In-Situ Nanomechanical Testing of One-Dimensional Materials

H. Ghassemi, Y. K. Yap, R.S. Yassar

Mechanical Engineering-Engineering Mechanics Department,

Michigan Technological University,

1400 Townsend Dr., Houghton, MI 49931

Abstract

One-dimensional nanomaterials including nanotubes are building blocks for constructing various complex nanodevices. Boron nitride (BN) nanotubes with structure similar to carbon nanotube are known to be among the strongest insulators in the world. In this work, deformation of an individual BN nanotube is performed inside a high-resolution transmission electron microscope (TEM) using a piezo-driven atomic force microscope (AFM)–TEM holder. The mechanical properties of individual BN nanotubes are obtained from the experimentally recorded force-displacement curves.

***Keywords**:* TEM, AFM, STM, in-situ, Nanotube

1. Introduction

The discovery of various types of nanotubes has provided fertile ground for both experimental and complementary theoretical studies. A Boron Nitride (BN) nanotube has structural analogue similar to a carbon nanotube in nature, alternating B and N atoms entirely substitute for C atoms in a graphitic like sheet with almost no change in atomic spacing, as shown in Figure 1. For pure carbon nanotubes, theoretical calculations predict that mechanical properties depend on radius and chirality of the nanotubes [1].

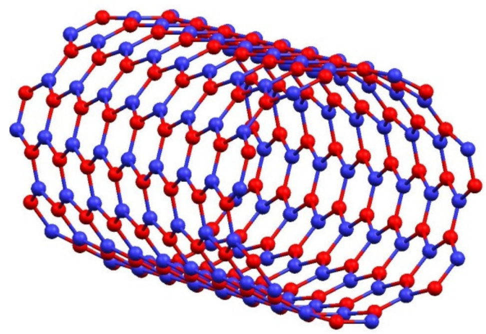

Figure 1 Schematic of a single wall BN nanotube

Different methods of BN nanotube synthesis are available such as arc-discharge [2], laser ablation [3], and substitution reaction [4]. But the most efficient method which is capable of synthesizing reasonable amount of BN nanotubes is chemical vapor deposition (CVD) [5]. Replacing strong covalent sp^2 bond between carbon atoms by B-N ionic bond is the main difference between BN nanotubes and carbon nanotubes. BN nanotubes show a preference for zigzag lattice formation, since an atomic layer of B has of considerably larger surface energy than one comprised of N atoms [6]. In fact, in the formation process of σ bonds of BN nanotubes, the considerable charge transfer would take place between the B atoms and the nearest N atoms because of their different electronegativities (2.04 and 3.04, respectively). This makes the distribution of charge density along the BN nanotube more non-uniform. In addition, the effect of isolated electron pairs localized at the N atoms or localized π electronic states in BN nanotubes quite substitute for that of the delocalized π electronic states in carbon nanotubes. These two inherent differences between BN nanotubes and carbon nanotubes can give rise to their different physical, chemical properties, and mechanical properties [7]. Another effect of ionic bonds may be destabilizing single-wall NT formation and strengthening so-called “lip–lip” interactions between adjacent layers in multi-walled BN nanotubes [8]. So it can be predicted that mechanical properties of BN nanotubes are as well as those of carbon nanotubes.

Golberg et al. [9] were the first time to perform the *direct* force measurements under bending of highly pure well structured MW-BN nanotubes of various diameters inside a high resolution field emission TEM. The force measurement experiments were carried out using a new state of-the-art atomic force microscopy (AFM) holder, which was placed into the TEM column.

In this paper, we analyzed the force-displacement curve of a BN nanotube using an in-situ AFM-TEM holder to measure the compression force and the elastic modulus properties.

2. Experimental

BN nanotubes were prepared via Plasma-enhanced chemical vapor deposition, at 1200 ^{o}c for 90 min inside a conventional furnace. Molar ratio of 2:1:1 was fixed for B:MgO:FeO as precursor. 200 sccm of NH_3 gas was flowed into the chamber as source of N. As shown in Figure 2a, SEM image shows effective growth of BN nanotubes grown by Yap et al [10]. The length of nanotubes are in range of few µm. Figure 2b shows TEM image of well-structured multi-walled BN nanotube that is in contact with the tip of AFM. The outer diameter of the nanotube in contact with AFM tip measured ~130 nm while the inner diameter is around 80nm.

