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Focusing on nanoparticles based photomultiplier in n-CARs

Satinder K. Sharma[1,a)], Mohamad G. Moinuddin[1], Midathala Yogesh[2], Shivani Sharma[1], Manoj Sahani[1], Subrata Ghosh[2], and Kenneth E. Gonsalves [2, b)]

[a)]*satinder@iitmandi.ac.in ;* [b)] *kenneth@iitmandi.ac.in*

[1]*School of Computing and Electrical Engineering (SCEE), Indian Institute of Technology (IIT)-Mandi,MANDI-175005(Himachal Pradesh),India*
[2]*School of Basic Science (SBS), Indian Institute of Technology (IIT)-Mandi,MANDI-175005 (Himachal Pradesh),India.*

ABSTRACT

To meet the International Roadmap for Device and Systems (IRDS), the development of an advanced lithography process for next-generation (NG) technology node is a vital and challenging task, as we are reaching to its physical limits. In the progress of high volume manufacturing (HVM) at sub-12 nm node, it is very important that resist materials should possess low line edge roughness (LER) and high sensitivity (E_0) using extreme ultraviolet (EUV) and its analogous exposure systems. Apart from standard chemically amplified resist (CAR), acid-free non-CAR has been studied immensely as a potential candidate for NG patterning. To achieve sub-12 nm patterns, a complete study for newly developed n-CAR is required to make sure that developed resist formulation is performing optimally. Aside from the n-CARs, we adopted a novel patterning approach using He^+ ion beam lithography with less proximity effect. Here we present the metallic nanoparticle photo-multiplier (high optical density materials for λ ~13.5 nm) embedded with n-CAR for better photo-absorption and high-resolution pattern development. The silver (Ag- OD 12 w.r.t Carbon) nanoparticles (NPs) with ~2 nm regime were embedded into MAPDST homo-polymer ((4-(methacryloyloxy)phenyl) dimethylsulfonium trifluoromethanesulfonate). To investigate the high-resolution patterning synthesized photoresist was exposed to e-beam (E_e) and Helium ion (E_{He}) beam lithography. The patterned samples were developed in aqueous solution and revealed the negative tone with the sensitivity of 172 $\mu C/cm^2$ and 50.4 $\mu C/cm^2$ for E_e and E_{He} respectively. The MAPDST-Ag resist found stable for more than 1 year, which clearly suggests that there is no sign of Ag-NPs agglomeration in the formulation. Thence, evidently, prove the considerable shelf life of developed resist formulation and can be used in NG semiconductor device HVM and other electronic device applications.

Keywords: *Ag Nanoparticles, helium ion beam lithography, electron beam lithography, non-chemically amplified resist, shelf life, and Critical Dimensions (CD).*

1-INTRODUCTION

Recently metal nanoparticles have been adapted by semiconductor and electronics industries due to its broad range of applications such as variable electronic band-gap and photoabsorption enhancement.[1] Further materials with high absorption cross-section for extreme ultraviolet (EUV) photons such as Au, Ag, Sn, Ni, and Sb have also shown the plasmonic effect (absorb-excite-emit) at nanometer regime, where absorbed photon by nanoparticles have emitted as plasmons. These plasmons further interact with the nearby regime and enhance the kinetics, called as photo-multiplication.[2] These photomultiplications can also use in next-generation lithography (NGL) to promote the high volume production (HVM).[3]

In the recent past, next-generation lithography comprises ~13.5 nm EUV energy aimed towards HVM. At the sub-10 nm node, it is also required to provide the materials which are capable to reach few nm features to provide a stable skeleton for the underneath material.[3,4] When most of the materials are transparent to EUV irradiation it is required to provide the materials consisting of decent absorption as well as patternable also.[5] In this context, chemically amplified resist (CAR) along with EUV sensitive photoacid generators (PAG) has already patterned sub-13 nm half-

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pitch at very low energy, however line edge roughness (LER) optimization is still a challenging task for these CARs.[6]

