Effect of Precursor Sticking Coefficient on Electron Beam Induced Deposition and Etching.

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Electron beam induced deposition and etching (EBIDE) can be utilized to directly write patterns and fabricate structures with nanometer scale dimensions and positional accuracy. In this process, precursor gases are locally decomposed by a focussed electron probe in a scanning electron microscope (SEM) to produce by-products that selectively deposit or etch nano-structures onto a substrate. The EBIDE reaction rate for a given precursor gas and substrate is determined by the surface coverage of the precursor molecules and the current density of the electron beam. In this work the role of the precursor molecule's sticking coefficient and surface diffusion on EBIDE process have been systematically investigated.

All EBIDE measurements were conducted using a FEI XL30 Environmental scanning electron microscope (SEM) operating between 20 and 30 kV with a short working distance to minimise gas-phase scattering of the primary electron beam. A series of straight chain alkanes (pentane, hexane, heptanes, octane and hexadecane) with increasing carbon chain lengths were used as the reaction gases. Rather than using a traditional injection needle for localised gas delivery, the entire SEM chamber was filled with gas to ensure a uniform surface flux of precursor molecules onto clean Si substrates. The microscope chamber pressure was typically set to 0.2 torr using a separate dedicated gas delivery system consisting of a needle-valve and a MKS Instruments mass-flow controller. An oxygen scrubber was used to clean the SEM chamber prior to each experimental run. The morphology of EBIDE structures were measured using the FEI ESEM and a Zeiss Supra 55VP SEM as well as a Digital Instruments Dimension™ 3100 Atomic Force Microscope (AFM).

The formation of carbonaceous rings with a defocused electron probe suggests that surface diffusion is the dominate transport mechanism of the precursor vapor. No dissimilarity was observed in the size or growth rate of deposits formed using a focussed electron beam with pentane, hexane, heptane and octane. Conversely, comparison of carbonaceous deposits produced with hexadecane ($C_{16}H_{34}$) and hexane (C_6H_{14}) at 20 kV with a 6 nA focussed beam reveal significant differences. Here, the short chain alkane precursor was found to produce larger deposits which take around an order of magnitude longer reach their maximum size. These data confirm that the surface diffusion and sticking co-efficient of the precursor molecules can have a significant affect on EBIDE processing rates.

Under equivalent EBIDE conditions a transition between deposition and etching was observed when the chamber pressure was changed over to high vacuum mode (~ 10^{-6} torr) from variable pressure ESEM mode (0.2 torr) without breaking vacuum. For example, with heptane at 30kV carbonaceous deposits were formed at 0.2 torr whereas switching over to high vacuum produced pits under equivalent conditions. Similar switching between carbon deposition and surface etching has been reported in a H_2O environment and attributed to an electron beam flux mediated process [1]. The hole depth was found to increase with irradiation time to a maximum depth of around 6 nm after 30 sec. Radiolysis induced electron beam hole drilling in SiO2 is well known. However, the pits etched by the beam are significantly deeper than the expected native oxide on Si substrate suggesting that there is also mass-loss from the Si substrate.

References:

[1] Milos Toth, Charlene J. Lobo, Gavin Hartigan and W. Ralph Knowles, *Electron flux controlled switching between electron beam induced etching, and deposition*, Journal of Applied Physics, 101, 054309, 2007