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Morphological and electrical study of FIB deposited amorphous W nanowires

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Abstract

Morphological and electrical measurements were carried out on W nanowires deposited by focused ion beam (FIB) onto the micro-hotplate of a micropellistor device. The *I–V* characteristics showed that the deposited W alloy wires have ohmic behaviour. Temperature dependent resistance measurements were carried out in vacuum (in situ in the chamber of the FIB/FESEM) and ex situ in air and in streaming nitrogen in the temperature range of 112–412 °C, using the micro-hotplate for temperature control. The first heat-up in vacuum caused a slight decrease of resistance followed by an irreversible, abrupt drop down to 3% of the as-deposited value, this value was kept during cool-down. The ex situ heat-up in air and N_2 atmosphere caused increasing resistance at temperatures over the range 300–350 °C, after a similar, slight decrease in the range of 200–300 °C, like in the case of vacuum ambient measurements.

Keywords: W deposition; Resistance measurement; Temperature dependence

1. Introduction

With the integration of nanoobjects into electrical circuits and devices, the investigation of nanocontacting methods and materials became more and more wide spread. The fabrication of nanowires by deposition of W or Pt with EB or/and FIB has proved a useful and reliable contacting technology of individual nanodevices [1].

Several papers deal with different aspects of Pt deposition by focused electron (EB) and ion (FIB) beam, comparing the deposition rates versus beam parameters [2] as well as the amount of deposited Pt atoms [3]. The deposition efficiency was found [2] comparable for both EB and FIB induced deposition in the 10–30 keV range, while for lower beam energies, the values differ. The amount of deposited Pt atoms increased with the increase in ion or electron dose, due to the enhancement in the decomposition of precursor gas [3].

The electrical properties of Pt and W wires deposited by FIB were also studied [1,4]. The Pt contacts fabricated to nanodevices revealed ohmic behaviour and good stability as a function of time and applied current [1]. The W nanowires display an ohmic behaviour, low resistance, (only 20 times higher than the bulk) and a good stability [4]. Comparing the EB and FIB techniques the resistance of the EB deposited Pt nanowires was quite high in the as-deposited state and increased drastically when cooled down [5], while the resistance of the FIB deposited ones showed only a weak temperature dependence.

Our aim in the present work was to investigate the influence of temperature on the electrical behaviour of FIB deposited W wires. We used both in situ annealing in vacuum and ex situ annealing in air and nitrogen atmosphere. A special substrate with a built in micro-hotplate [6] was used for the annealing of the deposited wires.

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2. Experimental

The deposition and morphological characterization of nanometer sized wires were carried out, without opening the vacuum system in a LEO 1540 XB workstation, a cross-beam system consisting of a high resolution Gemini FEG SEM column, an Orsay Physics FIB column using a 30 kV Ga focused ion beam for milling, and a gas injection system (GIS) to perform electron or ion beam assisted deposition. When using the GIS, milling and deposition processes are in competition [4], so the deposition process parameters (FIB beam current, scan speed and gas flow) have to be optimized.

Six samples were prepared; two of them were electrically characterized in situ, (without opening the chamber) after the deposition. The other four were removed from the system, two of them were electrically characterized in air and the last two in streaming nitrogen.

The electrical characterization of the deposited wires was carried out on a special micro-hotplate [6], (Fig. 1) prepared by photolithographic methods with characteristic dimensions of 100 × 100 μm, consisting of a substrate, conductive Pt wire for thermal heating, insulator layer and Au electrodes. W nanowires with 500 nm width were deposited between the fingers of interdigitated Au electrodes, with the characteristic distance of 15 μm. The resistance of deposited W nanowire was measured with a precision Keithley 175 Autoranging Multimeter.

The temperature dependence of the resistance was measured in three sets by heating the micro-hotplate using a Keithley 225 current source with a 100 nA resolution in the output current. In the first set, the hotplate was heated up to 310 °C from room temperature (RT) by a current from 200 to 5000 μA (see the used current/temperature values in Table 1). In the second set, the hotplate was cooled down to RT and measured at the same temperatures as before. In the third set, the hotplate was heated up again and measured similarly until the W wire burned out. The heating current was kept constant until the resistance was stabilized. The temperatures were obtained from the heating current values using the calibration curve of the hotplate [6].

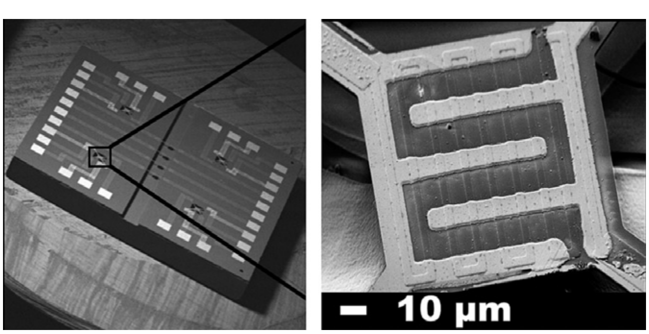

Fig. 1. The special micro-hotplate used for electrical characterization of deposited W wires.

