

A Study of Temperature Influence on Electron Beam Induced Deposition

Wei Li,^{*} and David C. Joy^{*,**}

^{*}Dept. of Materials Sci. & Engr, University of Tennessee, Knoxville, TN37996-2200

^{**}Oak Ridge National Laboratory, Oak Ridge, TN37831-6064

Electron beam induced deposition (EBID) is a novel technique for 3D nano-fabrication [1,2]. It employs an electron beam to dissociate the precursor molecules sticking to a substrate when a precursor gas is introduced into a vacuum chamber, leaving a non-volatile deposit on the substrate and a volatile by-product pumped out of the chamber. Unfortunately the mechanism is not yet well understood. One issue is whether the dissociation happens in the gas phase over the substrate surface or in a molecular layer adsorbed on the surface. The first assumption is substrate temperature independent while the second is temperature dependent since the residence time of the molecules adsorbed on the surface is a function of substrate temperature. The answer to this question is important because it can help people to optimize the deposition condition to easily achieve 3D nano-fabrication. This study was performed to verify which situation is true.

The experiments were carried out on a Hitachi S-4300SE/N SEM. The precursor, tungsten hexafluoride (WF_6), was introduced into the system using a wobble stick manipulator to position a hypodermic needle close to the substrate surface. The chamber pressure was 5.0×10^{-4} Pa and this increased to a range $5.5 \times 10^{-3} \sim 8.0 \times 10^{-3}$ Pa when WF_6 was admitted. An EMITECH K25X peltier cooling stage with a temperature range $-30^\circ C \sim 75^\circ C$ was also installed in the microscope chamber to give a control of substrate temperature. The experimental set-up is schematically illustrated in Fig. 1a. The deposition was carried on a germanium substrate to permit in-situ thickness determination to be made by X-ray spectroscopy without the problem of overlap between the Si and W lines. An array of W deposits, shown in Fig. 1b, was fabricated by exposing the substrate with an electron beam in spot mode in the presence of WF_6 . A beam energy of 20 keV and beam currents of 36, 42, 300, 383, 1240 and 1420 pA was adopted. For each beam current, a line of deposits was deposited with the same total dose (exposure time) but with different substrate temperatures. The exposure time was set to 45 seconds. The substrate temperature was changed from $75^\circ C$ to $-30^\circ C$ gradually and for each substrate temperature there was a stabilizing time of 3~5 minutes. Fig. 1b shows that as substrate temperature decreases the deposited volume increases. This supports the second proposed mechanism that the dissociation occurs in a molecular layer adsorbed on the surface because Fig. 1b reveals that the deposition rate varies significantly with the substrate temperature. Three types of deposits were found in this study. The first type, mostly found at the low beam currents of ~ 40 pA, have a cylindrical body and a hemispherical top, as shown in Fig. 2a. The second type, mostly found at the median beam currents of ~ 300 pA, have a cylindrical body and a sharp conic top, as shown in Fig. 2b. The third, mostly found at the high beam currents of ~ 1400 pA, have only a conic body, as shown in Fig. 2c. This indicates the beam current plays an important role in shaping 3D nano-scale features. The height of each deposit was measured by tilting the substrate by 45° and the volume of each deposit was calculated according to its shape. The relationship between the deposited volume and the substrate temperature was plotted in Fig. 3. This figure illustrates that for each beam current the logarithm of the deposit volume varies linearly with the inverse of the substrate temperature indicating an Arrhenius relationship between the deposition rate and the substrate temperature. The slope of Arrhenius plot is proportional to an energy barrier which the process must overcome. Here this barrier is the desorption energy which determines the residence time of the adsorbed molecules [3]. The desorption energies were calculated from Fig. 3. The measured desorption energies at ~ 40 , ~ 300 , and ~ 1400 pA were determined to be ~ 250 , ~ 150 and ~ 130 meV respectively. The desorption energies calculated in this study are comparable to the range between ~ 0.01 eV (physisorption energy) and ~ 1 eV (chemisorption energy), which suggests that the adsorption-desorption process plays a significant role in interpreting the growth mechanism and thus endorses the assumption that the dissociation occurs in the layer of molecules adsorbed on the surface.

