

In situ nanowire growth for electrical interconnects

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Abstract

We report a technique to grow a single tungsten nanowire at a predetermined location by using field emission in a low-pressure tungsten carbonyl atmosphere. A sharp tip is first made to contact the intended location of growth. Electrical current is then passed through the contact. When the contact is broken, the resulting localized plasma discharge at the point of breakage causes a nanowire to initiate. Continued growth at low currents results in a single nanowire a few nanometres in diameter and up to tens of micrometres in length. The nanowire is overcoated by a thin carbon layer which protects the metal core from oxidation and corrosion. Electrical measurements of nanowires grown between two pads show resistivity one to two orders of magnitude higher than that of bulk tungsten. The technique can be applied to the interconnection of nanostructures to electrodes on a die.

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1. Introduction

The many interesting phenomena that occur at low dimensions have resulted in the development of many techniques to fabricate nanosized objects. 2D structures are commonly fabricated by thin film techniques which are relatively established. For example, epitaxy allows for precise control over the thickness and quality of the film and is used for the manufacture of devices, such as laser diodes and high electron mobility transistors. 1D structures are of even more interest since they represent the minimum dimensionality for electrical conduction. However, fabrication of 1D structures is also more difficult since it requires additional control over lateral dimensions and is normally accomplished by lithographic techniques. Unfortunately, for dimensions below 100 nm, the number of lithographic options is limited. One popular technique, electron beam lithography, allows the fabrication of 1D structures of less than 5 nm in width [1]. An alternative to patterning the object to achieve the required dimensions is to utilize objects such as fibres or whiskers that are of a naturally long cylindrical shape with nanoscale diameters.

Many techniques have been developed to grow nanowires [2]; however, manipulating them into position and providing electrical contacts pose a difficult challenge because

of their small dimensions. The most popular technique for providing electrical contacts to an *ex situ* grown nanowire is by dispersing the nanowire on a substrate followed by electron beam lithography and metallization to fabricate gold electrodes to the nanowire [3] or by direct focused ion beam induced deposition of the electrodes [4]. Other techniques exist to provide the contacts, including electric field assisted assembly in solution [5] and conducting AFM [6]. An alternative would be to directly grow the nanowire on the substrate. For example, Zhang *et al* [7] demonstrated the growth of aligned carbon nanotubes on a substrate by using the electric field between two biased electrodes. Calleja *et al* [8] used an AFM to form a gold nanowire between two electrodes by field-induced mass transport using a conducting AFM.

We had previously reported a new technique [9] to achieve controlled single nanowire growth on pointed objects by field emission in an organometallic ambient. Here we report an adaptation of the technique to achieve localized single nanowire growth at a predetermined location (such as metal electrodes) and initial electrical characterization of the nanowires. While the technique described in this paper is used to grow tungsten nanowires, nanowires of other materials have been similarly grown.

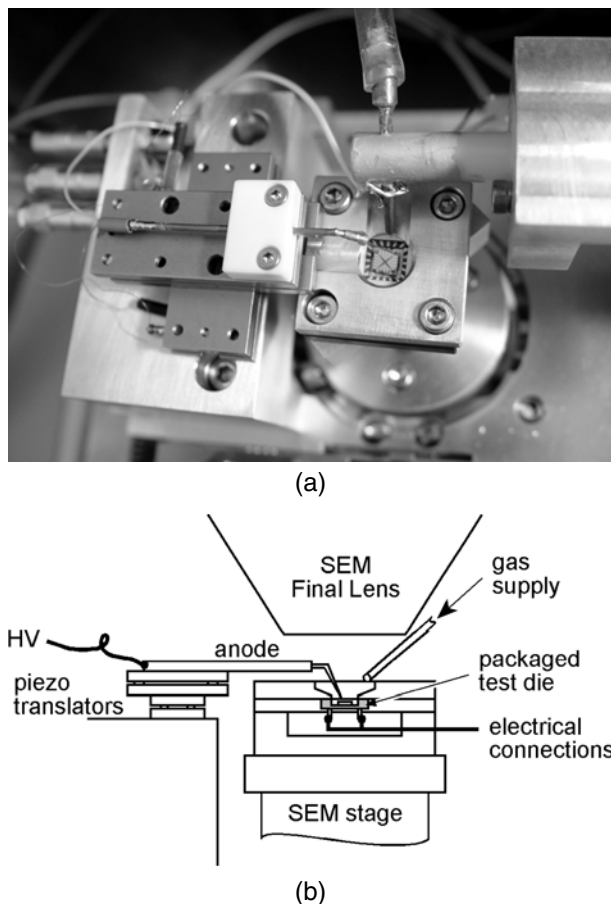


Figure 1. (a) Experimental set-up in SEM, (b) schematic.

