Structural and electrical studies of tungsten nano-electrodes prepared by FIBID (focused ion beam induced deposition)

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The use of an ion focused beam to induce the deposition of materials from metalorganic precursors has recently emerged as an important technique to fabricate nanowires and integrated circuit devices [1-3]. Focused-ion-beam-induced deposition (FIBID), based on the local decomposition of gaseous precursor by the ion beam, is a maskless direct writing technique allowing the deposition of sub-micronic patterns. We have studied the properties of electrodes (or wires) formed by FIBID from a tungsten hexacarbonyl $W(CO)_6$ precursor and subjected to an electrical treatment. These electrodes are supported on a structure also elaborated by FIBID. We present conductivity measurements and structural analysis of these electrodes before and after electrical treatment.

The experimental setup consists of a cross beam system coupling a scanning electron microscope (SEM, JEOL) and a focused ion beam (FIB, Orsay Physics), equipped with a gas injection system (GIS, Orsay Physics). Using a current–voltage (I–V) picoammeter, the electrical characteristics of the wires can be followed *in situ* during the deposition process and the electrical treatment.
The electrodes, prepared for subsequent analysis in the TEM, were deposited on a diamond like carbon (DLC) thin membrane (3x10 µm² ; thickness 50-100 nm). The carbon membranes are prepared by FIBID from a phenantren ($C_{14}H_{10}$) precursor and are used to bridge two aluminium electrodes *(Fig.1)*. These membranes are insulating (resistivity: 100 Ω.cm), very hard (Young modulus : 600Gpa) and are transparent to electrons.[4-5]

The structure and the morphology of the electrodes were characterized by atomic force microscopy (AFM) and high-resolution transmission electron microscopy (HRTEM). Their dimensions are : length 20 µm, width 45-60 nm and thickness 15-30 nm (function of the deposition time). Before electrical treatment, the microstructure of the wires shows no obvious long range order implying the deposited material exists in an amorphous like state. These nanowires are a mixture of tungsten (75%), carbon (10%) and oxygen (5%) resulting from the precursor decomposition and gallium (10%) coming from the ion beam.[6-8] During the deposition process, a constant voltage of 50 mV was applied between the aluminium electrodes and the current through the wires was measured *(Fig.2)*. We obtained electrodes with resistances between 10 and 100 kΩ for deposition time between 5 and 60s.

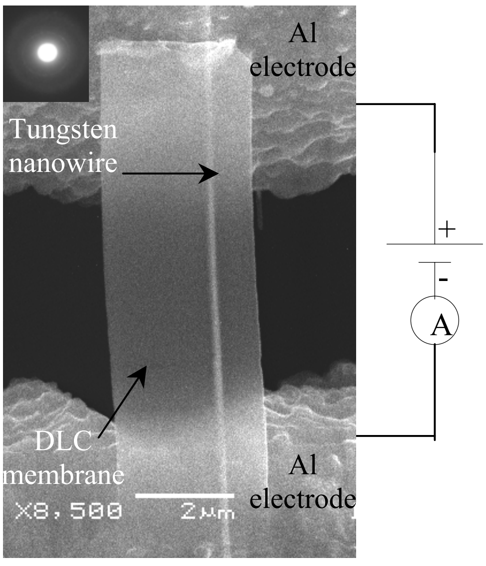


***Fig-1** :SEM view of the experimental support*

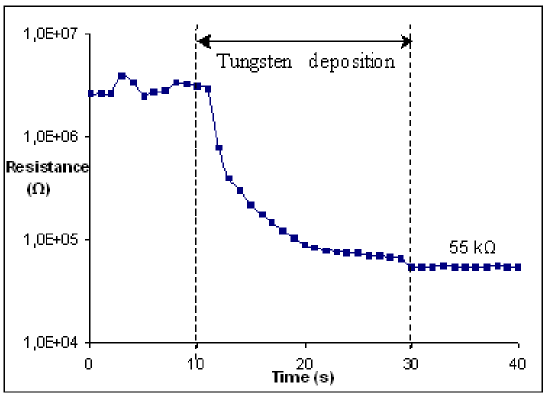


***Fig-2** : Electrode résistance during deposition process*

We have observed that simple electrical treatment can substantially improve the wire resistivity.[9] It consists in the application of voltage ramps to the wire *(Fig.3)*, producing a high current density and inducing an electrical annealing. This treatment first induces a segregation of Ga atoms forming droplets. The level of impurities in the wire decreases, and the wire becomes metallic. Then, as high temperature is reached (~600°C) the Ga droplets evaporate and a reorganisation of the microstructures occurs in the Ga-free wire. Figures 4 shows high magnification SEM and TEM images for the nanowire after electrical annealing. The wavelength of the modulation in the HRTEM image is about 0.22 nm, which corresponds to the interplanar distance of (110) tungsten's lattice planes (bcc ; a=3,16Å). The associated electron diffraction pattern in the inset of the figure reveals the (110) reflections. The periodicity of both the lattice planes and the associated electron diffraction pattern indicate that the treated nanowires are crystallized (crystal 5-20nm).