On the other hands, Xiaoqing Shi *et al.* have shown He^+ ion beam patterned fullerene C_{60} colloidal with 6.5 nm features and decent LER and 40 μC/cm^2.[7] It is also found hard to achieve the required LER in CARs due to ~1.7 nm PAG-PAG separation and generation of access H^+. Sulfonium ($SO3^-$) ion moieties are widely used in PAG for CARs, whereas Hengpeng Wu *et al.* has reported Sulfonium ($SO3^-$) based organic compound 4-(methacryloyloxy)phenyl)dimethylsulfonium trifluoromethanesulfonate (MAPDST) with for high-resolution patterning with low LER.[8]

To avoid the usage of PAGs in NGL resist, the metal nanoparticles (NPs) can be used for the same function. Instead of PAGs, these high absorbance sub-5 nm nanoparticles such as Ag, Au, Pd can stimulate the photosensitive polymers for patterning under EUV and analogous techniques as shown in Figure 1a and 1b.[5] Highly accelerated electrons have been projected as patterning tool similar to the photon-based lithography (DUVL, EUVL). The confinement of charge at high acceleration provides a suitable condition to pattern sub-10 nm features.[9] Despite the electron beam lithography, the helium (He^+) and neon (Ne^+) ion have also shown great potentials to pattern the resist, these ion-based patternings are closer to the EUVL such as required sensitivity and throughput.[3,10,11]

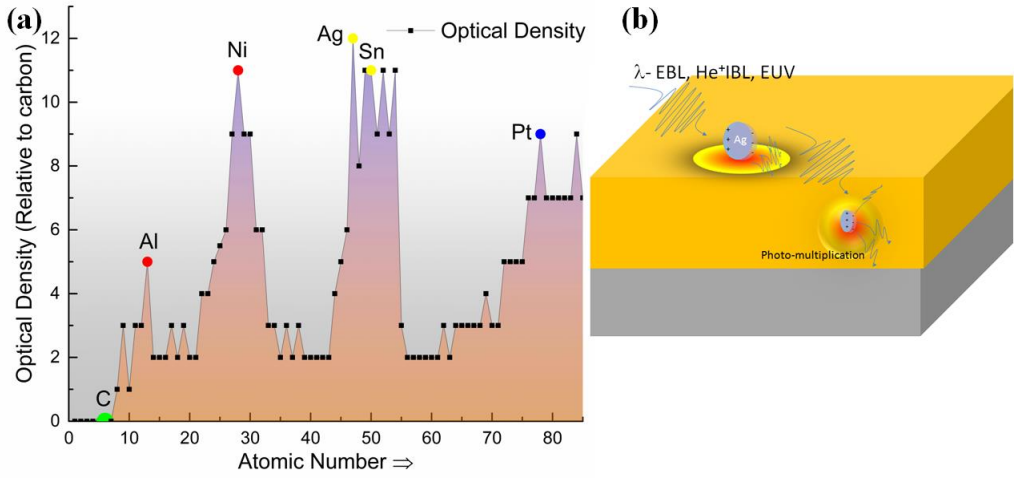


Figure 1 (a) Graphical representation of periodic table material's optical density adapted from Roberto Fallica *et. al.* SPIE JM3; 2018[5]; highlighted materials are capable of nano-particle/colloidal formation, **(b)** a schematic presentation of photon interaction with nanoparticles and Plasmon effect, the affected area has been highlighted here.

Herein, we demonstrate the synthesis of Ag nanoparticles and its blending inside the MAPDST-Homopolymer matrix as a photomultiplier. Nanoparticles were synthesized using water-in-oil emulsion technique and encapsulated with thio-dodecane to enhance the solubility. MAPDST-Homopolymer was synthesized using a free radical reaction. The blended solution has been shown the enhanced UV absorption which may possibly due to the Ag-NP's photo-multiplication. The photoresist patterned using e-beam lithography and found capable of pattern sub-15 nm. Further, a confined helium ion beam was irradiated over the thin film; exposed sample was developed and analyzed down to a critical dimension (CD) of 11.5 nm. The defect-free patterns on MAPDST-Ag claim the parametric enhancement to support the next generation patterning node down to sub-12 nm.

