In Situ Transport Properties Measurements of FEBID $Cu(II)(hfa)_2$ During Annealing

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Focused Electron Beam Induced Deposition (FEBID) using organometallic precursors often results in large carbon / low metal content deposit material [1]. Recently, post growth and in-situ purification techniques were applied successfully to improve upon the metal content in Pt-C deposits [2,3,4,5], W-C [2, 6], Ru-C [7], Cu-C [8]. In this work, post growth conventional annealing in vacuum was used to decrease the resistance of Cu-C lines deposited from $Cu(hfac)_2$ precursor $Cu(C_7HO_2F_6)_2$ by FEBID at room temperature with a 25kV, 1nA electron beam.

Ten micrometer long lines of Cu-C were deposited across four gold electrodes on a 200nm SiO_2 layer on Si substrate, see figure 1. A four point prober with Au covered probes was used to measure the changes in the resistance during heating. The experiment was performed inside of SEM at the pressure of ~$5x10^{-5}$ mbar for 3 different final temperatures, 140, 180 and 220 °C. The as-deposited Cu-C lines have about 7 to 14 at.% of Cu homogeneously dispersed in a polymeric carbonaceous matrix containing the ligand elements oxygen and fluorine as measured by EDX, as well as probably some hydrogen. They show high electrical resistance in the GΩ range, see figure 2. Increasing the temperature induces a substantial decrease of the line resistance probably due to the precipitation of copper nanocrystals and a change of reticulation of the carbon network inside the matrix. In-situ TEM annealing of a FEBID deposited lines on a carbon TEM grid, indicating that they became nanocrystallines , see fig. 3. The X-ray diffraction is ongoing to reveal the nature of this nanocrystals. A detailed discussion of the FEBID conductivity will be given in this contribution.

References:

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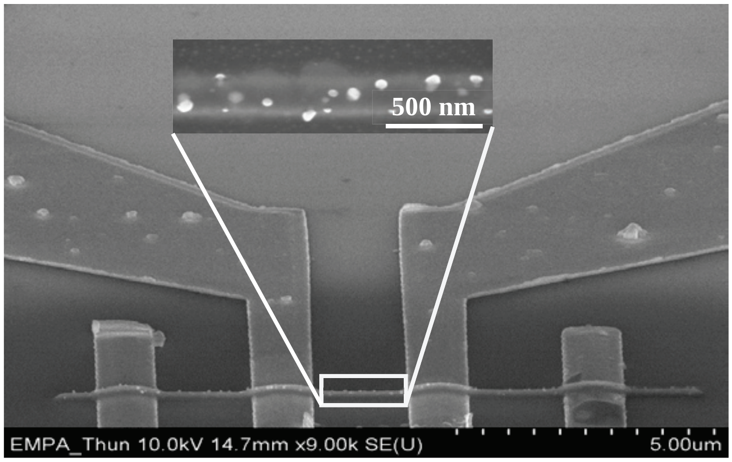

Figure 1: SEM tilt image of a post-growth annealed (180°C) FEBID line deposited with Cu(hfa)$_2$ on SiO2 and gold electrodes at room temperature.Inset: Top view SEM image zoom showing the line with precipitated copper crystals and the remaining carbon matrix after annealing.

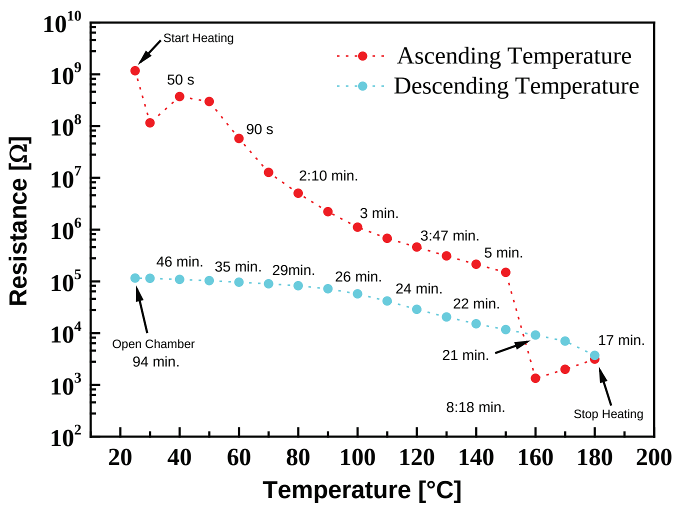

Figure 2: Monitoring of line resistance during heating up (red dots) and cooling down (cyan dots) in vacuum.

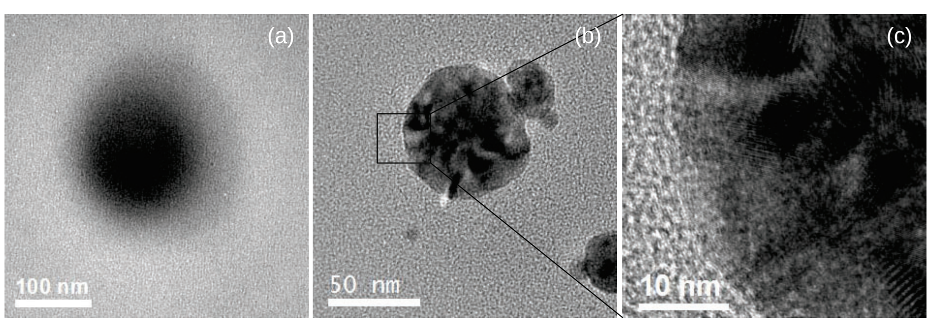

Figure 3: High-resolution TEM imaging reveals that the FEBID deposited Cu nano-particles are amorphous in (a). (b) and (c) show that the particles become crystalline after annealing in-situ in TEM at 200 °C only for ten minutes.