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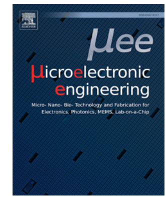
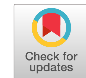


Research paper

Organic ice resists for 3D electron-beam processing: Instrumentation and operation



William Tiddi, Anna Elsukova, Marco Beleggia, Anpan Han *

DTU Danchip/Cen, Technical University of Denmark, Ørsteds Plads, Building 347, 2800 Kgs. Lyngby, Denmark

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ABSTRACT

Organic vapors condensed into thin layers of ice on the surface of a cold substrate are exposed with an electron beam to create resist patterns for lithography applications. The entire spin- and development-free lithography process requires a single custom instrument. We report the design, material choice, implementation and operation of this apparatus. It is based on a scanning electron microscope fitted with an electron beam control system that is normally used for electron beam lithography in a multi-user open-access laboratory. The microscope was also equipped with a gas injection system, a liquid nitrogen cooled cryostage, a temperature control system, and a load-lock. Three steps are required to initialize the apparatus for organic ice resist processing, and two steps are required to restore the apparatus for routine multi-user operations. Five steps are needed to create organic ice resist patterns. Finally, by stacking nanoscale patterns made in organic ice we created 3D structures using two complementary cyclic condense, expose and sublimate processes.

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1. Introduction

Electron beam lithography (EBL) is used to create patterns for the fabrication of nanodevices. The EBL process for a negative-tone resist consist of four steps: i) resist spin-coating of the sample, ii) sample baking to remove the resist solvents, iii) exposing resist to a focused beam of electrons that locally modifies the resist chemistry, iv) immersing the sample with exposed resist into a developer that selectively dissolves the unexposed resist, while the exposed areas remain. Other complementary e-beam based patterning methods are focused electron beam induced deposition (FEBID) [1], ice lithography (IL) [2–4], and e-beam processing with organic ice resists (OIR) [5]. In FEBID, a precursor gas is injected near the sample held in high vacuum; the precursor gas molecules adsorb onto the sample surface, and the e-beam electrons react with them; as a result, a non-volatile deposit is formed. In IL, the sample is cooled down to 110 K so that it condenses injected water vapor to a layer of water ice coating the sample. Then, the e-beam is used to locally remove the ice to define patterns. These patterns in the water ice can then be transferred into metal patterns by an in-situ cryogenic metal deposition and "lift-off" process. Instead of using water ice, Balhke et al. showed that carbon dioxide ice was compatible with large area processing of organic thin-film semiconductors, which is challenging with established lithography methods [6]. In e-beam processing of OIR [5], organic vapors are frozen to ice layers on a cold sample, and the e-beam is used to expose the organic ice. Examples of organic vapors are isopropanol, nonane, anisole. After ice sublimation, a non-volatile product is found in the exposed areas. This allows OIR to be used as negative-tone EBL resists for making nanodevices. Other advantages of OIR are e.g. 3D lithography capabilities, patterning on very fragile non-planar samples, plasma etch selectivity comparable to photoresists, several orders of magnitude faster than FEBID, and the user is not exposed to any chemicals [5]. However, it is several orders of magnitude slower than EBL. In this report, we describe the apparatus for processing OIR, operation of the apparatus, and two complementary processing methods for the fabrication of 3D OIR structures.

2. The apparatus

Our OIR instrument (Fig. 1), inspired by the ice lithography system [7], is based on a LEO SEM from Zeiss, which is equipped with an e-beam lithography system (ELPHY Quantum from Raith Gmbh, Germany). The SEM performance was evaluated using a gold on carbon sample, and the stage vibrations are about 30 nm, which made the system only suitable for EBL patterning of structures larger than 100 nm. The SEM base pressure is $5 \cdot 10^{-6}$ mbar. On the exterior of the OIR instrument, we added a load-lock chamber for sample transfer and warm-up. Also visible on the instrument exterior is the gas injection system (GIS), which is a manifold assembly consisting of a UHV leak valve and 3 HV angle valves (VAT, Switzerland). When all valves are shut, a cell with fixed volume containing gas is formed. The gas pressure in the cell is measured using a convection enhanced Pirani gauge (Kurt J. Lesker, USA). For the GIS, all fittings and vacuum parts are of HV type. A residual gas analyzer (RGA) or mass spectrometer (Pfeiffer Vacuum,

* Corresponding author.
E-mail address: anph@dtu.dk (A. Han).

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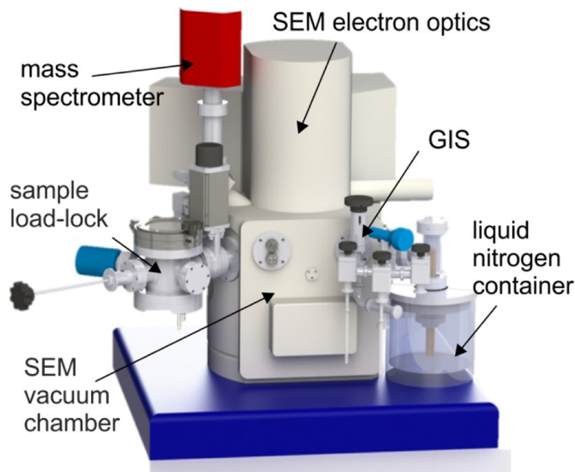

Fig. 1. OIR instrument. The Zeiss SEM is modified with custom-made assemblies to accommodate the cryogenic operation and gas inlet needed to condense the organic vapors inside the chamber. Load-lock is added for rapid sample exchange and ice sublimation. The mass spectrometer monitors the SEM vacuum.

