Accurate electrical testing of individual gold nanowires by *in situ* scanning electron microscope nanomanipulators

Yong Peng,[1,a)] Tony Cullis,[2] and Beverley Inkson[1,b)]
[1]*Department of Engineering Materials, Sheffield University, Sheffield S1 3JD, United Kingdom*
[2]*Department of Electronic and Electrical Engineering, Sheffield University, Sheffield S1 3JD, United Kingdom*

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This work presents an accurate measurement of electrical properties of individual gold nanowires, directly measured by nanomanipulators *in situ* in a scanning electron microscope. The electrical testing of 55 nm width gold nanowires, with a bamboo-type polycrystalline micorstructure, shows that individual gold nanowires have an ideal resistivity of about 2.26 $\mu\Omega$ cm and remarkably high failure current density of 4.94×10^8 A cm^{-2}. The measurement of resistance (R) versus nanowire length of individual nanowires shows that the intrinsic conductivity of the gold nanowire is 4.45 $\times10^7$ Ω^{-1} m^{-1}. There is no evidence that the polycrystalline grain structure, 55 nm width and 500–2800 nm length, generates any size-induced electrical effects. The accurate electrical testing of gold nanowires should be significant for nanodevices and nanoelectronics using them as building blocks or interconnects. © *2008 American Institute of Physics*. [DOI: 10.1063/1.3005423]

Metallic nanowires have extensive applications as interconnecting leads and functional building blocks in nanodevices and nanoelectronics[1] because of their unique electrical properties. There is already significant effort on investigating the transport behavior[1–12] of individual metallic nanowires by means of various techniques, including prepatterned electrodes over deposited nanowires and use of scanning probes.[12] However, a common problem is that it is hard to obtain good and reproducible Ohmic contacts[6] at patterned electrode/nanowire and nanoprobe/nanowire interfaces during the measurement, which is believed to be the main reason why there is huge scatter in the electrical properties of individual metallic nanowires in reported work.[1–8,10,11] Nanomanipulator-based techniques are widely adopted using tungsten tips[4,6,9,10] as nanoprobes/nanotips. Unfortunately, tungsten tips are unavoidably covered by a thin layer of tungsten oxide during preparation or later during usage.[13] The isolating and uncontrollable oxide layer is the reason why there is huge scatter in bias voltages for individual gold nanowires in reported work.[4,6] However, if the Ohmic contact problem between nanoprobes/nanotips and nanoobjects could be resolved, nanomanipulators have some unique advantages for electrical testing at the nanoscale, such as direct connection connecting to samples at chosen locations, tip-resistance calibration, and avoidance of involved electrode fabrication steps.

Here we have developed a nichrome alloy nanoprobe which reliably forms a good Ohmic contact between nanoprobes/nanotips and nanowires. In this work, we report detailed and accurate electrical testing of individual gold nanowires by means of *in situ* scanning electron microscope (SEM) nanomanipulators equipped with the nichrome nanoprobes/nanotips. Gold nanowires are of major interest because they exhibit excellent electrical conductivity, stability in oxidizing environments, and are widely used for electrodes and interconnections in the semiconductor industry[2] and nanoelectronics research.[8,10] It is significant and essential to study accurately and reliably the electrical characterization of individual gold nanowires for their application in nanoscale devices and electronics, whether they are fabricated by top-down[2] or bottom-up techniques.[8]

Gold nanowires were fabricated by electrodeposition into nanoporous anodic aluminum oxide (AAO) templates as detailed in our previous work.[6] The morphology and microstructure of the electrodeposited gold nanowires were analyzed using a SEM (JEOL JSM 6500F, Japan) and a high resolution transmission electron microscope (TEM) (JEOL JEM-2010F, Japan). Individual Au nanowires were isolated by dissolving the AAO templates using 0.2*M* NaOH and rinsing with distilled water. The Au nanowires fabricated had a uniform size in both diameter and length. A representative TEM image of an individual Au nanowire can be seen in Fig. 1(a), which shows that the nanowires have a uniform diameter of about 55 nm. Individual nanowires have a polycrystalline bamboo-type microstructure with grain sizes of 20 nm to several micrometers long along the nanowire axis.[6] In the 55 nm Au nanowires investigated, only the fcc lattice structure was detected [Figs. 1].

