Control of Liquid Film Thickness and Concentration *in-situ* for Focused Electron Beam Induced Deposition from Aqueous Solutions

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Liquid-phase focused electron beam induced processes (LP-FEBIP) are being investigated due to the potential benefits over the gas phase technique. In this method, deposition or etching occurs at the interface between a substrate and a bulk liquid. However, controlling the thin liquid film in partial vacuum are still a serious challenge. Previously, we used shallow microwells to produce a meniscus[1] which yields a thin liquid layer close to the center of the well. Although good copper deposition was achieved, control of the precursor concentration and the film thickness remained challenging due to the ultra-small liquid volume.

Here we increase control over the liquid layer by creating deep micro-wells connected to a shallower well via a channel. The micro capillary effect of the channel results in pulling the liquid solution from the larger reservoir well into the shallow well. The second microwell and the channel have controlled depth suitable for patterning. In this way we expect to provide larger areas appropriate for patterning. Moreover, since the precursor in the channel is connected to the solution in the large well, we can more accurately control the concentration of the solution.

The modified well configuration was created by deep reactive-ion etching into the silicon wafer is shown in Figure 1. The initial precursor was loaded into the 50 µm deep well *ex-situ* using a custom liquid injection system. The solution was further hydrated by controlling the temperature and pressure in the ESEM with a water vapor ambient. As liquid fills the deep well it starts to flow through the channel toward the other well (Figure 2). Both the channel and second microwell have a depth of 3 µm.

We used this modified surface to evaluate copper deposition from aqueous solutions containing copper sulfate ($CuSO_4$).[2] The solution also contained a surfactant, Triton X-100, to improve wetting and provide stable flow in the channel. Having control over the concentration and film thickness enables us to study the effect of refresh time, dose, number of cycles, and concentration on copper deposition as shown in Figure 3.

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[1] Esfandiarpour S., Noubani A., Hastings J. T., EIPBN 2015.
[2] Esfandiarport et al. Nanotechnology 28, no. 12(2017): 125301.

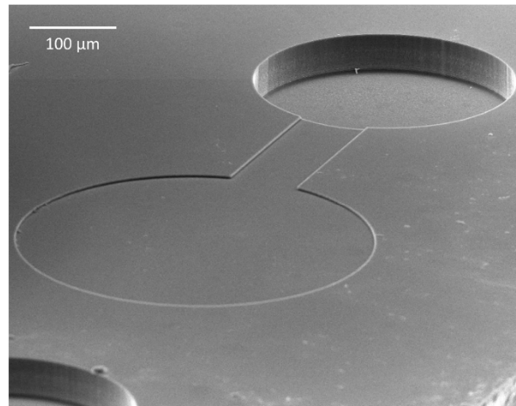

Figure 1: (a) 60° tilt-view of etched micro-wells on a silicon substrate.

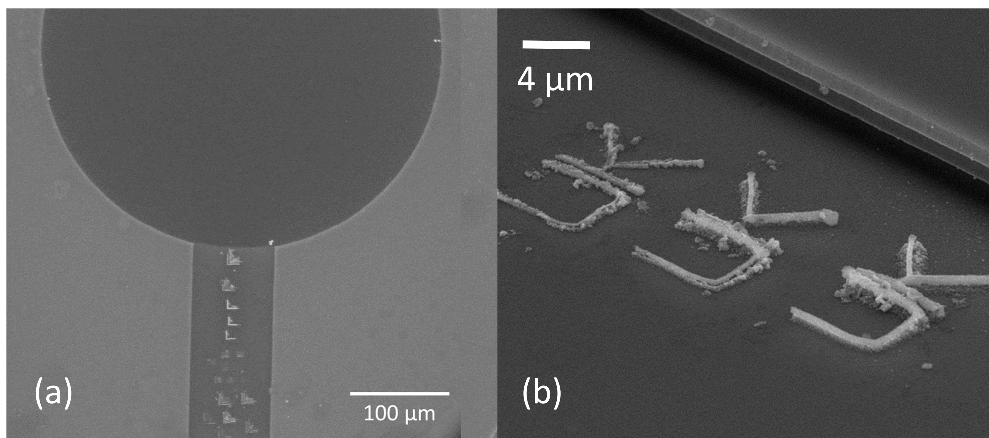

Figure 2: (a) Hydrated substrate and liquid flow from reservoir toward the channel, (b) 60° tilt-view of an arbitrary pattern deposited in the channel with the dose of 5 µC/cm, 10 µC/cm and 15 µC/cm from left to right.

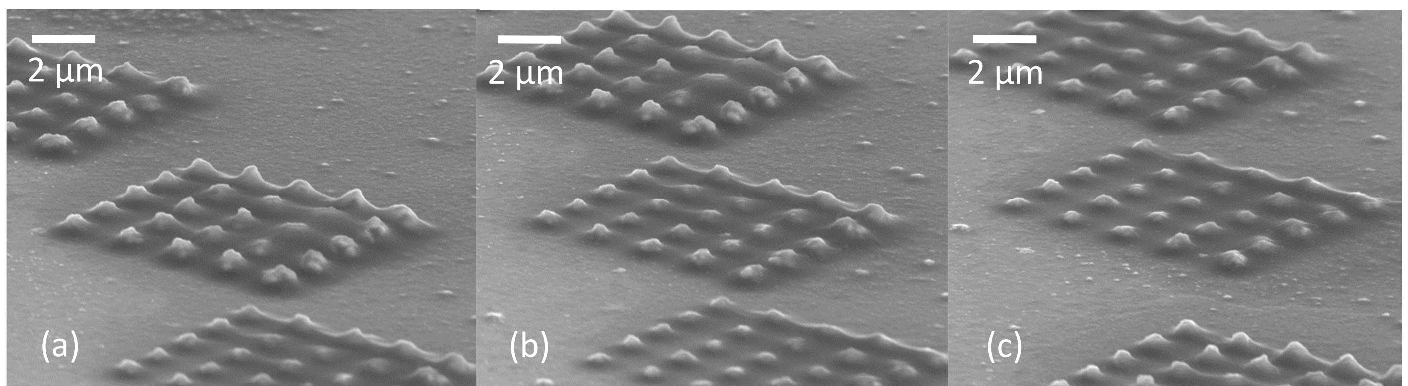

Figure 3: Micrograph of 5 x 5 array of copper dots deposited from CuSO4: H2SO4: Triton X-100 solution with the dose of 10 pC/dot and 20 cycles with refresh time of (a) 200 ms, (b) 100 ms and (c) 50 ms. Larger volume are deposited with longer refresh time.