# Attaching a Nanotube to a Zyvex S100 Nanomanipulator End Effector

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## Introduction

Over the past few years, researchers have developed many novel materials at the nanometer scale, including different types of structures, including nanotubes, coils, and wires. These structures will play an important role in the creation of stronger and lighter materials, as well as in the engineering of molecular devices. With the advent of new materials, there is also the need for new characterization tools. The Zyvex S100 Nanomanipulator System is designed specifically for manipulating and characterizing nanostructures. It can also serve as a platform to engineer nanoelectromechanical devices.

The purpose of this application note is to describe a method for mounting a multiwalled carbon nanotube (MWNT) to an end effector (or probe). This note presents a discussion of the necessary materials, the set-up of the experiment, and the detailed procedure for mounting the nanotube. The term "end effector" and "probe" are used interchangeably in this document. Researchers can use a similar procedure to mount nanotubes and nanowires.

The first step to performing any of these tasks is to physically manipulate a free-standing structure.

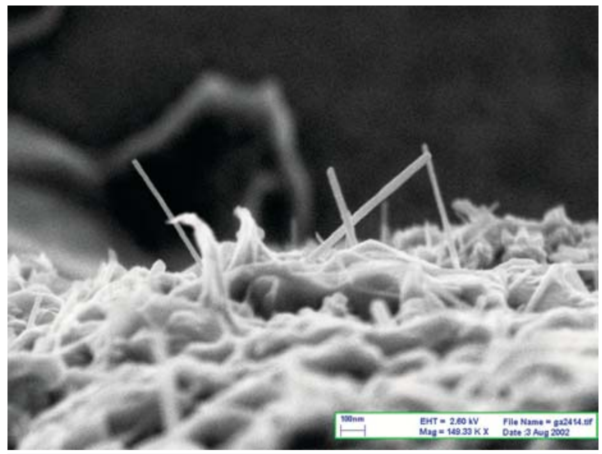


**Figure 1** Multiwalled carbon nanotubes (Alfa Aesar)

## Materials

The researcher can mount structures made up of different shapes, sizes, and materials. The structure described here is a multiwalled carbon nanotube (MWNT): the probes are sharpened tungsten wires (or silicon AFM tips). Later, we will explain how to affix the nanotube to the end effector using electron beam induced deposition (EBID).

### Multiwalled Carbon Nanotubes

We will use MWNTs in this demonstration. The nanotubes will usually need some processing to loosen and untangle them before they are adequate for manipulation. There are many ways to achieve this (i.e., sonication and treatment with surfactants), so we will not discuss that here. See Reference [1] and [2] for some other ideas.

The nanotubes should be on a wire or flat wafer surface. It is important that the long axis nanotubes are normal to the electron beam of the microscope and protrude from the surface in free space. This allows the probe to gain access to the nanotubes. It will also help the user to image the tubes under the SEM (see Figure 1).

### End Effectors

End effectors are defined as the "tools" at the business end of the manipulator. The user selects the type of probe/end effector according to their specific application.

- Tungsten wires are good (for manipulation tasks that require probes with a high precision radius of curvature) since they are easy to etch to fine points (<15 nm radius of curvature).
- Platinum wires are well suited for electrical measurements
- Cantilevered probes are good for mechanical characterization [3].

See Figures 2 and 3 for examples of end effectors.

*A variety of end effector probes are available for purchase from Zyvex Corporation. Call 972.235.7881 (ext. 244) for more information.*

## Set-up

It is crucial to success of the experiment to set up the S100 before starting. Take an extra minute to make sure all of the probes are in place and are making electrical contact through the feed-throughs. If possible, also make sure that the probes have not been dagamaged while handling.

The nanotubes need to be on a surface that is accessible to one or more of the probes. The rotational stage is a good place to mount the tubes (as opposed to mounting them in one of the end effector plugs).

In either case, the tubes should protrude from the surface and be normal to the electron beam and clear of obstructions. This will allow the probe to approach them while imaging at high resolution.

Once the S100 is in the SEM chamber, verify that the feed-through connectors are on properly. Always follow the procedure for handling and operating the S100 as prescribed in the "S100 User's Manual."

