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In situ Electrical Property Characterization of Individual Nanostructures Using a Sliding Probe Inside a Transmission Electron Microscope

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Abstract*—A sliding probe technique has been developed for the in situ electrical property characterization of individual nanostructures inside a transmission electron microscope (TEM) using a nanomanipulator. Experimental investigation into the transport measurement of copper-filled carbon nanotubes, carbide nanowires, and carbon microfiber has shown the effectiveness of this method. Comparing with conventional 4-point methods, the proposed setup is simple and agile and it can be readily combined with TEM-based imaging and analysis. Comparing with conventional 2-point methods, the sliding probe method are characterized by (1) the contact resistance can be partially eliminated and (2) sectional measurement using this method is particularly adaptable to non-uniform structures or hetero-structures.

***Index Terms* — Sliding probe technique, nanostructures, electrical property, in situ technology, nanomanipulation**

I. INTRODUCTION

The interest in transport property characterization of individual nanostructures such as nanotubes (NTs) and nanowires (NWs) has been greatly stimulated by their potential applications in electronic, electromechanical, sensing, actuation, and electrochemical systems [1-3]. Most often, individual nanostructures are electrically bridged between two nanofabricated electrodes. Two-point [2, 4] and four-point [1, 5-7] methods using either fixed electrodes or movable probes have been typically applied in the measurements. Two-point techniques do not allow the determination of the intrinsic resistance due to the contacts that lie in the loop, but it has advantages for *in situ* characterization, especially for electromechanical coupling property characterization, due to its simplicity and agility when a manipulation probe is applied. A standard solution to eliminate the contribution of contacts is the four-point measurement. However, the application of this technique to an individual nanostructure is challenging, since it is difficult to fabricate nanoelectrodes or probes with nanoscale separations and to make contacts of four probes to an individual nanostructure, particularly when it is free standing. Here we propose a sliding probe technique, in which a manipulation probe is used together with a fixed electrode or probe for the electrical property characterization as schematically shown in Fig. 1.

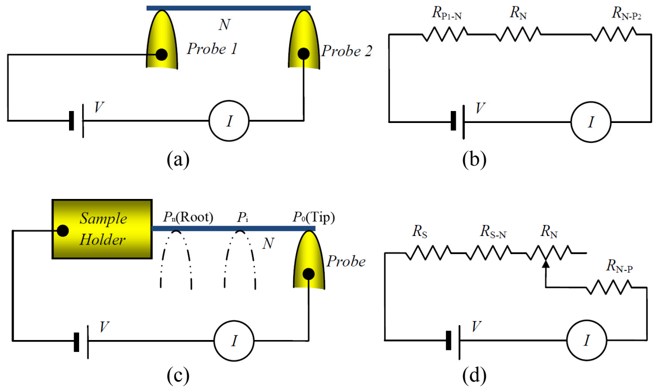

Fig. 1. Schematic of electric property characterization of an individual nanostructure. (a) Conventional two-point method for *in situ* electric property characterization. (b) Equivalent electric circuit of the conventional two-point method, where $R_{P1\text{-}N}$, R_N, and $R_{N\text{-}P2}$ represents the contact resistance of probe 1 to the nanostructure, the resistance of the nanostructure, and the contact resistance of probe 2 to the nanostructure, respectively. (c) Sliding probe technique for *in situ* electric property characterization. By contacting a probe to different points (P_0 (tip), P_1, P_2, …, P_i, …, and point P_n (root)) on an individual nanostructure, the resistance of the nanostructures is measured. (d) The equivalent circuit of the measurement loop, where R_S, $R_{S\text{-}N}$, R_N, and $R_{N\text{-}P}$ represents the resistance of the sample holder, the contact resistance of the sample holder to the nanostructure, the resistance of the nanostructure and the contact resistance of the probe to the nanostructure, respectively.

Conventionally, *in situ* electric property characterization is performed by contacting two probes on the two ends of a nanostructure as schematically illustrated in Fig. 1(a). In this setup, the contact resistance between the probes and the sample is included inside the measurement loop. The measurement accuracy is determined by the ratio of $(R_{P1\text{-}N} + R_{N\text{-}P2})/R_N$ (Fig. 1(b)), where $R_{P1\text{-}N}$, $R_{N\text{-}P2}$, and R_N represents the contact resistance between the probes 1 and 2 and the nanostructure, and the intrinsic resistance of the nanostructure, respectively. Technically, it is possible to improve the contact by coating the probes with low-resistance materials, soldering the nanostructure onto the

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probes, or applying a compressing force using the probes to the nanostructure. However, when the resistance of the nanostructure is close to the magnitude of the contact resistance, the conventional two-terminal method fails.

