

***In situ* monitoring and control of material growth for high resolution electron beam induced deposition on thin membranes**

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Electron-beam-induced deposition (EBID) is a versatile, direct, and resistless fabrication technique. It has demonstrated the highest resolution presently achieved for lithography with electron beams. Structures with an average size as small as 1.0 nm have been fabricated [1]. EBID has brought to light several kinds of non-linearities occurring at this near-molecular level. For instance, when an array of deposits is created with a fixed dwell time, the amount of mass varies from deposit to deposit. This distribution of masses becomes relatively wider as the dwell time decreases and the deposits contain less material [2]. Another non-linearity is a proximity effect, where the amount of deposited material depends strongly on the geometry of the surface at the irradiated position [3]. In our efforts to improve the understanding and control of the deposition process, we have developed a technique to monitor the growth of deposits *in situ*.

Experiments were performed in an environmental Scanning Transmission Electron Microscope (FEI Tecnai G², minimum probe size 0.2 nm), using W(CO)₆ as a precursor. The structures were deposited on 10 nm thick membranes. The imaging and monitoring of the growth were performed with the annular dark field (ADF) signal (Fig. 1a). This signal has a strong Z-contrast component and the signal strength is assumed to be approximately linearly dependent on deposited mass. Patterning and growth monitoring were executed with scripting software available on the machine. For each beam position that was defined in the pattern, the corresponding position on the sample was irradiated for 10 ms after which the ADF signal value was collected. This sequence of irradiating and collecting data was continued until a preset ADF signal value was reached.

The deposits in Fig. 1b with an average diameter of 2.2 nm were created using this script. The growth curves of some of these dots in Fig. 1c show that the script terminates the growth after the first measured value above the preset threshold (dashed line). It is observed that the growth process is different for each dot. Estimates indicate that the precision of the monitoring technique is in the order of tens of precursor molecules per deposit on average. The procedure has not yet resulted in a narrower distribution of deposited mass, probably because the fine electron beam probes the deposit only partially and not completely (indicated with the grey area in Fig. 1a).

The tilted line in Fig. 2a was deposited with a fixed dwell time per pixel. The line was 160 nm long and tilted over 20 degrees. Since the substrate was a transparent membrane, deposition occurred on both the entrance and exit surface. As the writing of the line proceeded (from left to right), more material was deposited and the height increased. This is due to a proximity effect. The tilted line in Fig. 2b was written with the same pattern, but now the growth was monitored and controlled with the ADF signal. The dynamic growth control compensated for the proximity effect, resulting in a 4.0 nm wide line of uniform height.