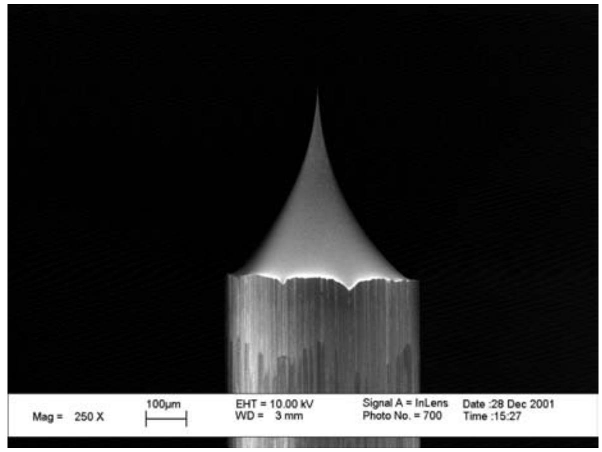


**Figure 2** Electrochemically etched Tungsten wire

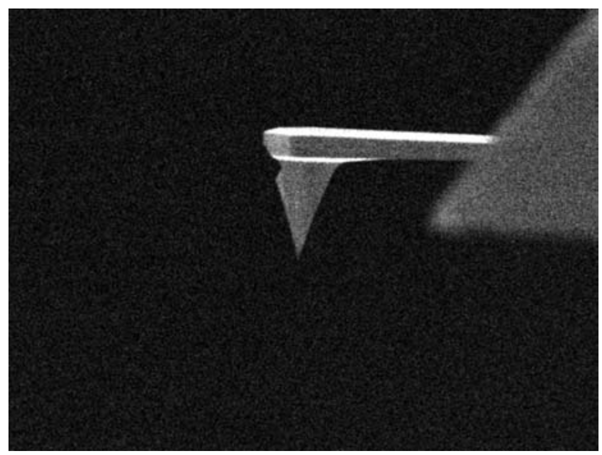

**Figure 3** Atomic force microscope tip (Non-Contact/ TM, MikroMasch)

## Approaching a Nanotube with an End Effector

This section will describe the procedure for making contact to the nanotube. We also list some tricks to optimize the visual feedback.

### SEM Modes of Operation

There are different modes of operation for each microscope (i.e., reduced scan or spot mode). One can observe the motion of the manipulator and the nanotubes with high resolution in near real-time by utilizing the 'reduced scan size' operating mode.

The ideal mode of operation for applying EBID is 'spot mode,' where the electron beam is fixed upon a single point. The next operating mode choice is the reduced scan mode, where the electron beam raster scans across a smaller area. Generally, this area is adjustable.

## Approaching the CNT

The best way to start approaching a CNT is to have a target nanotube. A good candidate nanotube tends to be long, relatively thin, and disentangled from bundles or neighboring tubes. There are some excellent experiments that could be performed on tangles of nanotubes or on tubes that are "stuck" together. However, for the purposes of this application note, try to use individual nanotubes. The reason for choosing a thin nanotube has to do with overcoming shadowing effects from the nanotube as you perform EBID. Once a good nanotube is found, select the positioner that has an end effector.

Begin by using the coarse positioners to bring the end effector within the range of the fine positioners (100 µm X and Z; 10 µm Y using the 'Local Coordinate System.'). See Figure 4.

The best way to approach the nanotube is from below (See Figure 5).

Using the fine positioner in the Z axis, slowly bring the end effector into contact with the nanotube (See Figure 6). Verify contact by seeing the nanotube deflect.

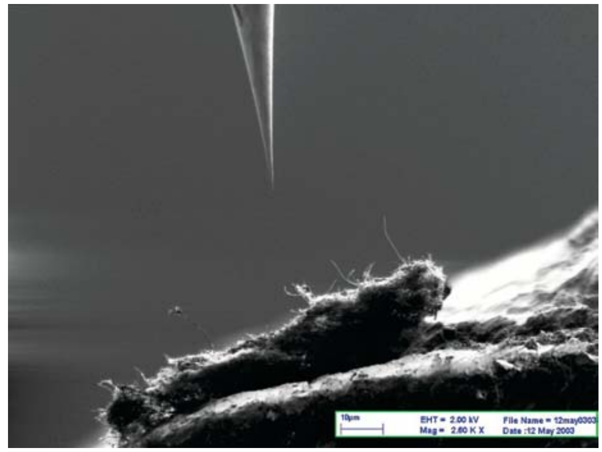

**Figure 4** On approach to the nanotube, which is circled, using the coarse positioners (Mitsui CNTs, Leo 1550 FE-SEM, Zyvex S100)

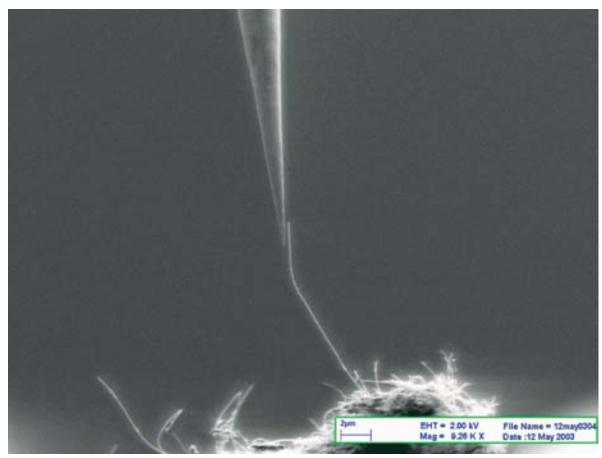

**Figure 5** Fine positioner adjustments to maneuver close to the nanotube

If the nanotube is loose enough, the user can remove it from the surface via *van der Waals* forces alone. At this point, retract the nanotube. If the nanotube needs to be bound more strongly to the probe, try performing electron beam induced deposition to affix the tube to the probe.

