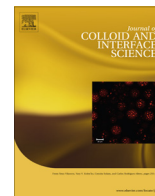




Contents lists available at ScienceDirect

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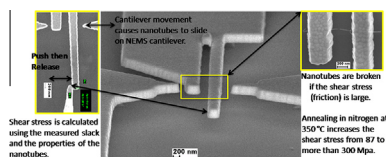


Short Communication

Interfacial shear stress between a single-walled carbon nanotube and a gold surface after different physical treatments

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GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 21 September 2014

Accepted 13 January 2015

Available online 3 February 2015

Keywords:

Shear stress

Dielectrophoresis

Single-walled nanotubes (SWNTs)

Annealing

e-Beam irradiation

Nanoelectromechanical systems (NEMS)

ABSTRACT

The interfacial shear stress between gold and dielectrophoretically assembled single-walled carbon nanotubes can be increased by annealing in N₂, by e-beam irradiation, or by e-beam deposition of carbon. For the first time this increase has been measured, using a technique developed by this group that is based on NEMS cantilever measurements combined with modeling. Annealing increases the shear stress by more than a factor of 3 over its value of 87 MPa for untreated gold surfaces, while e-beam irradiation increases the shear stress by more than a factor of 2 and carbon deposition increases the shear stress by a smaller amount.

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1. Introduction

It has been shown that the electrical contact resistance between metallic pads and SWNTs can be reduced by 3–4 orders of magnitude by either annealing in nitrogen [1,2] or by e-beam irradiation [2,3]. Nitrogen annealing at 600–800 °C for a period of 30 s reduces electrical contact resistance between a MWNT and Ti–Au electrodes by 3–4 orders of magnitude to the range of 0.5–50 KΩ [1]. Electrical contact resistance between a SWNT bundle and gold electrodes decreases by 3 orders of magnitude to be in the range of 10–100 MΩ with either nitrogen annealing at 350 °C for 5 min or electron-beam irradiation [2]. It is also reported [3] that with

an e-beam irradiation dose of 0.7 C/cm², the electrical contact resistance between a MWNT and a gold electrode decreases by 4 orders of magnitude from an initial value greater than 100 MΩ to ~30 KΩ. Furthermore e-beam induced carbon deposition has been used for clamping CNTs onto AFM cantilevers [4,5], but no tests have been conducted to measure how effective that clamping is.

The above processes [1–3] are typical methods used to improve the electrical contact between CNTs and metallic substrates. With the adhesion or friction between tubes and substrates affecting the fabrication and the performance of SWNT-based nanodevices [6–12], it is also very important to develop an understanding of how these processes affect the mechanical shear stress between CNTs and gold surfaces.

In our previous work, a new technique based on simple NEMS cantilever beams, a nanomanipulator, and a SEM, along with a theoretical model was developed to study the interfacial shear stress

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between SWNTs and gold surfaces with and without alkanethiols [13,14]. These results show that we can vary the shear stress by a factor of 20 by functionalizing a gold surface with different alkanethiols. With an average shear stress (τ) of 87 MPa between SWNTs and untreated gold, the shear stress can be increased to 142 MPa by functionalizing the gold surface with 2-aminoethanethiol or reduced to 7.2 MPa by functionalizing the gold surface with 2-phenylethanethiol prior to the assembly of SWNTs. In this paper, the same measurement technique is used to investigate how the shear stress between SWNTs and a gold surface is affected by annealing the devices in N_2 , by e-beam irradiation, and by e-beam induced carbon deposition after the SWNTs are assembled.

2. Experimental methods

2.1. Preparation of the devices

The details of the micro/nanofabrication process for the NEMS cantilever device are presented in Refs. [13,14]. The final released cantilever structure, shown in Fig. 1, consists of two cantilevers with two triangular electrodes on each side. One cantilever is longer and narrower than the other, which makes it more flexible. As described in [14], an individual SWNT bundle is dielectrophoretically assembled on the two cantilevers between the two triangular side electrodes, followed by a carbon dioxide critical point drying process. The bundle is then cut off from the two triangular side electrodes using an electron beam in the SEM, so that the SWNT bundles only contact the two NEMS cantilevers. SEM images of the device before and after nanotube cutting can be seen in Fig. 3 in reference [14]. After dielectrophoretic assembly of the SWNTs, the devices are annealed in N_2 , irradiated with the e-beam in the SEM, or have e-beam induced carbon deposited on them in order to investigate how each of these processes affects the interfacial shear stress.

