# Nano-scale Mechanical Test of MEMS Structures by Atomic Force Microscope

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***Abstract*** **– This paper focuses on nano-scale analysis of mechanical properties of polymer and carbon nanotubes (CNT) embedded MEMS devices using the probe tip of the Atomic Force Microscope (AFM).The mechanical properties of surfaces of layered materials were investigated by using nanoindentation produced with tips of an AFM. Experiment results indicated the bending characteristics of the device and could also indicate the Young's Modulus of the CNT embedded micro structure. Our objective is to determine the nano-scale mechanical properties and piezoresistivity of bulk carbon nanotubes using the local probe manipulation.**

## I. INTRODUCTION

The evaluation of mechanical properties of multi-walled carbon nanotubes is undergoing intensive investigation. Carbon nanotubes have grown in potential applications since the discovery of their extraordinary electronic and mechanical properties [1] – [2]. Several tests for the mechanical properties of single strand carbon nanotubes have been carried out by several research groups [3] – [5]. Because of their outstanding properties, carbon nanotubes could be used, for example, in nanometer-sized electronics or to strengthen the tensile strength and elastic modulus of composite polymer materials. Several experiments proved out that embedded carbon nanotubes lead to effective stress transfer [6] – [9]. Compared to carbon fibers which typically have Young's moduli of 0.1 – 0.8 TPa [10], the elastic moduli of carbon nanotubes are in the range of 1 – 5 TPa [11].

Conventionally, tensile tests have been widely used to evaluate both the elastic and plastic properties of bulk materials. For small strain $\varepsilon$ in the elastic regime, Hooke's law applies ($\sigma = E\varepsilon$), where $\sigma = F / A$, $F$ and $A$ represent the applied force and cross-sectional area, respectively, and $E$ is the Young's modulus. Typical characteristics such as the hardness and Young's modulus of elasticity are measure by the indentation method. The application of force by an indenter will cause deformation on the surface of materials and the amount of deformation produced can be estimated.

When the applied load lies between $10^{-5}$ and $10^{-2}$ $N$, microhardness measurements of metals tends to increase as the size of indenter reduced [12]; this is attributed to the limited range of dislocation movement available when very small volumes are involved. The plastic flow caused by the indentation on metals is accompanied by the slipping of atomic planes. Thus, better control is achieved over the local features by minimizing the size of the indentation. Deformation characteristics can be studied by comparing the same area before and after indentation.

The study of the nano-scale mechanical properties of surfaces to forces has become possible with the atomic force microscope [13] – [15]. Many techniques have been developed which take full advantage of the nanometer three dimensional resolution offered by the ultra sharp AFM tip. AFM images are produced by the scanning of a probe, with a sharp tip at the end of a cantilever beam, across the surface of a sample using piezoelectric scanners. The high resolution imaging capability of AFM allows accurate localization of indentation sites and measurement of indentation depth. The AFM can also be operated under a force mode in order to perform indentation test. In this case, a force curve is produced, which is a plot of tip deflection as a function of the vertical motion of the scanner. This curve can be analyzed to provide information on the local mechanical response [16] – [18]. The nanoindentation technique utilizes the force curve obtained by driving a sharp probe into the sample surface to extract the mechanical properties of the materials. The main feature of AFM based nanoindentation is that the force applied to the surface is achieved by bending of a cantilever and therefore the magnitude of force is usually smaller than that of traditional nanoindenter. Forces down to *pN* allow probing of very thin surface films and offer opportunities for examining mechanical properties of polymers.

A recent experiment has shown that mechanical deformation can significantly change the electronic behavior of carbon nanotubes [19]. Based on an order of estimate calculation, we project the gauge factor of carbon nanotubes to be ~1000, which is at least an order of magnitude higher than conventional Silicon based strain sensors. Motivated by this possibility, our ongoing work is to perform mechanical bending tests of bulk carbon nanotubes embedded in MEMS structures and to examine their bulk mechanical properties.

