Condensed Phase Electron Beam-Induced-Deposition: 3D Lithography and Growth Rate Enhancement using Cryogenic Cooling

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Electron beam-induced-deposition (EBID) is a site specific and maskless deposition process where a gaseous precursor adsorbs onto a substrate and is decomposed in the presence of an electron beam [1]. EBID growth rates are typically hindered by mass transport limitations [2] caused by the molecular characteristics of the precursors such as low sticking coefficients, adsorption times and surface diffusion coefficients, and by surface site saturation by precursor adsorbates. Through the use of cryogenic cooling these parameters can be manipulated, allowing for a gaseous precursor to be condensed onto a substrate. Subsequent electron exposure and re-heating of the substrate can be used to generate 2D and 3D nanostructure geometries. The condensed phase or 'cryo' EBID yields growth rates dramatically higher than room temperature EBID, since the exposure rate is reaction rate limited rather than mass transport limited. Complex 3D structures have been realized using single and multilayer processes by controlling condensate thickness and electron ranges in the condensates.

Experiments were performed using an FEI Nova 600 Nanolab dual beam system with a LN_2 cryogenic stage, enabling substrate temperatures of -155±5^{o}C. A standard EBID platinum precursor ($MeCpPtMe_3$) was first condensed onto the cooled substrate using a capillary-style gas injection system (GIS), as shown in Fig. 1A. Condensate thicknesses between 100nm and 3µm were achieved by adjusting the GIS-substrate gap and precursor crucible temperature. Next, gas flow was terminated and the condensate was irradiated with an electron beam to induce precursor decomposition (Fig 1B). When the substrate was returned to room temperature, unreacted precursor desorbed and was removed by the pumping system, while irradiated regions showed clear evidence of successful deposition. The morphology of the deposited material depends on the electron exposure conditions, and exhibits several reproducible, distinct types (Fig. 3), unlike conventional (room temperature) EBID [3].

3D structures were fabricated by single and multilayer deposition methods. Both methods exploit electron penetration ranges to selectively expose regions of the condensate. Fig. 1 shows a multilayer process flow used to create an incorporated gap structure. A first precursor layer was condensed (Fig. 1A) and two regions were exposed to act as anchors to the substrate (Fig. 1B). A second condensate was then created with a thickness selected such that the electron beam could penetrate only to the top of the underlying (first) condensed layer, and was exposed between the two anchors as shown in Fig. 1C. After reheating, the non-exposed precursor desorbs, leaving the exposed regions intact (Fig. 1D). Figure 2 shows a SEM image of a cross-section of the completed structure with the incorporated gap, demonstrating the feasibility of this approach. The cryo-EBID formation mechanism will be discussed in light of these results and the surface morphologies shown in Fig. 3.

References:

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2. **D. A. Smith, J. D. Fowlkes & P. D. Rack. Simulating the effects of surface diffusion on electron beam induced deposition via a three-dimensional Monte Carlo simulation. *J. Vac. Sci. Technol. B* 19, 415704-415714 (2008).**
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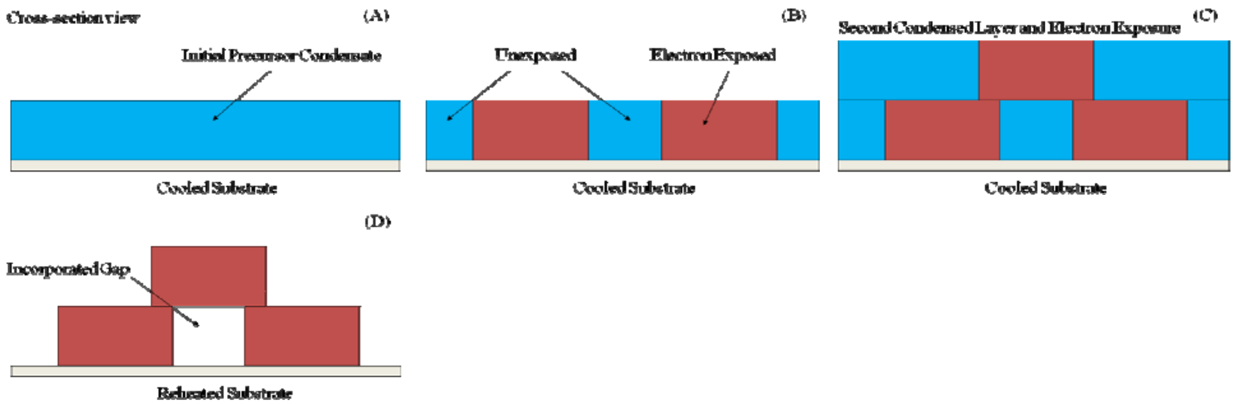

Fig 1. Multilayer process flow for the fabrication of incorporated gap 3D structures. A) A first precursor layer is condensed onto a cooled substrate. B) Two regions are electron irradiated to form posts. C) A second condensate layer is produced and the region between the posts is exposed to cap the posts. D) The sample is reheated to room temperature, causing desorption of non-exposed precursor.

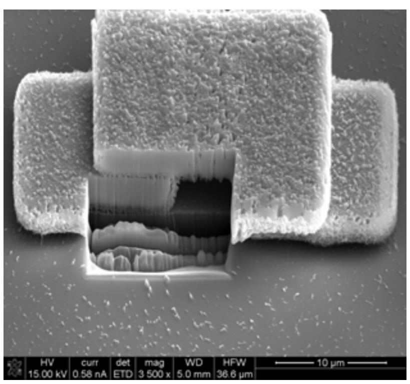

Fig 2. SEM micrograph (45^{o} tilt) showing a Focused Ion Beam (FIB) cross-section of an incorporated gap 3D structure fabricated by the process shown in Fig. 1.

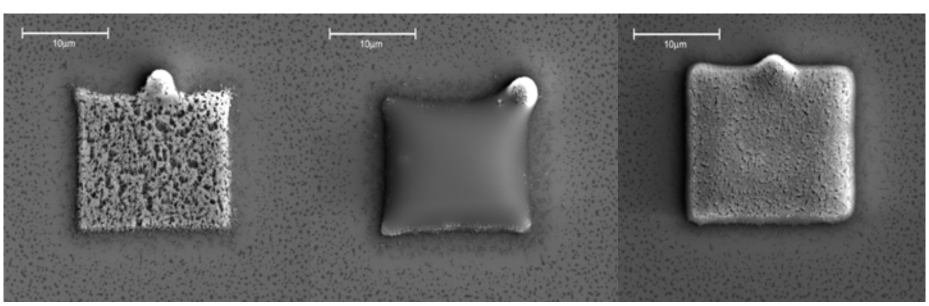

Fig. 3 Changes in surface morphology caused by the electron fluence used for cryo-EBID. Electron exposure was performed using a 15 keV, 0.71 nA beam, and irradiation times of 5 (left), 10 (middle) and 15s (right), yielding a nanoporous mesh, a smooth surface and a rough surface, respectively.