To improve the accuracy, we have developed a different process, which is named sliding probe methods as schematically shown in Fig. 1(c). By contacting a probe to different local positions (e.g. the tip or the root) of an individual nanostructure, the resistance of the nanostructure can be measured by finding the difference between the two measurements, and the uniformity of the resistance or the sectional resistance can be obtained using three or more local positions, which is also of particular interest for hetero-nanostructures. Fig. 1(d) shows the equivalent circuit of the measurement loop, where R_S, $R_{S\text{-}N}$, R_N, and $R_{N\text{-}P}$ represents the resistance of the sample holder, the contact resistance of the sample holder to the nanostructure, the resistance of the nanostructure and the contact resistance of the probe to the nanostructure, respectively. The overall resistance when the probe contacts to the tip and the root can be expressed as $R_{Tip} = R_S + R_{S\text{-}N} + R_{N,\,Tip} + R_{N\text{-}P,\,Tip}$ and $R_{Root} = R_S + R_{S\text{-}N} + R_{N,\,Root} + R_{N\text{-}P,\,Root}$, respectively. So, the resistance of the nanostructure between the point of the tip and root is $R_N = R_{N,\,Tip} - R_{N,\,Root} = R_{Tip} - R_{Root} - R_{N\text{-}P,\,Tip} + R_{N\text{-}P,\,Root}$. It can be seen that the resistance of the sample holder and the contact resistance between the sample holder R_S and $R_{S\text{-}N}$ are eliminated. Hence, the difference between the two measurements when the probe contacts to the tip and the root of the nanostructure reflects the resistance of the nanostructure (between the tip and the root) supposing that the contact resistance between the probe and the nanostructure are the same for the two cases. Comparing with the conventional two-terminal method as shown in Fig. 1(a) and (b), only one side of the contact resistance is brought into the measurements. Hence, this sliding probe method holds higher accuracy for *in situ* electric property characterization whereas keeps simplicity as comparing with 4-terminal method. Furthermore, it is more feasible to keep the contact resistance between the probe and the nanowire at different positions (tip and root) to the same scale ($R_{N\text{-}P,\,Tip} \approx R_{N\text{-}P,\,Root}$) than to eliminate the contact resistance ($R_{P1\text{-}N} \approx 0$ and $R_{N\text{-}P2} \approx 0$) at all.

On the other hand, due to the simplicity of this technique, it can be readily combined with transmission electron microscopy (TEM) and analytical technologies based on TEM. The limited space inside a TEM holder had constrained the application of 4-point measurement. The proposed method provides the unique capability to correlate the internal atomic structure or mechanical strain to the transport properties of the same nanostructure, which is of growing interest due to the dependence of the transport properties of nanostructures on their morphology, structural uniformity, defects, etc.

II. EXPERIMENTAL SETUP

The electrical properties of individual copper-filled carbon nanotubes (Cu-filled CNTs) [8], carbide nanowires (NWs) [9], and carbon microfibers are characterized using an STM-TEM (scanning tunneling microscope-transmission electron microscope) holder (FM2000E, Nanofactory Instruments AB). Our experiments were performed in a TEM (JEOL 2200FS) with a field emission gun. The raw materials were attached to a 0.35 mm thick Au wire using silver paint, and the wire was held in the specimen holder. The probe was an etched 10 μm thick tungsten wire with a tip radius of approximately 100 nm (Picoprobe, T-4-10-1 mm). To improve the conductivity, the probes were coated with Au thin film (thickness: c.a. 21 nm). The probe can be positioned in a millimeter-scale workspace with subnanometer resolution with the STM unit actuated by a three-degree-of-freedom piezotube, making it possible to select a specific object.

III. TWO-POINT SLIDING PROBE MEASUREMENTS

Fig. 2 is the current-voltage (IV) characterization of an individual Cu-filled CNT using the sliding probe method. The Au-coated STM probe contacts to the tip (Inset: top left) and the root (Inset: bottom right) of the nanotube in two measurements, respectively. A bias is applied between the sample holder and the probe and swept from -500 mV to 500 mV. The resistance is found to be 68.1 MΩ at 500 mV. It can be confirmed by comparing the two images that no obvious changes have occurred on the encapsulated materials during the measurement.