Germany) is installed to monitor the SEM vacuum quality. The liquid nitrogen (LN2) for cooling the cold finger and cryostage is kept in a standard 3 l LN2 vacuum flask. The flask is placed on the SEM table, which is vibration insulated from the SEM vacuum chamber and electron optics.

Inside the SEM chamber, we added a cold finger, a cryostage, and a stainless tube that is connected to the GIS, which is on the outside the SEM. The cold finger and cryostage is mounted onto the same vacuum flange (Fig. 2). The cold finger is 2.5-mm-thick, up to 50-mm-wide and 340-mm-long. It is mechanically clamped to a copper rod feedthrough, whose ambient end is immersed into LN2. For vibration and thermal insulation, the copper rod is silver-soldered onto a stainless steel flexible bellow. The cold finger is mechanically clamped to a flexible copper braid whose other end is clamped to the cryostage. Hence, the cold finger and the cryostage are cooled with the same LN2 source. The temperature increases along the cold path due to thermal losses. This arrangement is different from the ice lithography instrument, in which the cryostage and cold finger are mounted onto two separate flanges and cooled by separate LN2 sources, which enables separate temperature control. Our solution is compact and suits microscopes with fewer ports than the JEOL 7001F SEM used for IL [7]. The cold finger temperature can be adjusted with a 1/8 in. diameter cartridge heater (Firerod, Watlow, USA). The cryostage temperature is regulated independently with an additional heater. The temperature feedback control system is custom built using standard temperature control parts including a low noise linear power supply. Switched mode power supplies must be avoided, because they will generate excessive ripple electromagnetic fields, which would distort the SEM imaging and deteriorate lithography performance. The cold finger e-beam aperture is aligned with the electron optics, and the cold finger is then secured to the cryosystems flange with the alignment fixture made of thermal insulating stainless steel and PEEK washers. The cold finger is about 20 K colder than the cryostage, which allows efficient trapping of undesired condensable gasses. Using the RGA, the water partial pressure is measured to 10^{-7} mbar which is 10 times higher than the IL instrument.

Thermal insulation of the IL instrument relied on three pins centering the cryostage. From experience, this design occasionally caused mechanical instability during sample transfer. To improve the mechanical stability, we adopted a different design: our cryostage is mounted onto the SEM stage dove tail, and it can be easily removed (Fig. 3). The cold part of the cryostage, which is connected to the copper braid via a clamping ring, is thermally insulated from the warm part of the cryostage via 4 pillars made in PEEK. The warm part of the cryostage is an adapted SEM dove tail. On the SEM dovetail is also mounted a Faraday cup for e-beam current measurement.

We carefully tested the performance of the cryosystems. Two hours after immersing the copper rod in LN2, the cold finger and cryostage cooled to 110 K and 130 K, respectively. The lowest possible temperature of the cryostage is 120 K, which is 10 K warmer than the IL instrument. The cooling rate is similar to the IL instrument. The PEEK pillar design proved to be significantly better regarding to mechanical stability and robustness during operation. The warm part of the cryostage is 9 K lower than ambient temperature, which did not have any impact on the SEM stage movement.

The sample transfer mechanism is of the bayonet type, which is similar to the IL instrument. The sample holder surface measures 25 $\times$ 20 mm^2, and up to 3 samples can be clamped onto it. The sample clips were standard SEM clips. Another dedicated sample holder allows 90 degree tilting of two samples. The sample holders have a hollow design that allows them to be held firmly down by a cantilever spring (Fig. 3b). The hollow design also reduces thermal mass for a faster sample holder cool down.

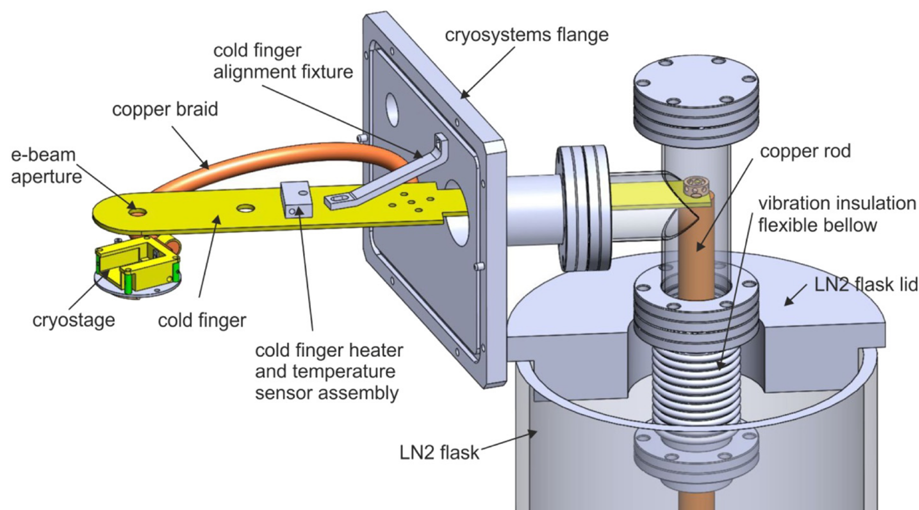

Fig. 2. Cryosystems flange and assembly. The cold path starts from the copper rod that is immersed in liquid nitrogen in the dewar. The cold finger is attached to the vacuum end of the copper rod, and the cryostage is connected to the cold finger through a copper braid.

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