2-EXPERIMENTAL DETAILS

2.1 Photoresist synthesis

The photoresist formulation was adapted in two separate syntheses. The synthetic procedure for the monomer of MAPDST was adapted from the following literature.[8,12] To realize these polymers as n-CAR, a highly sensitive Sulfonium salts group has been introduced into the polymer units. Adding sulfonium groups inside the polymer,

provide the photosensitive bond cleavage where polarity change occurs in MAPDST-Homopolymer during high energy beam irradiation (13.5 nm EUV, 20 keV e-beam and 30 keV He^+ ion).

The Ag nanoparticles were synthesized through water-in-oil (W/O) microemulsion methods. The 0.005-0.02 M of silver nitrate ($AgNO_3$) salt, 0.05 M of the AOT as the surfactant and 0.16 M of Hydrazine Hydrate (NH_2NH_2OH) as the reducing agent has been used for synthesis at room temperature to avoid the agglomeration due to thermal effect. Firstly the AOT was mixed with $AgNO_3$ and diluted with a non-polar solvent such as toluene under constant stirring. Finally, 20 µL hydrazine hydrate was added and stirred for 1 hour for the reduction process of Ag salt to Ag-NP. It is observed that the synthesized Ag nanoparticles were capped with an organic moiety which helps in dissolution inside the polymer chain rather than agglomeration. A similar approach for synthesis high OD nano-particles (Sn, Sb and etc.) was also reported elsewhere.[13]

Figure 2 shows the schematic of photoresist formulation with the blending of in-house synthesized materials. Here Ag-NPs in toluene were doped inside the polymer solution in vol/vol ratio. The resist formulation was established 2wt % MAPDST Homopolymer blended in 76 vol% acetonitrile and 24 vol% Ag-NPs (toluene). Both organic solvent (Acytonitrile and toluene) were found partially soluble in each other, further stirring provide a homogeneous solution.

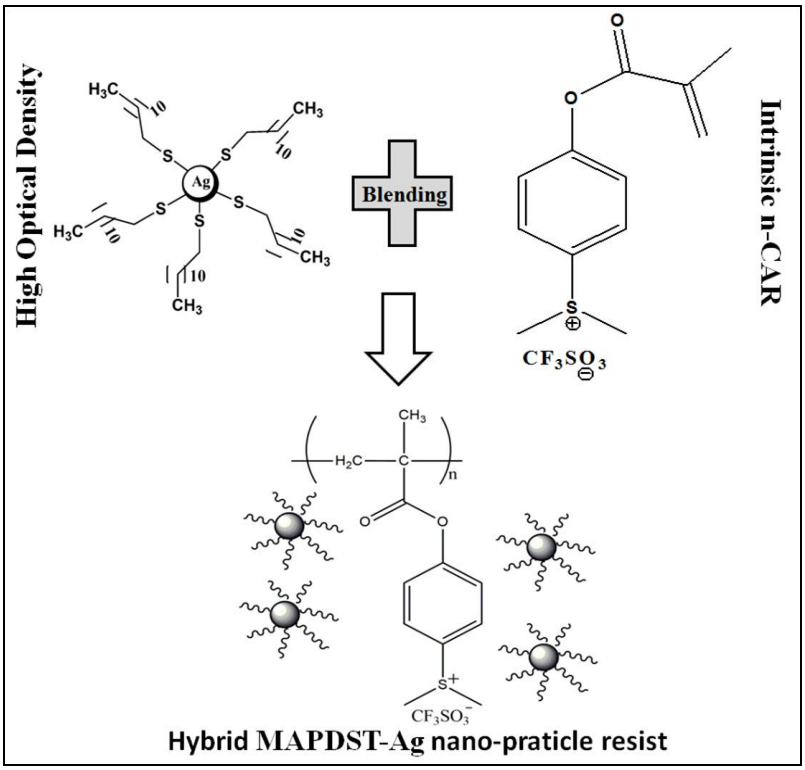


Figure 2 Schematic synthesis of MAPDST-Ag

2.2 Thin film formation and exposure

The blended resist formulation was filtered with 0.22 µm PTFE filters for 2-3 times which confirms removal of all agglomerated Ag-NP from the resist solution. The filtered solution was spin-coated on RCA cleaned Si (100) wafers. The ~25-30 nm smooth films were achieved at 3000 rpm and prebaked at 90 °C for 90 seconds. Thin films were examined under microscopes and profilometry to analyze the defect prior to the exposure, no dewetting was found inside the thin films even after 1 year of storage.