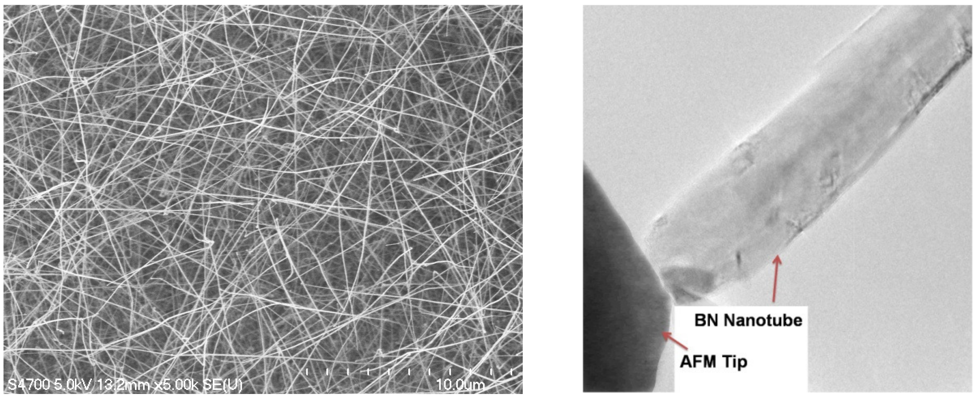

Figure 2 a) SEM image of effective grown BN nanotubes and b) TEM image of a BN nanotube that is in contact with the AFM tip

Figure 3 shows the AFM setup inside a JEOL JEM-4000FX TEM operated at 200 kV. The BN nanotubes on a wire were inserted into the movable part (Sapphire ball) of the piezo-driven side entry TEM holder, with the nanotubes exposed outwards. The length of the wire manipulated precisely to minimize the gap between the tip of AFM and nanotubes, due to limited movement range of hat. Inside TEM, the piezo-driven part was delicately moved in the *X* and *Y* directions so that the nanotube was first brought toward the sharp tip and then the *z*-height and wobbler function adjust AFM tip and nanotubes at the eucentric height. Force-displacement curve can be drawn by adjusting the displacement range which we put 100nm and continued to reach to highly bended nanotube, i.e. within each step of experiment, the wire and the AFM tip are 100nm closer to each other. Using camera connected to the software, the changes in the structure of BN nanotube can be captured and monitored, instantly.