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In this paper, we will show that CNT-embedded micro bridges can be bent repeatedly by using the tip of an AFM. The evaluation of mechanical properties of carbon nanotubes is essential for the design of potentially nanometer-scale piezoresistive sensors. We envision our sensors to eventually sense the input force by the change of conductivity due to mechanical bending of the embedded bundled carbon nanotubes, which are sandwiched by 2 layers of polymer (Parylene) thin films. The fabrication process of our CNT-embedded MEMS sensors were recently reported in [20] and [21].

## II. EXPERIMENTAL PROCEDURES

Nanoindentation experiments were performed with a Nanoscope III atomic force microscope at ambient conditions by using silicon nitride microfabricated cantilevers. Applied forces, $F_n$ are given by

$$F_n = k \times d\,, \tag{1}$$

where $k$ is the spring constant of the cantilever and $d$ is the cantilever deflection. The surface deformation, $\delta$, is calculated from $\delta = z - d$, where $z$ is the displacement of the piezoelectric scanner.

Prior to indentation, the surface is imaged using contact-mode AFM to locate the region of interest. The amplitude of oscillating cantilever was set to zero before indentation. The sensitivity of the cantilever was calibrated by acquiring the cantilever deflection versus the displacement of the piezoelectric scanner curves on the surface. By setting the feedback gains to zero during indentation, indentation is performed using the AFM. During indentation, depth as a function of load, as well as a function of time, is simultaneously recorded.

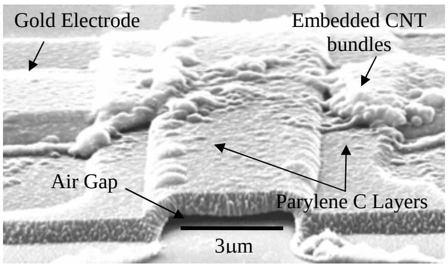


Fig. 1. SEM image of a CNT-embedded MEMS polymer bridge structure

The nanoindentation technique was applied to the bending test of the MEMS structure embedded with bulk carbon nanotubes as shown in Fig. 1. As described in the schematic diagram in Fig. 2, the test was initiated by first imaging the MEMS structure with the AFM tip in contact with the sample under a constant applied load, which allows the identification of the carbon nanotube-embedded micro bridge structure from the image. The AFM tip was then positioned above the centre of the micro bridge. The bridge was pushed towards the bottom of the trench by moving the AFM tip downward. There were five load levels used to indent on each particular area of the sample, using the same probe and operating conditions. Each step of AFM tip-movement iteration was 139*nm* in our experiments.

The nominal force, $F_n$, and the maximum vertical displacement of the bridge, $z$, were calculated from the following equations [22],

$$F_n = \left(\left(V_0 + V\right) / s \times k\right), \tag{2}$$

$$\delta = z - V / s\,, \tag{3}$$

where $V_0$ is the initial differential voltage produced by the deflection of the cantilever before the bending test, $V$ is the differential voltage during the test and $s$ is the ratio of the differential voltage obtained from the cantilever's deflection to the displacement of the piezoelectric actuator in $z$-direction.

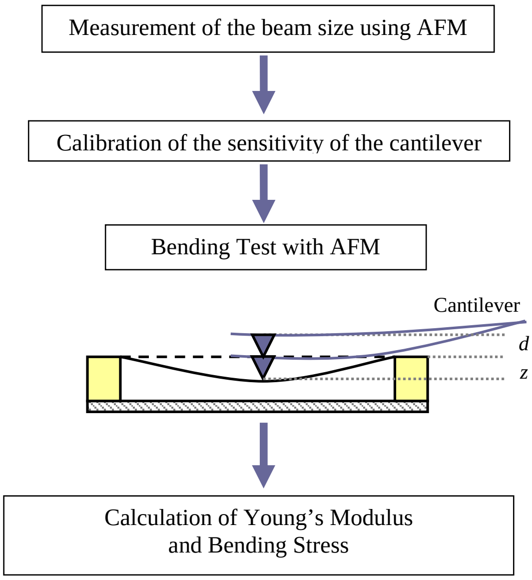


Fig. 2. Schematic diagram of the bending test of CNT-embedded MEMS structure

Based on the above experimental procedure, we have also used the AFM to characterize the deformation characteristics of different regions of the CNT-embedded MEMS sensor.

