Characterisation of electron-beam deposited tungsten interconnects

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Abstract. Electron beam deposition of tungsten from W(CO)₆ has been studied for a range of different height deposits. The resistance of the deposited tracks was found to decrease with increasing height implying that thicker deposits have a higher metallic cross section. It was also found that when the current limits were increased to values in excess of 100 µA, the resistance decreased with successive voltage cycles and increasing current limit. This improvement continued until a point where the structure of the deposit appeared to start breaking down and its resistance increased slightly. Subsequently, the structure was found to break near to the contact. It was also found that as the resistance of the deposit decreased, the structure of the deposited tracks changed implying that ohmic heating induced high enough temperatures within the deposit to be able to cause the structure of the material to change.

1. Introduction

As microelectronic circuits have decreased in size and increased in complexity, research interests in nanotechnology has grown. The need for new techniques, capable of fabricating structures with nanometer dimensions at specific locations has increased. One such technique which is currently attracting considerable attention is electron beam induced deposition (EBID), because it is a direct-write, maskless procedure which has a simple manufacturing process suitable for prototyping. It is also capable of fabricating structures using a wide variety of organometallic materials containing tungsten [1], chromium [2], platinum [3] and copper [4]. The structures are deposited using an electron beam within a high vacuum system that dissociates an adsorbed precursor gas into a non-volatile solid deposit on a substrate and a volatile by-product, which desorbs from the surface and is subsequently pumped away.

In order for EBID to be accepted as a mainstream nanofabrication technique, it is necessary to first achieve a high degree of understanding and control of the deposition process. Therefore, most of the research that has been carried out to date has focused on understanding the factors that influence the resolution limits [5, 6] and the deposition rates [1, 7, 8]. In particular it has been found that for a high deposition rate, a high beam voltage (30 kV) and current (5400 pA) are required, while for a minimum linewidth a reduced accelerating voltage (15.8 kV) and low beam current (60 pA) with a long dwell time (420 µs) need to be used [9]. It has also been found that decreasing the scan rate results in lower resistivity deposits [1]. Within this project different height EBID tungsten strips are fabricated from the precursor W(CO)₆ and they have been electrically and structurally characterized in order to determine their suitability for use as an interconnect material in nanoscale applications.
2. Experimental Method

The depositions were carried out using a FEI, Nova Nanolab 600 Dual-Beam Focused Ion Beam and the precursor W(CO)6, using an 10 keV electron beam energy, 0.54 nA beam current, 3.4 nm spot size, 200 µs dwell time and 10 nm pitch. The chamber base pressure was measured using a cold cathode ion gauge and was better than 4 x 10^-6 mbar before deposition and rose to ~4 x 10^-5 mbar during deposition. For the electrical characterization, strips 8 µm by 200 nm were deposited onto silicon dioxide substrates so as to bridge pre-patterned gold pads which had been pre-fabricated ~5 µm apart using UV lithography. Rectangles 12 µm by 200 nm were also deposited onto 100 nm thick silicon nitride membranes, which again were pre-patterned with gold pads with a separation of ~8 µm to allow simultaneous electrical and structural characterization.

After the deposition, the silicon dioxide substrates were transferred to a Digital Instruments 3100 Atomic Force Microscope (AFM) operated in tapping mode, to allow the size of the deposits to be measured accurately. The samples were then electrically characterized using a FEI, Quanta 200 Environmental Scanning Electron Microscope (ESEM), a custom manufactured, two-probe manipulation system and a Keithley 4200 Semiconductor Parameter Analyzer. The ESEM was also fitted with a STEM detector to allow for the deposits on the membranes to be simultaneously electrically and structurally characterized. Further structural characterization was carried out in a Philips CM 200 Transmission Electron Microscope (TEM), which was operated at an accelerating voltage of 200 kV and had a Gatan Imaging Filter (GIF 2000 with a 1 k x 1 k Charge Couple Device). TEM images of the deposits were made on samples before and after their electrical characterization.

3. Results and Discussion

Initially, the height of the strips was measured as a function of dose by varying the number of passes of the beam. The height scaled linearly with dose, such that the height of the structures (nm) was equal to the total dose (µC/m^2) multiplied by 0.27 (figure 1(a)). It was also found that one pass equated to a height of 1.1 nm. The samples were then transferred into the ESEM to allow resistance measurements to be made. For these measurements the voltage was initialized to 0 V, then increased up to a maximum positive voltage before being decreased to a maximum negative voltage and then returned to 0 V. Current limits of 50 µA were set to prevent premature destruction of the structures and the resulting I-V characteristics were linear and symmetric around 0 V. Figure 1(b) shows the measured resistivities of a range of different height deposits. As can be seen from the graph, the resistivity initially decreased as the height was increased, while for heights above 55 nm, the resistivity was constant. Repetition of this investigation demonstrated that these values were reproducible. TEM analysis of several deposits showed that they consisted of amorphous carbon and tungsten and that there was a greater amount of carbon than tungsten in the structures (see later). This analysis may help to explain the results in figure 1(b), as it is possible that the initial changes in the resistivity are due to increasing height and hence amounts of tungsten slowly forming a more continuously conducting film in the structures. The constant resistivity values for heights above 55 nm, may then be thought to be due to the structure and resistance scaling linearly with the increasing height.

