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Ice Resists for 3D Electron Beam Processing: Instruments in Denmark and China

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3D printing has had a revolutionary impact in mechanical engineering, and downscaling efforts has made significant progress. Most noticeable is two photon polymerization that allows fabrication of 3D structures down to 200 nm [1]. Unfortunately, this technology is inherently slow because of the nature of the unlikely 2 photon reaction which must be enhanced with a well-controlled localized polymerization process. Scalability is also difficult.

For 3D nanoscale patterning, electron beam processing, such as focused electron beam induced deposition (FEBID) is well known and enables beautiful and highly complex nanoscale 3D structures in a few nanometer sizes. Another newer but fundamentally different electron beam processing method towards 3D nanofabrication is ice lithography (IL), where ice serves as a resist for electron beam lithography. While FEBID is an electron-gas-surface interaction, IL is an electron-solid-surface interaction. The first dedicated IL instrument was reported in 2011 [2], which could be used to fabricate 3D nanostructures on fragile freely hanging single-walled carbon nanotubes [3]. Since the IL instrument is not available commercially, it usually takes a very long time to build up a dedicated system.

As collaborating groups, we report the design, material choice, implementation and operation of two IL instruments; one at Technical University of Denmark (DTU), Denmark, and one at Zhejiang University (ZJU), China. They are maybe the only two IL instruments that work well in the world. As shown in Figure 1, the Danchip's research group at DTU has repurposed a Zeiss LEO SEM equipped with a Raith Elphy Quantum EBL module [4,5]. The SEM vacuum chamber was fitted with a liquid nitrogen cooled cryostage for sample cooling and cold finger to condense vacuum contaminants. Through a custom gas injection system (GIS) organic molecule, e.g. anisole, is injected into the SEM chamber and condensed onto the sample. The GIS controls the deposition rate and final ice film thickness. A load-lock allows fast sample transfer and exchange while maintaining cryogenic conditions in the process environment. By stacking nanoscale patterns made in organic ice, 3D nanostructures are created through complementary cyclic condensation, exposure and sublimation processes (Fig. 1). Here, organic ice is usually a negative resist and the patterns after exposure are hardly removed even when placed in mild acids and bases. Additional etching process is necessary to realize pattern transfer for organic ice.

Prof. Qiu's group at ZJU has modified a Zeiss Sigma SEM (Fig. 2). The instrument is similar in principle to that at DTU, which is equipped with a gas injection system, liquid nitrogen cooled cryostages, and a load-lock chamber. In ZJU instrument, water ice is formed onto the sample and acts as a positive resist. It can also deposit metals at cryogenic temperature for pattern transfer. Due to the very low sensitivity of water ice resist, a bridge-like nanostructure could be obtained by modulating the dose during one-time exposure.

References

[1] X. Zhou, Y. Hou, and J. Lin, *A review on the processing accuracy of two-photon polymerization*, AIP Adv. 5, (2015) 030701.

[2] A. Han, J. Chervinsky, D. Branton, and J. Golovchenko, *An ice lithography instrument*, Rev. Sci. Instru. 82, (2011) 065110.

[3] A. Han, A. Kuan, J. Golovchenko, and D. Branton, *Nanopatterning on nonplanar and fragile substrates with ice resists*, Nano Lett. 12, (2012) 1018–1021.

[4] W. Tiddi, A. Elsukova, H. T. Le, P. Liu, M. Beleggia, and A. Han, *Organic Ice Resists*, Nano Lett. 17, (2017) 7886-7891.

[5] W. Tiddi, A. Eluskova, M. Beleggia, and A. Han, *Organic ice resists for 3D electron beamprocessing: instrumentation and operation*, Microelectron. Eng., 192, (2018) 38-43.

