# Sliding Probe Methods for *In Situ* Nanorobotic Characterization of Individual Nanostructures

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Abstract—Sliding probe methods are designed for the *in situ* characterization of electrical properties of individual 1-D nanostructures. The key to achieving a high resolution is to keep the contact resistance constant by controlling the contact force and area between the specimen and the sliding probe. We have developed several techniques and tools including differential sliding, flexible probes, and specimen-shape-adaptable probes using nanorobotic manipulation. Compared with conventional methods, these sliding probe methods allow *in situ* characterization with a higher resolution than conventional methods. Furthermore, they are superior for local property characterization, which is of particular interest for heterostructured nanomaterials and defect detection.

*Index Terms*—Electrical transport property, individual nanostructures, *in situ* nanotechnology, nanorobotic manipulation, sliding probe methods.

# I. INTRODUCTION

T HE *in situ* characterization of electrical transport properties of individual nanostructures is of growing interest for accelerating the development of novel nanomaterials, correlating their transport properties with their atomic structures and selecting suitable building blocks for electronic, sensing, actuation, electromechanical, or electrochemical systems [1]–[6]. Furthermore, since the characterization is an important application of the robotic techniques, the investigation of the nanorobotic *in situ* characterization on the nanomaterials is an important start for the development of large-scale/automated nanomanipulation-based nanoassembling or molecular manufacturing [7].

Manuscript received October 27, 2014; revised September 5, 2014; accepted October 11, 2014. Date of publication January 15, 2015; date of current version February 4, 2015. This paper was recommended for publication by Associate Editor S. Régnier and Editor B. J. Nelson upon evaluation of the reviewer's comments. This work was supported by the National Science Foundation (NSF) under Grant IIS-1054585, the National Natural Science Foundation of China under Grant 51002138, the Zhejiang Provincial NSF of China under Grant LR13E020002, the NSF (CMMI-0968843, CMMI-0824728, CMMI-0653651), the Qianjiang Talent Project (2010R10029), the "Qianjiang Scholars" program and the project sponsored by the Project sponsored by SRF for ROCS (2010609), SEM.

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Digital Object Identifier 10.1109/TRO.2014.2367331



Fig. 1. Schematic of electrical transport property characterization of an individual nanostructure. (a) and (b) Conventional two- and four-terminal methods. (c) Sliding probe methods for *in situ* electric property characterization. The resistance of the nanostructure is measured by contacting a probe to different points  $[P_0 \text{ (tip)}, P_1, \ldots, P_n \text{ (root)}]$  on an individual nanostructure. (d) Equivalent circuit of the measurement loop, where  $R_H$ ,  $R_{H-S}$ ,  $R_S$ , and  $R_{S-P}$  represent the resistance of the sample holder, the contact resistance of the sample holder to the sample (a nanostructure), the resistance of the sample, and the contact resistance of the probe to the sample, respectively. The method can be used to investigate a variety of nanostructures, particularly suitable for local transport measurement for a heterostructure (e), a structure with local defects/doping (f), or a nonuniformed one (g). Enhanced techniques such as multipoint continuous sliding or differential ( $n \rightarrow \infty$ ) sliding, together with flexible probes (h) and further improve its accuracy.

Conventionally, two-terminal methods [2], [8], as shown in Fig. 1(a), and four-terminal methods [1], [9]–[15], as shown in Fig. 1(b), using either fixed electrodes or movable probes, have been applied in such measurements. Theoretically, as shown in Fig. 1(a), two-terminal methods do not allow the determination of the intrinsic electrical transport properties due to the contact resistance between the electrodes/probes and the sample that lies inside the measurement loop. The measurement accuracy is determined by the ratio of  $(R_{P1-S} + R_{S-P2})/R_S$ , where  $R_{P1-S}$ ,  $R_{S-P2}$ , and  $R_S$  represent the contact resistance