To apply EBID, use either a reduced scan size or the spot mode of operation. While in focus on the nanotube and probe, scan the reduced area for several minutes. The electron beam will deposit amorphous carbon material, which tends to be ubiquitous even in high vacuum environments. This material with bind the nanotube to the probe.

## An Alternative to EBID

Scientists have reported that an effective method for attaching nanotubes to probes is to dip the probes in glue *before* making contact with nanotubes. For example, Dai, et. al. demonstrated attachment of nanotubes to SPM probes by bringing the probe tip in contact with carbon tape *in situ* [5].

To use this method, use carbon tape (acrylic adhesive) in addition to the aforementioned materials.

Apply the carbon tape to an unsharpened wire and place it into one of the inputs on the end effector plug. It can also be placed on the center stage. Bring the probe into contact with the adhesive. Retract the probe, and approach the target nanotube. The drawback of using the adhesive is less control over where, when, and in what state the nanotube sticks to the probe. It may also be difficult to ensure ohmic contact to the probe, even though the carbon tape tends to be electrically conductive.

If the SEM has lithography capabilities, such as a focused ion beam, utilize them to put down metals as the binding material. The focused ion beam works in a similar manner as EBID, but one should follow the manufacturer's instructions. Having an integrated lithography system can be very useful. Materials other than carbon can be put down, and in addition, one can pattern the deposition. This way, researchers can perform experiments on patterned electrodes.

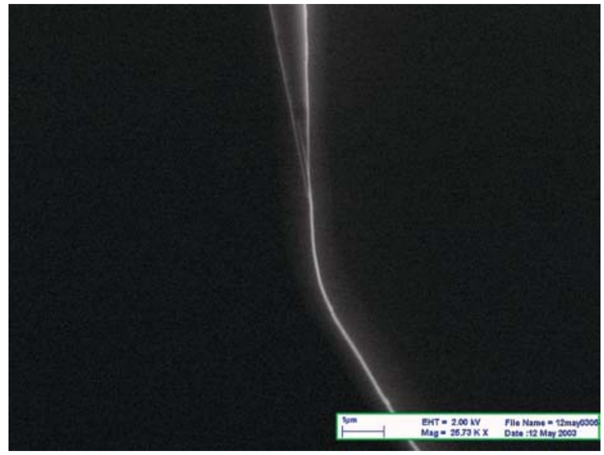


**Figure 6** Using the end effector to contact the nanotube

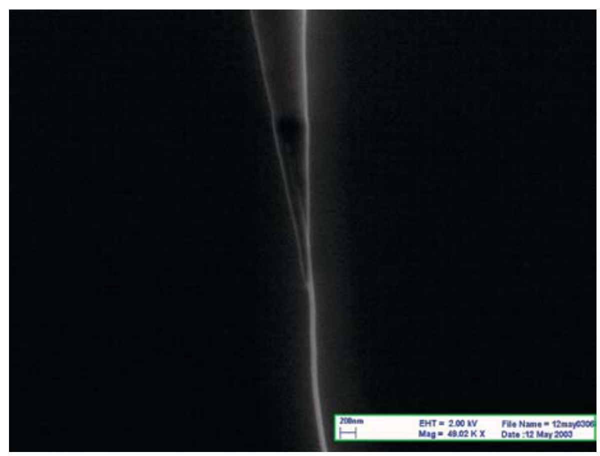


**Figure 7** Apply EBID

## Electron Beam Induced Deposition

Electron beam induced deposition (EBID) is caused by the dissociation of surface adsorbed molecules by high-energy electrons. EBID layers will act as a binder as the deposited amorphous material coats the nanotube.

### Optimizing the Deposition

Koops, et. al., have investigated this phenomenon for a number of years. From the literature, we see that the density $N$ of adsorbed molecules on the substrate surface varies with time:

$$dN/dt = g * F(1-N/N_0) - N/\tau - q * N * f, \qquad \textbf{(AppEq 1)}$$

using $g$ as the sticking coefficient, $F$ as the molecular flux density arriving on the substrate, $N_0$ as the molecule density in a monolayer, $\tau$ as the mean lifetime of the adsorbed molecule, $q$ as the cross section for dissociation of the adsorbed molecules under electron bombardment, and $f$ as the electron flux density.

