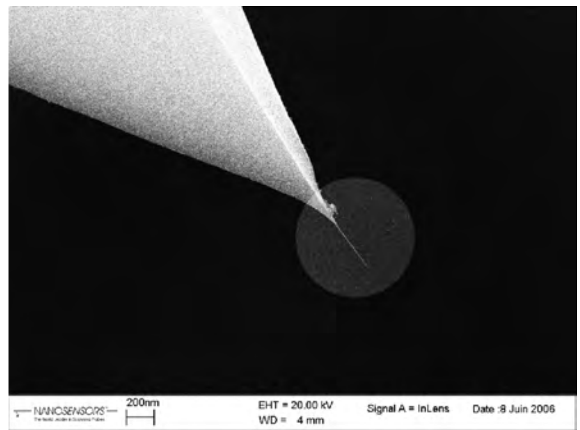
How to use a Single / Double Wall Carbon Nanotube AFM probe

Please read these instructions carefully



This short manual is intended to present briefly some guidance for Carbon Nanotube (CNT) probe imaging. The presented methods have been tested on Nanoscope™ systems but generally they can be easily extended to other systems.

Introduction

Most failures to image using Carbon Nanotube probes result from a lack of control of the dynamics of the Nanotube at the tip apex and consequently lead to an unstable tip-surface interaction. Note that it is rarely the cause of a faulty Nanotube or a blunt tip.

Before using the Carbon Nanotube AFM probe please imagine briefly what will happen to the CNT whilst engaging the probe and imaging: a Carbon Nanotube is a cylinder which is extremely strong when stressed in axial direction, but through its outstanding aspect ratio (diameter of some Nanometers, length of up to some hundred Nanometers) it can be bent very easily in radial directions.

Therefore, the forces applied to the CNT have to be adjusted very carefully and controlled properly. At first, a relatively small cantilever oscillation amplitude (oscillation amplitude much smaller than CNT length) should be chosen. Secondly, the tip-sample interaction force has to be big enough to ensure a proper contact but small enough that the CNT might be bent but does not buckle or flex. NANOSENSORS™ CNT AFM probes are recommended for use in Tapping Mode only. Single and Double Wall CNTs are very flexible and can bend very easily. Therefore they are not suitable for use in Contact Mode

Here, we describe two different methods of using NANOSENSORS Carbon Nanotube SPM probes. Method A is the easier way to image with a CNT probe. Method B allows the user to adjust the tip-sample forces more directly with the risk of buckling the CNT more easily. Eventually both methods have to be repeated several times to get a stable image.

METHOD A

A.1 Tuning Tune the cantilever with an amplitude of about 0.6 to 1.0 Volts.

A.2 Engage Set the scan size to 0 nm. Engage Setpoint 1.0. Engage the tip. When the tip is in contact with the surface increase the scan size slowly to the desired scanning size and scanning speed.

A.3 Adjusting the tip-sample forces

(1) If the image is not stable or free of artefacts due toCNT bending decrease the interaction force slightly (enlarge the Amplitude Setpoint). Do only increase the Amplitude Setpoint whilst the tip is engaged (enlarging the forces in this method will be only done by reducing the Engage Setpoint whilst the tip is withdrawn). Let the CNT stabilize. If there are still artefacts decrease interaction again. Repeat this procedure until the image stabilizes or the tip is loosing contact.

(2) If contact is lost withdraw the tip, set the scan size to 0nm again and reduce the Engage Setpoint from 1.0 to 0.95V.

(3) Repeat this procedure with reduced Engage Setpoint (down to about 0.8V) until you get a stable image.

METHOD B

B.1 Tuning Choose ther cantilever oscillation amplitude much smaller than the length of the CNT. For a NCH cantilever in air, a free amplitude of about 0.5V in most photodiode meters is recommend. Tune with light interaction. Tune by a 10% offset on the left side of the resonance peak.

B.2 Engage Set the scan size to 0nm. Set the Engage Setpoint value at 2.0 (false engage). Press Engage and let the tip approach. When the approach is completed, the Z-voltage is scaled out (retracted). Reduce the Setpoint slowly and carefully until the tip approaches the surface (Z-voltage becomes stable). Increase the scan size and scan speed to the desired values (possibly higher than 10μm/sec).

B.3 Adjusting the tip-sample forces

When engaged control if trace and retrace are tracking properly. If the tracking is

(1) **GOOD**	Start imaging.
(2) **POOR**	Let the CNT scan for 5 to 15min. Often the CNT will stabilize after some minutes.
(3) **still POOR**	Increase the tip-sample interaction by decreasing the amplitude setpoint. Decrease the setpoint slowly down to a minimum of about 0.2V.
(4) **still POOR**	Withdraw and reengage the tip.
(5) **still POOR**	Determine the amplitude setpoint by performing the working-point determination procedure.
(6) **still POOR**	Increase continuously the tip-sample interaction.