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between probes 1 and 2, 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 probes, or compressing the nanostructure using the probes [16]. Two-terminal methods were applied to the *in situ* characterization, especially for the electromechanical coupling property, due to their simplicity and flexibility when a manipulation probe is used [2], [8]. However, when the resistance of the nanostructure is close to the magnitude of the contact resistance, the conventional two-terminal method fails. A standard method that eliminates the effect of contact is the four-terminal measurement, but it is challenging to apply this technique to an individual nanostructure. First, it is difficult to fabricate nanoelectrodes or probe arrays with separation in the nanoscale [labeled as S in Fig. 1(b)]. Second, additional degrees of freedom are needed for orienting the holder of the array so that the four probes can make contact with an individual nanostructure, which is difficult to achieve using even the most up-to-date nanorobotic manipulators. Third, it is particularly difficult to simultaneously make contact with a low-dimensional structure such as nanotubes (NTs) or nanowires (NWs) in four points, especially when they are free-standing. In a conventional method, the fourprobe method was carried out on the nanostructures with lattice stage underneath the nanostructures [2], [17]-[19], which functions as a supporting stage as well as the electrodes. The lattice stage is fabricated by the conventional nanolithography method, which usually takes one to several weeks of lithography works in a clean room. Therefore, it takes a long time to prepare (i.e., fabricating the supporting lattice stage) before the characterization. Therefore, although using four manipulators to position four separate probes onto a nanostructure would be ideal, it can be very costly and may not be attainable due to the limitation of space inside a transmission electron microscope (TEM).

The concept of a sliding probe method is illustrated in Fig. 1(c), in which a manipulation probe is used together with a fixed electrode or another probe. By attaching a probe along an individual nanostructure at different positions (e.g., the tip and the root points), its electrical transport properties can be characterized. The resistance of the sample (nanostructure) can be measured by finding the difference between any two measurements.

#### **II. SLIDING PROBE METHODS**

Fig. 1(d) schematically illustrates the equivalent circuit of the measurement loop in Fig. 1(c), where  $R_H$ ,  $R_{H-S}$ ,  $R_S$ , and  $R_{S-P}$  represent the resistance of the sample holder, the contact resistance of the sample holder to the sample, the resistance of the sample, and the contact resistance between the probe and the sample, respectively. The overall resistance when the probe makes contacts with any two points  $P_i$  and  $P_j$  (i, j = 0, 1, ..., n and i < j) are expressed as  $R_i = R_H + R_{H-S} +$  $R_{S,i} + R_{S-P,i}$  and  $R_j = R_H + R_{H-S} + R_{S,j} + R_{S-P,j}$ , respectively. Therefore, the resistance of the sample between points  $P_i$  and  $P_j$  is  $R_{ij} = R_i - R_j = R_{S,i} - R_{S,j} + R_{S-P,i} R_{S-P,j}$ . It can be seen that the resistance of the sample holder and the contact resistance at the fixed end between the sample holder  $R_H$  and  $R_{H-S}$  are eliminated. Hence, the difference between the two measurements when the probe contacts to any two points  $P_i$  and  $P_j$  of the nanostructure  $(R_{ij} = R_i - R_j)$ reflects the intrinsic resistance of the nanostructure between them  $(R_{S,i} - R_{S,j})$ , assuming that the contact resistance between the probe and the nanostructure is the same for the two cases  $(R_{S-P,i} = R_{S-P,j})$ . Different from the conventional twoterminal methods shown in Fig. 1(a), only one side of the contact resistance is involved in the measurements of the sliding probe method, and it is more feasible to keep the contact resistance between two contact positions constant or at least similar  $(R_{S-P,i} \approx R_{S-P,i})$  than to eliminate them at all in a single measurement. Hence, this sliding probe method yields a higher accuracy for the in situ characterization of electric property than the two-terminal methods and holds simplicity as comparing to four-terminal methods.

The feasibility of basic sliding probe methods including two and three discrete contacting points has been demonstrated elsewhere using a nanorobotic manipulation inside a TEM [20]. In this report, we propose several enhanced techniques and tools for improving the uniformity of the contact resistance between the probe and the sample at different positions  $(R_{S-P,i} \approx R_{S-P,j})$ . These include: 1) multipoint continuous sliding or differential  $(n \rightarrow \infty)$  sliding; 2) flexible probes as shown in Fig. 1(h); and 3) specimen-shape adapting of probe tips as shown in Fig. 1(i). The methods can be used to investigate a variety of nanostructures, particularly suitable for local electrical transport property measurements of a heterostructure as illustrated in Fig. 1(e), a structure with local defects/doping sites as shown in Fig. 1(f), or a nonuniformly shaped structure as illustrated in Fig. 1(g).

## **III. EXPERIMENTAL SETUP**

The experiments were performed in a TEM (JEOL 2200FS) equipped with a field emission gun. The raw materials were attached with silver paint to a 0.35-mm-thick Au wire, which was fixed in a specimen holder. The probe was an etched  $10-\mu$ m-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 a thin Au film (thickness ca. 21 nm). The motion of the probe was controlled by a scanning tunneling microscope (STM)—TEM holder (FM2000E, Nanofactory Instruments AB). The probe can be positioned inside a millimeter-scale workspace with a subnanometer resolution, with the STM unit actuated by a three-degree-of-freedom piezotube. This makes it possible to select a specific object and to take the multipoint sliding probe measurement of different nanostructures.

