REVIEW OF SCIENTIFIC INSTRUMENTS **77**, 095105 (2006)

Method of characterizing electrical contact properties of carbon nanotube coated surfaces

Onnik Yaglioglu, A. John Hart, Rod Martens, and Alexander H. Slocum
Precision Engineering Research Group, Department of Mechanical Engineering, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, Massachusetts 02139

(Received 19 December 2005; accepted 8 August 2006; published online 25 September 2006)

We present a method for electromechanical characterization of carbon nanotube (CNT) films grown on silicon substrates as potential electrical contacts. The method includes measuring the sheet resistance of a tangled CNT film, measuring the contact resistance between two tangled CNT films, and investigating the dependence on applied force and postgrowth annealing. We also characterize Au-CNT film contact resistance by simultaneous measurement of applied force and resistance. We measure a contact resistance as low as 0.024 Ω/mm^2 between two films of tangled single-wall carbon nanotubes grown on a polished silicon substrate and observe an electromechanical behavior very similar to that predicted by classical contact theory. © *2006 American Institute of Physics.* [DOI: 10.1063/1.2349300]

INTRODUCTION

Power handling capability (low resistance) and heat dissipation are important issues for contact surfaces for many industrial applications including microsystems. Due to their excellent electrical and mechanical properties,[1] carbon nanotubes appear to be promising contact materials which may also provide long life and wear resistance. However, while high current carrying capacity ($\sim 10^9\ \mathrm{A/cm}^2$) and near-ballistic conductance ($R \approx 6.5\ \mathrm{k}\Omega$) have been demonstrated along the length of an individual carbon nanotube (CNT),[2,3] properties of CNT films at the micro- to macroscales need further investigation to determine suitability for use as contact surfaces.

An important limitation for microsystems employing planar contact surfaces is the low forces generated by the actuation mechanisms which result in contact between only limited number of points.[4,5] This problem may be addressed using CNT coatings which provide highly dense nanoscale contact regions, enabling continuous and well-conformed contact surfaces. However, one must understand the reversible mechanical contact properties of nanotube-nanotube interfaces as well as nanotube-metal interfaces to fabricate high-performance contacts. Most of the literature on carbon nanotube-metal contacts reports on connections for applications such as transistors[6] and interconnects,[7] where a permanent bond is formed between the CNTs and the metal. Theoretical studies have also been carried out to study carrier transport at carbon nanotube-metal heterojunctions.[8,9] A recent study[10] demonstrated improvement in reversible contact resistance between a copper electrode and a copper surface by using an interfacial CNT layer. Reversible contact properties of a CNT coated metal wire and CNT coated electrode have also been reported.[11] But the studies on reversible contacts discussed above employ at least one electrode as one of the contact surfaces and do not represent the planar and parallel contact geometries required for both contact surfaces, such as in relays and switches. Here, we present a versatile method to characterize two contacting parallel planar CNT coated surfaces as well as metal-CNT surfaces. The flexibility of the method allows changing of substrate material and the contact area, which are important parameters for the characterization of candidate surfaces for the specific application.

EXPERIMENTAL METHOD

Chemical vapor deposition is used to grow high quality tangled films ($\sim 1\ \mu\mathrm{m}$ thick) of primarily single-walled CNTs from a $\mathrm{Mo/Fe/Al_2O_3}$ thin-film catalyst which is deposited on highly doped silicon by e-beam evaporation. The highest-yield films are grown using 85%/15% $\mathrm{CH_4/H_2}$ at 825 °C and have a G/D ratio of over 25 as measured by Raman spectroscopy[12] (Fig. 1).

First, we measure the sheet resistance of the films using a standard four-point test. As control values, the resistance across samples of catalyst only (with no CNTs) is hundreds of kilohms, and in some cases there is no conduction. Next, two CNT coated rectangular substrates are brought together in cross configuration such that the CNT surfaces are in contact [Fig. 2(a)]. This four-point test configuration (Kelvin

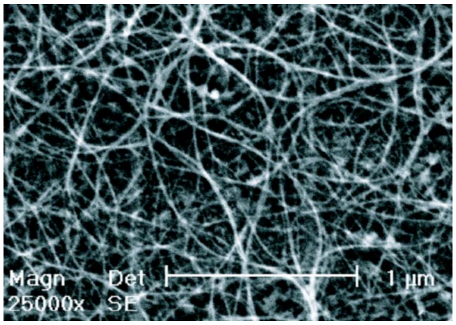

FIG. 1. Scanning electron microscopy (SEM) image of tangled CNT film grown on polished silicon surface using $\mathrm{CH_4/H_2}$.

