OPEN ACCESS

Nanomanipulation and electrical behaviour of a single gold nanowire using in-situ SEM-FIBnanomanipulators

To cite this article: Y Peng et al 2008 J. Phys.: Conf. Ser. 126 012031

View the article online for updates and enhancements.

You may also like

- <u>A platform for *in-situ* multi-probe electronic measurements and modification of nanodevices inside a transmission electron microscope</u> T T Xu, Z Y Ning, T W Shi et al.
- Investigating the impact of SEM chamber conditions and imaging parameters on contact resistance of *in situ* nanoprobing Juntian Qu, Martin Lee, Michael Hilke et al.
- <u>Characterizing the surface forces between</u> <u>two individual nanowires using optical</u> <u>microscopy based nanomanipulation</u> Hongtao Xie, James L Mead, Shiliang Wang et al.





DISCOVER how sustainability intersects with electrochemistry & solid state science research



This content was downloaded from IP address 18.223.196.211 on 08/05/2024 at 20:21

Journal of Physics: Conference Series **126** (2008) 012031

Nanomanipulation and Electrical Behaviour of a Single Gold Nanowire Using In-situ SEM-FIB-Nanomanipulators

Y Peng^{1, 2*}, I Luxmoore², M D Forster¹, A G Cullis², and B J Inkson¹ ¹Department of Engineering Materials ²Department of Electronic and Electrical Engineering, Sheffield University, Sheffield

² Department of Electronic and Electrical Engineering, Sheffield University, Sheffield S1 3JD, UK

E-mail: Y.Peng@sheffield.ac.uk

Abstract. Gold nanowires were successfully fabricated by a DC electrodeposition technique into Anodic Aluminium Oxide (AAO) templates. The microstructure of 55nm gold nanowires released from AAO templates was observed by SEM and TEM to be polycrystalline, with a bamboo-type structure and grain sizes 20nm to several micrometers. Individual gold nanowires were picked up from bundles of gold nanowires using a super-sharp W tip attached to an insitu Kleindiek nanomanipulator fitted in a SEM-FIB. The picked-up gold nanowires were then deposited onto a silicon wafer, or connected between two nanomanipulator tips, to fabricate single nanowire nano-circuits for electrical testing. The electrical properties of single manipulated nanowires are compared to that of bundles of gold nanowires for the two circuit types. The lowest resistance is achieved by connecting the gold nanowires between two FIB-milled tungsten tips.

1.Introduction

Scanning Electron Microscopes (SEM) equipped with Focused Ion Beam (FIB) and nanomanipulators offer exciting opportunities for in-situ nanocharacterisation, nanofabrication, nanopatterning, nanomanipulation and precise integration of nanoscale materials and systems. Here SEM-FIB nanomanipulation has been applied to the characterisation of electrical properties of individual gold nanowires.

2. Experimental Methods

AAO templates with about 55nm pore diameter were fabricated by anodising 99.999% Al sheets in 0.6M oxalic acid electrolyte for three hours at room temperature with continuous stirring. The detailed fabrication method has been detailed in our previous work [1]. Electrodeposition of gold into the AAO templates was performed using a VoltaLab 80 (Radiometer Analytical, France). To deposit the gold the electrodeposition electrolyte was composed of 5g/1 AuCl₄ (Aldrich, 99.99%), and 30g/1 H₃BO₃ (Aldrich, 99.5+%). A pulsed signal, of which each period is composed of two sequential potentials - 0.4V for 30s and 0V for 0.2s (both referring to Ag/AgCl reference electrode), was applied to prepare the gold nanowires. The typical total deposition time for each specimen was ~30 minutes, and the electrodeposition electrolyte was continuously agitated throughout by a magnetic stirrer.

The morphology and microstructure of the electrodeposited gold nanowires were analysed using a SEM (JEOL JSM 6500F, Japan) equipped with a FIB supplied by Orsay Physics, and a high-resolution TEM (JEOL JEM-2010F, Japan). SEM samples were prepared both by fracture of the

Electron Microscopy and Analysis Group Conference 2007 (EMAG 2007)	IOP Publishing
Journal of Physics: Conference Series 126 (2008) 012031	doi:10.1088/1742-6596/126/1/012031

templates, and by dissolving the AAO templates using 0.2M NaOH and rinsing with distilled water. Nanowires completely dissolved out of the templates were dispersed on prepatterned-electrode-pads on a $Si/SiO_2(100nm)$ wafer and holey carbon film coated grids for electrical testing and nanomanipulation. The electrical testing was carried out using a Keithley measurement system.