The coated samples were exposed under charge based lithography tools. The e-beam exposures were done at Raith e-line PLUS tool with 20 kV acceleration, 10 μm aperture and current of 25 pA. The samples with standardized parameters were subjected to the He^+ beam lithography (He^+IBL) exposure with 30kV beam acceleration and 1pA current. With beam current parameter it can be observed that He^+ beam concise lesser nos. of charge interaction than EBL, but has a greater impact on the resist. The exposed samples were post baked at 70 °C for 90 seconds. An aqueous tetramethylammonium hydroxide (TMAH) developer was used to develop the pattern for 150 seconds. A low normality 0.026N, TMAH was used keeping pH ~11.3 for high-resolution pattern realization.[14] Deionized water were used as development stopper, a rigorous rinsing provides the removal of unexposed homopolymer and blends.

2.3 Characterization and analytical modeling

The newly synthesized Ag-NPs were characterized by transmission electron microscopy (TEM form FEI) and direct light scattering (DLS from Malvern Panalytical) spectroscopy. The chemical investigation of the blended resist was performed under x-ray photoelectron spectroscopy (XPS from Thermo Scientific). The effect of Ag-NPs over the photoabsorption of resist formulation was analyzed using ultraviolet-visible absorption microscopy (UV-Vis from PerkinElmer).

The high-resolution patterns were analyzed under atomic force microscopy (AFM) from Bruker icon and Zeiss Orion Helium ion microscope (HIM). The analyzed patterns micrograph was subjected to the SUMMIT® software for analytical analysis of patterns. The power spectral density (PSD) of 3σ variance for LER value has been analyzed for the effect of Ag-NPs over pattern formation using SuMMIT®.[15]

3-RESULTS AND DISCUSSION

3.1 Realization of nanosized Ag nanoparticles

The synthesized nanoparticles were examined for different feed ratios (0.005 to 0.02 Molar) of source slat ($AgNO_3$). To achieve sub ~5 nm uniform particles; the synthesis was established for a homogenous distribution of Ag-NPs. in 5 sets. Figure 3 shows the size distribution of Ag-NPs, figure 3a, and 2b shows the TEM micrograph of synthesized Ag-NPs with an average dimension of ~2.5 nm. The TEM micrograph reveals the crystallized Ag core capped with organic moiety. Similarly, figure 3c shows the particle size distribution of DLS spectra for all 5 sets of samples, DLS spectra suggest most of the sample having NPs of dimension 2-5 nm which suggest the self-assembly of Ag inside the microemulsion. These Thio-dodecane capped Ag-NPs were found DUV active and well soluble in organic solvents such as toluene.