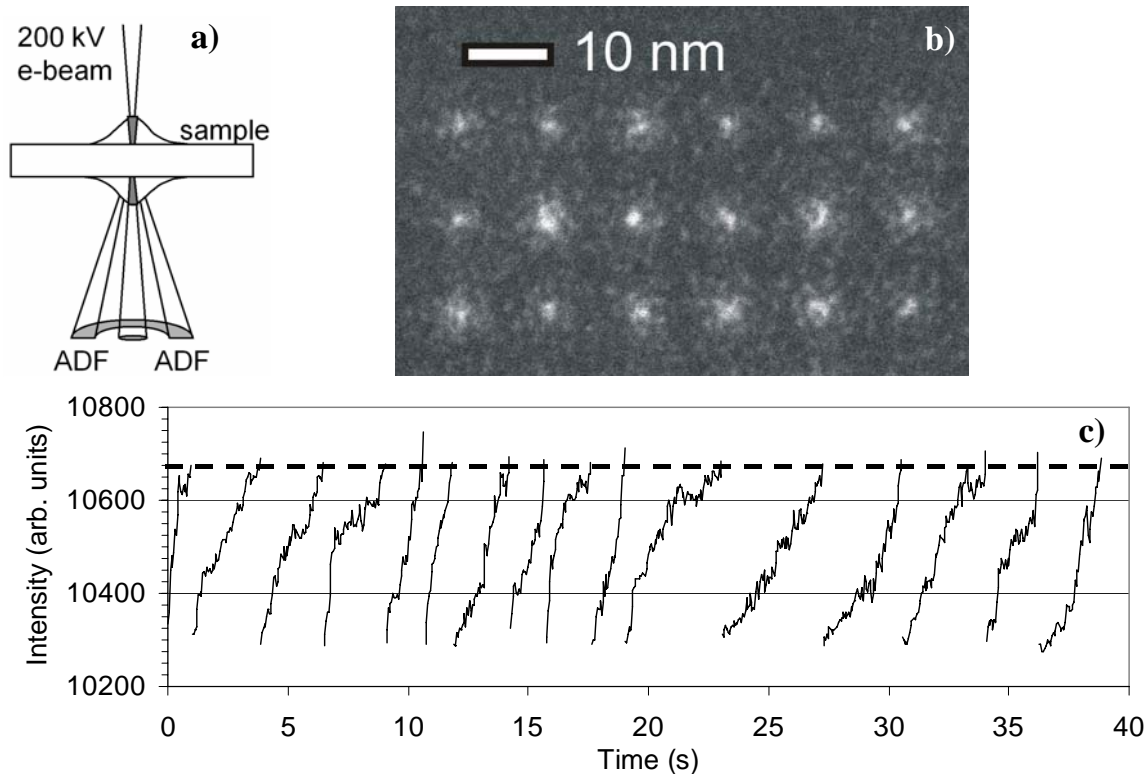


Fig. 1. (a) Schematic drawing of the experimental setup. Imaging and monitoring of the deposits is performed with the ADF signal. (b) An array of deposits created with the monitoring method. Average deposit diameter is 2.2 nm. (c) Growth curves of some of the dots in the array.

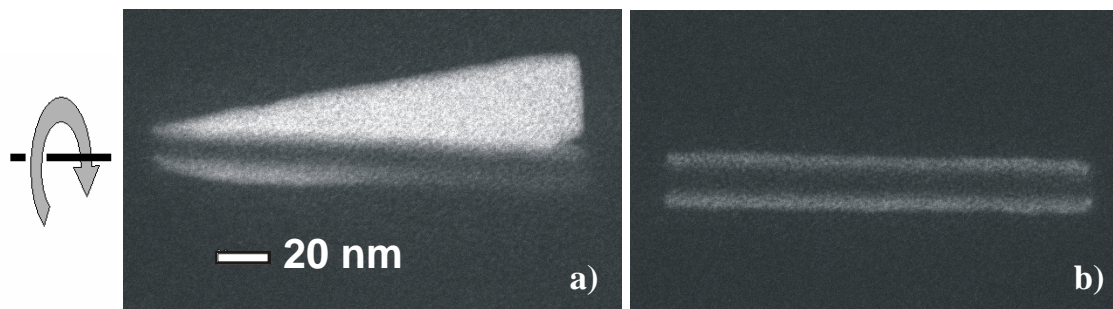


Fig. 2. (a) A 160 nm long tilted line, deposited with a fixed dwell time. The tilt direction is indicated. Since the substrate was a transparent membrane, deposition occurred on both the entrance and exit surface. A proximity effect caused an increase of the height as the writing of the line proceeded (from left to right). (b) A tilted line with the same pattern, but now the growth was monitored and controlled with the ADF signal. The dynamic growth control compensated for the proximity effect.

¹ W.F. van Dorp, B. van Someren, C.W. Hagen, P. Kruit, P.A. Crozier, *Nano. Lett.* 5 (2005) 1303

² W.F. van Dorp, B. van Someren, C.W. Hagen, P. Kruit, P.A. Crozier, *J Vac Sci Technol B* 24 (2006) 618

³ W.F. van Dorp, S. Lazar, C.W. Hagen, P. Kruit, P.A. Crozier, *J Vac Sci Technol B* (2007, submitted)