Organic ice resists: condensed small molecules as spin-free volatile E-beam resists

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Publication date:
2017

Document Version
Publisher's PDF, also known as Version of record

Citation (APA):
Tiddi, W., Lê Thanh, H., Elsukova, A., Beleggia, M., & Han, A. (2017). Organic ice resists: condensed small molecules as spin-free volatile E-beam resists. Abstract from 43rd International conference on Micro and Nano Engineering, Braga, Portugal.

Organic ice resists: condensed small molecules as spin-free volatile e-beam resists

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Keywords: electron-beam lithography, negative organic resist, condensed films

We developed a novel technique using simple organic molecules for lithography applications (Figure 1). The chosen chemical (Figure 2) is condensed from its vapor phase at cryogenic temperature to form a thin frozen layer which demonstrates resist-like capabilities.

We repurposed a Zeiss LEO scanning electron microscope (SEM) by fitting it with an electron-beam lithography (EBL) system, a custom-made liquid nitrogen cryostage, a gas injection system (GIS), and a load-lock for sample exchange and processing (Figure 3). After the substrate cools down, the GIS is used to condense and deposit thin films ranging from <10 nm to >100 nm on the sample surface.

The e-beam exposure defines the desired patterns into this organic ice resist (OIR). The patterned features consist of non-volatile, carbonaceous deposits, while the unexposed ice sublimates away as soon as the sample is brought back to room temperature (Figure 4a-b). A minimum electron dosage is required to successfully create the deposits, as in a negative tone EBL resist (Figure 4c). No spin coating or development steps are needed: the entire lithography process happens in a single tool. The resulting nanopatterns are stable in air and they are compatible with conventional downstream processing and characterization techniques.

Patterns can be extended effortlessly across the entire writing field (0.5 x 0.5 mm^2) which could be completely exposed in less than 3 hours. Limited by the size of our cryostage, OIR layers were uniform across the 2 x 2 cm^2 areas. We could also pattern areas near the sample perimeter, which would suffer from edge-bead formation with spin-coated resist. E.g. Figure 4b shows patterns reaching to the silicon chip edge. During our experiments we also deposited and patterned OIR on top of 5 nm-thick silicon nitride membranes in small samples just 3 mm in diameter. These results are beyond reach of the standard EBL process.

The lithography steps in Figure 1 can be iterated multiple times for more complex lithographic processes. Multilayer stacks (Figure 5) can be obtained by patterning and depositing additional layers in sequence before sublimating the film. Double or triple patterning for high density patterning can be similarly performed, by sublimating each layer before proceeding with a new deposition. In both cases, OIR have the distinct advantage of the sample never having to leave the tool across the cycles. The deposition setup can be easily modified to accommodate multiple precursors, which would allow depositing OIRs with distinct properties at each iteration, giving further versatility to this technique. These represent first steps toward true 3D EBL with functional materials.

Patterned OIR shares similarities with polymeric resists. It was stable when placed in mild acids and bases, and even organic solvents. We used it as an etch mask to transfer the features into the underlying silicon substrate by using an SF6-based ICP process. The resulting finFET-like structures are shown in Figure 6. The simple elemental composition of OIRs means oxygen plasma can easily strip remaining OIR patterns from the sample surface.

Eager to investigate further the potential offered by OIRs, we are now aiming at achieving a deeper understanding of the beam-organic ice interaction, from both a chemical and physical point of view. We are also assessing the ultimate achievable resolution limit, envisioned to be comparable to the molecule size, with targeted experiments in the transmission electron microscope where the spot size can be as small as 0.1 nm. Novel approaches and materials could then be the key to unlock the full potential of this attractive nanopatterning method.

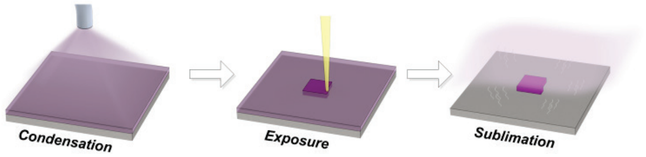

Figure 1. Organic ice resist process: organic vapor is condensed on a cryogenically cooled substrate to form a thin film. The e-beam exposure is used to define the desired features. When the sample goes back to room temperature, the unexposed parts of the ice layers sublimate away and are removed by the tool pumps.

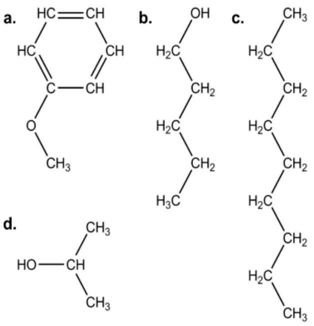

Figure 2. Various molecules tested as OIR: anisole (a), 1-pentanol (b), nonane (c), 2-propanol (d).

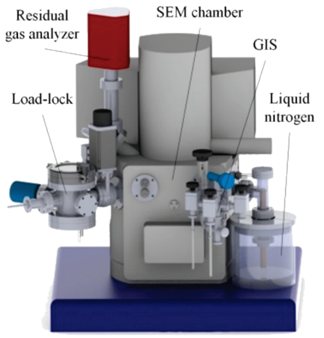

Figure 3. Experimental setup: EBL-ready SEM, modified with custom add-ons for the in-situ cryogenic process.

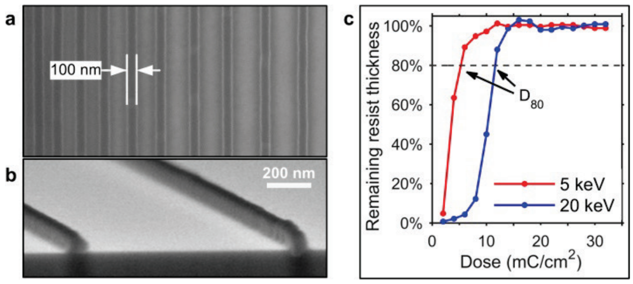

Figure 4. Lithography on OIR: SEM images of patterned nonane OIR lines after sublimation (a, top view, and b, titled view) and contrast curve for nonane at different acceleration voltages (c).

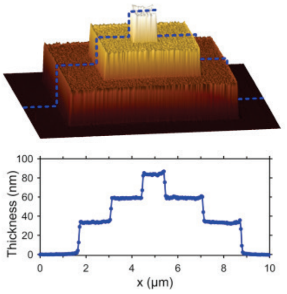

Figure 5. 3D OIR structure: tri-layer stack obtained by three consecutive anisole OIR deposition and patterning sequences.

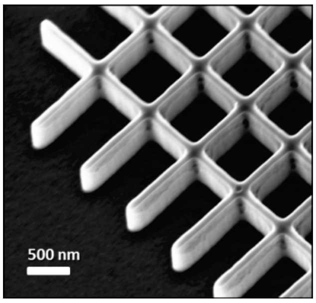

Figure 6. ICP etch of OIR patterns: silicon nanowires obtained using patterned nonane OIR as an etch mask. Resulting features are 400 nm deep.