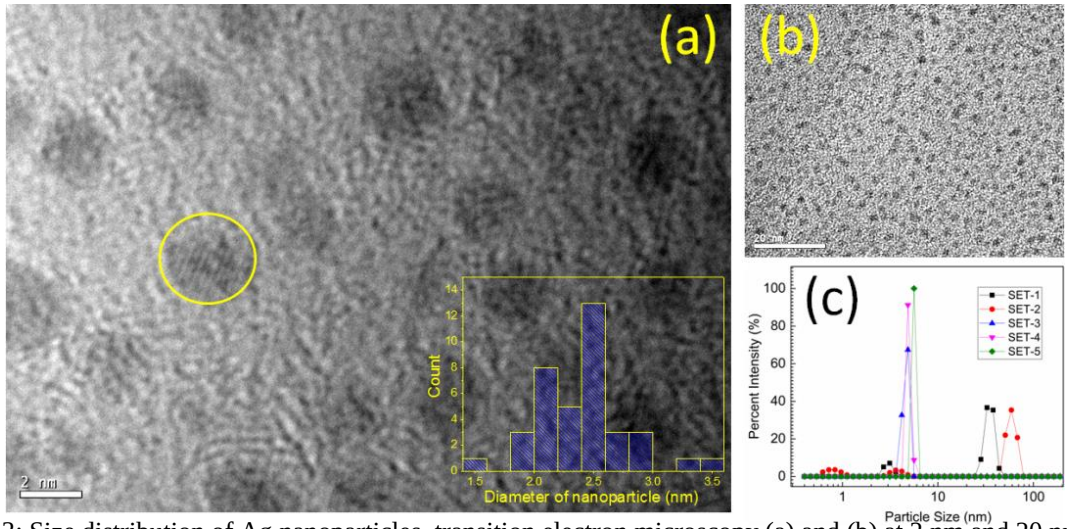
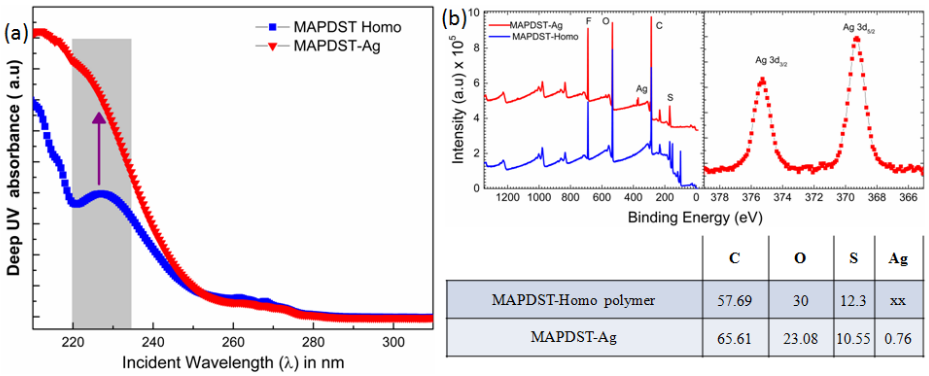


Figure 3: Size distribution of Ag nanoparticles, transition electron microscopy (a) and (b) at 2 nm and 20 nm scale respectively; the size distribution analyzed from 1(a) shows uniform distribution of 2-3 nm in (c)

3.2 Effect of Ag-NP inside the blend resists

The influence of Ag-NPs has been analyzed under UV-Visible absorption through this photoresist solution. Figure 4a shows the enhancement in absorption due to Ag-NPs due to which suggest a significant contribution of Ag-NP in UV absorbance. The <5 nm Ag-NPs provide plasmonic resonance for electromagnetic irradiation (photonic or mobile charge), these Ag-NPs interact with EM irradiations and dissociative electron excitation take place. The generated multiple excitons transfer the energy to the nearby polymers (MAPDST) and further enhance the sensitivity of resist.[16] It also confirms that the Ag-NPs have absorbance bellow λ<300 nm, which could be due to more physical confinement of Ag and due to capping organic ligands (thio-dodecane). Similarly, Figure 4b shows the chemical distribution of MAPDST-Homopolymer and Ag-NPs blended MAPDST-Ag. The chemical distribution shows an increased carbon percentage inside the matrix explaining the availability of carbon at the periphery of the Ag core. These carbon moieties provide a good desolation post blending process rather than just composite formation. Ag core binding energy reveals the partial Ag^0 state in crystalline form (similarly marked in figure 3a), responsible for plasmon field and photomultiplication.[13]



	C	O	S	Ag
MAPDST-Homo polymer	57.69	30	12.3	xx
MAPDST-Ag	65.61	23.08	10.55	0.76

Figure 4 UV absorbance of MAPDST-Homopolyer and MAPDST-Ag samples (a) Doped Ag shown better absorbance beyond the DUV regime. (b) XPS of MAPDST Homo and MAPDST- Ag; Ag3d core spectra; chemical distribution of the MAPDST-Homo and Ag