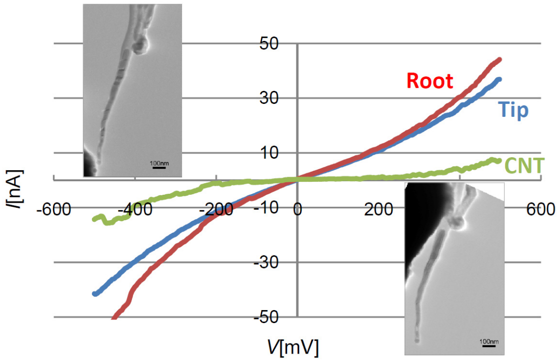

Fig. 2. IV characterization of a Cu@CNT.

Fig. 3 is the IV characterization of an individual TaC nanowire. The result using the sliding probe method at the tip and root is shown in Fig. 3(a). The STM probe contacts to the tip (Inset: top left) and the root (Inset: bottom right) of the nanowire in two measurements, respectively. A bias is applied between the sample holder and the probe and swept from -500 mV to 500 mV. The resistance is found from the slope of the linear IV curves. The diameter and the length of the nanowire are 292.0 nm and 10.0 μm, respectively. The resistivity of the nanowire is then calculated to be 38.1 μΩ·cm in average based on 5 measurements. This is comparable to the resistivity of bulk TaC materials [10] (137 μm thick and 4.6 cm long: 32.7-117.4 μΩ·cm for a variety of compositions). The fact that the measured value is closer to the lower limit can be contributed to the composition similarity and/or scaling effects. Fig. 3(b) shows the results using conventional 2-terminal method. A TaC nanowire is picked up onto an STM probe at the root of the nanowire,

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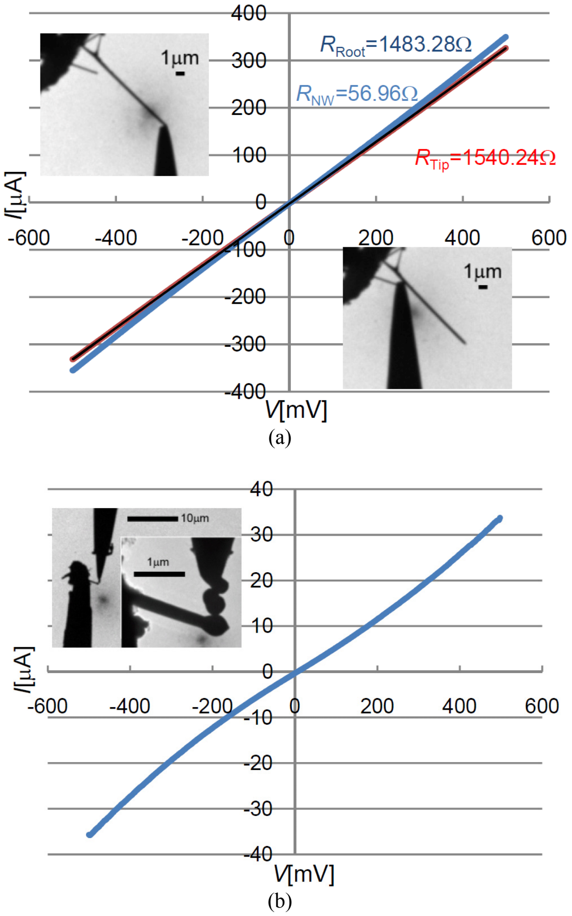
and then made contact to another STM probe at the tip (Inset, with an enlarged central part). Both probes are coated with Au (Thickness: 21 nm). A bias is then applied between the two probes and swept from -500 mV to 500 mV. The diameter and the length of the nanowire are 240 nm and 1.8 μm, respectively. The IV curve can be fitted perfectly with a degree 3 polynomial: $I = 5.0\times10^{-8}V^3 - 3.1\times10^{-6}V^2+5.8\times10^{-2}V - 0.3$, where I and V are in μA and mV, respectively. The resistivity of the nanowire at 500 mV is calculated to be 37.5 mΩ·cm in average. This is significantly different from that measured by tip-root method and previous measurements [10]; showing that the contact resistance occupied a large portion, and the measured value has much less accuracy than that obtained from sliding probe technique.



