Direct carbon deposition by EBID at low substrate temperatures

J.J.L. Mulders
FEI Electron Optics, Achtseweg Noord 5, 5651GG, Eindhoven, the Netherlands

Electron beam induced deposition (EBID) is a technique based on the local decomposition of a precursor gas using a focused electron beam. A variety of common precursor materials is used nowadays for the deposition of a range of materials such as Pt, Au and Co[1, 2]. Today much EBID research focuses on the purity of the material[3, 4]. However, none of the reported EBID processes can be applied at a substrate temperature in the range -50 to -160 ºC because the sticking coefficient of the precursor on the substrate approaches unity and hence the precursor freezes onto the surface as a thin film. Low temperature EBID is a technique that is required in the process for the FIB creation of a TEM lamella under cryogenic condition, which is required to preserve a vitrified biological sample (fast frozen with no ice crystal formation). In the TEM lamella creation process, it can serve two purposes: a local conducting path to reduce charging and -more important- as a temporary "glue" for the TEM lamella when transferred from bulk material on to a grid or on a dedicated TEM holder. In both cases an EBID process for a substrate at very low temperature is required. This paper reports on the use of an alkane carbon deposition precursor at very low temperature.

As a guide to the possible applicability of a precursor for a EBID process, its melting temperature compared to the substrate temperature can be used as a first indication. For linear chain alkanes C_nH_{2n+2} the melting points can be plotted as a function of n (Figure 1). From this it can be seen that hexane C_6H_{14} has a melting point of -95 ºC. At -85 ºC hexane can be used to create very small beam defined structures (Figure 2), as in a regular EBID process. To study the EBID process as a function of temperature, point depositions have been made at -50 ºC and at -80 ºC (Figure 3 and 4 respectively). These figures show the growth as a function of deposition time and the decay as a function of the time after closure of the supply line. It can be seen hat at -80 ºC the process is more efficient and that the decay is slower as compared to the process at – 50 ºC. This phenomenon is attributed to an increased sticking coefficient for the hexane molecules at lower temperature. However at -95 ºC the precursor freezes onto the substrate and the EBID process is lost (Figure 5). At this point the sticking coefficient is very close to 1.

For even lower temperatures then -95 ºC use was made of butane and propane. At a temperature of -150 ºC, which is well below the temperature to preserve a vitrified TEM sample, propane was used to create a 200 nm wide EBID point deposition (Figure 6). Even at this very low temperature the EBID process is effective and can be used in a similar way as a regular EBID process at room temperature.

[1] S Graells, R Alcubilla, G Badanes, R Quidant, Appl. Phys. Lett **91** 121112 (2007)
[2] A. Fernandez-Pacheo, J M de Teresa, R Cordoba, M R Ibarra, J Phys D: Appl. Phys. **42** 055005 (2009)
[3] A. Botman, J.J.L. Mulders and C.W. Hagen, Nanotechnology **20**, 32700 (2009)
[4] J D Barry, M Ervin, J Molstad, A Wickenden, T Brintlinger, P Hoffmann, J. Melngailis, J Vac Sci Technol B **24** (6) 3165 (2006)

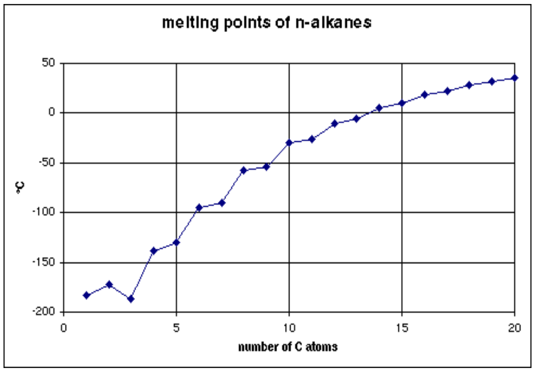


Figure 1: The melting point of the linear-chain alkanes C_nH_{2n+2} as a function of n