2. Experiment

The experiment is set up in a Philips XL-30 FEG environmental scanning electron microscope (ESEM) for accurate positioning as well as *in situ* observation of the growth. An electrochemically etched tungsten tip is used as an anode to localize the electric field to the point where the nanowire is to be grown and as a probe to initiate the growth. The substrate where the nanowire is to be grown is mounted on the SEM stage. The experimental set-up is shown in figure 1. The anode tip is mounted on a piezo-stage for lateral movement. Vertical movement is accomplished by moving the substrate up and down on the SEM stage. A gas nozzle with a diameter of about 2 mm, positioned by micropositioners close to the die, is used to introduce the precursor vapour. The nozzle is connected to a heated canister of tungsten carbonyl via a needle valve to regulate the flow. The SEM is used to position the substrate and the anode tip. The anode tip is connected to a Keithley 237 high-voltage source measure unit (SMU) through a 500 M Ω series resistor. This resistor serves two purposes: (i) it protects the sensitive electronics of the SMU from damage in the event of an arc discharge, and (ii) given the relatively slow response of the SMU, it also helps maintain a constant current while field emitting since an increase in current through the resistor will automatically result in a voltage reduction at the anode.

The substrate used for the experiment is a custom-made die with multiple pairs of gold-coated aluminium electrodes

(600 nm thick), each 5 μm wide and separated by a gap of 0.8 μm , on PECVD silicon oxide (400 nm thick). The die is bonded to a small IC package (20LCC) and the package is either grounded to a SEM specimen stub via carbon tape or inserted into an IC socket for *in situ* electrical measurements. The nanowire is to be grown on one electrode and allowed to fall under its own weight onto the adjacent electrode. However, most of the wires tend to land on the oxide instead. This is due to the charging of the oxide surface by the SEM electron beam during viewing which attracts the grown wires to fall there when the anode bias is terminated. To increase the chance of a nanowire landing on an opposing electrode, a patterned gold layer on the opposing electrode is shaped like a U, enclosing the electrode where the nanowire is grown.

To initiate the growth, the probe is first made to contact the substrate. This is accomplished by first introducing a positive bias of a few tens of volts to the anode tip, which manifests itself as voltage contrast in the SEM image. The substrate is then raised to meet the anode tip. When contact is established between the two, the anode becomes grounded and the voltage contrast disappears. The current limiting resistor (500 M Ω) prevents an excessive amount of current from flowing. The SMU is then set to source a current of 1 μA with a compliance voltage of 1000 V. A potential of 500 V will be dropped across the 500 M Ω buffer resistor. Tungsten carbonyl vapour is introduced into the chamber via the nozzle till the chamber pressure is about 5×10^{-5} mbar. However, the pressure at the nozzle itself is estimated to be approximately 10^{-2} mbar. The die is then slowly lowered till the contact between the metal electrode and anode probe is broken. Unfortunately this coarse contacting technique often results in mechanical damage of the electrode in the form of a shallow crater by the sharp tungsten anode, but this problem can be minimized by a finer vertical positioning mechanism (such as a piezo translator) for the die. The damage is reduced if a sharp anode tip is used. However, the growth process also causes dendritic formations on the anode by electron beam induced deposition. If growth were to be repeated on another electrode, the deposits on the anode will fall off the anode onto the electrode, thereby placing a limit on the number of times the anode tip can be re-used.

Upon breaking the contact, a potential of 500 V immediately forms between the metal electrode of the die and the anode probe, resulting in a discharge. The discharge results in a raised protrusion forming at the metal electrode (cathode). The formation of a nanotip can be observed as a rapid reduction of the voltage at the SMU, indicating that a field emission source had formed at the electrode. The voltage from the SMU is immediately terminated and a short and sharp field emission tip has now been formed at the electrode. The actual nanowire growth can then be continued at a different current if desired.

