14
Thermal K

It is sometimes necessary to know the spring constant of an AFM probe. This chapter reviews the calibration method using Thermal K to determine spring constants.

Atomic Force Microscopy (AFM) is a powerful technique that can be used to quantify the elastic properties of materials or to measure single molecule unfolding or unbinding interactions on the picoNewton scale. AFM probes are composed of flexible, triangular or rectangular cantilevers with a sharp tip near the end of the cantilever. They can be manufactured from a variety of materials, but most AFM probes are made from silicon and/or silicon nitride (Si_3N_4) wafers using semiconductor-based etching processes.

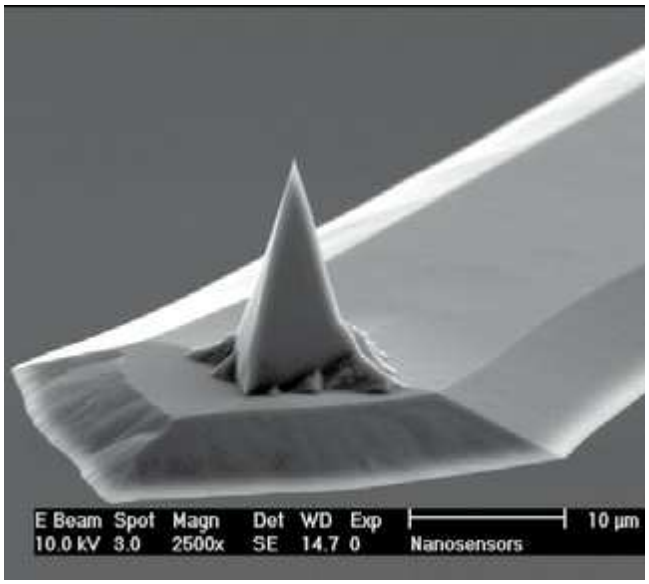

Figure 14-1 Scanning electron microscopy image of an AFM probe

An AFM probe's sensitivity, or spring constant (k), is the force required to bend the cantilever per unit distance (usually reported in Newtons/meter; N/m). It is an important factor in AFM probe behavior and performance for imaging applications and an essential parameter when attempting to quantify intra- or inter-molecular interactions or the compliance of materials with the AFM.

Nominal spring constant values are commonly reported by AFM probe suppliers. However, because of manufacturing variability, the spring constants of individual cantilevers can vary greatly; even between probes in the same batch or from the same wafer. Consequently, in order to accurately quantify material compliance or molecular interactions with the AFM, the spring constant for each probe should be determined by empirical or semi-empirical methods.

Thermal K Calibration

Thermal K is an option for the Keysight AFM that allows the user to quickly and easily calculate the spring constant of an AFM probe. The Keysight Thermal K method calculates AFM probe spring constants by describing the motion of the cantilever as a harmonic oscillator through the use of the equipartition theorem from fundamental thermodynamic theory.

The theory states that the kinetic energy stored in a system at a momentum coordinate, which is the deflection of a cantilever from its equilibrium position, is equal to one half of the thermal energy of the system.* The deflection value from the resting state of the cantilever is considered to be small for AFM probes, and it can be assumed to be linearly related to the force required to deflect the cantilever according to Hooke's Law:

$$F = -\kappa \cdot x$$

For an ideal spring with a spring constant value of k, the thermal noise according to its position allows the spring constant to be determined. In principle, simply measuring the deflection value of the cantilever and temperature would permit its spring constant to be calculated. However, an AFM cantilever is not an ideal Hookean spring, so its potential energy can not accurately be described in such a simple manner. Consequently, the area of the resonant peak in the Power Spectrum Density (PSD) spectra of the vibrational noise permits a more accurate determination of k.

Thermal K Setup

The Thermal K method requires installation of an additional data acquisition card. To enter the acquisition card address, open the Setup section of the Controls drop down menu and select Components. Enter

*S.M. Cook, T.E. Schaffer, K.M. Chynoweth, M. Wigton, R.W. Simmonds, and K.M. Lang, *Nanotechnology* Volume 17, 2135 (2006).

the address in the Acquisition Card Address field and select the Acquisition Card (Thermal K) check box to activate the card.

Before the spring constant can be computed, the **Deflection Sensitivity** has to be known. This number can be entered directly in the Spectroscopy Advanced tab, or it can be computed as described in the "Spring Constant Calibration section below.

A rigid sample like mica will also be needed to obtain a good force curve.

NOTE

In order to obtain accurate spring constant values with Thermal K, the Z closed loop scanner is recommended. Also, in order to maintain the integrity of the tip of the AFM probe, it is often preferable to perform the calibration after all Force vs Distance sweeps have been performed. PicoImage is required for post-processing. The **Cantilever Settings** button in PicoImage allows the user to set (or calculate) the Sensitivity, enter a spring constant after the fact, and then display the plot with V, nm or nN vertical scale units.