B.4 Force Calibration plots - working-point determination

What kind of information can be obtained from a force distance curve:

Is there a Carbon Nanotube? How long is it?

What interaction regime / amplitude setpoint should be used for imaging (working point determination)?

Attention: the working-point determination of the CNT probes is recommended for experts only! Do not perform any standard Force Calibration operations. This will destroy the CNT.

Before using a CNT tip, exercise yourself in this technique by trying this procedure first with a used standard silicon probe. Performed in a wrong way this procedure may destroy the CNT.

Go to the Force Curve menu. View the amplitude channel (if possible). Choose single ramps (ramp size 50nm) with a very low ramp speed of about 0.5Hz. Figure 1 shows a Force Calibration plot for a standard silicon probe (large ramp size for the sake of clarity).

Procedure with a CNT probe: start with a small ramp far above the surface (1000nm, fig 2a). Image a single ramp. If no damping event is monitored (straight horizontal lines, or slight sinusoidal horizontal curves) bring the tip towards the surface (increase the z-start value by 25nm (much smaller than the CNT length), fig. 2b-d. Image a single ramp…

Continue until the curve is starting to drop (contact to surface) or showing any non-linear behavior (CNT bending). Continue very carefully until the zero amplitude horizontal line is showing up.
DO NOT DECREASE THE Z-START VALUE ANY FURTHER (marked as "NO GO" z-values in fig. 3! This will damage or destroy the CNT because the tip is scratching over the surface, thereby eroding the silicon apex that is the support of the CNT tip. Increase the z-ramp size window to measure the approximate length of the CNT

In order to determine the working amplitude setpoint choose a voltage for which the approach curve as well as the retract curve have only a single solution (therefore avoiding multistable behavior, which is the source of a lack of control). Go back to the image mode. Enter the determined amplitude setpoint. Start imaging.

Please note that Carbon Nanotubes can be easily destroyed by electron irradiation whilst imaging them in an electron microscope. Also, force curve imaging will destroy the CNT due to heavy loads!

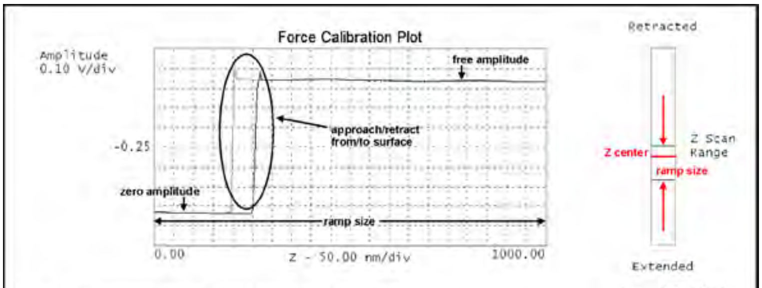


Figure 1: Force Calibration Plot of a standard silicon probe

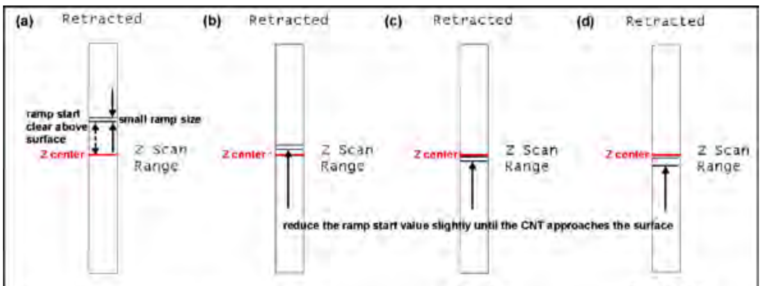


Figure 2: the z-piezo, ramp size and ramp start

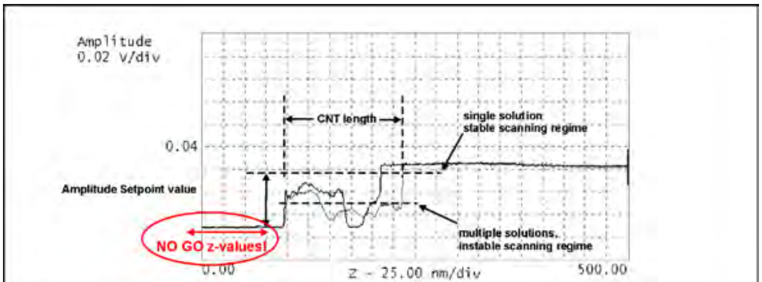


Figure 3: Force Calibration plot of a CNT probe

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