We had previously observed that low current growth (<100 nA) on field emission tips results in single nanowire growth whereas higher current growths tend to result in multiple (forked) nanowire growth. However, growth on the metallic pads on the dies while sourcing a constant current almost always results in single nanowire growth. We believe that the nanowire length away from the tip is electrostatically shielded by the substrate and thus suppresses field emission from nascent side branches. Low current growth gives rise to nanowires of a few nanometres in diameter that will fall

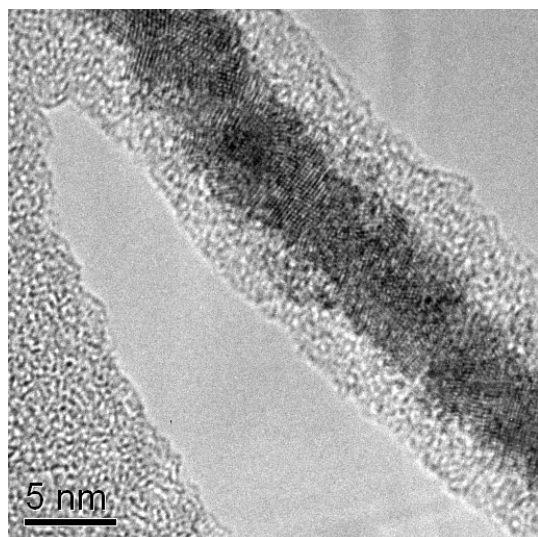


Figure 2. High resolution TEM of a tungsten nanowire shows its polycrystalline morphology.

under their own weight onto the substrate after growth. Higher current growth (a few microampere), however, results in a thicker nanowire with diameters of a few tens of nanometres that is normally capable of standing upright if the length is not too long. High-resolution TEM observation of the nanowires show that the nanowires are polycrystalline with disordered regions (figure 2). The nanowire is also overcoated with a layer of carbonaceous material believed to be a by-product of the growth process.

3. Electrical measurements

Electrical measurements on the grown nanowires straddling the two metal pads (figure 3) were performed in high vacuum inside a SEM to help preserve the integrity of the nanowire against oxidation as the nanowire heats up. The SEM also allowed real-time observation of the nanowire while it is being tested. Both electrodes were patterned by electron-beam lithography, gold metallization and liftoff, being placed on top of or connected to aluminium metal lines leading to external bond pads. The measurements, carried out with an externally connected parameter analyser (HP4155A), are essentially two-point measurements and thus include the effect of the contact resistance between the nanowire and the gold metallization. The resistance of the large-scale metal lines can be neglected.

Initial measurement shows the nanowire to have very high resistance. However, it was later established that the high resistance originates from the contact of the nanowire and the metallic pad rather than the nanowire itself. Previous studies on conducting AFM [10] show that a considerable amount of pressure is needed on the tip before good electrical contact can be established. Since our nanowire is simply resting on the electrodes and is furthermore overcoated by a carbonaceous layer, poor electrical contact is not surprising. To overcome the high contact resistance, current is forced through the contact until electrical breakdown occurs. This is accomplished by slowly ramping the voltage and setting a low current compliance limit. On the first voltage ramp from

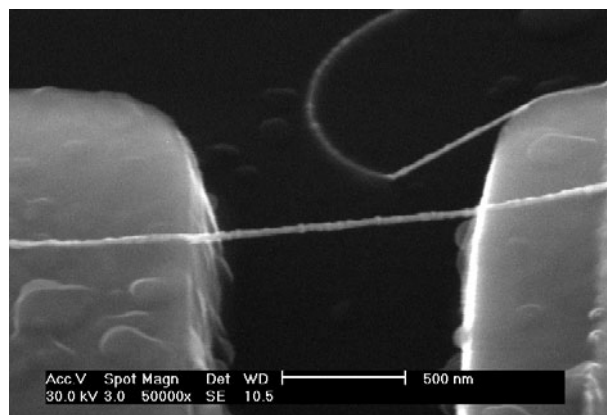


Figure 3. Nanowire straddling two metal electrodes. The wire appears thicker due to contamination build-up during SEM viewing.