(a)

(b)

Fig. 3. IV characterization of a TaC nanowire using the sliding probe method and conventional 2-point method. (a) Sliding probe method: an STM probe contacts to the tip (Inset: top left) and the root (Inset: bottom right) of the nanowire in two measurements, respectively. A bias is applied between the sample holder and the probe and swept from -500 mV to 500 mV. The resistance is found from the slope of the linear IV curves. The diameter and the length of the nanowire are 292.0 nm and 10.0 μm, respectively. The resistivity of the nanowire is then calculated to be 38.1 μΩ·cm in average based on 5 measurements. (b) Conventional 2-point method: a TaC nanowire is picked up onto an STM probe at the root of the nanowire, and then made contact to another STM probe at the tip (Inset, with a magnified central part). A bias is then applied between the two probes and swept from -500 mV to 500 mV. The diameter and the length of the nanowire are 240 nm and 1.8 μm, respectively. The resistivity of the nanowire at 500 mV is calculated to be 37.5 mΩ·cm in average.

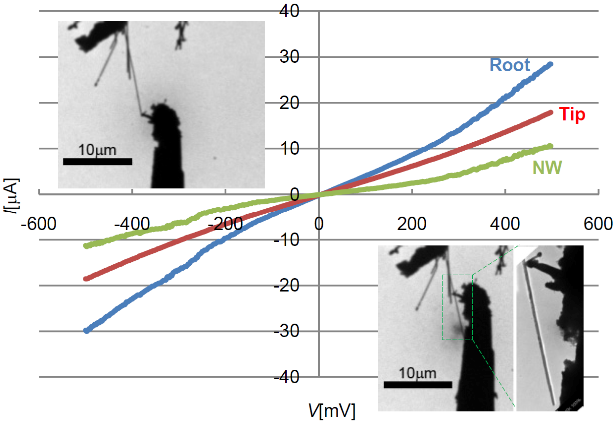


Fig. 4. IV characterization of a NbC nanowire. An STM probe contacts to the tip (Inset: top left) and the root (Inset: bottom right, with a magnified central part) of the nanowire in two measurements, respectively. A bias is applied between the sample holder and the probe and swept from -500 mV to 500 mV. The resistance of the nanowire is found from the difference of the IV curves. The diameter and the length of the nanowire are 146.3 nm and 4.9 μm, respectively. The resistivity of the nanowire is then calculated to be 15.5 mΩ·cm in average based on 5 measurements.

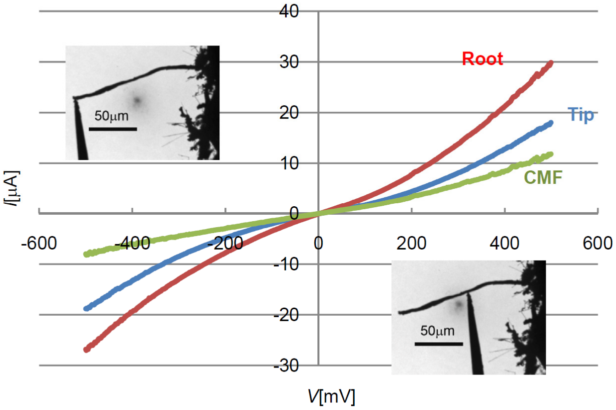


Fig. 5. IV characterization of a carbon microfiber from which NbC nanowires have been grown. An Au-coated STM probe contacts to the tip (Inset: top left) and the root (Inset: bottom right, with a magnified central part) of the carbon microfiber in two measurements, respectively. A bias is applied between the sample holder and the probe and swept from -500 mV to 500 mV. The resistance of the carbon microfiber is found from the difference of the IV curves. The diameter and the length of the microfiber are 3.3 μm and 72.5 μm, respectively. The resistivity was calculated to be 493.6 mΩ·cm in average based on 5 measurements.

The sliding probe can be applied in a variety of nano and microstructures. Fig. 4 is a typical result on a NbC nanowire. The diameter and the length of the nanowire are 146.3 nm and 4.9 μm, respectively. The resistivity of the nanowire is then calculated to be 15.5 mΩ·cm in average based on 5 measurements. Fig. 5 shows the IV curves obtained with tip-root method of carbon microfibers (CMFs) from which NbC nanowires have been grown. The average diameter and the length of the microfiber are 3.3 μm and 72.5 μm, respectively, and the resistivity is calculated to be 493.6 mΩ·cm. It can be found by comparison that the conductivity of TaC NWs is three orders of magnitude better than that of the NbC NWs, and both types of NWs are better conductors than the CMFs.

