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Electron-beam patterning organic ice resists

Anna Elsukova, William Tiddi, Anpan Han, and Marco Beleggia

DTU Danchip/Cen, Technical University of Denmark, 2800 Kongens Lyngby, Denmark

e-mail: annaels@dtu.dk

Ice lithography is a method for patterning nanostructures using a thin frozen layer of beam sensitive material (for example, water ice [1] or organic ice resists [2]). The precursor gas is introduced into the lithography instrument and condenses on a cold sample surface (Fig 1 (a)). After electron-beam (e-beam) patterning (Fig 1 (b)), the unexposed resist is removed simply by heating up the substrate to room temperature (Fig 1 (c)). This allows to bypass the spinning, baking and development steps required in conventional e-beam lithography. Thus, the entire process takes place in a single instrument, does not require cleanroom environment or any additional chemicals and is free of residual resist contamination.

In our work we demonstrate the advantages of ice lithography and propose new resist options, while investigating the resolution limits of ice lithography on organic ice resists (OIR).

Most experiments were performed in our custom-built instrument [3], consisting of a modified scanning electron microscope (SEM) equipped with gas injection system (GIS) and a liquid nitrogen cooled cryostage. The sample was cooled down in SEM vacuum and the organic gas was directed through the GIS onto the cold sample surface.

We have shown that frozen hydrocarbon nonane C_9H_{20} and anisole (methoxybenzene, C_7H_8O) can be used as negative-tone resists for nanopatterning. Our results demonstrate that OIR can provide several advantages over other lithography methods. First, we are able to deposit uniform ice layers on non-planar and fragile substrates without adjusting the resist deposition process, as is the case when spin coating is involved. To showcase this advantage we have patterned OIR on a Si_3N_4 membrane with $2.5\ \mu m$ holes for transmission electron microscopy (TEM), and on a small $2 \times 2\ mm^2$ diamond chip used for nitrogen-vacancy centers for quantum technology applications. Second, thick ice layers can be processed ~ 3 orders of magnitude faster than in focused electron induced deposition (FEBID), which makes ice lithography a potentially favorable method for 3D e-beam patterning. These results were recently published in *Nano Letters* [2].

Finally, we demonstrate ice lithography capabilities for patterning structures on a sub-10 nm scale. To this purpose, we have used an environmental transmission electron microscope (ETEM) equipped with a built-in gas cell and cryo-holder to introduce OIR gas precursors to the microscope and condense a $\sim 10\ nm$ thick ice layer on a Si_3N_4 TEM membrane. Besides nonane, we have patterned other hydrocarbons such as octane C_8H_{18} , undecane $C_{11}H_{24}$ and tetradecane $C_{14}H_{30}$. Fig. 2 shows patterned undecane lines with average linewidth $8 \pm 1\ nm$. As hydrocarbons consist of a set of short (1-2 nm) carbon chains, we expect to improve further on this result by carefully optimizing experimental conditions, most critically beam current and resulting dose.

[1] Han, A., Kuan, A., Golovchenko, J. A., Branton, D. *Nano Lett.* (2012), 12, 1018–1021.

[2] Tiddi, W., Elsukova, A., Le, H.T., Liu, P., Beleggia, M., and Han, A. *Nano Lett.* (2017), 17 (12), 7886–7891

[3] Tiddi, W., Elsukova, A., Beleggia, M., Han, A. *Microelectron. Eng.* (2017), submitted for publication.

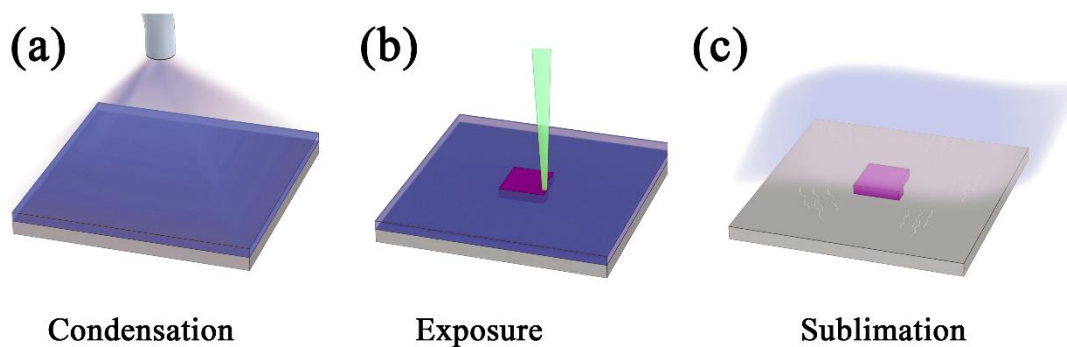


Fig. 1 Organic ice resist patterning. (a) A vapor of organic precursor condenses on a substrate cooled down to cryogenic temperature. (b) During e-beam exposure, the chemical structure of the exposed area is altered resulting in the formation of a non-volatile product. (c) After heating the substrate to a room temperature, the unexposed resist sublimates, while the exposed area remains.

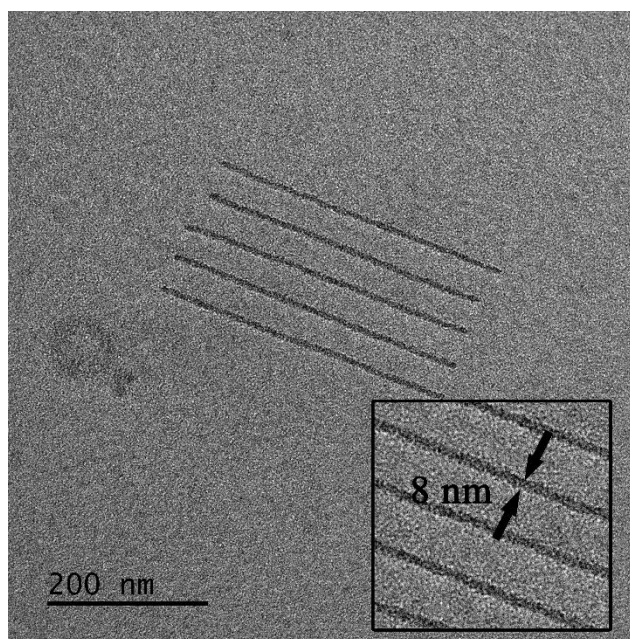


Fig. 2 Patterned undecane ($C_{11}H_{24}$) lines with average line widths of 8 ± 1 nm. Patterning was carried out in an environmental transmission electron microscope operated at 80 kV in scanning mode with an estimated beam diameter of ~ 1 nm and a beam current of ~ 0.53 pA.