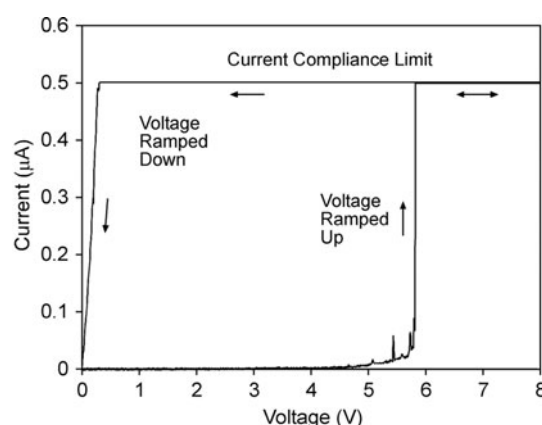
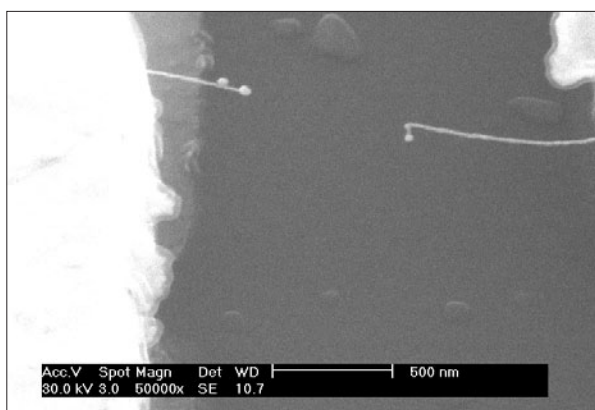
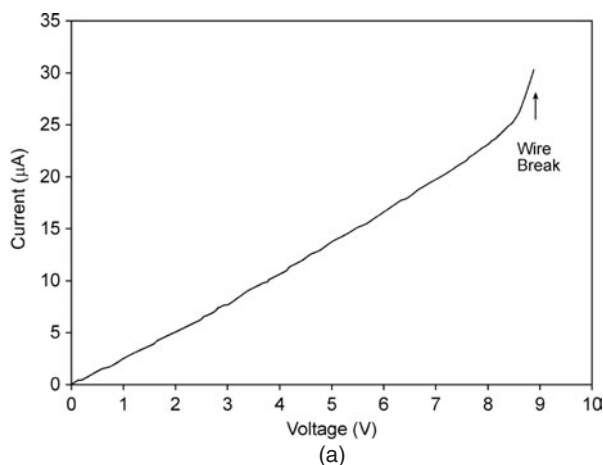


Figure 4. Initial I - V sweep of a nanowire lying on a gold electrode. Contact resistance breakdown occurs during voltage ramp-up, with the current reaching a preset current compliance limit of $0.5 \mu\text{A}$.

0 V to typically around 10 V, the current rises slowly until it jumps suddenly (figure 4). The low current compliance ensures that wire is not destroyed by the current surge. On the backtrace, and subsequent retrace, the wire shows ohmic characteristics. This observed behaviour is thought to be due to the tunnelling through, and finally breakdown of, the poorly conducting overcoat layer between the metal nanowire core and the gold electrode on which it is lying. The current compliance is slowly increased and the process repeated until there is no further change in resistance with increasing current.

The diameter of the nanowire could not be measured directly by height measurements in a tapping-mode AFM as the metal electrodes tend to obstruct the AFM. Instead, diameter measurements are taken from separately grown nanowires. For such measurements, a nanowire is grown from a bare electrode on a substrate to a sufficient length such that it falls onto the surrounding flat oxide after the removal of the electric field. AFM scans are carried out and the height is determined as an average of several line scans across the wire. This average height then represents the wire diameter inclusive of the overcoat. For small geometries, the lateral dimensions returned by the AFM are not accurate due to the convolution of the tip shape with the much smaller geometry the tip is measuring. The smallest tungsten wires grown at 20 nA field-emission current have a diameter between 7 and



(b)

Figure 5. (a) I - V characteristics of a nanowire tested to destruction. (b) Formation of balls at the ends of the nanowires, indicating the nanowire had melted during testing.