Spring Constant Calibration

In the PicoView software main window:

1. Choose **Mode >Contact**.
2. Choose **Controls >Spectroscopy** to open the Spectroscopy window.
3. In the Spectroscopy window, confirm **Force vs Distance** for the plot type (Figure 14-2).
4. In the Spectroscopy Basic tab, click on the **1** button [1] to do a single sweep.

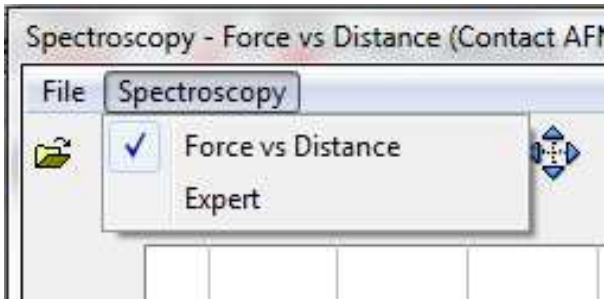

Figure 14-2 Spectroscopy window showing Force vs Distance selected

5 Obtain a force curve using a rigid substrate like freshly cleaved mica.

6 In the plot window, right-click to **Add Ruler to Plot** on a steep portion of the deflection curve. Click on the ruler line to move the second marker closer to the jump-to-contact point as shown below.

Figure 14-3 Force curve (Deflection vs distance) with ruler markers

7 Right-click on the ruler after the end points are set and select **Deflection Sensitivity** (Figure 14-4) to update the value in the Advanced tab.

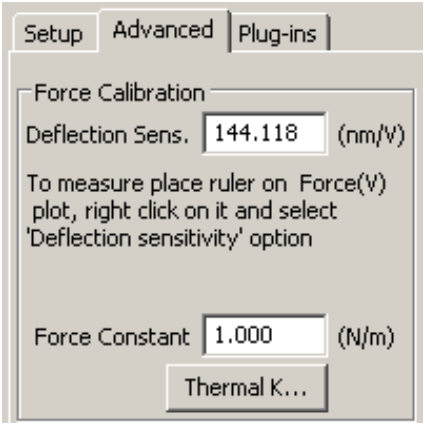

Figure 14-4 Advanced tab in the Spectroscopy window

8 Click the **Thermal K...** button in the Advanced tab of the Spectroscopy window.

A message window will open as a reminder to withdraw the tip.

9 Click **OK**.

10 Click on the Withdraw button at the top of the main window to withdraw the tip.

This will open the Thermal K plot window, similar to that shown below.