## IV. RESULT AND DISCUSSION

Using multipoint sliding, the transport properties can be accurately characterized by fully removing the contact resistance on the fixed end and erasing a large portion resistance on the sliding side, which is suitable for measuring the heteronanostructure [20]. Previous works have been carried out in characterizing the electrical properties of the carbon microfiber and the



Fig. 2. (a) Cu@CNT and the probe. (b) Soft probe fabrication. By using the EMBD, a copper stick is deposited on the tip of probe, and the stick serves as a new probe to acquire more accuracy. (c) Differential sliding along a single CNT. The probe slides on an NT in a uniformed speed (about 10 nm/s) under the bias of 500 mV, and the resistance of it along the moving direction is found during the sliding process. (d) I-t curve recorded by the differential sliding method. During the sliding, the current value steadily decreased along the length of the CNT, described precisely about the increasing of resistance.

individual niobium carbide NW [21], [22]. To further improve the measurement resolution, the contact force and area between the probe and the sample should be controlled to keep the contact resistance constant. To keep the force constant, an elastic contact is preferred over a stiff one. To keep the area constant, a shape-adaptable probe tip is superior to a sharp one due to its ability to maintain the contact and its higher average effect over the contact area.

A novel flexible sliding probe method is proposed in this section for keeping the contact force constant during the measurement. This method is particularly suitable to the measurement of nonuniform nanostructures, as shown in Fig. 2, such as an irregularly shaped CNT. A copper NW is fixed on the probe tip by using electromigration-based deposition (EMBD) [see Fig. 2(a) and (b)] [23]. The encapsulated metal can be delivered out of the carbon shells, which creates a nanoscale soft probe attached to the tip of the probe. Since the volume of the nanoscale flexible probe is much smaller than the STM probe (tip radius: 100 nm, root radius: 10  $\mu$ m), the surface-force induced binding between the sample and the probe during sliding will be increased accordingly (the attractive surface force largely depends on the volume of the contact objects). Thus, it is easier to keep a constant contact force by using the nanoscale flexible probe. Furthermore, in previous investigations using either a



Fig. 3. Impacting test of a CNT. (a) Blue line is a reference line to the CNT, and the red line shows the top position of it. The distance between the tip of the CNT and the reference line before the impact is 5.14 nm. After the impact, the distance increased to 8.82 nm. Therefore, the deformation here is 3.68 nm, and the corresponding impact force is 200.7 pN. According to the change of resistance during this approach  $(-0.95 \text{ k}\Omega)$ , the parameter  $\sigma$  is calculated to be  $-4.73 \Omega/\text{pN}$ . (b) Resistance versus length curve during the sliding process. The *R*-*L* curve represents the resistance before the elimination of the impact force influence versus the length of CNT, which can be fitted by a one-order curve with the appropriateness of 0.8211. After the elimination of the impact force influence, the correspondent  $R_{ei}$ -*L* curve has an improved fitting appropriateness of 0.9357.

fixed electrode or a movable probe, contact has been made only on several positions on a sample. The limited number of contact points can provide data to describe the general characteristics of the nanostructure but not local properties; therefore, a differential sliding technique is proposed for using the soft probe. Fig. 2(c) shows that when the soft probe continuously slid on a copper-filled carbon nanotube (Cu@CNT) at a uniform speed (about 10 nm/s) under a bias of 500 mV, the resistance along the direction of movement was measured continuously. The current value steadily decreased along the length of the CNT, while the resistance increased accordingly [see Fig. 2(d)].

This differential sliding method has the advantage that the changes in the contact resistance  $R_c$  can be neglected during the measurement and the stick-slip effect is also largely eliminated with soft probe sliding. Using the current–time (*I*–*t*) curve and a real-time video generated by the TEM during the sliding process, the sliding speed, bias between the probe and the CNT, and resistance versus length curves can be obtained. In the experiment, the original data fit a straight line with fitness  $X^2 = 0.8211$ . If the resistivity of the CNT is constant, the increased resistivity will match the increased length of the CNT more closely. However, as illustrated in Fig. 3(a), the measured resistance is affected by the impact of the probe during the sliding,