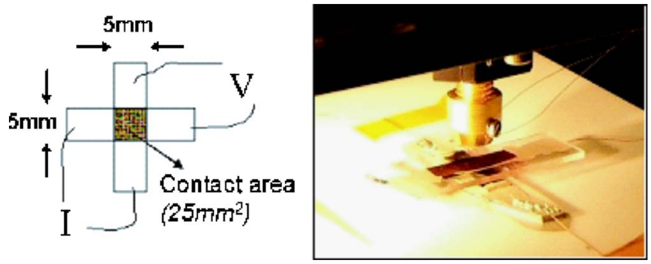

FIG. 2. (Color online) (a) Schematic of Kelvin structure setup used to measure contact resistance. (b) Measurement setup for measuring contact resistance between two CNT films. The substrates are brought together in a "cross" configuration such that two surfaces covered with CNT films come into contact.

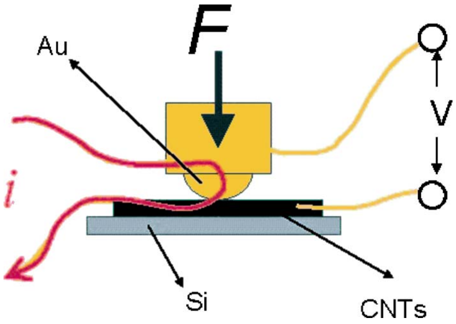

FIG. 3. (Color online) Measurement setup for CNT-Au contact resistance measurement. Current is sourced through the Au ball and the film, and the voltage drop between the Au ball and the film is measured.

configuration) is used to measure the contact resistance between two CNT films, where current is sourced into one leg of a sample, passes through the contact area, and then flows out one leg of the other sample, while the voltage drop across the contact area is measured. The tests are performed with a CETR UMT microtribometer[15] which allows simultaneous force/displacement and electrical contact resistance measurements. The samples are fixed on a glass slide, and electrical contacts to copper wires are made through colloidal silver paint and conductive epoxy [Fig. 2(b)]. As a control value, there is no measurable cross-configuration conductance across two catalyst only samples, and thus conduction is only through two contacting CNT surfaces. Because the substrate is highly doped and therefore conductive, in the Kelvin test configuration, the current flows through the silicon substrate in the contact region, instead of flowing through the CNT film. We found that conduction occurs through the thickness of the thin aluminum oxide underlayer supporting the catalyst film, which enables electrical connection between the CNT film and the conductive substrate.

Next, we measure the contact resistance between a metal surface (Au) and the CNT film using a similar setup, as shown in Fig. 3. This enables us to determine the contact resistance between the two contacting surfaces by simultaneously measuring the current flowing trough the contact and the voltage drop at the contact. We approach the CNT coated surface with a Au-plated ball ($d \sim 4$ mm) and record force and resistance simultaneously.

RESULTS AND DISCUSSION

The sheet resistance of the tangled CNT film is between 100 and 180 Ω/square, corresponding to resistivities between 1×10^{-4} and 1.8×10^{-4} Ω m. This agrees with published values for single-wall carbon nanotube (SWCNT) films produced by other methods.[13] Data are taken across different lengths of CNT film. The trend is linear indicating bulk transport through the CNTs and resistance dominated by a large number of individual CNT-CNT contacts within the film. The samples are then annealed at different temperatures in Ar (99.9999%, Airgas) up to 500 °C and no significant change in sheet resistance is observed.

The lowest measured contact resistance between two contacting CNT films is 0.6 Ω (0.024 Ω/mm^2), and the values are typically between 0.6 Ω and 1.4 Ω with an average of 0.87 Ω. The dependence of the contact resistance on the contact force is shown in Fig. 4. Upon oscillation of the load from a light to a firm load (up to 5 kg), the resistance values oscillate; however, only the first few cycles are needed to reach a steady-state resistance. During these first cycles of compression, it is likely that increased CNT-CNT adhesion by van der Waals forces is modifying the nature of the CNT-CNT contacts. However, we observe that compressing the CNT films in areas that are not in contact with another CNT surface does not change the cross-configuration resistance; therefore, this compression mechanism only affects the CNT-CNT contact resistance and not the sheet resistance. We also observe that the contact resistance between two CNT films greatly increases (up to four times) after annealing at temperatures above 300 °C, possibly due to oxidation of the CNTs. We next measure the contact resistance between the Au-plated ball and the CNT film while recording applied force. Figure 5 compares this resistance with Au ball and Au-plated flat surface for reference.