3. Nanowire morphology

Figure 1a and 1b show two representative SEM and HAADF-STEM images of the nanowires, from which the AAO template has been completely dissolved. The gold nanowires are uniform in diameter and length, indicating a good quality deposition. The average size of these gold nanowires is about 55nm in diameter and 10 μ m in length. A corresponding TEM image of the same gold specimen is shown in Fig. 1c. The nanowires have a bamboo-type structure with non-uniform grain size along their length. TEM analysis shows that grain sizes have a range distribution from 20nm to several micrometers. As an example, a nanowire in Fig. 1c is composed of four grains indicated by four brackets with the three arrows pointing out the grain boundaries. SAED analysis confirms that the gold nanowires are polycrystalline with the fcc structure (Fig. 1d).



Fig. 1 (a) SEM image of gold nanowires on a gold film replica; (b) STEM image of a bundle of gold nanowires; (c) TEM image of two Au nanowires with grains A-D marked; (d) Electron diffraction pattern of a $<101>_{ZA}$ orientation grain circled in (c).

4. Nanomanipulation of Au nanowires



Fig. 2 Manipulation of an individual gold nanowire using the end of a FIB-refined tungsten tip: (a) a selected single gold nanowire; (b) contact of the single nanowire with the tungsten tip; (c) nano-weld of the single nanowire to the end of the tip; (d) separation of the nanowire from holey carbon film copper grid; (e) lifting and moving away the single nanowire from the grid by mechanically manipulating the support tip; (f) approach to a Si wafer with a trench; (g) assembling the picked-up nanowire on the Si wafer; (h) separation of the nanowire from the W tip by FIB cut; (i) removal of the tungsten tip.

Figure 2 shows the whole manipulation process by which a single gold nanowire was picked up from a holey carbon film grid and placed on a Si wafer. After a single nanowire was targeted as shown in Fig. 2a, a tungsten tip was manipulated to contact the end of the chosen nanowire (Fig. 2b). It was

Electron Microscopy and Analysis Group Conference 2007 (EMAG 2007)	IOP Publishing
Journal of Physics: Conference Series 126 (2008) 012031	doi:10.1088/1742-6596/126/1/012031

attempted to attach the chosen nanowire to the end of the tungsten tip and manipulate away from the substrate by using Van der Waals force. However, the nanowire was found not to adhere on the tip end even if we focused the e-beam on the contact point for more than ten minutes. This indicates that the Van der Waals force between the metal nanowire and tungsten tip is very weak and grown carbon contamination [2] from e-beam exposure could not attach the nanowire to the tip apex. This phenomenon is different from similar experiments carried out with multiwall carbon nanotubes, where the Van der Waals force between carbon nanotubes and tip was strong enough to pull a carbon nanotube out from a bunch of multiwall carbon nanotubes even if there was no any e-beam radiation.

Here, for the Au nanowires, e-beam deposition of platinum was introduced to nanoweld the gold nanowires to the W tip as shown in Fig. 2c. The chosen gold nanowire was separated from the substrate by a FIB cut or simple mechanical pulling (Fig. 2d) and then withdrawn mechanically from the grid by manipulating the tungsten tip as shown in Fig. 2e. The picked-up gold nanowire was then assembled on the Si wafer. Fig. 2f shows the picked-up gold nanowire approaching Si wafer patterned with a trench. E-beam platinum deposition was applied to fix the end of nanowire to the Si wafer (Fig. 2g). After a FIB cut, the picked-up nanowire was separated from the tungsten tip (Fig. 2h), and the tungsten tip was moved away from sample (Fig. 2i).

5. Electrical properties of Au nanowires

The electrical properties of a single gold nanowire and a bundle of gold nanowires have been studied by two methods (i) using substrate electrodes, and (ii) direct measurement by two nanomanipulators. The Keithley measurement system used was calibrated by a standard 12.6Ω resistance.

Fig. 3a and 3b show two examples where a bundle of 10μ m long gold nanowires and a single gold nanowire have been connected to two substrate gold electrodes by e-beam Pt deposition, and their electrical behaviour are shown in Fig. 3e and 3f. One can see that the resistance of the circuit containing the bundle of five nanowires is 6970 Ω . If all contacts in this case were ohmic behaviour, the resistance purely belongs to gold nanowires, and each gold nanowire is homogeneous, the resistivity of each gold nanowire can be calculated:

$$\rho = \frac{n \cdot R \cdot A}{L} = \frac{n \cdot R \cdot \pi \cdot r^2}{L} \approx 8.6 \times 10^{-6} \,\Omega \cdot m$$

Where, n (5 in this case) is the number of contacted nanowires in this bundle, $L = 10\mu m$, $R = 6970\Omega$ and nanowire radius r = 28nm. This value is about 380 times the bulk gold resistivity at room temperature [3], and 120 times the resistivity of a 15nm diameter and 2.2 μ m long gold nanowire fabricated by the same technique [4]. This value indicates that contact resistance and the intrinsic resistances of Pt deposits [5] and gold electrode pads are serious problems for obtaining the intrinsic resistivity of gold by this simple substrate electrodes method. Fig. 3b and 3f shows an example where a single gold nanowire was bridged by much less Pt deposition. A 471M Ω resistance was obtained for this circuit. It is obvious that the connection between the nanowire and the gold electrode pads is insulating, indicating that high quality platinum bridge wires are essential for the electrical testing of single nanowires.