![Figure 1](image_url)  
Figure 1. (a) Height as a function of total dose for electron beam deposited tungsten. (b) Resistivity as a function of height for electron beam deposited tungsten.
These results are in agreement with the experimental results of Hoyle et al. [1] who also investigated the dependence of resistance on the exposure dose. They indicate that for electron beam deposited tungsten, deposited using these parameters, to be used as a contact material with a low resistivity, then more than 50 nm of material needs to be deposited. However with this process, as the dose or the number of passes made by the beam is increased, so does the amount of time required to complete the deposition. Both of these restrictions are not compatible with either the fabrication of nanostructures with minimal material or with the IC industry.

As described earlier the I-V measurements that were made to calculate the resistances used current limits of up to 50 µA. Within this current range the structures remained stable and did not break down. To determine the currents that the deposits were able to carry before breaking, the current limit was gradually increased. As the current limit was increased towards 100 µA, the I-V characteristics gradually became non-linear but remained symmetric with respect to 0 V and showed no hysteresis. When the current limit was increased from 100 to 200 µA, a large change in the resistance occurred as shown in figure 2(a). Repeated cycling of the voltage at this current limit caused the resistance to continue decreasing until it reached a reproducible non-linear I-V characteristic, which was again symmetric with respect to zero voltage and showed no hysteresis (figure 2(b)). This behavior was seen each time the current limit was raised, with the size of the change in resistance gradually decreasing until at ~1 mA, the I-V characteristics regained their linearity as shown in figure 2(c). The onset of non-linearity in the I-V characteristics may be due to heating within the structures, while the permanent change in the resistance (figure 2(a)) may be due to structural changes induced by the heating. The return to linear I-V characteristics (figure 2(c)) may then be due to the strips forming more robust metallic cross-sections such that the structures are not ohmically heated during the I-V sweeps. All of the deposited tungsten strips followed this behavior, with the thicker structures maintaining their original resistance for higher current limits.

![Figure 2.1-V characteristics of a 28 nm high strip showing (a) hysteresis and non-linearity due to changes in the resistance and structure due to heating (b) non-linearity due to heating and (c) linearity after all of the structural changes have occurred.](image)

AFM images of the deposits taken during and after the electrical measurements showed that as the resistance of the deposits changed so did their size. In particular it was found that the height decreased while the width increased. This suggests that the change in the resistance is due to a change in the structure of the deposits as the material is heated ohmically above its sublimation temperature.

To understand the effects of this behavior on the structure, the experiment was repeated using a 100 nm thick tungsten strip deposited onto a silicon nitride TEM grid. No significant differences in the electrical behavior were found when changing to this substrate. Scanning-TEM images taken in the ESEM at 10 kV during the electrical characterization showed that as the resistance changed, the deposit took on a rippled structure. TEM analysis of an as-deposited strip is shown in figure 3(a) and the deposit after electrical characterization is shown in figure 3(b). Following the resistance measurements, it was found that the as-deposited amorphous structure had transformed to contain tungsten clusters embedded in a carbon graphitized network (as evidenced by the carbon k-edge,
obtained using EELS and the 002 fringes (~0.34 nm) observed in the HREM images (not shown here)). It was also noticed that the tungsten clusters (dark dots in figure 3(b)) only occurred in the defined deposition area, whereas the carbon (light grey area) extended beyond it and that during the electrical characterization, the membrane had been damaged so that the nitrogen had been burnt away. No structural changes were observed during TEM imaging, including HREM or EELS imaging. By overlaying the scanning-TEM images onto the TEM images it was also possible to see that the higher parts of the deposit in the scanning-TEM images were related to the tungsten clusters. This structural change is similar to that seen by Botman et al. in reference [10], who found that by annealing EBID deposited platinum and gold pads at temperatures up to 500ºC they could remove some of the carbon in the structures as well as transforming them from an amorphous to a partially crystalline structure. This result reinforces our hypothesis that the changes in the resistance and the structure must be due to ohmic heating within the deposited strips.

![TEM image](image.jpg)

**Figure 3.** TEM images of a 110 nm high tungsten strip deposited using ~400 µC/m² (a) Bright field image of the as-deposited strip (b) Carbon EFTEM map. The carbon is graphitised (light grey area), as confirmed by filtered EELS and HREM. The dark dots are tungsten clusters.

4. Conclusions
In conclusion, we have shown that the resistance of the deposits decreases with increasing height and that the structure and electrical characteristics of the deposits remain constant for currents up to 100 µA. However, when the current is increased to very high values the structure of the material appears to alter, implying that ohmic heating of the deposit occurs.

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