Mechanical Measurement of Individual Carbon Nanotubes Using MEMS and the S100 Nanomanipulator

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Introduction

Carbon nanotubes' (CNTs) unique physical properties of high strength, low weight, high thermal and electrical conductivity, and high surface area have made them a primary focus of nanotechnology research. But before CNTs can be utilized in engineering systems, their fundamental material properties must be well understood. Previously, nanotubes have been mechanically characterized as bulk bundles, or as composites with polymers. With the S100 Nanomanipulator System and custom designed microelectromechanical systems (MEMS) test structures, mechanical investigation of individual CNTs is possible. Nanotubes can be manipulated in 3D, attached to MEMS devices, and tested *in situ* using the S100 system and a scanning electron microscope (SEM). This application note reviews the methods for attaching CNTs to MEMS devices and measuring their force-deflection characteristics.

Materials

Many possibilities exist for measuring the mechanical response of CNTs. For this application note, multi-walled nanotubes were manipulated with tungsten probes and attached to surface micromachined polysilicon MEMS devices.

Multi-Walled CNTs

Nanotubes were received in suspension from the University of Kentucky (http://www.mrsec.uky.edu/direct/grulke.htm). To provide accessible free ends, the CNTs were rinsed using detergents and stored in high-purity methanol. A drop of the methanol/CNT mixture was placed on a glass slide, and allowed to dry. A separate glass slide with a small amount of carbon tape adhesive at its edge was prepared and dragged over the dried CNTs, creating available tubes as **(Figure 1)**.

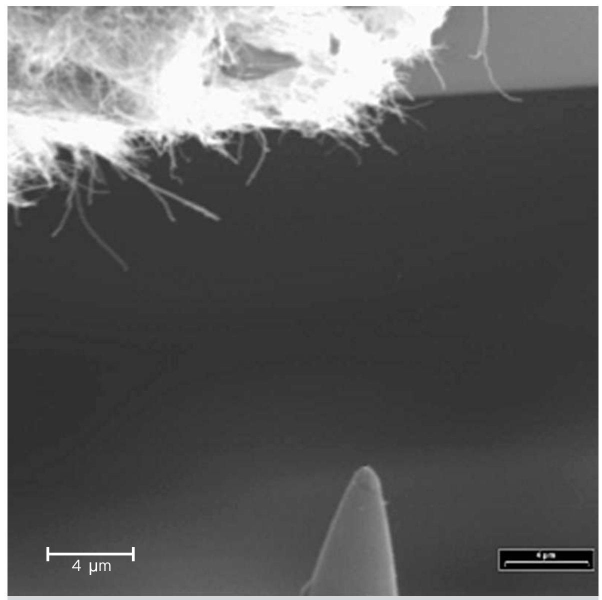

Figure 1 Multi-walled CNTs extending over the edge of a glass slide, with probe approaching at the bottom

Tungsten Probes
Standard microelectronic testing probes were installed as end-effectors on the S100 Nanomanipulator System. However, finer tipped Zyvex NanoEffector® probes will enhance dexterity for CNT samples that are densely packed.

Polysilicon MEMS Devices
Many types of MEMS test structures can be used for characterizing CNTs. A simple approach is to use calibrated atomic force microscope (AFM) cantilevered beams. In this custom application, commercially fabricated polyMUMPs (http://www.memscap.com/memsrus/ crmumps.html) devices were used. A 3D model of one such tensile testing device is shown (**Figure 2**).

Current supplied through the connection pads created differential, resistive heating in the thermal actuator. Thermal expansion caused the released actuator to deflect toward the right of the image. With a CNT connected between the actuator and movable gauge flexure, relative displacements of the tube ends were measured in the SEM against the stationary deflection metric.

Zyvex MEMS engineers can assist in designing other types of micromechanical test beds, utilizing methods like electrostatics for actuation and integrated sensing. This application note presents a simpler technique, using one S100 Nanomanipulator in place of the thermal actuator.