The characteristics of both CNT-CNT and Au-CNT contact measurements are very similar to classical contact theory (Holm theory),[14] where the contact resistance decreases as the force increases. Classical contact theory states that contacting surfaces first establish contacts through asperities, and these asperities form "a" spots which then become larger while new a spots are also formed as the contact force increases. According to Holm theory,[14] at a given contact force, the effective contact area, i.e., the sum of the areas of all a spots, is calculated using the yield stress of the contacting films. For the case of contacting CNT surfaces, however,

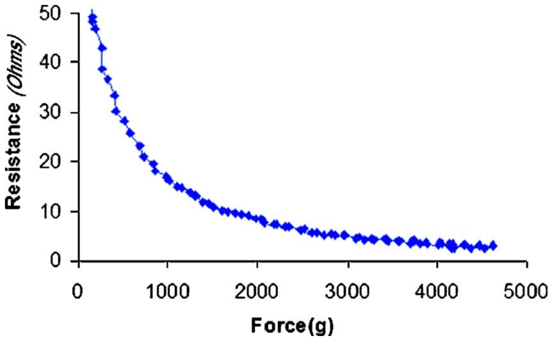

FIG. 4. (Color online) Contact resistance between two CNT films as a function of applied force. The contact area is 25 mm^2.

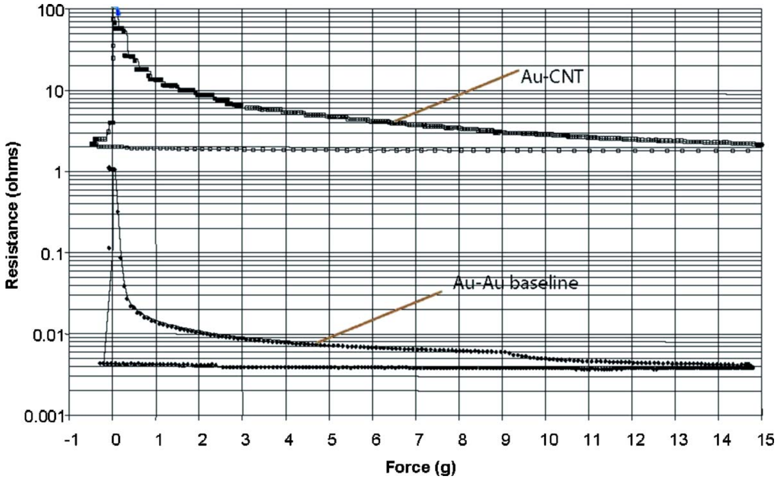

FIG. 5. Experimental results showing measured resistance values between two Au surfaces and between Au and CNT film as a function of the applied force.

the effective area calculation is not straightforward as the CNT film is made of "spaghettilike" carbon nanotubes (Fig. 1), and the theory is difficult to apply directly. We believe there are two mechanisms responsible for this behavior. First, as the contact force increases, the contact forces between two given carbon nanotube sidewalls increase, which facilitates electron transfer. Also, as the two CNT films are pressed together, the thickness of the film reduces and the effective CNT density increases due to bending of CNTs sideways, which results in more CNT-CNT sidewall contact points. Since the contact points act as parallel resistors, more contact points result in reduced overall resistance.

We also observe that the contact force required to obtain low contact resistance is much higher for two contacting CNT films. This can be explained by the fact that carbon nanotubes have very large elastic modulus[1] (terapascal range), and large forces are needed to bend them and obtain more contact points. We conclude that a tangled CNT film against a Au coated surface works better than two contacting tangled films. We believe the contact resistance is a strong function of the surface density of the carbon nanotubes, which affects the effective contact area. Although the CNT film conforms well to the Au surface, the contact resistance is still higher than Au–Au resistance (Fig. 5) due to the low surface density.

We present a method and experimental setup to characterize contact resistance between two CNT films. We also demonstrate a method to characterize the contact resistance between metal surfaces and CNT films. The method is versatile and can be used to characterize CNT films of different properties and morphologies and can serve as a platform for future research and investigation of the contact properties of carbon nanotube films with different densities (number of CNTs per area), different qualities of carbon nanotubes (G/D ratio), different CNT film thicknesses, as well as contact properties of CNT films with different metals.

ACKNOWLEDGMENTS

This work was funded in part by NSF Grant No. DMI-0521985 and in part by an Ignition Grant from the MIT Deshpande Center for Technological Innovation. One of the authors (A.J.H.) is grateful for a Fannie and John Hertz Foundation Fellowship. The authors are also thankful to Professor J. Lang and Professor J. Kong for their comments and to Professor Y.M. Chiang for sharing his laboratory for our nanotube growth facilities.

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