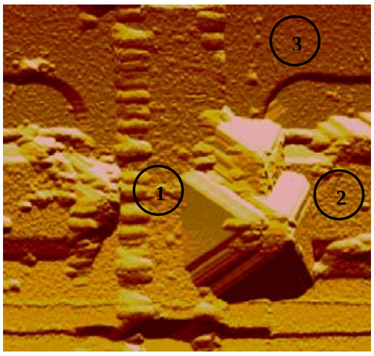
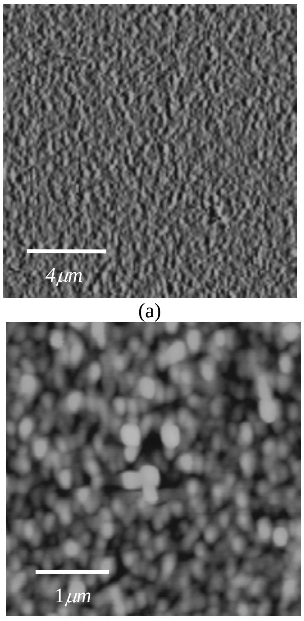


Fig. 3. Specific regions under the nanoindentation test

In this experiment, three different regions in the device were investigated as shown in Fig. 3. The first region is the center of the carbon nanotube bridge with 2 layers of Parylene thin films. The second region is the Parylene surface with gold underneath it and finally the surface of Parylene on a silicon substrate. Results of the experts described above are given in the next section.

## III. III. RESULTS AND DISCUSSION

Polymeric materials are easily penetrated by an AFM probe, especially at large tip forces. The penetration depth can be evaluated from the force curves. This provides a relative measure of local stiffness and also might be useful for estimating the depth of penetration of the AFM probe during the imaging of a sample. Moreover, the slopes of force curves provide information about the elastic properties of the samples.



(a)

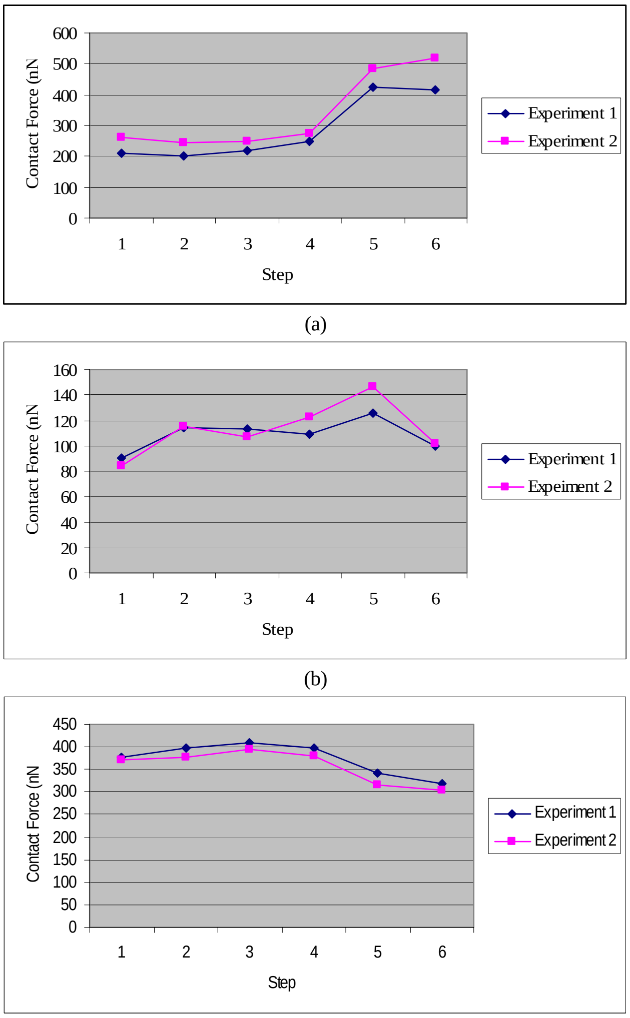
(b)

Fig. 4. Scanning images of Parylene surface before and after nanoindentation by AFM tip

When a sample surface is deformed, the spring constant of the cantilever was in series with the local surface compliance, changing the effective slope of the curve. Fig. 4(a) and (b) are the AFM scanning image of a sample with a layer of 300*nm* Parylene thin film on silicon substrate before and after nanoindentation, respectively. The nanoindentation was performed at ambient conditions by using a silicon nitride cantilever. The force applied on the surface is calculated to be 1.59 $\mu N$ and the depth of penetration is 14.69*nm*. When the same force was applied to the gold surface, no indentation was formed as the hardness of gold is comparatively much higher than that of Parylene. In order to perform nanoindentation on harder surface, steel cantilever with diamond tip should be used to apply a larger nominal force on the surface.