9 nm. After taking into consideration the thickness of the carbonaceous coating, the inner metallic core is estimated to be about 5 ± 2 nm, in general agreement with TEM measurements for nanowires grown on holey carbon grids.

The measured resistance values are typically a few hundred kilo-ohms to just under $1 \text{ M}\Omega$ for wire lengths of several microns, corresponding to a resistivity of one to two orders of magnitude higher than the bulk value of $5.4 \mu\Omega \text{ cm}$ for tungsten. It is reiterated that the measured resistance also includes residual contact resistance that has not been totally eliminated. Higher accuracy four-point measurements are being attempted. The resistance of nanowires has been known to increase as diameters are decreased [11] and is attributed to grain boundary and surface scattering. Resistivities of disordered metals are also known to be much higher than their crystalline counterparts. More detailed measurements are currently being carried out to understand the transport behaviour of these nanowires.

The maximum current before the nanowire is destroyed ranges from 10 to a $100 \mu\text{A}$ (figure 5(a)), corresponding to a current density of $5\text{--}50 \times 10^{11} \text{ A m}^{-2}$. SEM observations of the nanowires during destruction indicate that the wire melted (figure 5(b)). This is supported by the formation of balls at the ends of the nanowire after testing to failure.

The technique can be extended to initiate growth from a nanostructure that is typically vertically aligned. Provided the

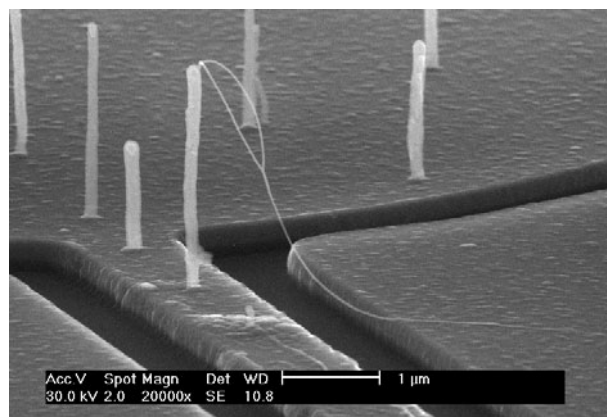


Figure 6. A carbon nanotube–nanowire junction was grown and allowed to straddle across two metal electrodes.

nanostructure is of a fairly small tip radius for field emission to take place, and reasonably conductive, the initiation of nanowire growth from the tip does not require physical contact of the anode and avoids mechanically damaging delicate nanostructures. However, it should be noted that the field-emission current flows through the nanostructure and thus the structure must be able to sustain this current without damage. This technique is demonstrated by contacting a vertically aligned carbon nanotube (CNT), thereby forming a CNT–nanowire junction in the process (figure 6). Various nanowires including iron and cobalt have been grown from a CNT tip in the same way.

4. Conclusions

A technique has been developed to grow and contact a tungsten nanowire across two bare metal electrodes. The nanowire growth is initiated by touching the first electrode with a biased anode and breaking the contact. The nanowire then grows towards the anode which can be positioned to lead the general direction of growth. Upon removal of the anode bias, the nanowire will fall onto the adjacent electrode. Smaller diameter nanowires are, however, susceptible to attraction by charged insulator surfaces. The diameter of the smallest nanowires is estimated to be in the range of 3–7 nm. Initial estimates of the nanowire resistivity indicate values about one to two orders higher than that of bulk tungsten. Alternatively, a similar approach can be adopted to grow nanowires on sharp pointed nanostructures that are capable of field emission without the need for anode contact, and thereby provide a method for interconnection to such structures.

M Multimedia is available from stacks.iop.org/Nano/15/687. The video clip, recorded in the SEM, shows the growth of a nanowire initiated by first contacting the substrate with a biased anode. A nanowire is seen to grow towards the anode as the latter is withdrawn from the surface. The noise in the video is due to the electrons that are field emitted by the growing nanowire, while the erratic image movement is due to SEM. Primary electron beam deflection by the changing anode bias to maintain constant current growth. This nanowire was grown under high currents (> 1 microamp) so that the relatively thick wire is visible under otherwise noisy imaging conditions.

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