Table 1
Heating currents and corresponding temperature values used for resistance measurements

Heating current (μA)	1st heat-up 1 min steps (°C)	Cool-down 1 min steps (°C)	2nd heat-up 30 s steps (°C)
200	112	112	112
500	174	174	174
1000	199	199	199
1500	212	212	212
2000	220	220	220
2500	231	231	231
3000	243	243	243
3500	257	257	257
4000	272	272	272
4500	290	290	290
5000	310	310	310
5500			330
6000			360
6500			379
7000			412 (break)
7500			460 (break)

The melting of the wire and sample breaking due to static charge introduced in the system when connecting was avoided using a special, ground free device [7].

3. Results and discussion

Morphological characterization of the samples was carried out after the deposition as well as after the electrical measurement in the cross-beam SEM/FIB system. Extreme care had to be taken when selecting the region of deposition and the deposited area. After heating up the W precursor reservoir and capillary the microscope was used in single beam (FIB) mode in order to avoid the EB deposition of W on the whole surface (usually scanned by the electron beam to monitor the FIB processes). The exact area of wire deposition had to be defined on a single scan FIB induced secondary electron (SEI) image. The W nanowire was deposited by irradiating the selected area by FIB. After the W wire deposition, a morphologically smooth surface could be observed on the SEI of the SEM. Care had to be taken to limit the current flowing through the nanowire during the resistance measurement, because if it was too high, the wire was getting narrower in the middle, and was finally molten into droplets (Fig. 2).

The annealing and electrical measurements were carried out on two samples for each case. In each case the samples showed the same ohmic behaviour. The resistance of the W nanowires was measured at room temperature and the relative resistance changes during the thermal cycles were normalized to this value (100%). The characterization temperatures are given in Table 1.

Relative resistance values measured during one heat-up cycle of in situ annealing are shown in Fig. 3. The resistance slightly decreased between RT and 272 °C (from 100% to 95%). At 290 °C the resistance suddenly decreased to 3.4%. Two experiments were made at this point. After it, the sample was cooled down slowly to RT, while its resis-

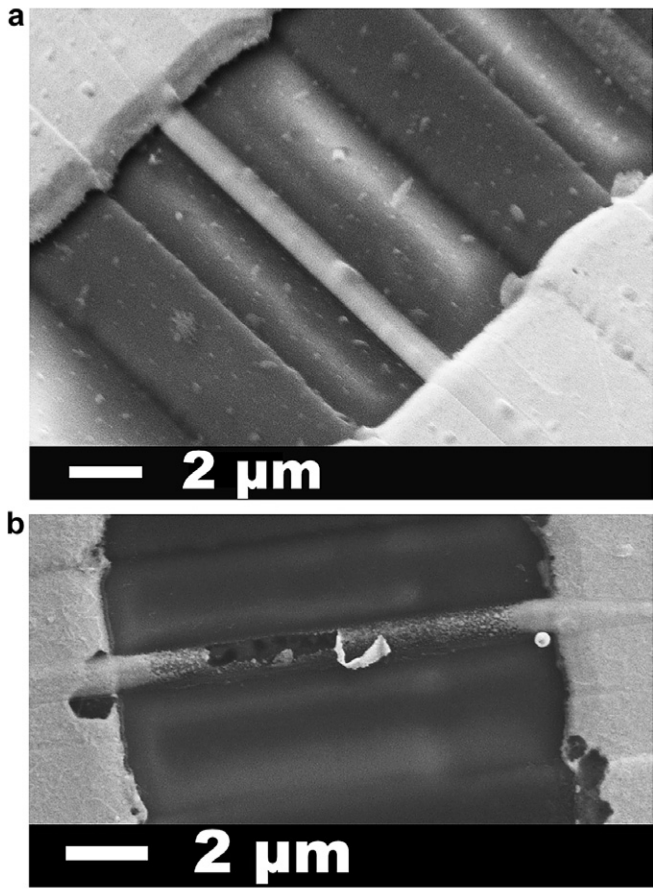

Fig. 2. SEM image of a 500 nm deposited W wire before electrical measurements (a) and after the full electrical characterization in nitrogen atmosphere. The broken wire and the molten drops along the wire are clearly seen (b).