Gold nanowires completely dissolved out of the AAO templates were dispersed on a SiO_2(100 nm)/Si wafer for the characterization of electrical properties. The electrical testing was carried out using two *in situ* SEM nanomanipu-

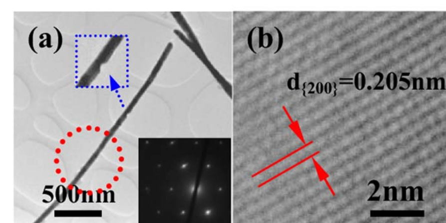

FIG. 1. (Color online) (a) TEM image of individual Au nanowires. The bottom-right inset shows the electron diffraction pattern of a $\langle 111\rangle_{ZA}$ orientation grain from red circle. The top-left indicates a local view of bamboo-type structure; (b) showing the fcc Au lattice structure, $d_{\{200\}}=0.205$ nm.

[a)]Electronic mail: y.peng@sheffield.ac.uk.
[b)]Electronic mail: beverley.inkson@sheffield.ac.uk.

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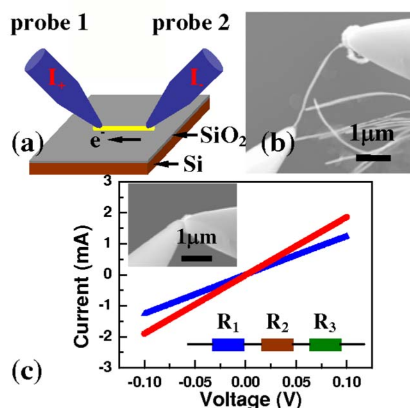

FIG. 2. (Color online) Resistance measurement of individual gold nanowires. (a) Schematic of the electrical measurement of a single nanowire between two nanomanipulator probes. (b) A typical SEM image of the electrical measurement of a single nanowire directly using two nanomanipulator nanoprobes. (c) *I-V* behavior of the gold nanowire (blue curve) shown in (b). A relevant resistance model is shown in the bottom-right inset, R_1–R_3. The red curve is the calibrated resistance of the two probes directly in contact as shown in the top-left inset.

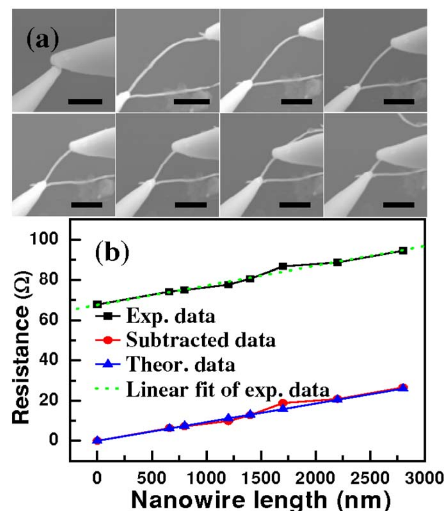

FIG. 3. (Color online) (a) SEM images of tip pair calibration and individual measurement positions used for determining the *R-L* curve in (b). All scale bars are for 1 μm; (b) resistance (R) vs nanowire length (L) taken and analyzed from the setup shown in (a). Black curve for the measured experimental data; green line for the linear fit of the experimental data; red curve for the analyzed data after subtracting the calibrated tip-to-tip resistance of 67.88 Ω; blue curve for theoretical values using the resistivity of bulk gold.

lators (Kleindiek Nanotechnik, Germany) equipped with a Keithley 6487 picoampermeter [Fig. 2(a)]. The data acquisition was automated using a dedicated multifunctional program. A typical SEM image of the experimental setup is shown in Fig. 2(b), where two conductive nichrome probes are tightly pressed on the nanowire body. In order to determine the correct nanowire resistance, we firstly measured the probe tip-to-tip contact resistance. An example of calibrated tip-to-tip resistance (53.80 Ω) is shown as the red curve in Fig. 2(c), which slightly changes for different probe pairs due to tip shape and other factors. The electrical transport data recorded from a single 55 nm diameter gold nanowire (blue line) are shown in Fig. 2(c), indicating a 79.01 Ω resistance. The current (I) versus voltage (V) behaviors of both the probes and nanowire circuit are linear.

