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Mechanical properties of suspended individual carbon nanotube studied by atomic force microscope

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ABSTRACT

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Keywords: Carbon nanotube Atomic force microscope Young's modulus Force–distance measurement This paper is a short review on the mechanical properties of individual carbon nanotubes (CNTs), which were studied by atomic force microscope (AFM). AFM force–distance measurements were applied to three different nano structures based on the suspended CNTs, those are a straight suspended CNT, a coiled CNT, and a torsional CNT. Force–distance measurements were done on the pick and valley position of the coiled CNT and the estimated elastic moduli was compared with the straight one. Nanoscale metal plate was fabricated on the middle part of the suspended CNT structure using conventional lithography and etching procedure. The torsional modulus of the suspended CNT was estimated from the force–distance measurement using AFM manipulation on top of the metal plate. The paper covers synthesis and preparation method of various suspended CNT structure and the analysis of the force–distance measurements appropriate to each CNT structure. Prospects of the AFM force–distance measurements on emergent nano scale materials as well as CNT are suggested in the end of this paper.

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1. Introduction

Because of its superior mechanical and electrical properties, carbon nanotube (CNT) has been widely studied and possible applications of CNT devices were also suggested in many directions during last two decades [1–4]. In the beginning stage of CNT studies, a lot of works have been focused on the electrical properties of CNTs and their application to future electronics since the electronic properties of CNT shows either metallic or semiconducting according to its chirality. However, the CNT electronic devices are not yet embodied into practical application to current electronic system because the chirality of CNT is still not perfectly controlled.

CNT based nano mechanical devices, in the other hand, have been grown later compare to electronic devices because a little more nano lithography techniques were necessary give a mechanical degrees of freedom to CNTs as well as electrical degrees of freedom. CNT is one of the best candidate materials for nanoelectromechanical system since it has high conductivity, extreme mechanical strength, and ultra-low mass density [5]. Intriguing mechanical properties individual CNT were reported since a suspended CNT structure were fabricated [6] and its elastic

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http://dx.doi.org/10.1016/j.synthmet.2015.09.014 0379-6779/© 2015 Elsevier B.V. All rights reserved. modulus value was estimated from force-distance measurements using atomic force microscope (AFM) [7].

Although there have been several reports so far on studying the basic mechanical properties of CNTs [8–10] and proposing possible applications of CNT based nano mechanical devices to sensitive mechanical sensors [11–13], there are still a lot of rooms to investigate the mechanical properties of CNTs with different aspects of mechanics as well as their various shapes. In this paper, we investigated the mechanical properties of suspended CNTs using AFM manipulations. Two different CNT structures, which is coiled CNT and doubly clamped suspended CNT, were prepared for this study. Elastic moduli of coiled CNT were estimated both on pick and valley positions by using AFM force distance measurements and compared the modulus values with straight CNT. Torsional force could be applied to CNT by pushing the nanoscale metal attached on the middle of the doubly clamped suspended CNT using AFM tip manipulations. Torsional modulus was estimated out of the force-distance curves measured along the various distance from the pivot point of metal plate.

2. Experimental

2.1. Synthesis of CNT and device fabrication

CNTs were synthesized by chemical vapor deposition method. 1 nm of catalyst metal (Fe) was deposited on the Si substrate which has around 1.5 nm of natural oxide layer. CNTs were grown in a







guartz tube furnace by flowing 20 sccm C₂H₂ and 100 sccm H₂, and 600 sccm Ar gas at temperature of 750 °C. The CNTs with \sim 20 μ m length and 20-50 nm diameter were synthesized for 15 min of growth time. The synthesized CNTs were dispersed by ultra-sonic agitation in a sodium dodecyl sulfate (SDS) water solution. One droplet of the CNT suspension was deposited on top of the predefined Au/Ti electrode with assistance of an ac dielectrophoresis (DEP) method. An ac bias signal with 13 MHz of frequency, 16 V_{pp} was applied for 30s for CNT deposition. Fig. 1 shows the synthesized CNTs aligned and attached to the metal electrode using DEP method. Coiled CNTs were found as indicated by arrow in Fig. 1 while the dispersed CNTs were observed using scanning electron microscope (SEM). It is known that ring or coiled CNT structures can be formed during synthesis [14–18]. The coiled CNTs need heptagon-pentagon pairs at a regular distance so that they can bend the direction in the hexagonal structure of CNT. There are several theoretical studies about the formation mechanism of coiled CNT [19,20] but the growth mechanism is still not clear and question remains why such defects appear periodically to form the coiled structures.