Set-up
The 3D nature of this application requires that the installation and setup of the S100 system, CNT sample, and MEMS device be considered carefully. The Nanomanipulator's large sample area allows great flexibility in mounting the CNT and MEMS samples. A standard SEM stub may be modified to provide access to both samples by simply filing one edge of the stub at 45 degrees. The nanotube sample may be bonded to the top of the stub using carbon tape or epoxy, and allowed to extend over the filed edge. The MEMS sample can then be attached to the inclined surface, directly below the overhanging CNTs. Silver filled epoxy may be used at the corners to ground samples to the stub.

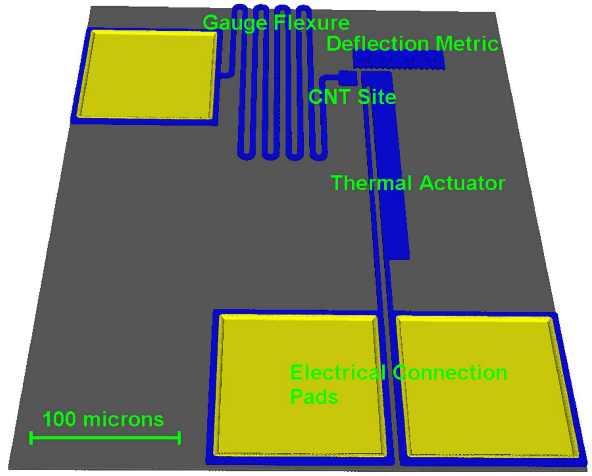

Figure 2 Three-dimensional model of a thermally actuated MEMS device for tensile testing of CNTs.

Due to the S100's large travel area, multiple MEMS and CNT samples can be used without breaking vacuum and exchange mounts. The probe was installed into the S100 quadrant facing the MEMS chip, and the manipulator was moved in all three dimensions to ensure that the samples were placed within the work volume of the system. After this check, the probe was centered near the desired CNT sample, the vacuum chamber was closed, and the SEM activated.

CNT Attachment, Manipulation, and Characterization

Using the S100 System, it was possible to select an individual CNT from the prepared sample, attach it to the probe using electron beam induced deposition (EBID), remove that tube from the sample, and attach it to a tensile testing structure.

Attachment

Trace gases present in the SEM vacuum chamber can be deposited onto surfaces being struck with the electron beam. The tungsten tip was positioned against an exposed CNT end and the electron beam was focused, in spot mode, at the point where the tube and tip meet. Electron beam deposits "welded" the CNT rigidly to the probe. The tube was then pulled from the CNT sample and manipulated freely in three dimensions. The Zyvex application note "Attaching a Nanotube to a Zyvex S100 Nanomanipulator End Effector" provides an excellent, detailed description of this process. An example of a tube being extracted from a CNT sample using the S100 is shown in **Figure 3**. The image has been color inverted for clarity. **Figure 4** shows a close-up image of a nanotube that has been EBID-welded onto a tungsten probe.

Manipulation

After the nanotube was removed from the sample, the probe was further retracted to place it (approximately) over the MEMS device. The focus and position of the SEM image was changed until the desired MEMS site was clearly visible, then the probe was lowered with the S100 Nanomanipulator until it came into focus. Using the S100, it was possible to position the free end of the tube at any location on the MEMS structure. In this application note, the tube was attached to the movable gauge flexure.

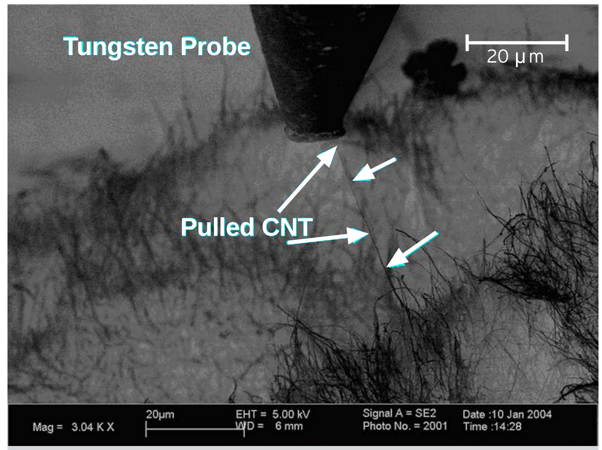

Figure 3 Tungsten probe end-effector for the S100, used to pull a single tube from the CNT sample.