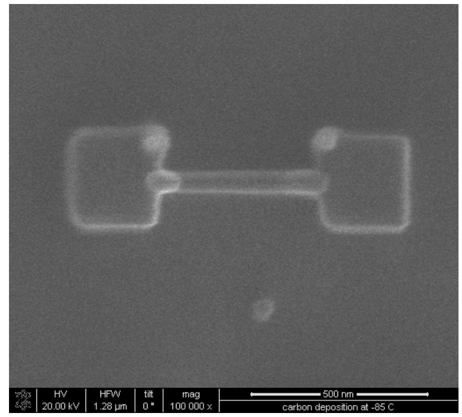


Figure 2: Deposition using hexane at – 85 ºC. At this temperature the precursor works as a standard precursor at room temperature.

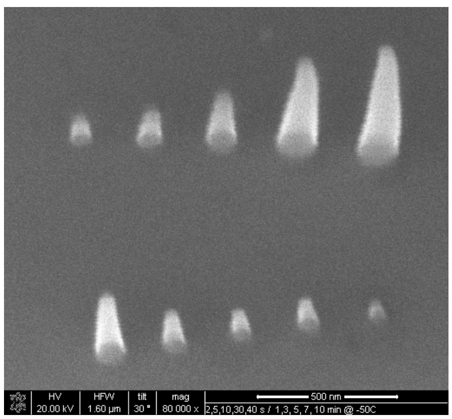


Figure 3: Point depositions made with hexane at - 50 ºC. Top: precursor ON, deposition time 2, 5, 10, 30 and 40 s. Bottom: deposition time 60s each, at 1, 3, 5, 7 and 10 min. after precursor OFF.

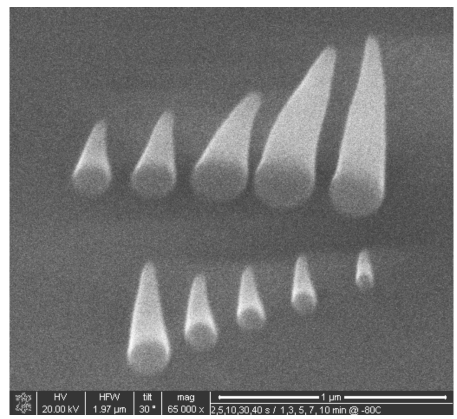
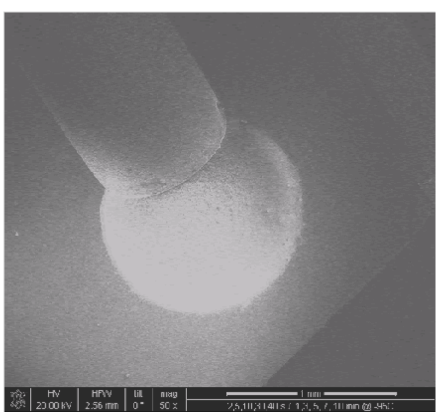


Figure 4: Point depositions made with hexane at -80 ºC. Top: precursor ON, deposition time 2, 5, 10, 30 and 40 s. Bottom: deposition time 60 s each at 1, 3, 5, 7 and 10 min after precursor OFF,



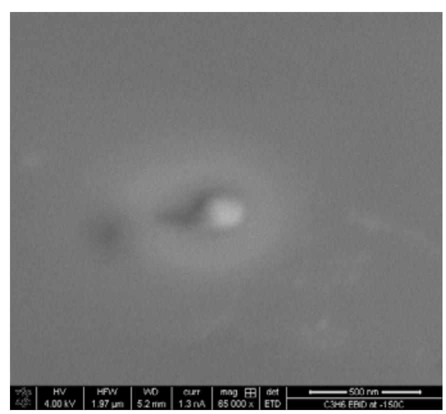


Figure 5: Hexane at -95 ºC substrate temperature. Quick scan, low current, low magnification image, showing direct freeze onto the substrate. No EBID possible

Figure 6: Point deposition of 180 nm width using propane as a precursor and a substrate temperature of -150 ºC.