Mechanical properties of CNTs were measured using atomic force microscope with three different CNT structures. First of all, a straight suspended CNT structures were produced using conventional nano fabrication method combined with ac DEP method. Detailed fabrication procedure for realizing the suspended CNT was previously reported [21]. The application condition of ac bias signal is almost same, except the density of CNT-water suspension, as depositing CNT and coiled CNT at the Au electrode, which is described at previous paragraph. The density of CNT suspension was significantly reduced down to $\sim 0.5 \,\mu$ g/ml so that only one individual CNT can be caught between the two electrodes. In the case of the coiled CNT deposited on the substrate, secondly, the force-distance measurements could be directly performed since this structure itself naturally has suspended areas. Lastly, a suspended metal paddle was defined on the CNT to study the torsional modulus of CNT. To realize a suspended paddle structure, CNT was dispersed on the SiO₂ substrate. Two electrodes on the end and a nano scale plate on the middle part of the deposited CNT were patterned using electron beam lithography. 10 nm of Cr following 90 nm of Au were evaporated for electrodes and plate. Wet etching with HF buffer solution was applied for 2 min to remove the SiO₂ beneath the Au plate. Fabrication of the suspended CNT structure with torsional paddle was completed after rinsing and critical point drying process.

2.2. Determination of Young's moduli of various devices based on suspended CNT

Mechanical properties of the three types of CNT structures were studied by AFM force–distance measurements. First of all, a straight suspended CNT structure was fabricated and its Young's modulus was studied. Suspended nano structures were found by a tapping mode AFM scan. By zooming in the scanning area, the AFM tip was moved on top of the desired position of the CNT structures so that the force–distance curves could be obtained by pushing the AFM tip. The deflections of the suspended structures were estimated by extracting the deflection of the cantilever, which was obtained by measuring force–distance curve on SiO₂ substrate [22]. The force value on the *y*-axis of the force–deflection curve was estimated from a spring constant of the AFM tip specification, which was 0.65 N/m in this works [23].

Fig. 2 shows force–deflection curve of suspended CNT. The deflection of CNT according to the external force was obtained by extracting the deflection of cantilever from the original force–distance measurement results, which is shown at upper inset of Fig. 2. The elastic modulus of CNT can be estimated by applying the string model [24,25]. The force–deflection curve was well fitted to $F \propto \delta^3$. The elastic modulus (*Y*) of the suspended CNT was estimate to be 0.3 TPa by the relation of

$$F = 8YA\left(\frac{\delta}{l}\right)^3$$

where A is the cross section of a CNT $(3.2 \times 10^{-18} \text{ m}^2)$ and l is the length of the suspended CNT (1 μ m).

Secondly, mechanical properties of a coiled CNT were investigated using AFM manipulation. As shown in the Fig. 1, the diameter of the coiled CNT is thicker than the straight one even though it was found in the same batch. However, the coil was not tightly wound and the diameter is rather thin if it is compared to the previous works on the coiled CNT which was synthesized on purpose [18]. Fig. 3 shows the AFM measurement results on the coiled CNT. The cross section analysis of the AFM height image revealed that the distance of pitch is around 400 nm and the height of CNT coil is around 200 nm.

AFM force–distance measurements were done on the three different areas where, the peak, valley of coiled CNT, and SiO_2



Fig. 1. Scanning electron microscope (SEM) image of carbon nanotubes deposited by ac dielectrophoresis (DEP) method. Arrow indicates a coiled CNT.



Fig. 2. Force-deflection curve of the suspended CNT. Upper inset: Force-distance measurement result of the suspended CNT using AFM manipulation. Lower inset: SEM image of the CNT under test.

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