Focused Electron Beam Induced Deposition of Copper from Aqueous Solutions in Micro-wells

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Focused electron beam induced deposition (FEBID) is a nano-scale patterning technique that allows the direct deposition of functional materials. This process has a variety of applications such as nano-scale rapid prototyping, lithographic mask and imprint template repair, interconnection to chemically synthesized nanostructures and circuit editing. Liquid-phase processes, in which deposition occurs at the interface between a substrate and a bulk liquid, are being investigated because they have potential for higher purity deposits, faster processing rates, simpler precursors, and access to new materials. However, for practical applications, one prefers to conduct liquid-phase processes in an environmental scanning electron microscope (ESEM) without using a sealed liquid cell. Thus, controlling thin liquid layers in partial vacuum becomes a serious challenge.

Here we increase control over the liquid layer by creating micro-wells on the substrate. The wells were patterned using photolithography and SU-8 resist, as shown in Figure 1. The liquid precursor is loaded into the well *ex-situ*, and then rehydrated in the ESEM. A liquid meniscus forms in the well with a thin central region in which high-resolution patterning can be conducted. The wells also isolate different liquid precursors on the same substrate because hydration/condensation occurs only inside the wells, not on top of the SU-8.

We used the microwells to evaluate copper deposition from two aqueous solutions containing copper sulfate ($CuSO_4$). The first solution also contained a surfactant, sodium dodecyl sulfate (SDS), to improve wetting and reduce the liquid layer thickness required to form a stable meniscus. The second solution contained sulfuric acid (H_2SO_4) which also had some stabilizing effect and prevented precipitation of $Cu(OH)_2$. In both cases, a ~25-nm thick copper layer was sputter-coated on the silicon substrate to improve deposit adhesion and eliminate complexity associated with deposition on a dissimilar material. Energy-dispersive x-ray spectroscopy (EDS) was used to estimate the liquid thickness in the target area and to quantify the purity of deposits.

As shown in Figure 2, we deposited dense copper lines from both solutions. Deposits from the acidic solution exhibited lower sulfur contamination and less undesired deposition around the pattern. We also conducted four-point resistivity measurements of copper nanowires deposited from $CuSO_4$:SDS solutions on oxidized silicon substrates with no copper film. An example wire, shown in Figure 3, exhibited resistivity within an order of magnitude of that of bulk copper.

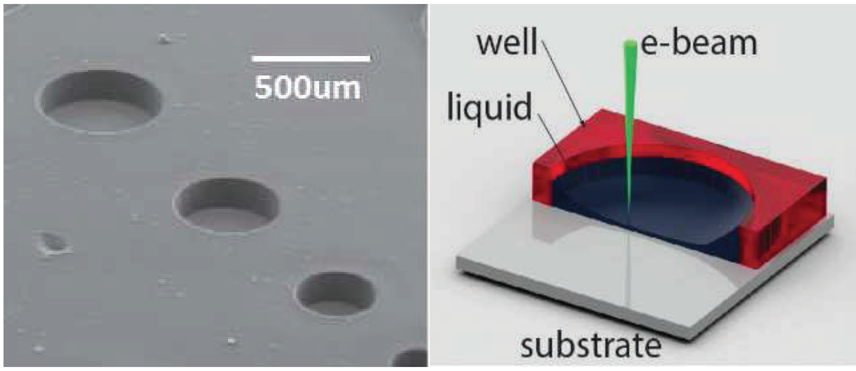

Figure 1: (a) Tilt-view of SU-8 micro-wells on a silicon substrate. (b) Schematic of electron-beam induced deposition through the liquid meniscus inside the well.

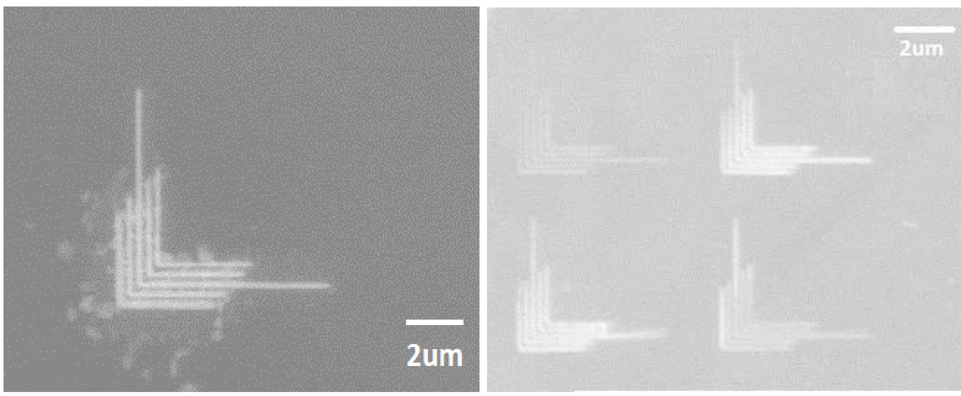

Figure 2: Patterns deposited in micro-wells. (a) 200-nm half pitch nested line deposited from an aqueous solution of $CuSO_4$:SDS and (b) 100-nm half pitch nested lines from $CuSO_4$:H_2SO_4 (aq) at different doses.

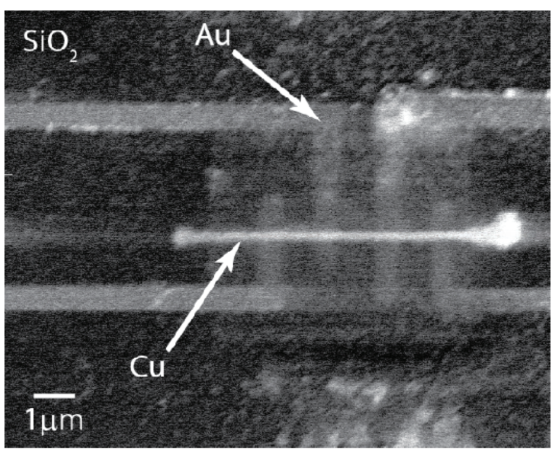

Figure 3: Cu nanowire deposited across a gold four-probe structure for resistivity measurement. Preliminary measurements indicate the resistivity of the deposit is on the order of 10^{-5} Ω·cm compared to a bulk copper resistivity of 2×10^{-6} Ω·cm.