3.3 High-resolution pattern generation using e-beam lithography

e-beam lithography has paved the way to the nano-patterning down to sub-15 nm due to confined beam focused over the localized area.[17] Herein, figure 5 shows the patterned resist micrograph for different feature sizes and line spacing. The blended MAPDST-Ag showed pattern generation down to 15 nm line when exposed under 20kV e-beam irradiation. Enhance sensitivity and LER has been calculated for these patterns and found Ag-NPs support to reduce the LER while maintaining the line-sensitivity-roughness (LSR) trade-off. Figure 5 emphasizes the 15 nm L/2S feature which is well resolved with good contrast. The thickness of ~35 nm with ~ 15 nm L/2S resist patterns suggest a great potential for pattern transfer with a high aspect ratio. Small buckling for the same thickness patterns can be removed while reaching the lower thickness and feature node.

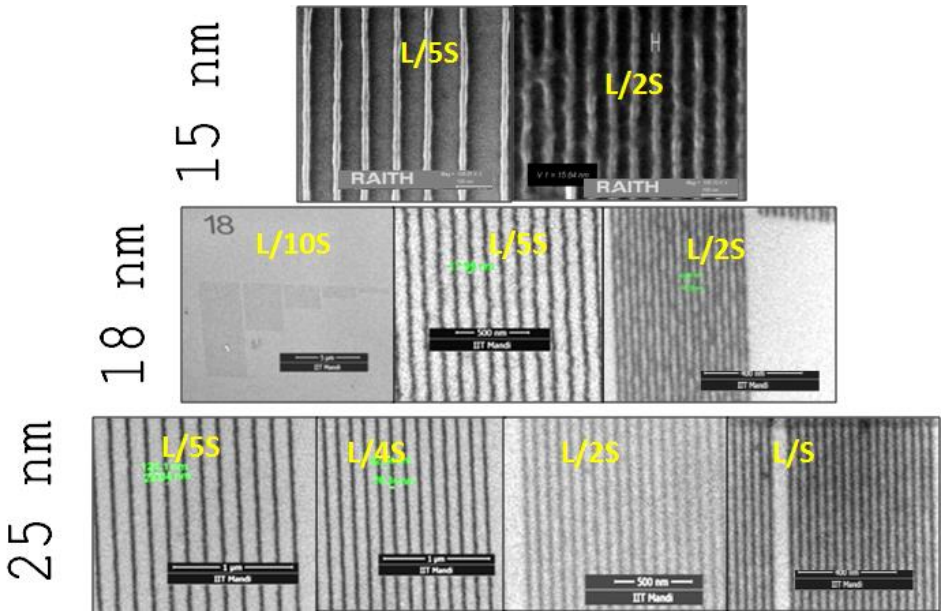


Figure 5 E-beam pattern exposed with dose 200 μC/cm^2 with different feature size and spacing; 15 nm with L/5S and L/2S; 18 nm L/10S, L/5S and L/2S; 25 nm L/5S, L/4S, L/2S, L/S patterns

3.4 High pitch sub-15 nm patterning using He+ ion beam lithography

With optimized parameters of EBL patterns, the MAPDST-Ag was exposed to check the HIBL sensitivity.[18] He^+ ion consisting of high energy found patterning the resist more precisely than the e-beam due to the high stopping power of mobile ion inside the substrate. The MAPDST-Ag found sensitive to the 50.4 μC/cm^2 for 11.5 nm critical dimension (CD) pattern. Figure 6 shows the HIM images of exposed MAPDST-Ag with line-space od 20, 30, 50 and 100 nm. Isolated 15 nm lines (figure 7) show very low LER of 2.12±0.5 nm confirms the candidature of Ag-NPs doped blend-resist to the extreme ultraviolet lithography. The CD pattern 11.5 nm shows minimal breakage/damage on pattern with Ag-NPs incorporation. Improved pattern feature and in He^+IBL pattern shows the scope of ultra-low LER dense pattern. It is also clear that the Ag-NPs dimension has a great role over the pattern quality and defect generation. These results suggest that the Ag-NPs are dissolved inside the MAPDST matrix rather than composite formation as discussed in section 3.1.