which makes the resistance–length (*R*–*L*) curve fail to fit a firstorder equation. In order to find the relation between the impact force  $\Delta F$  and the resistance  $\Delta R$ , we correlate them with

$$\Delta R = \sigma \cdot \Delta F. \tag{1}$$

Here,  $\sigma$  is the relation between  $\Delta F$  and  $\Delta R$ , and

$$\Delta F = \frac{6 \cdot E \cdot I \cdot \Delta f}{x^2 (3L - x)} \tag{2}$$

where  $\Delta f$  is the bending deflection of the CNT, *E* is the Young's modulus of the CNT, which is 1 TPa [24], *I* is the moment of inertia the CNT, *x* is the distance between the end of CNT to the contact point, and *L* is the length of the CNT.

Then, the relation between  $\Delta R$  and  $\Delta f$  can be given by

$$\Delta R = \delta \cdot \Delta f \tag{3a}$$

where

$$\delta = \frac{6 \cdot E \cdot I \cdot \sigma}{x^2 (3L - x)}.$$
(3b)

To obtain the parameter  $\delta$ , we consider the situation that the probe impacted the CNT without the sliding movement. The effect of the sliding process can thus be ignored. The current is measured continuously during this approach. Therefore, the current fluctuation can be recorded. Before the impact, the distance between the tip of the CNT and the reference line is 5.14 nm [see Fig. 3(a)]. After the impact, the distance increases to 8.82 nm. The deformation is measured to be 3.68 nm. The corresponding impact force is calculated to be 200.7 pN based on (2)  $\left(I = \frac{\pi(D^4 - d^4)}{64}\right)$ , D = 29.5 nm, d = 22.3 nm, E = 1 TPa, L = 1141.5 nm, and x = 1069.6 nm in this measurement). Since the resistance change during this approach was measured as  $-0.95 \text{ k}\Omega$ , the parameter  $\sigma$  was calculated to be  $-4.73 \Omega/\text{pN}$ , and then, the parameter  $\delta$  for each sliding position x could be achieved.

As a result, the resistance of the CNT after eliminating the influence of the impact can be expressed as  $R_{\rm ei} = R - \Delta R$ . Fig. 3(b) shows the  $X^2$  of the one-order-fitted curve reaches up to 0.9357 as we use parameter  $\delta$  to compensate for the rest differential sliding measurements that stem from the force impact influence. This is a significant improvement on the fitness over the previous approach. The fitted equation is given by

$$R_{ei} = 0.1445L + 65.824. \tag{4}$$

Moreover, since the differential sliding process consisted of infinitly small steps and each step represented a single measurement of the nanostructure, based on the basic resistivity equation, the resistivity of a single measurement can be given by

$$\rho_i = \frac{\Delta R_i}{\Delta L_i} \cdot A_i. \tag{5}$$

The resistance increases gradually as the probe slides along the CNT and the corresponding *RL* curve fits to a first-order function. Therefore, the  $\frac{\Delta R}{\Delta L}$  is the same for each measurement, which can be expressed as a differential formula  $\frac{dR(L)}{dL}$ . Then,



Fig. 4. (a) Cu@CNT and the probe. (b) Inner copper flowed out from the CNT. (c) Shape-adapted probe fabrication: By repeatedly attaching the CNT to the copper stick, the deposited copper tip was reheated and reshaped to a perfectly adapted shape of the tip to the specimen. (d) Shape-adapted sliding process, which improves the accuracy by keeping a constant contact resistance.

the average resistivity  $\rho'$  is given by

$$\rho' = \frac{dR(L)}{dL} \cdot A' \tag{6}$$

where A' is the average cross-sectional area (390 nm<sup>2</sup>) of the CNT. From (4),  $\frac{dR(L)}{dL}$  is calculated to be 0.1445 k $\Omega$ /nm. The average resistivity is then obtained as  $5.6 \times 10^{-5} \Omega$ ·m. This value is comparable with the four-point measurements of a supported multiwalled CNT with a cross-sectional area of about 200–314 nm<sup>2</sup> [25].

# V. ADAPTED PROBE MEASUREMENTS

Measuring the nonuniform nanostructure by using a soft probe differential sliding method is a good approach with the advantages of high accuracy and simplicity; however, to the nanostructure with local defects or to the nanostructure that is heterostructured, the soft probe method may not precisely reveal the electrical properties of the nanostructure. Due to the changing of contact area during the soft probe sliding, the contact resistance was variable, which means that the measured electrical property was influenced as well.