The bending test of the MEMS structure embedded with carbon nanotubes was carried out using an AFM system. This MEMS structure can be potentially used as a novel piezoresistive CNT-based sensor. The nominal contact force of the tip and sample is calculated and the results show that forces applied to various materials (e.g., Au, Si, and Parylene polymer) remained consistent for continuous pushing by the AFM tip. Figure 5(a), 5(b) and 5(c) represent the force applied to region 1, 2 and 3 with continuous downward pushing by the AFM tip, respectively.

However, the contact forces were found to be different for the same material on different substrates. The force measure at the center of the carbon nanotube bridge with 2 layers of Parylene films was in the range of 200*nN* – 500*nN*, where the



(a)

(b)

(c)

Fig. 5. AFM contact force data on bending the CNT-embedded MEMS structure with continuous pushing

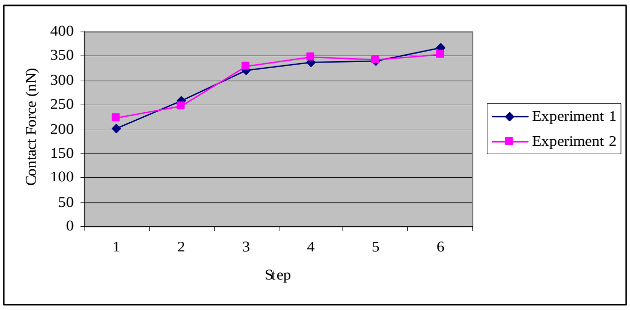


Fig. 6. AFM contact force data on bending the CNT-embedded MEMS structure with discrete pushing

forces measured on the Parylene surface with gold and silicon underneath ranged within 100*nN* – 150*nN* and 300*nN* – 400*nN*, respectively. The sudden increase of contact force in Fig. 5(a) was determined to be the deformation of the bridge structure. Although the top layer in each region is the same, the force varied when there were different materials underneath the layer.

To further test the bending characteristics of the CNT-embedded bridge, the micro bridge was pushed towards the bottom of the trench and then the AFM tip was retracted. This experiment was repeated with different downward distance of the AFM tip. After each step, the travel distance of the cantilever was increased by 139*nm*. The applied force by the cantilever at different stage is shown in Fig. 6. The force measured was smaller than that of continuous pushing on the surface. This implied that in order to perform the bending test on discrete level, large nominal force has to be applied on the force as to obtain the same level of displacement of the structure during continuous manipulation.

## IV. CONCLUSION

AFM nanoindentation technique was evaluated as a viable technique to determine the mechanical properties of polymer thin films. From the results obtained to date, this technique is capable of measuring modulus of thin films down to the submicron levels and gain substantial additional information concerning the true response of a material to indentation at the nanometric scale. The force applied on the surface was controlled by the differential voltage applied to the cantilever and the mechanical properties of the cantilever. Changing of the parameters would alter the size and depth of the indentation.

The nano-scale mechanical properties of the carbon nanotube-embedded structure were investigated and the micro-scaled hardness of different parts of the device was calculated. The bending test was applied to further investigate the Young's modulus of the CNT-based sensor. Once the bridge made contact with the bottom of the trench, the contact force increased, which allowed us to determine the depth of the channel (which matched the design expectations). During the experiment, the AFM cantilever was recorded. The mechanical properties of the micro bridge were calculated by the recorded contact force and the deflection of the bridge. The deflection of the bridge at its centre point was evaluated from the cantilever deflection signal. As the initial tip-surface and the travel range of the AFM tip were controlled, the micro bridge was deflected to various degrees and allowed us to study the effect of mechanical deformation on the bundled CNT sensing element.

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