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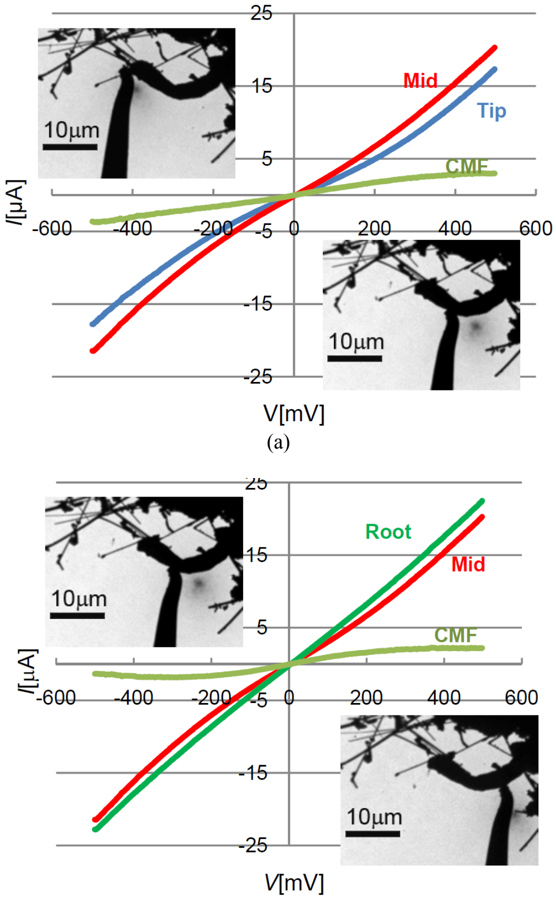
IV. MULTI-POINT SLIDING PROBE MEASUREMENTS



(a)

(b)

Fig. 6. IV characterization of a carbon microfiber (CMF) from which TaC nanowires have been grown. (a) Tip-mid-root 3-point method: An STM probe contacts to the tip (Inset: top left) and the mid (Inset: bottom right) of the CMF in two measurements. A bias is applied between the sample holder and the probe and swept from -500 mV to 500 mV. The resistance is found from the slope of the linear IV curves. The diameter and the length of the carbon microfiber are 3.0 μm and 6.1 μm, respectively. The resistivity of the carbon microfiber at 500 mV is calculated to be 17.9 Ω·cm in average based on 5 measurements. (b) An STM probe contacts to the mid (Inset: top left) and the root (Inset: bottom right) of the carbon microfiber in two measurements. A bias is applied between the sample holder and the probe and swept from -500 mV to 500 mV. The resistance is found from the slope of the linear IV curves. The diameter and the length of the nanowire are 2.9 μm and 7.7 μm, respectively. The resistivity of the CMF at 500 mV is calculated to be 19.4 Ω·cm in average based on 5 measurements.

For non-uniform or hetero-nanostructures, more measurement points can be applied to understand the sectional properties. Fig. 6(a) and (b) are the results on a microfiber from which TaC nanowires have been grown by using 3-point sliding probe method. The diameter and the length of the microfiber from the tip to mid is 3.0 μm and 6.1 μm, respectively, and the resistivity at 500 mV is calculated to be 17.9 Ω·cm in average based on 5 measurements. The diameter and the length of the microfiber from mid to root is 2.9 μm and 7.7 μm, respectively, and the resistivity is calculated to be 19.4 Ω·cm in average based on 5 measurements.

V. CONCLUSIONS

In summary, we have proposed a sliding probe method for the electrical property characterization of individual nanostructures inside a TEM. Experimental investigation into the transport measurement of copper-filled carbon nanotubes, carbide nanowires, and carbon microfiber has shown the effectiveness of this method. Comparing with conventional 4-point methods, the proposed setup is simple and agile and it can be readily combined with TEM-based imaging and analysis. Experimental investigation on TaC NWs has shown that higher measurement accuracy can be obtained using the sliding probe method than conventional 2-point method because the contact resistance can be partially eliminated. Sectional measurement using multi-point sliding probe method is particularly adaptable to non-uniform and hetero-structures.

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