87 ± 0.23 nm for 45 nm lines with a development time of 120 seconds. Development in *n*-heptane leads to reduced resist sensitivity, as evidenced by the higher EBL exposure dose requirement, while simultaneously decreasing LER due to its low polarity and non-swelling behaviour. However, *n*-heptane provides a narrow development window to resolve line-patterns below 45 nm *HP* and limited solubility contrast between exposed and unexposed regions. In addition, its high volatility and flammability, along with challenges in process control and compatibility with high volume chip manufacturing, limit its practical implementation despite the observed improvements in resist sensitivity [15].

Developer: n-heptane for 120 s @ 800 $\mu C/cm^2$

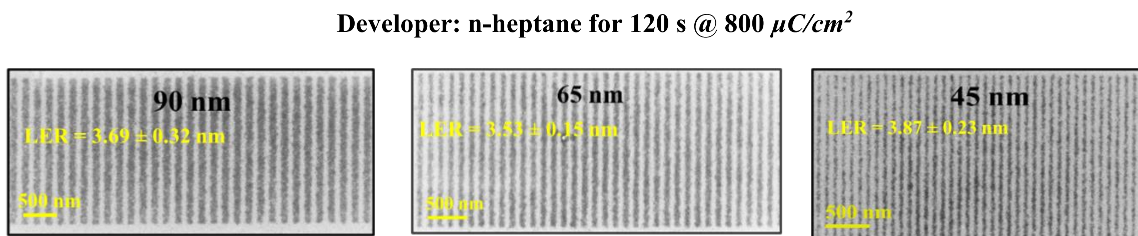


Figure 2. FESEM micrographs of Sb-mTA resist patterns generated using EBL, showing (a) 90 nm half-pitch (HP), (b) 65 nm HP, and (c) 45 nm HP line patterns, using n-heptane as a developer.

3.1.3 Lithographic Performance of Sb-mTA Resist (developed with iso-octane)

To further examine pattern fidelity, the EBL-exposed Sb-mTA resist thin films were developed using iso-octane, and the resulting patterns are shown in Figure 3(a–c). *HP* line features of 45, 65, and 90 nm were obtained with a development time of 120 seconds at an e-beam exposure dose of ~ 4000 μC/cm^2. Similar to *n*-heptane development, 25 nm *HP* features could not be resolved. The measured LER values were 6.26 ± 0.14 nm for 90 nm *HP* lines, 4.51 ± 0.28 nm for 65 nm *HP* lines, and 5.73 ± 0.48 nm for 45 nm *HP* lines, indicating increased edge roughness and reduced pattern fidelity compared to *n*-heptane development. Iso-octane exhibits limited solvency toward the exposed Sb-mTA resist system, resulting in inefficient resist dissolution and a substantial reduction in resist sensitivity, as reflected by the significantly higher EBL exposure dose requirement. In addition, curtailed clearing and non-uniform dissolution at the pattern boundaries contribute to the increased LER and reduced development contrast, consistent with previously reported behaviour for low-polarity hydrocarbon developers [16].

Developer: iso-octane for 120 s @ 4000 *μC/cm^2*

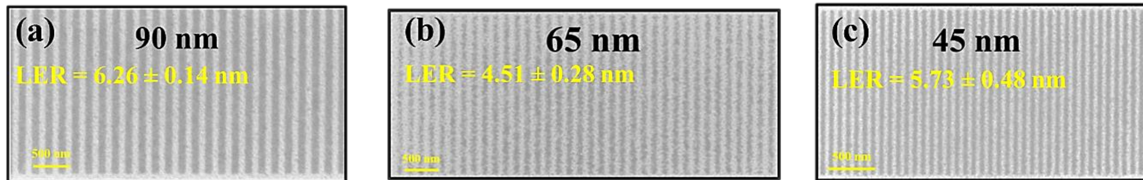


Figure 3. FESEM micrographs of Sb-mTA resist patterns generated using EBL, showing (a) 90 nm half-pitch (HP), (b) 65 nm HP, and (c) 45 nm HP line patterns, using iso-octane as a developer.

The corresponding contrast curves are shown in Figure 4, where 500 nm×500 nm NRT boxes were exposed under e-beam with a dose range of 30-3000 μC/cm^2. Under EBL, the Sb-mTA resist developed in DI water exhibited a contrast value greater than one (γ_EBL = 1.53) and a sensitivity of 495 μC/cm^2. These values indicate a clear difference in dissolution behaviour between the exposed and unexposed regions of the resist. The higher contrast suggests a well-defined development threshold when DI water is used, allowing the resist to be removed selectively during development while preserving the patterned features. As a result, uniform and well-defined patterns were obtained at a moderate exposure dose. Largely, the contrast and sensitivity values confirm that DI water provides a reliable development medium for Sb-mTA based resist formulation under EBL exposure.