As a result, a shape-adapted probe sliding method was proposed. The adapting of the shape of a probe tip is significant for keeping a constant contact area between the probe and the specimen. Here, we show that by using the same method as in the soft probe fabrication, the copper inside the CNT would flow out from an NT against the specimen [see Fig. 4(a) and (b)]. It is possible to reheat and reshape deposited copper, which is not close to the probe [26] by repeatedly attaching the CNT to the copper stick.

After the copper is cooled down, it would be possible to fabricate the tip of the probe to perfectly reflect the shape of the specimen [see Fig. 4(c)]. The adapted probe sliding method is a new approach for the electrical transport characterization of irregular shaped nanostructures. With the combination of the adapted probe tip and the soft probe differential sliding method, the experiment results strongly suggest that the stick-slip motion can be avoided, and the contact force and area can be controlled [see Fig. 4(d)]. This approach will make it possible to keep a constant contact resistance between the sliding

		Two-terminal methods	Four-terminal methods	Sliding probe methods
Conventional characterization	Advantages	Simplicity	Accuracy	Fast and comparatively accurate; could be applied in any position
	Disadvantages	High-error (caused by contact resistance); the initial supporting- stage/electrodes that fabricated by lithography method is needed	Complicate in operation; the initial supporting- stage/electrodes that fabricated by lithography method is needed	Some errors during the measuring
In situ characterization	Advantages	Simplicity	Accuracy	Fast and comparatively accurate; could be applied in any position
	Disadvantages	High error rate (caused by contact resistance)	Difficult to be realized	Some errors during the measuring

 TABLE I

 Comparison Between Various Characterization Methods

probe and the specimen and, hence, significantly improve the measurement resolution. The feasibility and the accuracy of this method enable us to explore the detail of electrical properties of one single nanostructure in a high efficient mode.

# VI. COMPARISON BETWEEN VARIOUS CHARACTERIZATION METHODS

As we introduced three different kinds of characterization methods, including two-terminal methods, four-terminal methods, and sliding probe methods, we found that those three methods could be both applied in the conventional characterization or *in situ* characterization. Each method has its advantages and disadvantages. We have composed a table to illustrate the comparison between these methods in Table I.

# VII. CONCLUSION

In summary, to characterize the transport properties of individual nanostructures, we have developed several enhanced in situ techniques and tools for sliding probe methods including differential sliding, flexible probes, and specimen-shape adaptable probes based on nanorobotic manipulation inside a TEM. Using a copper-NW-tipped probe, we have demonstrated that a flexible probe facilitates the contact force control. Adapting the shape of the tip is essential for keeping a constant contact area between the probe and the specimen. This has been implemented by using a copper tip with a shape resembling the profile of the specimen. The tip was prepared by flowing copper from a Cu@CNT against the specimen. By controlling the contact force and area, it becomes possible to keep a constant contact resistance between the sliding probe and the specimen, which significantly improves the measurement resolution. Sliding probe methods are in situ techniques characterized by a higher resolution and simplicity in setup compared with conventional two- and four-terminal methods, respectively.

In addition, the investigation in the nanorobotic-based *in situ* characterization also advances the robotic techniques. First, the sliding probe method enables the fast characterization of the nanomaterials without the fabrication of the supporting-stage/electrodes as in the conventional *in situ* characterization techniques. The application of the nanorobotic manipulator in

the sliding probe method provides the precise positioning and the specific nanomaterials selection during the characterization, which opens a new ground for the application of the robotic technologies. Due to the development of the new nanomaterials in recent studies, the nanorobotic-based *in situ* characterization techniques such as sliding probe method will become more important in the future investigation.

Second, the sliding probe method is not only an *in situ* characterization method, which correlates the electrical/mechanical property with the structure evolution during the characterization, but also an important start for the future large-scale/automated nanomanipulation-based nanoassembling or molecular manufacturing [7]. Since the nanostructures are extremely sensitive to the flaws that exist on their components (e.g., NT, NW, fullerene), the precise characterization of each nanomaterial used as a component for the nanostructure is important. In this point, the nanorobotic-based *in situ* characterization methods are more applicable than the conventional supportingstage/electrodes-based *in situ* techniques.

Third, based on the technical reserve on the vision-based manually rectification during the nanorobotic *in situ* characterization as well as the developing of the visual tracking software specifically for the nanorobotic manipulators, we believe that the vision-based automatic nanorobotic manipulation will be realized soon.

#### ACKNOWLEDGMENT

Z. Fan would like to thank J. Doroshewitz for the final refinement of this paper.

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