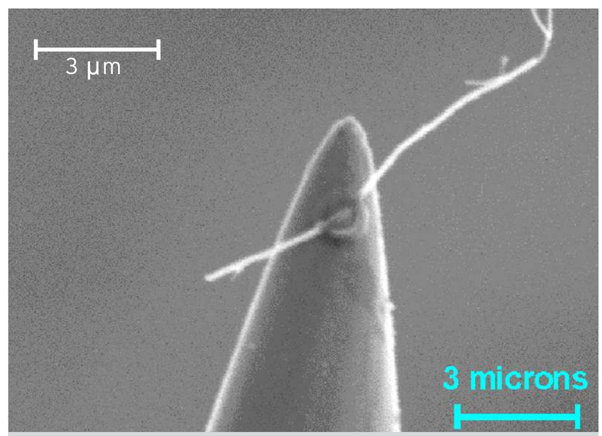

Figure 4 Close-up image of the electron beam weld used to attach a CNT to a tungsten probe.

As the tube end came into contact with the polysilicon surface, an electrostatic attraction suddenly held the CNT against the MEMS device. To test exactly where the tube was in contact, the fine positioning controls of the S100 were used to move the probe back and forth, paying attention to the region of the CNT over the MEMS surface. As the tube was moved, it appeared to rotate around a fixed spot, which defined the point at which the two surfaces touched.

In spot mode, the electron beam was focused at this point, welding it in place. The beam dwell time was equivalent to that for the weld onto the probe. This ensured that the tube-to-MEMS weld was as strong as the tube-to-probe weld. **Figures 5 and 6** show a nanotube being positioned on a MEMS device and welded into place, respectively. **Figure 7** shows a 3D AFM scan of the region where a nanotube was welded to the surface.

Characterization

To measure the force-deflection characteristics of the attached nanotube, the S100 was moved away from gauge flexure. As the probe retracted, tension was created in the tube. This force transferred to the flexure and began to deflect it. The SEM was used to capture images between S100 movements, making sure to include the stationary deflection metric in each picture. **Figures 8 a-d** show a sequence of such images captured while moving the MEMS device with an attached CNT.

Beam bending analysis for approximate flexure geometries, or finite element simulations for more precise results, can be used to find the spring constant relating force to deflection for your particular gauge design. For the flexure used in this application note, analytical and simulated results agreed on a spring constant of 0.63 μN/μm. Based on this constant, and digital image analysis of flexure deflections, a tensile force of 2.4 μN was applied to the tube.

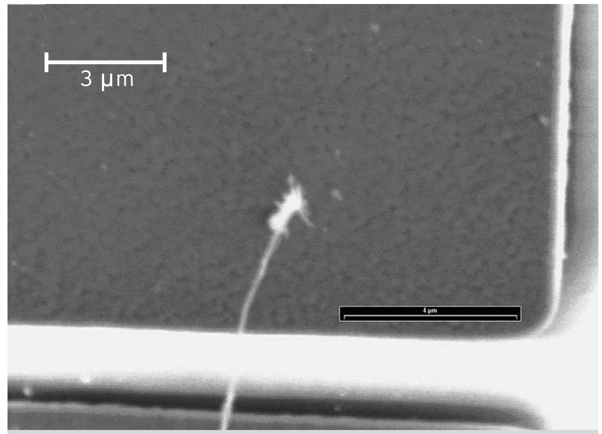

Figure 5 Free end of a carbon nanotube in contact with a polysilicon surface.