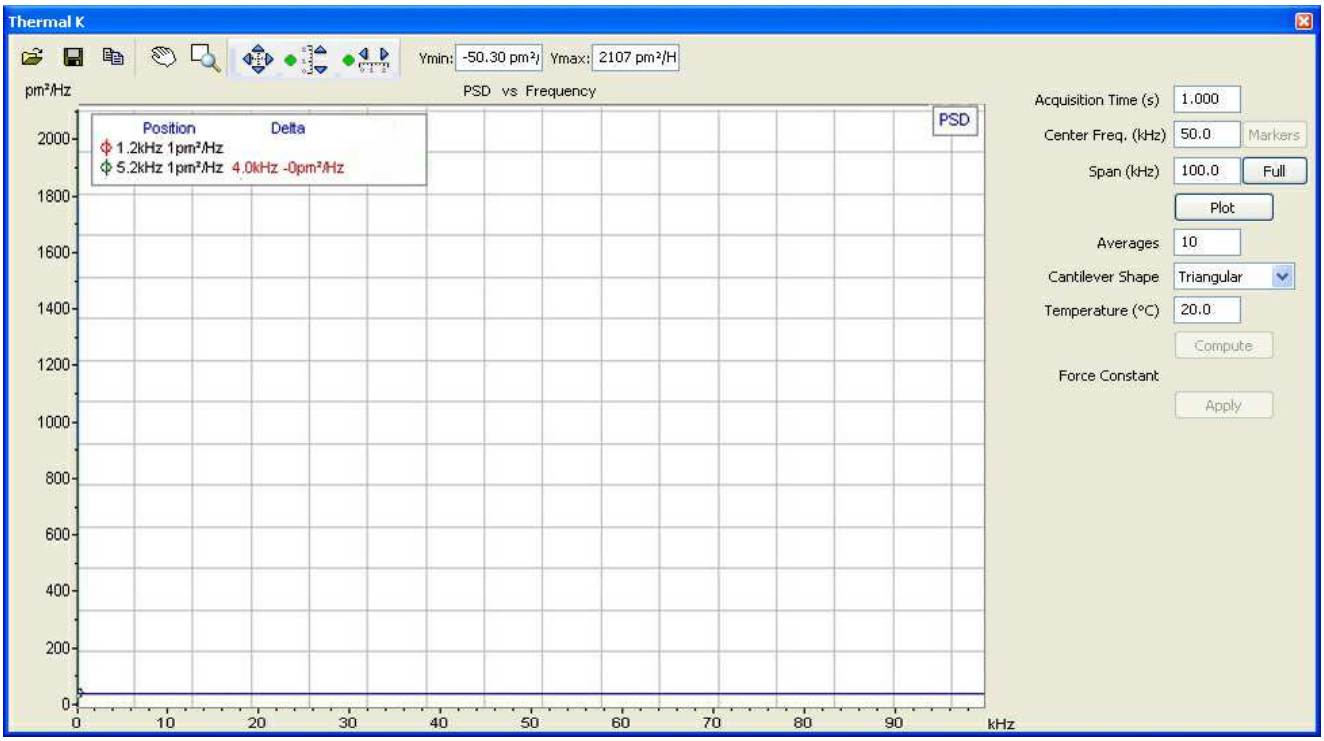

Figure 14-5 Thermal K plot window

11 Set the parameters:

- **a** Acquisition Time to **1.0 s.**
- **b** Averages to **10**.
- **c** Chose the correct cantilever shape from the drop-down menu.
- **d** Temperature to the correct ambient temperature in Celsius.

12 Click the **Full** button on the right side of the Thermal K window.

13 Click the **Plot** button.

14 A Thermal K PSD plot (Figure 14-6) should appear.

15 Click and drag the red and green circles to define the frequency range in which the resonance frequency should be found. The mouse scroll wheel can be used to zoom in the plot if needed.

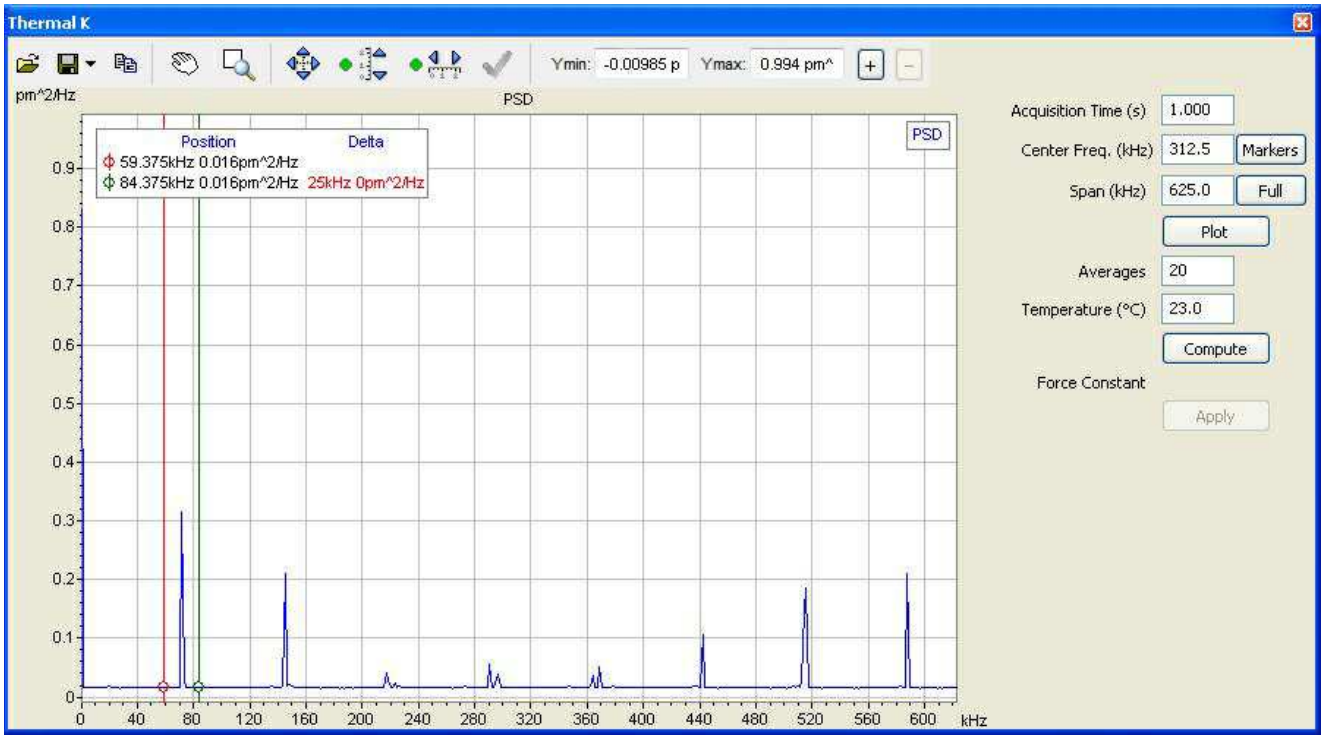

Figure 14-6 Thermal K PSD plot with markers set to define frequency range

16 Click the **Compute** button.

PicoView will acquire the selected number of scans and will average the frequencies within the selected range to complete the calculation. The Thermal K window will refresh, showing the PSD across the frequency range, and a best fit curve in red. The area under the curve is integrated