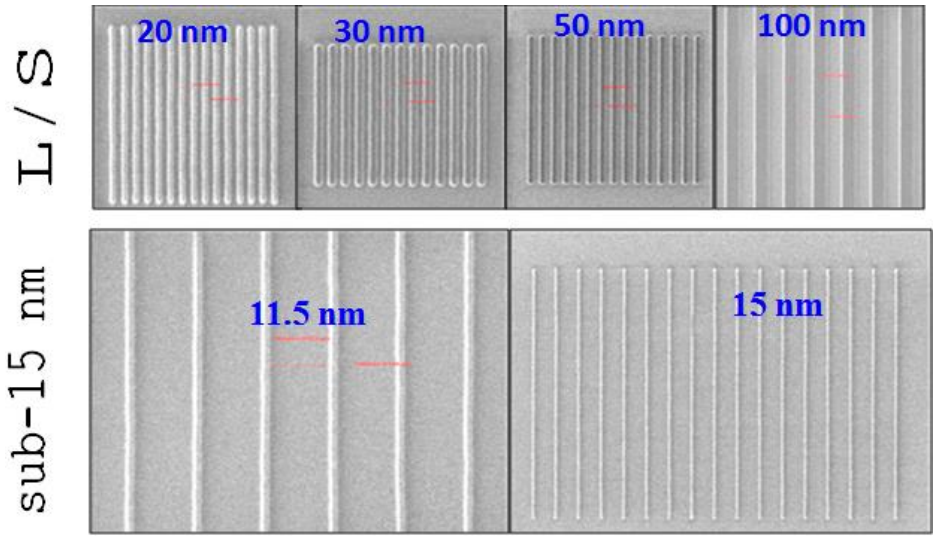


Figure 6 High-resolution patterning with He^+IBL; Line space pattern of 20 nm, 3o0 nm, 50 nm, 100 nm on MAPDST-Ag. Isolated lines of 11.5 nm and 15 nm with ultra-low LER

3.5 Defect-free complex pattern formation

To establish these Ag incorporated MAPDST as resist for NG logics and NEMS/MEMS application, we exposed samples with the different complex structures under EBL with line exposure and curved area patterns. The complex structure containing sharp edge (⌐) for dynamic random access memory (DRAM) and interconnects application; curve, stars and nano-rings gratin divergence for photonic crystal application; nano-boat (I> <I) for single-electron transistor and advanced quantum computation device applications. Figure 7 shows the micrograph of patterned complex structures such as critical dimension patterns with a sharp edge (labeled as 15 nm). These patterns elucidated no defects formation due to Ag-NPs at the sub-15 nm regime also. At corner sharp edges suggest proper development and adequate dose (line patterned – 300 pC/cm). The nano-boats have been exposed with a 2-dimensional matrix array with variable separation and dose to achieve the least feature size, the results elucidate 11.91 nm feature separation for 35 nm thick resist with an area of 70×100 nm^2. Different separation has been achieved when considering the energy dispersion inside the resist with increased dose. Photonic structures have been realized at different dimensions for guiding the different λ photons. These neat complex exposures on MAPDST-Ag resist showed its ability of 3D nanoconfinement (35 nm ×70 nm ×100 nm) with better edge smoothness and sensitivity as earlier reported data.[19]