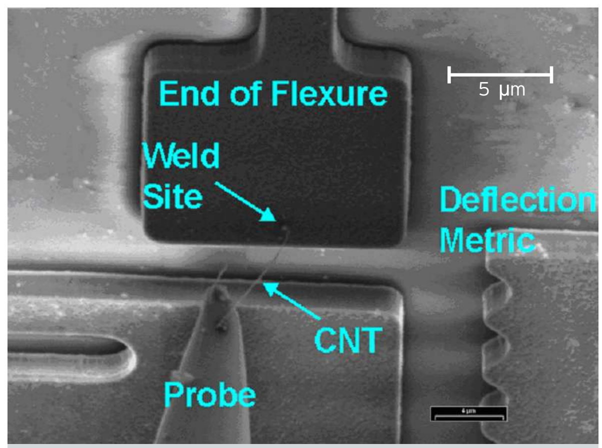

Figure 6 CNT welded between the movable MEMS flexure and the tungsten probe.

Conclusions

Although carbon nanotubes are a promising material for future nanotechnology projects, they still require some very fundamental material properties research before they can be included in engineered systems. However, such characterization has mostly been done on bulk clusters of tubes, or on tubes mixed with polymers. The challenges to CNT measurement are numerous. Their unique aspect ratio makes them difficult to study as individual structures. Diameters on the order of tens of nanometers require a fine positioning capability in three dimensions to select a single tube, while lengths of up to tens of microns require large travel for manipulation and deflection. Their overall size makes optical placement and measurement of tubes impossible, so an SEM-compatible system is essential. During the EBID process, specimen drift would allow a misplaced weld to release a nanotube on the surface, meaning that a very stable manipulator must be employed. The Zyvex S100 Nanomanipulator fits these stringent requirements, and using this system, it is possible to conduct ground-breaking mechanical experiments on individual nanotubes.

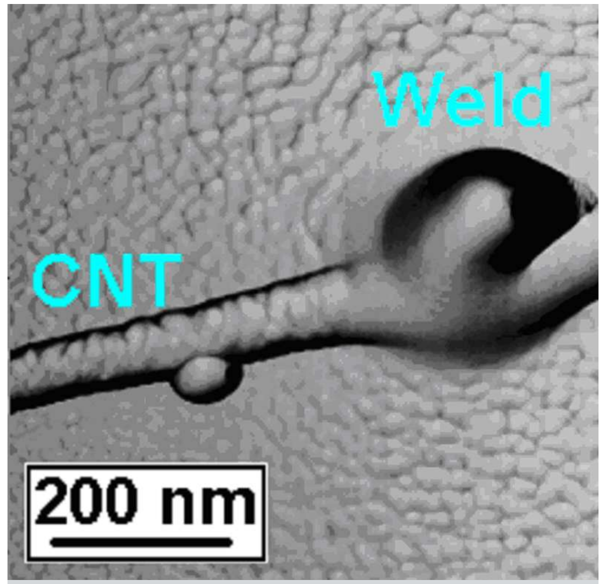

Figure 7 AFM image showing the smooth weld bonding a nanotube to a surface

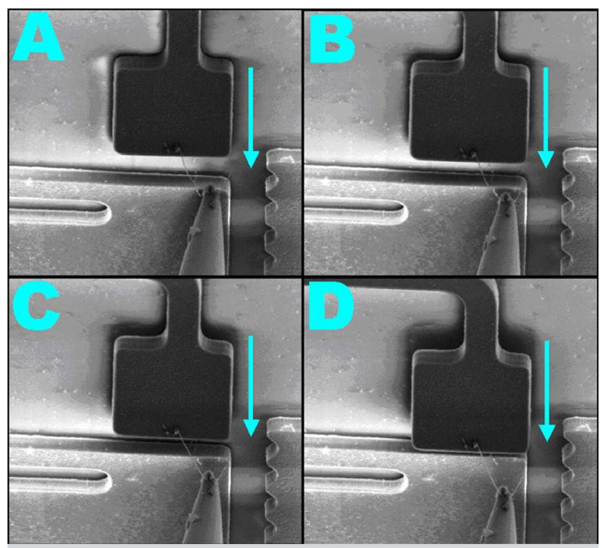

Figure 8 Image sequence showing a MEMS device being deflected by a CNT attached to an S100 Nanomanipulator

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