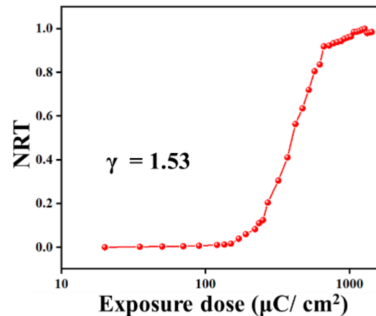


Figure 4. Normalised Remaining Thickness (NRT) vs. exposure dose for contrast measurement after development with DI water.

3.2 Mechanistic approach of resist formulation

To get insight into structural transformations after exposure of the resist material to DUV exposure, the XPS analysis was performed as shown in Figure 5. In XPS results, the Sb 3d core levels showed a systematic shift toward lower binding energies with DUV exposure, with a rise in atomic concentration and a decrease in the Sb 3d$_{5/2}$ peak from 531.58 to 531.38 eV and the Sb 3d$_{3/2}$ peak from 540.68 to 540.58 eV. Instead of total oxygen removal, these modifications suggest a change in the Sb coordination environment, which is compatible with a partial decrease of Sb species due to Sb-O bond breaking, followed by structural reorganisation. This interpretation is confirmed by the O 1s peak shift from 531.68 to 531.48 eV with a simultaneous increase in atomic percentage, suggesting the production of new oxygen-containing species, such as Sb-O-Sb interactions or hydroxylated structures, under DUV irradiation [17].

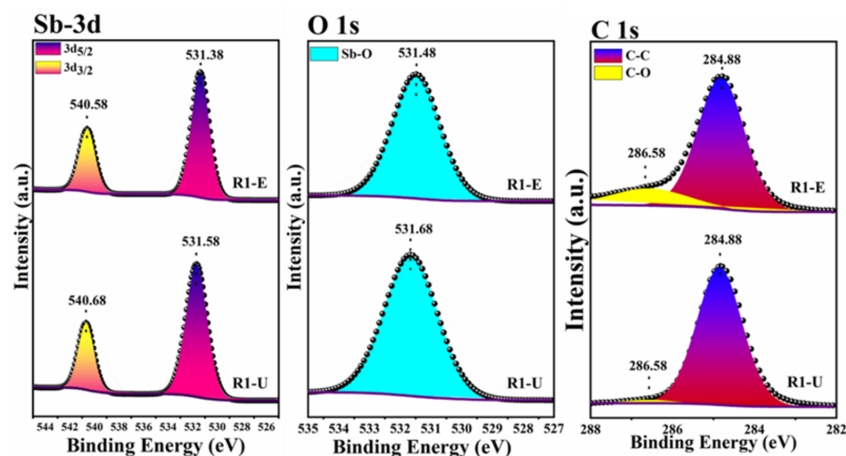


Figure 5. XPS results of Sb-mTA before and after DUV exposure (R1-U = unexposed Sb-mTA; R1-E = exposed Sb-mTA).

The C 1s components at 284.88 eV (C-C/C-H) and 286.58 eV (C-O/C=O), on the other hand, revealed a drop in atomic concentration following exposure but no discernible binding energy shift, suggesting selective photochemical reactions within the organic framework. While maintaining the carboxylate ($-COO^-$) coordination to Sb, this behaviour is compatible with carbon–carbon cross-linking and the cleavage of C=O functionalities linked to the meta-toluic acid group. Taken as a whole, the XPS data show that rather than total carboxylate ligand breakdown, DUV irradiation promotes inorganic–organic network densification through Sb-centred rearrangement and C-C cross-linking [18].

4. Conclusions

In conclusion, these lithographic performance studies reveal that the Sb-mTA MOC resist is highly influenced by developer selection, with a fab-compatible water-based developer providing clear advantages. A comparison of iso-octane, n-heptane, and DI water (pH 7.2) reveals that DI water allows better pattern fidelity, resolving ~ 25 nm half-pitch (*HP*) features at an exposure dose of ~ 700 µC cm^{-2}, while nonpolar hydrocarbon developers are only able to resolve about ~45 nm half-pitch patterns, even at higher doses. Apart from enhanced resolution, development in aqueous results in a strong contrast (γ_EBL = 1.53) with good sensitivity, suggesting a wider process window and a quick solubility switch. XPS investigation also demonstrates that exposure promotes carbon-carbon cross-linking and regulated coordination modifications, allowing for strong pattern formation without significant ligand loss. Together, these findings demonstrate the potential of antimony-based MOC resist for fab-friendly, next-generation lithography, with relevance to sub-16 nm patterning for EUV lithography and beyond, and establish DI water as an efficient and ecologically friendly developer for Sb-mTA resist formulation.

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