References

- [1] P. Hoffmann et al., 10th Int. Symp. "Nanostructures: Physics and Technology", St Petersburg, Russia, June (2002).
 [2] Z. Q. Liu et al., *Appl. Phys. A* 00 (2004) 1.
 [3] M. Ohring, *Materials Science of Thin Film*, Academic Press, San Diego, 2002.
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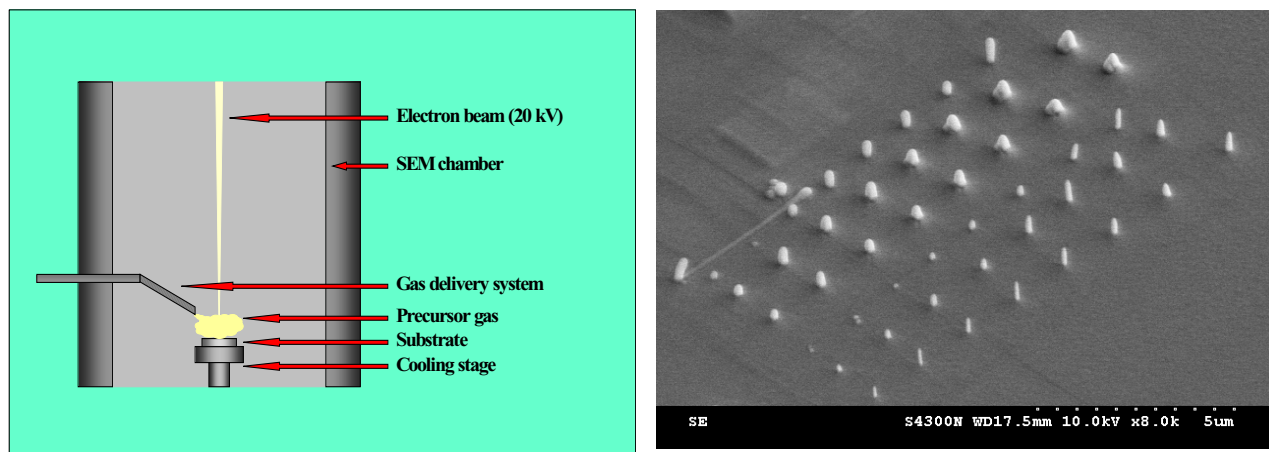


FIG. 1. Electron beam induced deposition. (a) Scheme of the experimental set-up, (b) An array of W deposits grown as the substrate temperature varying from 75°C to -30°C at 36, 42, 300, 383, 1240 and 1420 pA respectively.



FIG. 2. Types of the deposits. (a) Deposit of Type I grown at 20 keV, 36 pA and -28°C, (b) Deposit of Type II grown at 20 keV, 383 pA and 0°C, (c) Deposit of Type III grown at 20 keV, 1420 pA and 15°C.

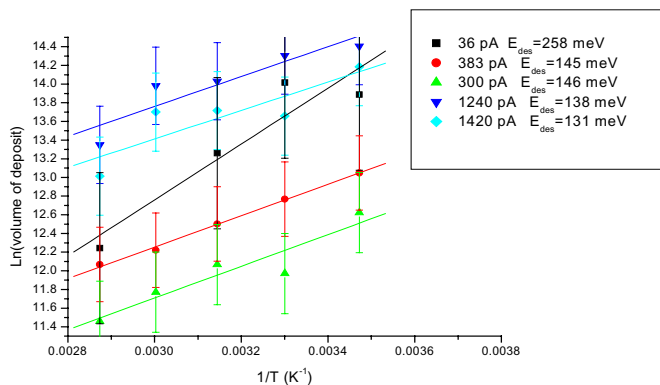


FIG. 3. An Arrhenius plot of the deposit volume and substrate temperature. The measured desorption energies at ~40, ~300 and ~1400 pA were determined to be ~250, ~140 and ~130 meV.