The total resistance was independent of the applied voltage (0–0.10 V) and the observed current levels from tens of nanowires with various lengths typically exceeded 0.10 mA at 100 mV bias, showing remarkable low-resistance Ohmic contacts. This measured value includes the gold nanowire intrinsic resistance (R_1), the resistance of all contacts (R_2), and the feed line resistance of circuitry (R_3). The calibrated tip-to-tip resistance should be the sum of R_2 and R_3 and the feed line resistance of circuitry (R_3). A real resistance R_1 of the gold nanowires, 25.21 Ω for the nanowire in Fig. 2, can be then determined. The values of contact resistance between nichrome tips and gold nanowires were not found to be detectably larger than that of tip touching tip during our experiments, so we take a simple treatment that the contact resistance of tips/nanowire is equal to that of tip touching tip. Using the geometry of the measured nanowire, a calculation of the resistivity ($\rho=R\cdot A/L$, where R is the subtracted resistance, A the cross-sectional area of gold nanowire, and L the nanowire longitudinal length) yields a value of $\rho=2.26\ \mu\Omega$ cm. This is the same as the standardized bulk value of 2.27 $\mu\Omega$ cm (Ref. 14) at room temperature. Repeated measurements of further individual nanowires confirmed the accuracy and repeatability of our measurements.

This result shows that the Ohmic contact problem between nanoprobes and nanomaterials has been fully resolved in this work. Furthermore, the resistance of the 55 nm gold nanowires does not appear to be affected by the bamboo-type polycrystalline grain structure or by size-induced effects. This finding is consistent with the observation of Liu *et al.*,[3] but, it is different from other recent reports[7,10] which may be due to the problem of Ohmic contacts.

In order to double confirm the above measurements, the nanowire resistance (R) versus nanowire length (L) was determined for an individual nanowire [Fig. 3(a)], and is shown in Fig. 3(b). The original measured *R-L* data are displayed by the black curve with square points in Fig. 3(b), where the point at zero length is the calibrated tip-to-tip resistance data. The resistance of each point on this curve was obtained from the corresponding *I-V* curve with >200 data points. In order to obtain the most accurate wire length, values on the curve were mainly measured from SEM images of the contact point, taken after the electrical measurement of each point had been finished and the right-hand probe moved away. As shown in Fig. 3, the calibrated tip-to-tip resistance is 67.88 Ω for this tip pair. The linear behavior of R versus L indicates that the contact resistances between tips and nanowire are largely reproducible. The extrapolation for the length $L=0$ ($R_0=67.41$ Ω, obtained by a linear fit of the experimental data, marked by green line) gives the sum of all contact and feed line resistances of the circuitry. This fitted R_0 is the same as the calibrated tip-to-tip resistance within error, confirming that our electrical measurement by means of the nichrome tips is very accurate and reproducible, and that the nanoscale Ohmic contact problem has been fully resolved in our experiments.

The resistance values of the gold nanowire with different lengths can be obtained using the experimental data (black curve) and subtracting the 67.88 Ω calibrated tip-to-tip resistance. The subtracted curve of *R-L* is shown in red with solid circle points in Fig. 3(b). One can see that this curve nearly perfectly overlaps with a theoretical curve marked in blue with solid up-triangular points, which is calculated using the

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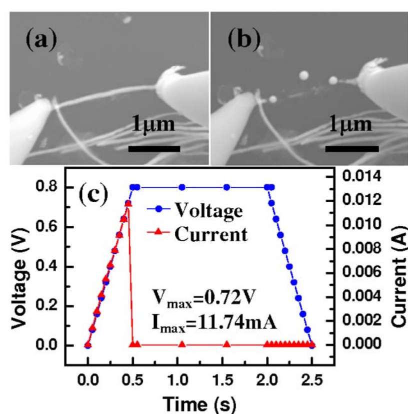