To circumvent the high line resistances arising from using e-beam Pt connections, two nanomanipulators were introduced to directly measure the electrical properties of the gold nanowires. The tungsten tips were milled by FIB to remove a surface oxide layer arising from the fabrication [6], then tip-to-tip contact was checked for ohmic contact. The linear I-V behaviour of the tip-tip contact with a constant 21.8 Ω resistance indicated a good ohmic contact. Two gold nanowires were measured as shown in Fig. 3c. By applying 10V for a few seconds, the bottom of a bundle of gold nanowires was nano-welded to one tungsten tip which had been checked to be conductive. This method ensures that the bundle of gold nanowires has a fully ohmic contact with one tip. Then another W tip was moved and connected to the end of two gold nanowires. A 6089 Ω resistance was obtained as shown in Fig. 3g. Using the above resistivity formula, a relative resistance value of $1.5 \times 10^{-5} \ \Omega \cdot m$ is calculated, which is double the value of $8.6 \times 10^{-6} \ \Omega \cdot m$ obtained for a bundle gold nanowires.



Fig. 3 Electrical testing of gold nanowires: (a) a bundle of gold nanowires and (b) a single gold nanowire, connected by e-beam deposition Pt to gold electrode pads on a $Si/SiO_2(100nm)$ substrate; (c) two nanowires and (d) a bundle of gold nanowires were measured by directly contact two nanomanipulator tips; (e)-(h) corresponding I-V behaviour of (a)-(d).

The most likely reason for the high values of resistivity in this case is extra contact resistance at the W tips contact with the gold nanowires. Possible reasons for this include a thin layer of tungsten oxide on the tungsten tips [6], the amorphous carbon and other contamination covering both tips and gold nanowires from the SEM chamber, and chemicals like sodium aluminate or sodium hydroxide from nanowire fabrication covering gold nanowire surface.

Both tungsten tips had been milled by FIB to remove surface oxide, and shortly thereafter were measured to have a fully ohmic tip-to-tip contact. However it is likely that a thin oxide/contamination layer formed afterwards in the SEM. It was found that scratching the tips against one another and then immediately contacting another bundle of gold nanowires as shown in Fig. 3d, resulted in the I-V behaviour shown in Fig. 3h. A 52 Ω resistance for this bundle of gold nanowires was obtained. A relative resistivity of $6.4 \times 10^{-7} \Omega \cdot m$ is obtained, 28 times of that of bulk gold. This data shows the dramatic effect of removing tip contamination on the measured resistance of the nanowire circuits.

6. Summary

In conclusion, gold nanowires were fabricated by AAO template method. Individual gold nanowires were polycrystalline, composed of sequential single crystal grains, in a bamboo-type structure. By means of nanomanipulators, single gold nanowires were picked up from holey carbon film copper grids and placed on a Si wafer. The electrical properties of gold nanowires were studied in circuits using both substrate electrodes and direct measurement using two nanomanipulators. The lowest circuit resistance is obtained by connecting the gold nanowires between tungsten tips whose surface oxide has been removed by FIB.

Acknowledgement

We would like to thank Mr. A Walker, Department of Electronic and Electrical Engineering, Sheffield University, for technical support. This work was supported by grant GR/585689 from the EPSRC, UK.

References

[1] Peng Y, Shen TH, Ashworth B, Zhao XG, Faunce CA and Liu YW 2003 Appl. Phys. Lett. 83 362.

[2] Jonge N de, Lamy Y, Kaiser M 2003 Nano Lett. 3 1621-1624.

[3] Wilms M, Conrad J, Vasilev, Kreiter M and Wegner G 2004 Appl. Surf. Sci. 238 490.

- [4] http://hypertextbook.com/facts/2004/JennelleBaptiste.shtml
- [5] Luxmoore I, 2007 PhD thesis, Sheffield University.

[6] Peng Y, Xu X J, Moebus G, Gay R, Cullis A G and Inkson B J 2006 IMC16 2 862.