

Calibrating AFM Cantilevers

The importance of a properly calibrated AFM cantilever is tantamount to the collection of reliable quantitative data. Therefore, all tips used in such applications should be calibrated before use. The following is a concise set of instructions to assist Ward group members in learning a simple method by which AFM cantilevers can be calibrated.

The principle of this calibration method stems from the fact that each cantilever will have a resonant oscillating frequency associated with its intrinsic spring constant. Adding a mass to the end of the cantilever effectively retards the resonating frequency and measurement of both the mass on the end of the cantilever and the resulting resonance allows extrapolation of a spring constant associated with the AFM tip. For further information, please see the following paper by Cleveland and Manne; *RevSciInstrum*, 1993, 64(2), 403-40.

This procedure can be used for standard, rectangular tapping mode tips or triangular contact mode tips.

(1) CANTILEVER SETUP

An AFM cantilever should be selected and mounted so that the additional tip (if there are more than one on the block) has been broken off. The remaining tip should be set up in air according to standard contact mode procedures. When the tip has been mounted and the laser has been carefully positioned, adjust the setpoint and the difference so that both read as close to zero as possible.

(2) CANTILEVER TUNING

Next, switch the AFM from contact mode to tapping mode and load the tapping mode profile in the AFM software.

Select the icon represented by a blue tuning fork on the toolbar or select “View->Sweep->Cantilever Tune” from the menu.

Several interface windows will appear. One will be labeled “Auto Tune Controls” and has four input boxes located in this window.

The input boxes on the left are labeled “Start Frequency” and “End Frequency”. These two inputs control the minimum and maximum frequencies over which the software will scan in an attempt to locate resonance. Different tip designs and sizes will resonate in different frequency regions and it is important to locate the lowest resonance peak. Triangular tips have a tendency to report very strong resonance from harmonic multiplication and the user should be careful to avoid these peaks which occur at frequencies higher than the base resonance. A typical frequency sweep size is from a

start frequency of .1 KHz to approximately 60 KHz. Note that more flexible tips, i.e. 200 μm narrow leg tips, will tend to resonate at approximately 15 KHz to 20 KHz and the sweep can be ended at approximately 30 KHz to avoid harmonic multiplying resonance.

The other two input boxes are labeled “Target Amplitude” and “Peak Offset”. Set the target amplitude to 0.50 Volts and the Peak Offset to 0.00%. These parameters can be left alone for the purposes of this procedure.

When the proper values in each input box has been set, click the button labeled “Auto Tune.” The software will execute a routine resulting in the identification of a resonant peak it feels is most likely the primary resonant frequency of the cantilever. If there are several multiplication frequencies, the routine will restrict the “sweep width” parameter in the “Sweep Controls” interface window so that the region associated with only one peak is displayed. Often this results in a confusing flat peak region. Change the “sweep width” parameter to a value of 30 KHz or larger to view the resonant patterns. Ideally, it is best to have one dominant peak with little multiplying resonance. If the sweep signal does not display a single or dominant peak adjust the laser so that it is aimed on a slightly different portion of the cantilever. The value displayed in the “Drive Frequency” input box is the resonant frequency for the cantilever. Record this value as the undamped resonant frequency.

(3) ADDING TUNGSTEN MASSES

The next step will involve adding a mass to the tip of the cantilever. A polydisperse Tungsten powder is used to add individual masses to the end of the cantilever. The powder can be purchased from Aldrich if we don't already have it in inventory. The location for this powder will be in the same box with the AFM calibration standards.

Attach a small glass coverslip to a magnetic AFM puck with tape or adhesive and sprinkle a SMALL amount of the tungsten powder on the glass such that there are several spheres sitting as individuals on the glass and not in clumps. Use the stage controls and up/down motor on the AFM control head and base to manipulate the position of the AFM cantilever so that a small tungsten ball is picked up and attached to the end of the cantilever. Use the up/down motor to raise the tip safely above the glass surface.

Set the AFM base to contact mode again and reset the setpoint and the difference so that they are both as close to zero as possible.

Switch the AFM base to tapping mode again and repeat the tuning procedure as described above.

(4) MEASURING THE TUNGSTEN MASS DIAMETER

When a resonant value has been obtained for the damped cantilever (which should be smaller than the undamped resonance), remove the AFM tip holder leaving the tip in the holder and measure the diameter of the attached tungsten sphere using the compound microscope. Convert the diameter of the tungsten sphere to a volume and use the density of tungsten to obtain a mass of the particle. Tungsten is 19.3 grams/cm³.

---Repeat steps (3) and (4) with a tungsten sphere of a different diameter.

(5) SPRING CONSTANT CALCULATION

$$M = k(2\pi\nu)^{-2} - m^*$$

Using the above relationship, the derivation of which can be viewed in the aforementioned paper by Cleveland and Manne, create an excel file plotting the mass of the attached tungsten sphere, M, against the corresponding values of $(2\pi\nu)^{-2}$.

A linear regression line of this data give the value of the slope, k , and this is the value of the spring constant of the cantilever.

Approximate spring constant values for a standard 4 tip Silicon Nitride AFM tip block are as follows

Wide leg 200 Micron:	.12 N/m
Wide leg 100 Micron:	.58 N/m
Narrow leg 200 Micron:	.06 N/m
Narrow leg 100 Micron:	.32 N/m

(6) CANTILEVER STORAGE

Finally, record the location of the tip block when it is stored and make note of the spring constant associated with this tip block and the specific cantilever that has been calibrated.

When the sensitivity is calibrated with this cantilever for contact mode, the sensitivity and the calibrated spring constant can be used together to calculate force vs. distance or force vs. voltage via the DI AFM software.