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# **Three-dimensional Electron Beam Lithography Using Ice Resists**

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**Abstract** Three-dimensional (3D) nanofabrication techniques are of paramount importance in nanoscience and nanotechnology. Here we propose a 3D nanofabrication method based on electron beam lithography using ice resists (iEBL) and fabricate 3D nanostructures by stacking layered structures and dose-modulated exposing, respectively. The whole process of 3D nanofabrication is realized in one vacuum system by skipping spin-coating and developing steps required for commonly used resists. This needs much less processing steps and is contamination-free as compared to conventional methods.

#### **1. Introduction**

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. Unfortunately, this technology is inherently slow because of the nature of the unlikely 2 photon reaction which must be enhanced with a wellcontrolled localized polymerization process.

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 e-beam processing method towards 3D nanofabrication is called ice lithography, where ice serves as a resist for electron beam lithography. The first dedicated iEBL instrument was reported in 2011 [1], which could be used to fabricate nanostructures on fragile freely hanging single-walled carbon nanotubes [2].

## 2. Instrumentation and Results

As collaborating groups, we have respectively one iEBL instrument at Technical University of Denmark (DTU), Denmark, and one at Zhejiang University (ZJU), China. They are probably the only two iEBL instruments that work well in the world. As shown in Figure 1, the research group at ZJU has repurposed a modified Zeiss Sigma field emission scanning microscope (SEM) equipped with a liquid-nitrogen dewar, a water vapor injector, an airlock chamber and a metal deposition chamber (MDC) [3]. In SEM, the sample holder is fixed on the cryo-stage, which is cooled down through a copper braid connected to the liquid-nitrogen dewar. The cold finger, which is also connected to the liquid-nitrogen

dewar, can absorb the vacuum contaminants by cryosorption. A similar cooling device including a cryostage and a liquid-nitrogen dewar is also installed in MDC. The sample holder can be transferred between two cryo-stage though magnetic drives.



Fig. 1. (a) and (b) Illustration of dedicated system at Zhejiang University. (c) Process of EBL using water ice resists.

3D nanostructures can be easily fabricated by iEBL through stacking layered structures. The process flow of a stepped pyramid is shown in Figure 2. The iEBL processes, including ice forming, e-beam patterning, and metal deposition, are repeated three times. At each time, the thicknesses of ice resist and Ag deposit are maintained 300 nm and 60 nm, respectively. In situ SEM images of the first ice layer on a silicon substrate before and after 20 keV e-beam patterning are shown in Figures 2a and 2b, respectively. Ag film is subsequently

deposited (Figure 2c), and a Ag stepped pyramid surrounded by ice/Ag multilayers is achieved by repeating above mentioned processes illustrated in Figures 2d-2i. Figures 2j-2l show SEM images and an atomic force microscope (AFM) line scan of the 3D nanostructure after lift-off. It is an extremely tedious process for fabricating such 3D pyramidal nanostructure by standard EBL, where at least 19 processing steps and 8 load-unload operations (in and out of the vacuum system) of the sample are required. While for iEBL, only 10 processing steps are needed here, and all in the same vacuum system except the final lift-off step. Overall, only single load-unload operation and one-off lift-off step are performed during iEBL, regardless of how many layers are fabricated. This technique effectively reduces possible contamination to the sample and time consumption caused by repetitive pumping and venting of the vacuum chamber.



Fig. 2. 3D pyramidal nanostructure fabricated by iEBL and in situ cryogenic SEM imaging. All scale bars are 1  $\mu$ m.

Due to the particular interaction between e-beam and water ice, it is possible to remove only the top part of ice resist within the exposure area during iEBL, meanwhile, the bottom part survives. This paves the way for iEBL to fabricate another kind of 3D nanostructure by carefully designing the dose distribution in the layout. The basic idea is shown in Figure 3a, where T-shape cross-section appears in the ice resist after a single exposure step. The thickness of resist after exposure is controlled by e-beam dose (Figure 3b), which is similar to the gray-scale lithography method. A 3D mushroom-shaped Ag nanostructure is realized after metal deposition and liftoff (Figure 3c), where a top-layer disk with 3-um diameter is supported on a 2 µm bottom-layer pillar with 170 nm height. In the same way, a bridge-shaped Ag nanostructure with a height of 250 nm, a span of 1.8 µm and a width of 300 nm, is fabricated (Figure 3d).



Fig. 3. 3D mushroom-shaped and bridge-shaped Ag nanostructures fabricated by iEBL. All scale bars are 1 µm.

In summary, we have developed a 3D nanofabrication method using water ice. As a modified technique based on EBL, iEBL can hardly fabricate arbitrary structures, but it works certainly better than conventional EBL in position registration, especially for the structures need overlayer exposure. Two types of 3D nanostructures are realized to demonstrate features of this approach. The iEBL technique needs much fewer processing steps and is almost contamination-free compared to conventional EBL techniques for 3D nanofabrication. It shows great potential in fabrication of complicated 3D nanodevices for almost all applications.

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