FIG. 4. (Color online) (a) Experimental setup before running a trapezoidal dc signal for the measurement of maximum transport current through a gold nanowire; (b) SEM image after running the dc signal showing the breakup and failure of the nanowire; (c) the recorded time-voltage and time-current curves during the whole experiment.

resistivity of bulk gold. There are two points on the curve having a very small deviation. The SEM images taken after the right-hand tip moved away did not show a visible contact point, indicating that relatively loose contacts of the right-hand tip on nanowire body could be the reason for the deviation. However, the *I-V* curves recorded from these two points were linear and stable although their Ohmic contacts were not ideal. We deduced that loose contacts in reported work[1–4,6–10,12] could be the reason why there were the huge scatters in the testing data of gold nanowires.

The intrinsic conductivity σ of the 55 nm diameter nanowire, $\sigma=\Delta L/\Delta R \pi r^2$, where $\Delta L/\Delta R$ is a inverse slope taken from the black curve of Fig. 2(b) and r is the diameter of gold nanowire determined from a high resolution TEM image of the same nanowire, is calculated to be $4.45 \times 10^7\ \Omega^{-1}\ \mathrm{m}^{-1}$. This is the same as the gold bulk conductivity value[14] of $4.52 \times 10^7\ \Omega^{-1}\ \mathrm{m}^{-1}$ within error. This result further indicates the finding that there are no detectable grain structure and size-induced effects on the electrical behavior of the 55 nm gold nanowires. Experimental data from more than ten individual gold nanowires support this finding.

The maximum transport current of the gold nanowires has also been quantified. In order to record accurately a maximum transport current, special software was developed which provides a trapezoidal voltage signal and synchronously records the relevant current at adjustable regular intervals (typically tens of milliseconds). A typical measurement is shown in Fig. 4. A failure current of 11.73 mA (recording a current reading each 50 ms) occurred on the 55 nm gold nanowire, of which the corresponding voltage was 0.72 V [Fig. 4(c)]. Repeated experiments showed the failure current mainly ranging from 9.5 to 11.73 mA. Small defects on the nanowire body influenced the maximum transport current of each particular gold nanowire. The experimental setups before and after running the trapezoidal dc signal for the measurement of maximum transport current are shown in Figs. 4(a) and 4(b). The maximum current density is calculated to be $4.94 \times 10^{12} \mathrm{A\ m}^{-2}$. This value is about 12 times that of a 15 nm diameter gold nanowire,[4] three times that of a 50 nm gold nanowire tested by AFM technique,[7] one and a half times that observed for 40–45 nm metallic NiSi nanowires,[5] and comparable to the value of single-walled carbon nanotubes.[15]

Three main reasons can explain why the measured maximum current density is higher and more accurate than others. Firstly, the Ohmic contact problem has been fully resolved in our experiments. Secondly, we have used real-time current detection during an exceptionally short total measurement time which effectively reduces the errors generated by Joule heating when current runs for a longer time. Thirdly, the direct contact of individual gold nanowires with nanomanipulators avoids the high contact resistance and contamination influence of prepatterned electrodes and metal-deposited interconnect wires which are usually used to form a circuit for resistance measurements. Like NiSi nanowires,[5] the failure of the individual gold nanowires also occurred in the middle, as shown in Fig. 4(b), indicating a Joule heating breakdown mechanism and electromigration[5] during the failure process. Here the determined values of J_{max} suggest that 50 nm gold nanowires are suitable to be used as interconnects or building blocks in bottom-up paradigm if integrated with Ohmic contacts.

In conclusion, we have demonstrated an accurate measurement of electrical properties of individual 55 nm gold nanowires using *in situ* SEM nanomanipulators. The resisitivity and conductivity have been obtained and found to be same as those of bulk gold at room temperature. There is no evidence that the polycrystalline grain structure, 55 nm width and 500–2800 nm length, generates any size-induced electrical effects. The electrical failure of gold nanowires has also been investigated. A remarkably high failure current density of $4.94 \times 10^{12}\ \mathrm{A\ m}^{-2}$ is measured. The accurate characterization of the transport properties intrinsic to the nanowires shown in this work is significant for nanowire applications in nanoscale devices and electronics.

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