The N_2 annealing was performed in a 7355B Bruce Furnace at 350 °C for 5 min. In the e-beam irradiation process, a contact area of $0.3 \mu\text{m} \times 1 \mu\text{m}$ is exposed to the e-beam with a beam current of 130 pA (30 μm aperture and 3 kV accelerating voltage) for 16 s to achieve an exposure dose of 0.7 C/cm^2 . It is noted that in addition to this intentional e-beam irradiation of the contact area, all tested devices are exposed to a certain amount of e-beam irradiation during SEM imaging before the manipulation tests. To minimize this additional e-beam irradiation, the exposure time during imaging is minimized. For an imaging area of $\sim 3.7 \mu\text{m} \times 5.7 \mu\text{m}$ at a magnification of 20 K \times , if the total exposure time is approximately 10 s, with a beam current of 130 pA, the overall exposure dose for the device area is $\sim 6 \times 10^{-3} \text{ C/cm}^2$, which is over 100 times

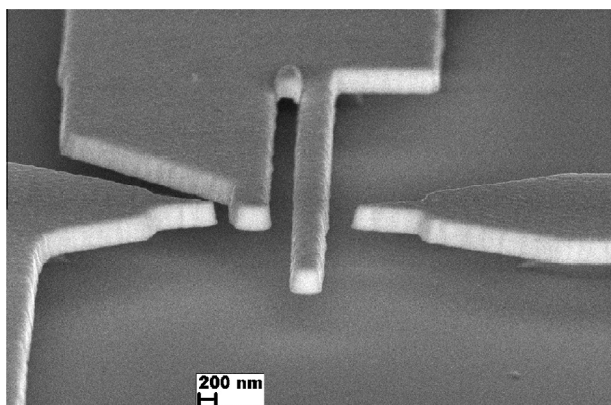


Fig. 1. SEM of the fabricated NEMS structure. (Single-column fitting image.)

lower than the intentional e-beam exposure dose used for the surface treatment described above.

For the e-beam induced carbon deposition, the electron beam is directed to an area adjacent to the cantilever surface to prevent e-beam irradiation of the nanotube/cantilever interface. This causes an amorphous carbonaceous deposit over an area extending several microns from the electron beam [3]. To modify the cantilever surface with this amorphous carbon layer, the tip of the triangular electrode close to the narrow cantilever is exposed to the e-beam for either 30 min or 1 h. The nanotube bundles grow bigger after a carbon deposition process, which indicates the presence of a carbon layer from the SEM.

2.2. Test method

A Zyvex nanomanipulator is used to push the flexible cantilever away from the stationary cantilever, thereby inducing axial tension in the suspended portion of the nanotube bundle. The axial tension increases as the gap between the two cantilevers increases. At some point, this induced axial tension causes the SWNT bundle to start to slip on the cantilever surfaces. When the axial tension increases to the value of the interfacial shear force corresponding to the width of the narrower cantilever, the SWNT bundle slips across the whole of the narrow cantilever and the axial tension stops increasing. After releasing the nanomanipulator from the flexible cantilever, the originally taut nanotube bundle ends up with a certain amount of axial slack (the increased length of the originally taut SWNT bundle in between the two cantilevers), which is a function of the shear stress that acted during the slipping of the nanotube on the substrate surface.

The entire nanomanipulation process is conducted inside the SEM, as discussed in [13,14]. After the nanomanipulation, the SWNT bundle is checked in the SEM and images of the bundle at two different angles (90° apart) are taken. These two SEM images give a 3-dimensional view of the bundle and allow further image analysis using MATLAB to determine the amount of axial slack in the tube bundle.

2.3. Modeling

A theoretical model was developed to determine the shear stress from the experimental measurements of nanotube slack. The details of the modeling are discussed in [14]. A Young's Modulus of $E = 1 \text{ TPa}$ is assumed for a hollow tube with a radius of 0.65 nm (dimension from the supplier) and a wall thickness of 0.34 nm from which the cross-sectional area (A) is computed. We assume the contact width (b) to be equal to the SWNT tube radius of 0.65 nm in order to convert the shear force per unit length to the shear stress. It is noted that the carbon deposition treatment may increase the effective contact width by depositing carbon near the interface between the SWNTs and the surface. However, as discussed in [14], it is really the force per unit length (shear flow) which is measured. Its interpretation as an effective shear stress facilitates its comparison with nanotubes of vastly different widths.

Furthermore, the assumption that all SWNTs interact with the substrate can make the number of SWNTs in a bundle irrelevant in the shear stress calculation [14]. The effective contact width based on this spreading assumption is close to the one based the hexagonal-packing lattice of a SWNT bundle. For the example of six SWNTs in one bundle, the effective contact width is equal to 3.9 nm based on the spreading assumption and equal to 3.2 nm based on the hexagonal-packing model in which the contact width is approximated as the total diameters of two SWNTs. Furthermore SWNTs usually spread out as several small sub-bundles on the

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