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Organic Ice Resist Lithography (OIRL) is a novel one-step method for patterning nanostructures using a thin frozen layer of beam sensitive organic material [1]. Fig. 1 sketches the basic implementation of OIRL. 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 recently published work [1] we have demonstrated the advantages of OIRL over other lithography methods, such as easy handling of fragile and non-planar surfaces and effective fabrication of 3D materials. OIRL was performed in our custom-built modified scanning electron microscope (SEM) [2].

Our goal is now to investigate the resolution performance of OIRL. To this purpose, we have used an environmental transmission electron microscope (ETEM) [3] equipped with a built-in gas cell and cryo-holder (Fig 2) to introduce organic gas precursors to the microscope and condense a ~10 nm thick ice layer on a Si_3N_4 TEM membrane. The optics offered by our ETEM, which is capable of achieving sub-atomic spatial resolution even in presence of gasses in the chamber, allows to minimize the instrumental limitation on the patterning resolution imposed by a broad beam spot or instabilities, which we encountered when using SEM [2].

As precursors, we used simple linear hydrocarbons with different molecular weights (n-octane C_8H_{18}, n-undecane $C_{11}H_{24}$, n-tetradecane $C_{14}H_{30}$). After adjusting deposition and beam exposure, we patterned parallel lines with the same optimal settings for all materials examined. The patterned ices were brought to ambient temperature, and directly imaged in bright field mode. As an example, Fig. 3 shows patterned undecane lines with average linewidth 8±1 nm. Patterning was carried out in ETEM operated at 80 kV in scanning mode with an estimated beam diameter of ~1 nm and a beam current of ~4.0 pA. Our experiments revealed that we could improve the linewidth by decreasing the beam current, as expected, but also by choosing precursors with smaller molecular weight, which is less straightforward to understand. With octane, we achieved stable **4.5 nm** parallel continuous lines, and even **3 nm** lines, which, however, tend to collapse or become discontinuous.

Coupling the observed linewidth vs. molecular weight and linewidth vs. beam current trends with the experimental contrast curves of each precursor (exposed thickness vs. dose), we are now developing a model of exposure mechanism of OIRL. In general, e-beam exposure leads to crosslinking and increase of molecular weight and, as a result, increase of melting/sublimation point of the exposed area. If the degree of crosslinking is sufficient, the exposed ice becomes non-volatile at room temperature. This approach connects electron beam-induced structural transformation of organic ice with its initial molecular structure and resulting patterning feature size and can reproduce semi-quantitatively the observed experimental linewidth trends. The model is an essential step towards developing an understanding of the beam-organic ice interaction and serves as a guideline for selection of organic precursors for OIRL.

[1] W. Tiddi, A. Elsukova, H.T. Le, P. Liu, M. Beleggia, and A. Han, Nano Lett. 17 (2017) 7886–7891.
[2] W. Tiddi, A. Elsukova, M. Beleggia, and A. Han Microelectron. Eng. 192 (2018) 38-43.
[3] http://www.cen.dtu.dk/english/Microscopes/Transmission-Electron-Microscopes/FEI-Titan-E-Cell-80-300-ST-TEM

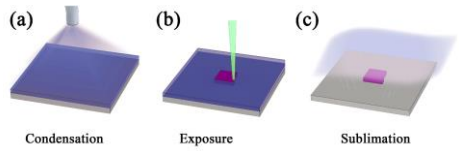

Figure 1. Basic implementation of organic ice lithography. (a) A vapor of organic precursor condenses on a substrate cooled down to cryogenic temperature. (b) During e-beam exposure, the chemical structure of the exposed area is altered resulting in the formation of a non-volatile product. (c) After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

Figure 2. Our environmental transmission electron microscope (ETEM) equipped with built-in gas injection system and gas cell, allowing operation at high pressures.

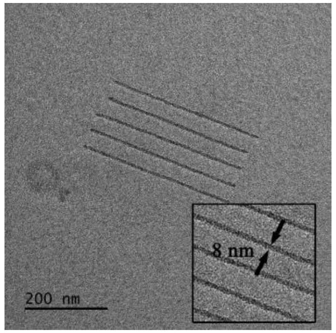

Figure 3. Patterned undecane ($C_{11}H_{24}$) lines with average line widths of 8 ±1 nm.