Technical University of Denmark

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Publication date:
2016

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Advances in Ice Lithography in Denmark and China

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Keywords: ice lithography, electron beam lithography, metal deposition, cryogenics, instrument research

Lithography plays a critical role for semiconductor industry: the ability to reliably reproduce high-quality nanometric patterns is a core requirement for next-generation nanodevices. Ice lithography patterns sub-10 nm features: a vapor-deposited film of frozen water acts as a resist for high-resolution e-beam lithography (EBL), followed by a metallization process in a dedicated vacuum chamber for subsequent "melt-off" [1]. Furthermore, this technology's process flow avoids spinning and development steps and complex chemistries, and it enables patterning on non-planar and fragile substrates, which is not compatible with resist spinning and baking [2]. As collaborating groups, we report advances towards development of more advanced ice lithography instruments in Denmark and China. We investigate further the capabilities of this technology and improve its performances for potential applications.

Danchip's research group at Technical University of Denmark (DTU) has repurposed a Zeiss LEO SEM equipped with a Raith Elphy Quantum EBL module (Fig.1). The SEM vacuum chamber was fitted with a liquid nitrogen cooled cryostage for sample cooling and "cold trap" to condense vacuum contaminants (Fig.3). Through a custom gas injection system (GIS) water vapor from hydrated salt 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.

Prof. Qiu's group at Zhejiang University (ZJU) has modified a Zeiss Sigma SEM. More advanced compared to the Harvard design, it is connected to a dedicated UHV thermal and electron beam evaporation system (Fig. 2) that allow deposition of both dielectrics and metals. An ion pump creates vacuum $< 2\times10^{-9}$ mbar. Samples with diameters up to 15 mm can be directly loaded into the system from the loadlock chamber, then transferred to the material evaporation chamber (MEC) through magnetic linear and rotary motion drives. A copper braid connects the MEC stage to a liquid nitrogen dewar for sample cooling during material deposition onto ice masks (Fig. 4). A tungsten filament within the MEC stage can be used to bake the sample and remove ice layer. The inverted MEC stage design may allow for "sublime-off" for extremely delicate nanostructures that are sensitive to liquid air interfacial forces.

Danchip successfully demonstrated patterning of thin ice-films. Ice is condensed onto a substrate maintained by the cryostage at < 160 K. The patterned ice layer is immediately imaged to inspect the resulting patterns without pattern deterioration (Fig.5). At the same time, Qiu's group realized metal deposition and in-situ morphology analysis by SEM. Ag is thermally evaporated onto a silicon substrate at room temperature inside the MEC with a deposition rate of 1 nm/min, as monitored by a quartz crystal thickness monitor. The sample can then be directly transferred into the SEM chamber while in UHV, contamination- and oxidation-free. This makes the instrument a powerful tool for material science study of metal thin-films that would otherwise oxidize in air. In the SEM, 'films' of nominal 3-nm and 10-nm thickness can be seen as discontinuous layers of multifaceted Ag particles (Fig.6).

The full ice lithography setup requires all the components interconnected to the same SEM chamber. The DTU and ZJU groups are collaborating on instrument design, knowledge sharing, student and scientist exchange. Together, we explore and reveal the full potential of ice lithography.

[1] King, G. M., Schürmann, G., Branton, D., & Golovchenko, J. A. (2005), *Nano letters*, 5(6), 1157-1160.
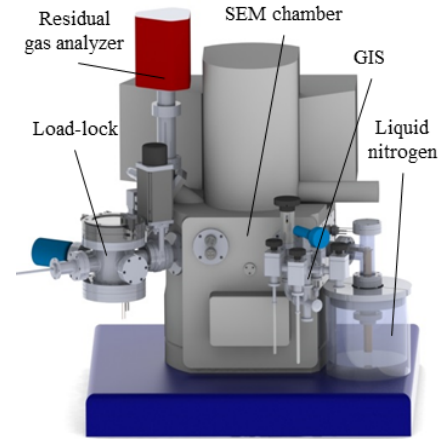[2] Han, A., Kuan, A., Golovchenko, J., & Branton, D. (2012), *Nano letters*, 12(2), 1018-1021.

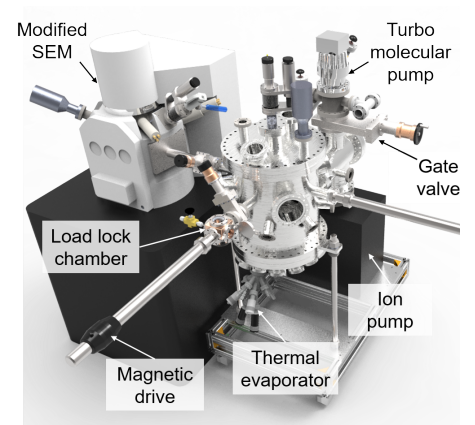Figure 1. Danchip's ice lithography system with its principal components.

Figure 2. Qiu's group's SEM integrated with the custom metal deposition system.
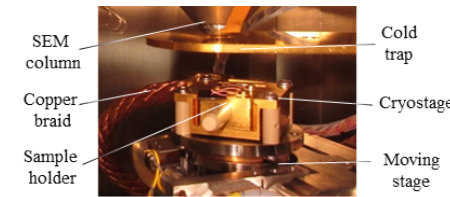

Figure 3. Danchip's SEM cryostage inside the SEM chamber. The sample holder is fully inserted.
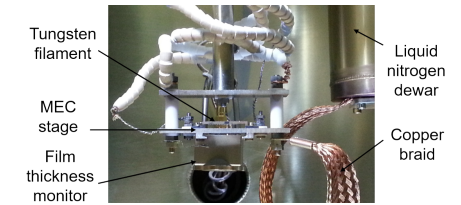

Figure 4. Qiu's group's MEC stage connects a copper braid to a liquid nitrogen dewar.
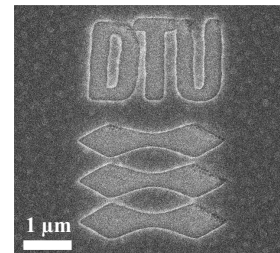

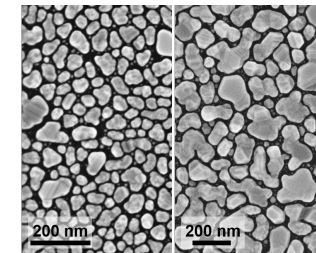Figure 5. EBL patterns on 150 nm thick ice layer.

Figure 6. In-situ SEM image of 3 nm (left) and 10 nm (right) thick silver deposited onto silicon.