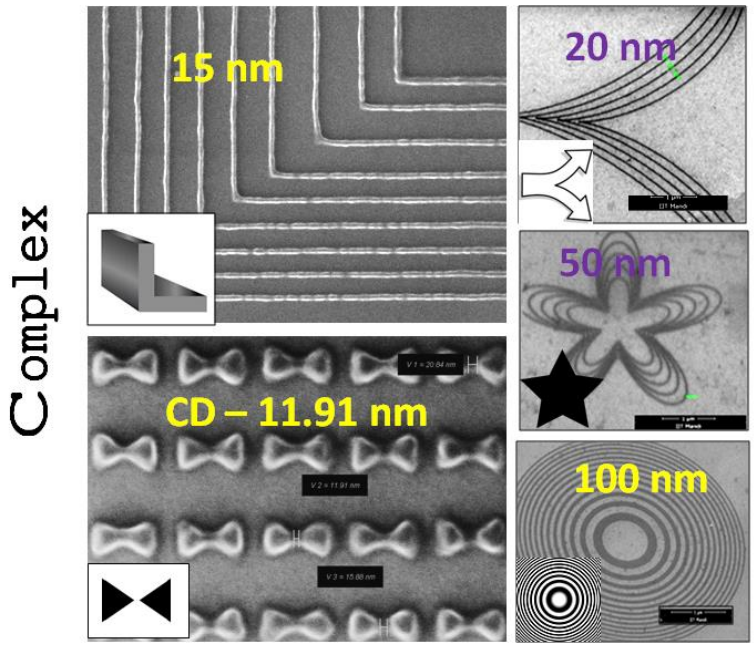


Figure 7 Complex patterning on MAPDST-Ag for different NEMS/MEMS applications; L shape, arrow, star, ring, and nano-boat; the inset shows images of design file patterned over MAPDST-Ag.

3.6 Stability of MAPDST-Ag Blend resists

Considering the issue of PAG in CARs which affects the pattern resolution and edge roughness, we found nanoparticles based photomultiplier can be used to enhance sensitivity and pattern quality (LSR). Therefore the stability and shelf life of blended resist have been analyzed for a year in the form of coated thin film and solution. No agglomeration of crystallization has been observed post shelf life (1 year). The fresh and 1-year-old blended MAPDST-Ag resist were exposed e-beam with a variable dose to find the sensitivity of the system. The exposed old MAPDST-Ag found as sensitive as a fresh one, the critical dose (E_o) found 172 μC/cm^2 and 243 μC/cm^2 for fresh and old samples respectively as shown in figure 8. The constant contrast (γ) in both samples suggest a small effect of humidity and temperature which further increases the sensitivity by 40%.[20] Moreover, the Ag-NPs don't have a direct effect on the stability of resist material which suggests a strong candidature of Ag-NPs as a photomultiplier.

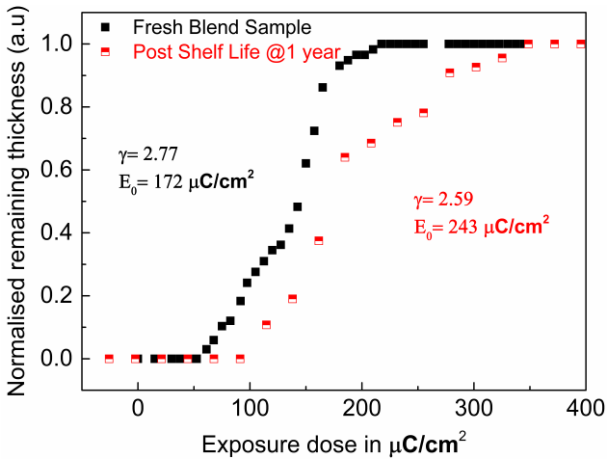


Figure 8 Normalised remaining thickness vs. sensitivity curve for MAPDST-Ag fresh sample and post shelf life (1 year)

4. CONCLUSIONS

In summary, we adapted nanoparticles as photomultiplier to enhance the sensitivity and pattern resolution of synthesized n-CAR. To achieve high resolution down to ~12 nm, the Ag nanoparticles of ~2 nm diameter were synthesized and characterized. Further doping of these photomultipliers have shown the reduction of sensitivity in MAPDST homo-polymer ((4-(methacryloyloxy)phenyl) dimethylsulfonium trifluoromethanesulfonate) exposed patterns under e-beam and helium ion with decent ~12 nm patterns have shown good agreement for its usage for NGL.

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