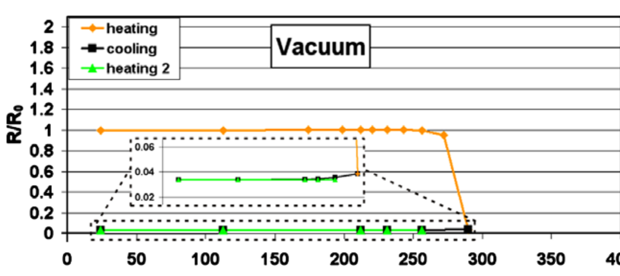

Fig. 3. Temperature dependence of the resistance of 500 nm W wire characterized in situ in vacuum. Note the resistance drop at 290 °C.

tance decreased further down to 2.2%. If it was heated further the Au and Pt electrodes were molten and the bridge of the hotplate was broken around 310 °C.

Results of one full (heat-up and cool-down) and a subsequent heat-up cycle of ex situ measurements in N_2 gas are shown in Fig. 4. The nanowire remained stable up to 310 °C showing a small decrease (100% → 93.7%) only. When cooling down the sample, the resistance increased to 98.5% at 175°C, then kept this value until RT. During the second heat-up cycle the resistance values were

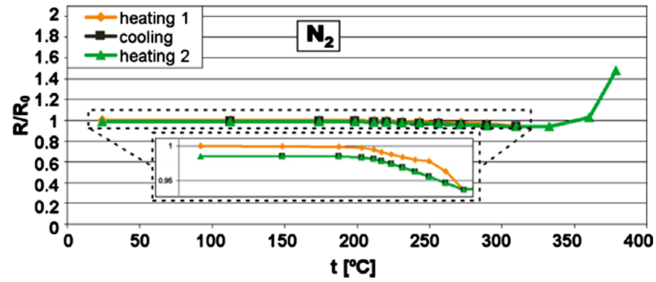

Fig. 4. Temperature dependence of the resistance of 500 nm W wire characterized in nitrogen atmosphere. No drop is observed. The cooling and second heating data overlap at slightly lower values than in case of the first heat-up.

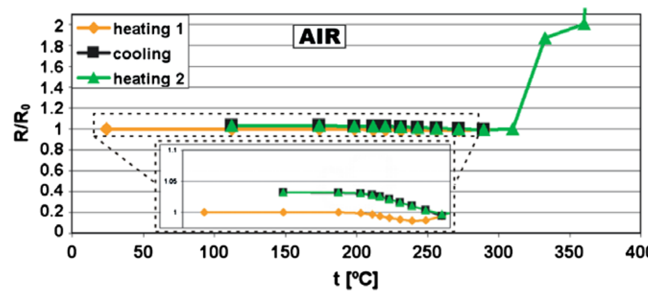

Fig. 5. Temperature dependence of the 500 nm W wire resistance measured in air. The wire molten at 379 °C. The cool-down and second heat-up data overlap at higher values than in case of the first heat-up.

approximately the same as during cool-down. Heating further, the wire was melt at 460 °C.

The results of a similar experiment in air are shown in Fig. 5. The resistance decrease during the heat-up was smaller (the resistance value at 310 °C was 97.8% of the original RT one) while it increased to a higher value (103.7% of the reference) when cooled down again to RT. The second heat-up cycle reproduced the resistance values measured during cool-down, similarly to the case of measurements in N_2. The wire was melt at 380 °C.

The observed differences in the temperature dependence of the W nanowires resistance measured in vacuum, nitrogen and air are tentatively attributed to recrystallization of the amorphous alloy followed by chemical reactions at the freshly formed grain boundaries in case of nitrogen and air. Further investigations are needed to gain a deeper insight in these processes.

4. Conclusion

The temperature dependence of the resistance of W nanowires deposited by FIB was studied. It was found that in each case the resistance decrease between RT and 270 °C is approximately 2–6% of the as-deposited value. From this point, the different experiments yielded different results.

The resistance of W wires measured in vacuum suddenly decreased to 3.4% at this temperature. This change seems to be irreversible, as when cooling down the sample to RT the resistance further decreased to 2.2% of the original RT value.

The ex situ experiments in air and N_2 atmosphere did not show such a radical change, showing that the gaseous ambient inhibits the process. The fact, that the heat treatment in air increased the resistance above the as-deposited value (while in N_2 the direction of the change was the same as in case of the experiment made in vacuum) suggests that the oxidation works against the resistance decrease. This can be the reason why the samples measured in air have the lowest burn-out temperatures, too.

The good reproducibility of resistance values measured after the first heat-up cycle is promising in respect of future applications, but it is clear, that to obtain good conductivity, the FIB deposited W nanowires have to be carefully annealed. Further systematic measurements, including compositional and structural characterization of the wires are planned to clarify the reason of the different behaviour of the wires annealed in different environments.

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