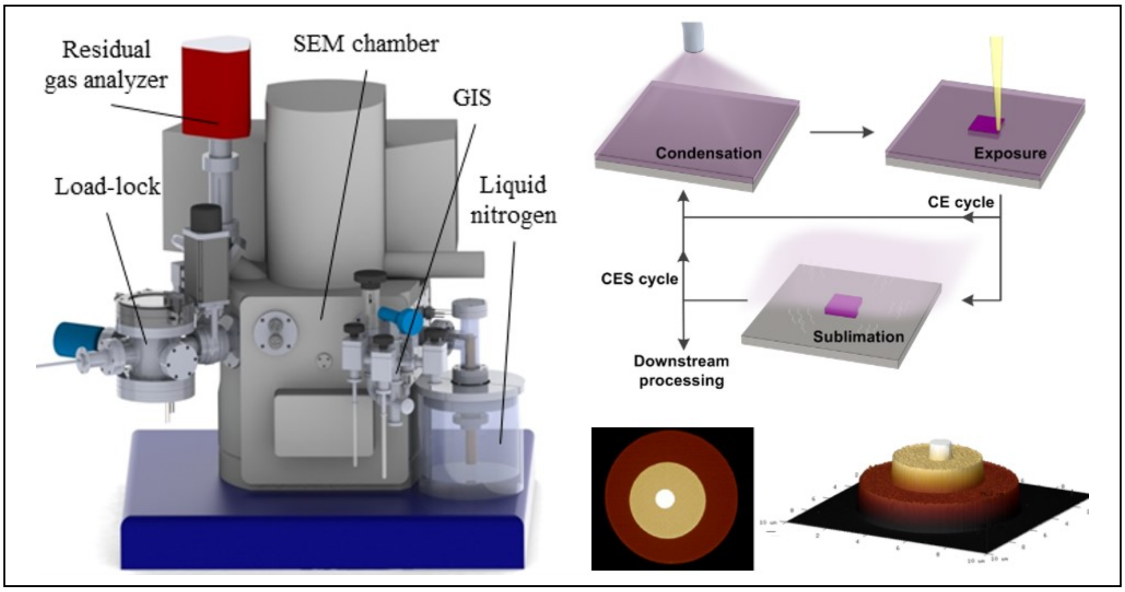

Fig. 1: DTU IL instrument (left) and organic ice resist processing sequence and AFM images of fabricated 3D structures (right).

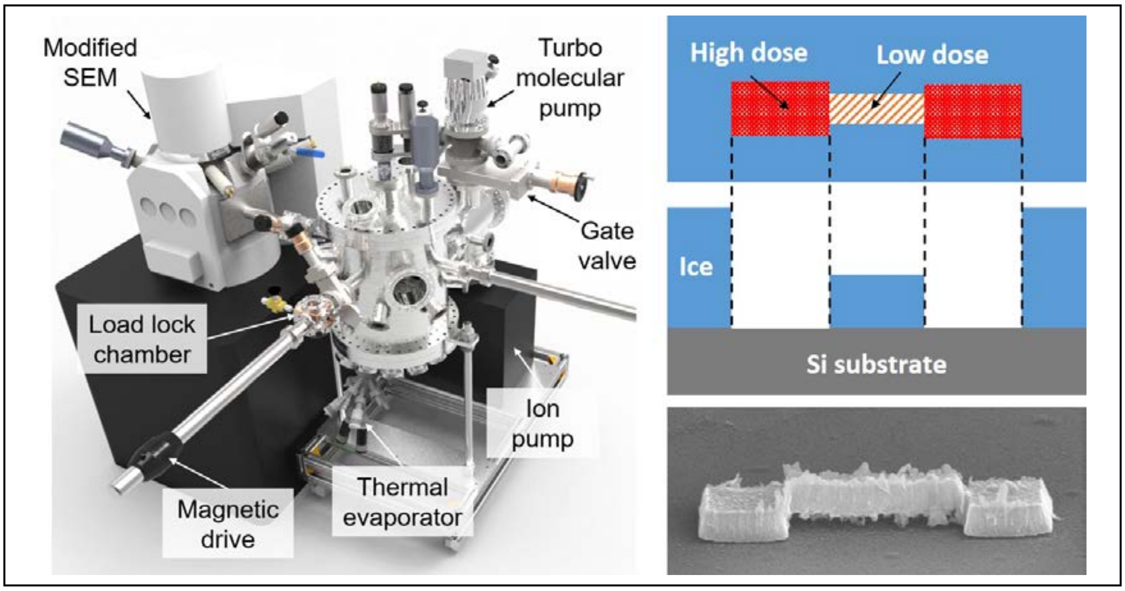

Fig. 2: ZJU IL instrument (left) and water ice resist under dose-modulated exposure and SEM image of fabricated 3D bridge-like structures (right).