The layer growth rate, R, is

$$R = v * N * q * f, \qquad \textbf{(AppEq 2)}$$

where $v$ is the volume occupied by a dissociated molecule or its fractions [4].

From (AppEq 2), we can see that the growth rate depends on the cross section for dissociation of molecules in the path of the electron beam. From quantum mechanics, we know that the differential (and total) cross section for scattering is inversely dependent upon the energy of the incident particle. Thus, one should adjust the accelerating (EHT) voltage of the electron beam to optimize the deposition efficiency. As a rule, the lower the EHT value, the higher the electron cross section, allowing for a higher probability of electron-molecule interaction. Also, a lower mean free path will increase the EBID efficiency for similar reasons. Higher pressures result in higher probability for electron-molecule collisions. Thus, if possible, perform the EBID growth at *relatively* high pressure (~$10^{-5}$ torr).

EBID is not a "fire and forget" technique. If the SEM image begins drifting, continue depositing material. This could ruin the experiment by applying EBID to inappropriate areas.

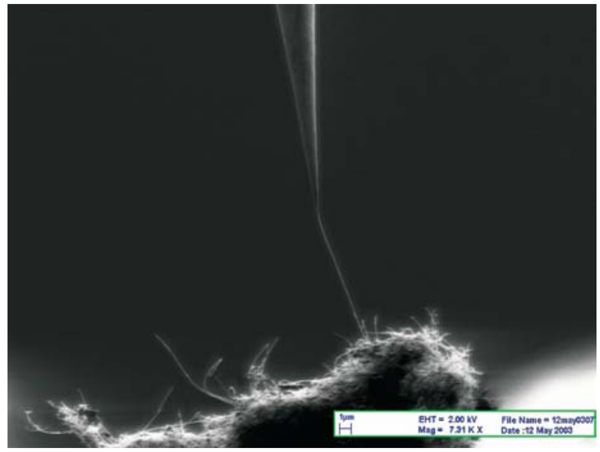


**Figure 8** Begin pulling the nanotube from the surface.

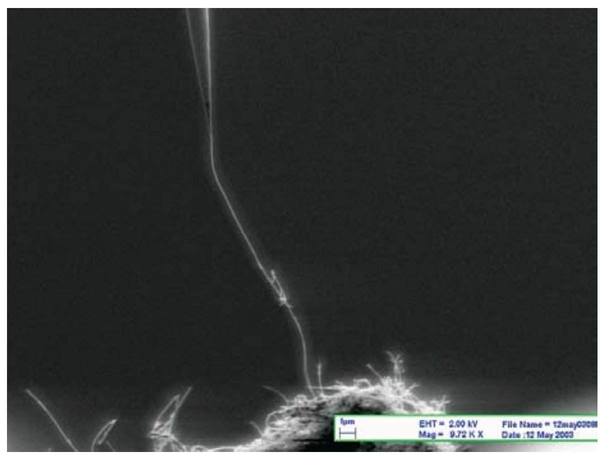


**Figure 9** The nanotube loosened and beginning to come out.

## Conclusions

The procedure for making contact with a nanotube is straightforward and makes use of the most fundamental capabilities of the S100. Once the user has mastered this procedure, a wide variety of experiments. Feel free to try new methods of CNT processing and of nanotube extraction. Also, feel free to contact Zyvex for support.

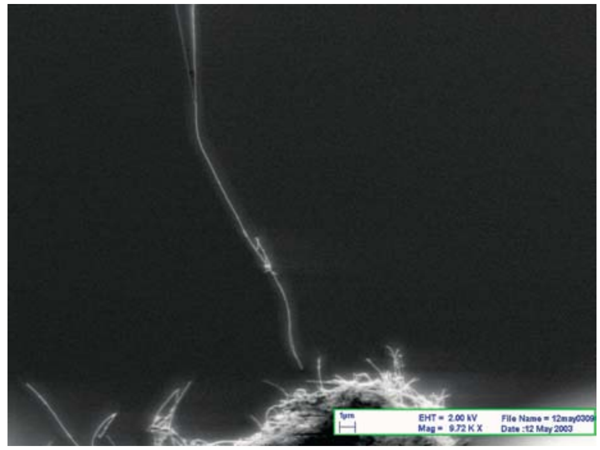


**Figure 10** The nanotube is free from the surface and attached to the probe

## References

[1] Y. Sato, et. al., J. Phys. Chem. B 105, 3387 (2001).
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