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Recent advances in ice lithography for 3D nanofabrication

Ding Zhao[a], Anna Elsukova[a], Marietta Batzer[b], Brendan J. Shields[b], Patrick Maletinsky[b], Marco Beleggia[a],
Anpan Han[c]

[a] DTU Nanolab, Technical University of Denmark, Kgs. Lyngby 2800, Denmark
[b] Department of Physics, University of Basel, Klingelbergstrasse 82, Basel CH-4056, Switzerland
[c] Department of Mechanical Engineering, Technical University of Denmark, Kgs. Lyngby 2800, Denmark
e-mail: dizhao@dtu.dk

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The flexible nanoscale positioning and writing afforded by scanning electron microscopes has prompted the development of a variety of electron-beam-based patterning methods. The most widely used technique is electron-beam lithography (EBL), which has been implemented for patterning micro/nano-structures with advantages of high resolution and reliability. Over the past few years, newer e-beam based methods have demonstrated robust 3D patterning capabilities that are unachievable by electron-beam lithography. Here, we present an emerging ice lithography (IL) technique, and focus on its advantages in 3D nanofabrication.

In IL, a vaporized material is condensed into an ice thin-film when it is sprayed on a substrate held at cryogenic temperature (Fig. 1a). The ice thin-film is then exposed by a focused e-beam to generate nanoscale patterns. This is a subtractive patterning process for water ice, where ice within exposure areas vanishes due to electron-stimulated decomposing of H_2O (Figs. 1b and 1c). Final pattern transfer is realized through metallization. For alkane ice, cross-linking occurs during electron-ice interaction. Only heavily cross-linked molecules within the exposure area are solidified and remain after heating the sample, acting as an additive process (Figs. 1f and 1g). The carbonaceous products are nonvolatile and very stable at ambient conditions. They can be used as masks for etching of the underlying material [1].

Ice lithography research is still in its infancy, and only a few carefully selected ice materials have been studied. Currently, only water ice is capable of acting as a positive-tone lithography resist, and it has been used to fabricate nanostructures with sub-10 nm features [2]. The plots shown in Fig. 2a show the contrast curves for water ice resist. For a 600-nm-thick ice resist exposed by a 20 keV e-beam, it is completely removed with dose over 0.7 C/cm^2. This critical dose for water ice is three orders of magnitude lager than EBL resists; hence, we are able to observe nanostructures under the ice resist through SEM imaging, while it is not possible with a sensitive EBL resist, as SEM imaging would expose the resist. This advantage for 3D nanofabrication is illustrated in Fig. 2b by patterning on a single nanowire, whereupon we can align nanoparticles neatly [3]. The diameter of the nanowire is 160 nm, and the alignment accuracy is less than 50 nm.

Organic ices condensed from simple organic molecules, e.g. alcohols, alkanes, and aromatic hydrocarbons, demonstrate negative-resist-like capabilities. The linewidth of organic ice resist (OIR) could be significantly reduced to less than 5 nm using an e-beam with a small diameter provided by a scanning transmission electron microscope [4], which is in pair with the smallest features made in EBL resists [5]. As illustrated in Fig. 3a, both tiny beam current and short molecular length of OIR contribute to decreasing the line width achieved in IL.

For the first time, we demonstrate the benefits of IL using OIR for 3D quantum sensing devices. After depositing an Al thin-film on premade diamond frustum, we first fabricate an OIR nanodisc on the top. Then, Al nanoparticles covered by OIR can be realized through HF etching (Fig. 3b), which will be used as hard masks for subsequent plasma etching for realization of diamond NV nanoprobes.

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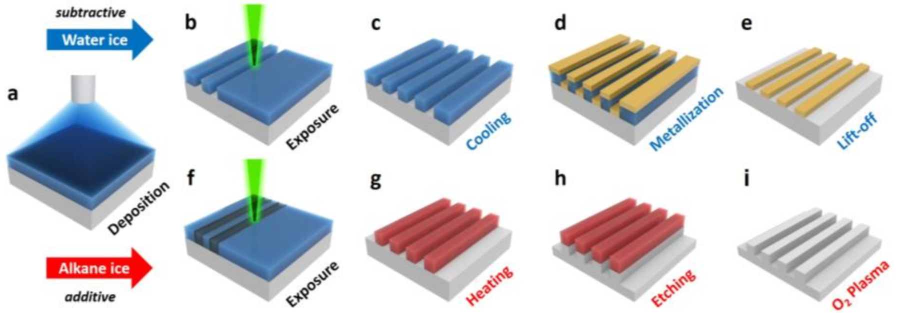

Figure 1. IL Process flow. (a) Vaporized material is frozen onto the cold sample to form a layer of ice resist. (b,f) Focused e-beam exposes ice resist for patterning. For water ice, the exposed ice vanishes (c) and pattern transfer is realized after metallization (d) and lift-off (e). For alkane ice, only the exposed areas remain after heating (g) and pattern transfer is finished after etching (h) and oxygen plasma (i).

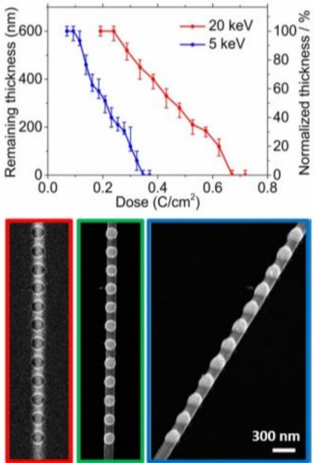

Figure 2. (a) Contrast curves with error bars for water ice resist. (b) Metallic nanoparticle arrays on a single Ag nanowire. SEM images with red and green frames show the nanowire after e-beam exposure and metallization, respectively.

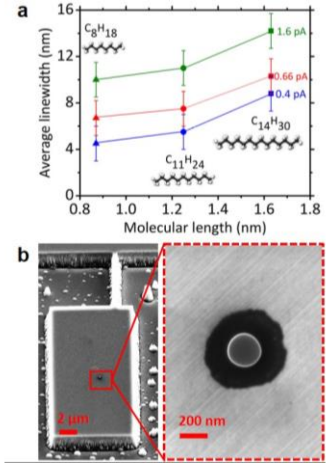

Figure 3. (a) Relationships among linewidth of patterned structure, beam current of TEM, and molecular length of ice resist. (b) Al nanoparticles covered by OIR on diamond frustum.