Our process allows the fabrication of polycrystalline tungsten nano-electrode with low resistivity (50µΩ.cm) being able to support high density current (5.10^7 A/cm²). These results open a wide range of applications for the FIBID technology. The high resolution images presented here show the most promising application of our carbon membranes as substrates for electronic transmission microscopy. Furthermore, the nano-electrodes can be used to probe and heat nano-objets in in-situ TEM preparations.

Electrical annealing

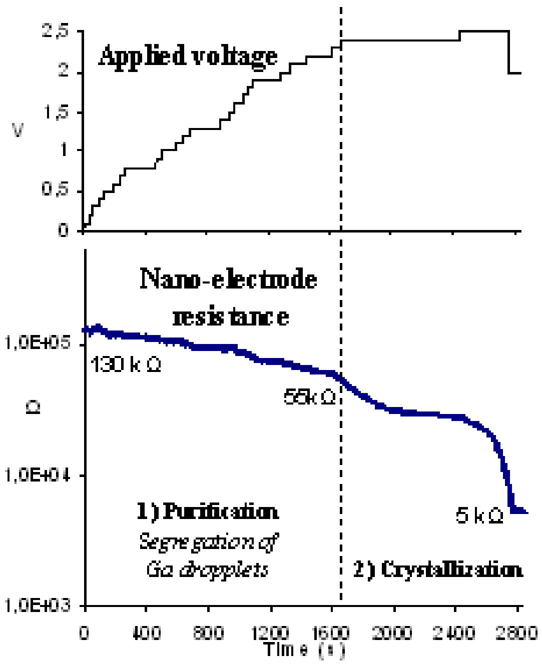
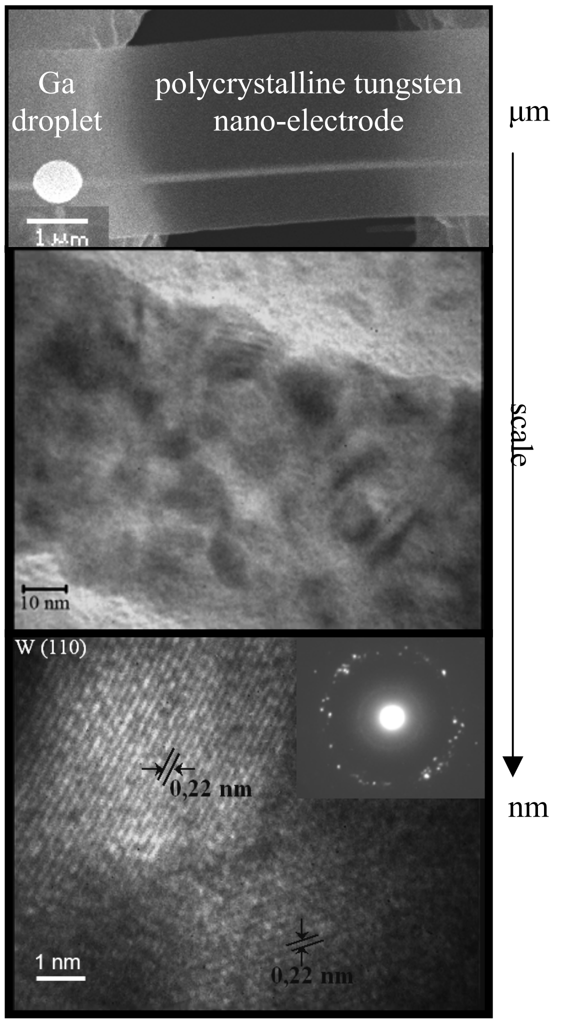


***Fig-3 :** Evolution of the nano-electrode resistance during the electrical treatment*

***Fig-4 :** SEM, TEM , HRTEM images and diffraction pattern of the annealed nano-electrode*



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