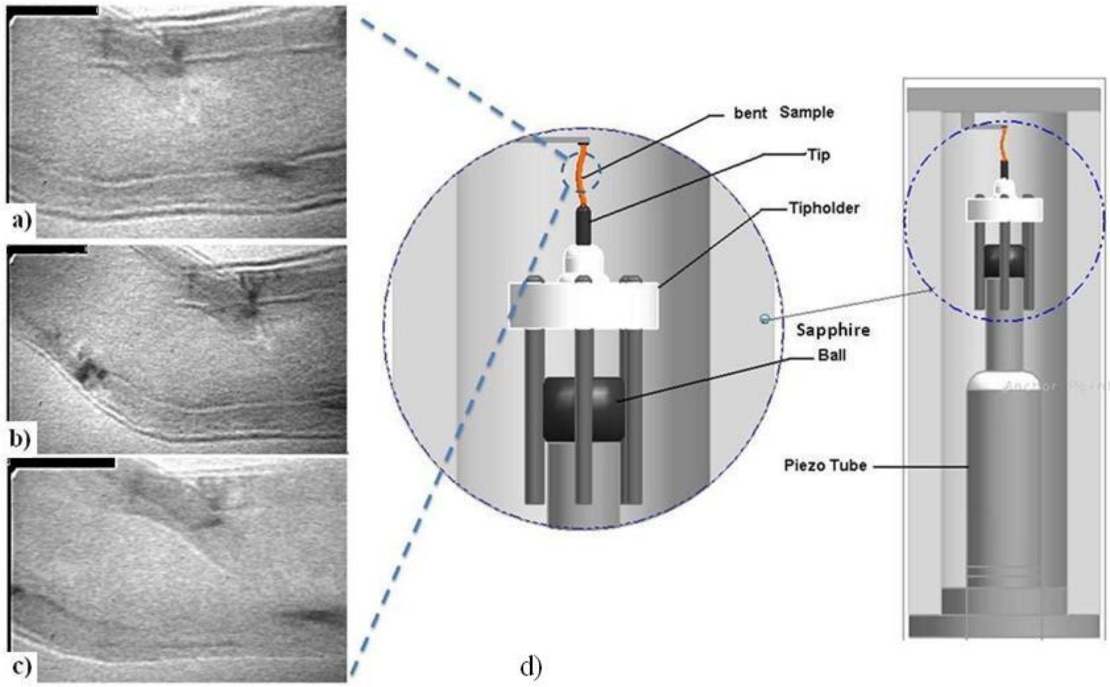

Figure 3. BN nanotube under deformation process inside TEM; a) with no applied force, b) bent BN nanotube and c) after releasing force. d) Schematic of AFM setup inside the TEM.

3. Results

Figure 4 shows the force-displacement plot of BN nanotube during deformation. Before starting the experiment, the applied force and displacement set to zero position. By approaching the wire to AFM tip, the force-displacement curve data has been collected and the nanotube was bended.

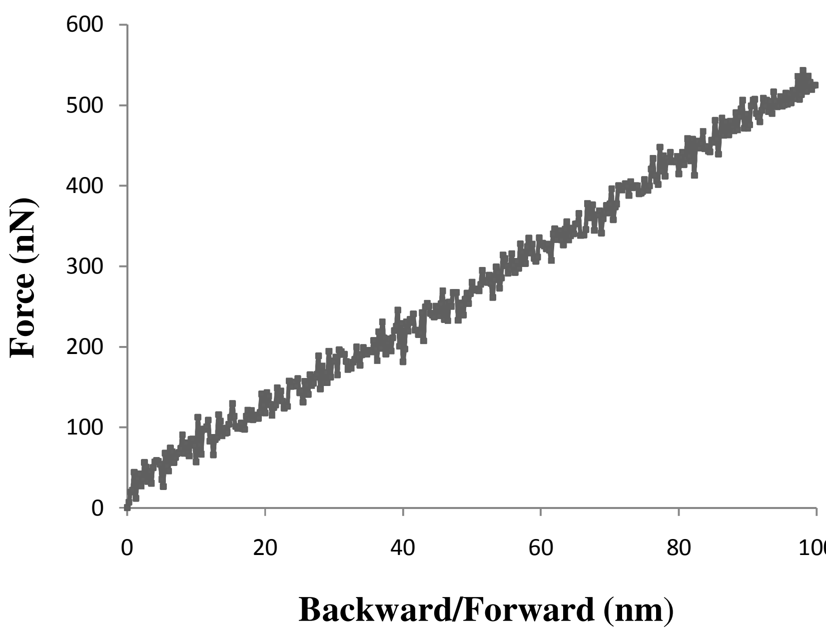

Figure 4 force-displacement of BN nanotube during deformation

As it can be seen in force-displacement plot, the slope is constant and it's worth mentioning that upon few next steps of applying force, the plot was still linear. Golberg et al

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[9] proved that BN nanotubes are very flexible nanotubes, which is shown in Figure 3 a-c. Figure 3a shows BN nanotube under no applied force. After applying force, Figure 3b, the nanotubes went under deformation and the force was measured, and after releasing the force nanotube recovered its initial shape with no residual stress or deformation.

Young's modulus of nanotubes is one of the important properties which can be measured in different methods. One of the methods is based on the Euler's formula. The elastic modulus of the BN nanotube can be calculated as [11]:

$$F_{Euler} = \frac{\pi^2 EI}{L^2} \qquad I = \frac{\pi(d_2^4 - d_1^4)}{64}$$

where, L was the length of the BN nanotube between the two contacts, d_1 and d_2 were the internal and external BN nanotube diameters respectively, E was the elastic modulus, and F_{Euler} was the force applied. The measured elastic modulus was found to be in the range of 0.4-0.5 TPa.

7. Conclusion

The force-displacement curve of BN nanotube has been investigated. We observed that these nanotubes are flexible at moderate applied force. Therefore, after a few cycles of loading and reloading, the nanotube can easily and totally recover its initial shape. Using force-displacement curve and data, we calculated the Young's modulus of nanotubes. Measured Young's modulus proves that these nanotubes as strong insulator nanotubes.

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