to determine the Thermal K value, which will be displayed as the Force Constant.

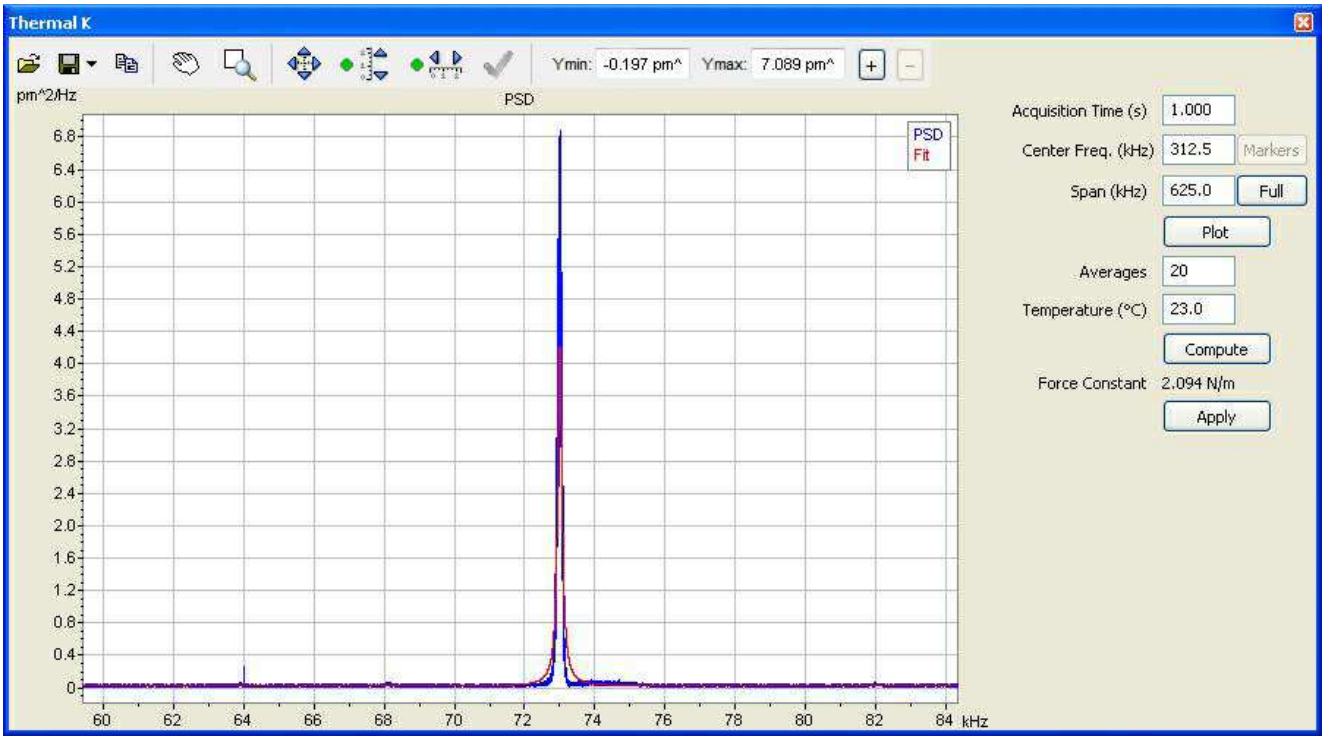

Figure 14-7 Thermal K window showing best fit curve in red for Thermal K calculation

17 Click the **Compute** button again.

As you accumulate more scans, the Thermal K calcuation should converge to a value. Using a small number of scans for this process lets you see how much the calculation is changing as you accumulate data.

18 Once the Force Constant value is stable, click the **Apply** button in the Thermal K window.

The Force Constant should now be updated in the Spectroscopy Advanced tab.

Change the plot window to display Force units if needed:

19 In the Spectroscopy plot window, click on the units and select nN from the drop-down menu as shown in Figure 14-8:

Figure 14-8 Select units after calibrating the Force Constant

Once the units have been selected, measurements can be taken on subsequent force curves as shown in Figure 14-9:

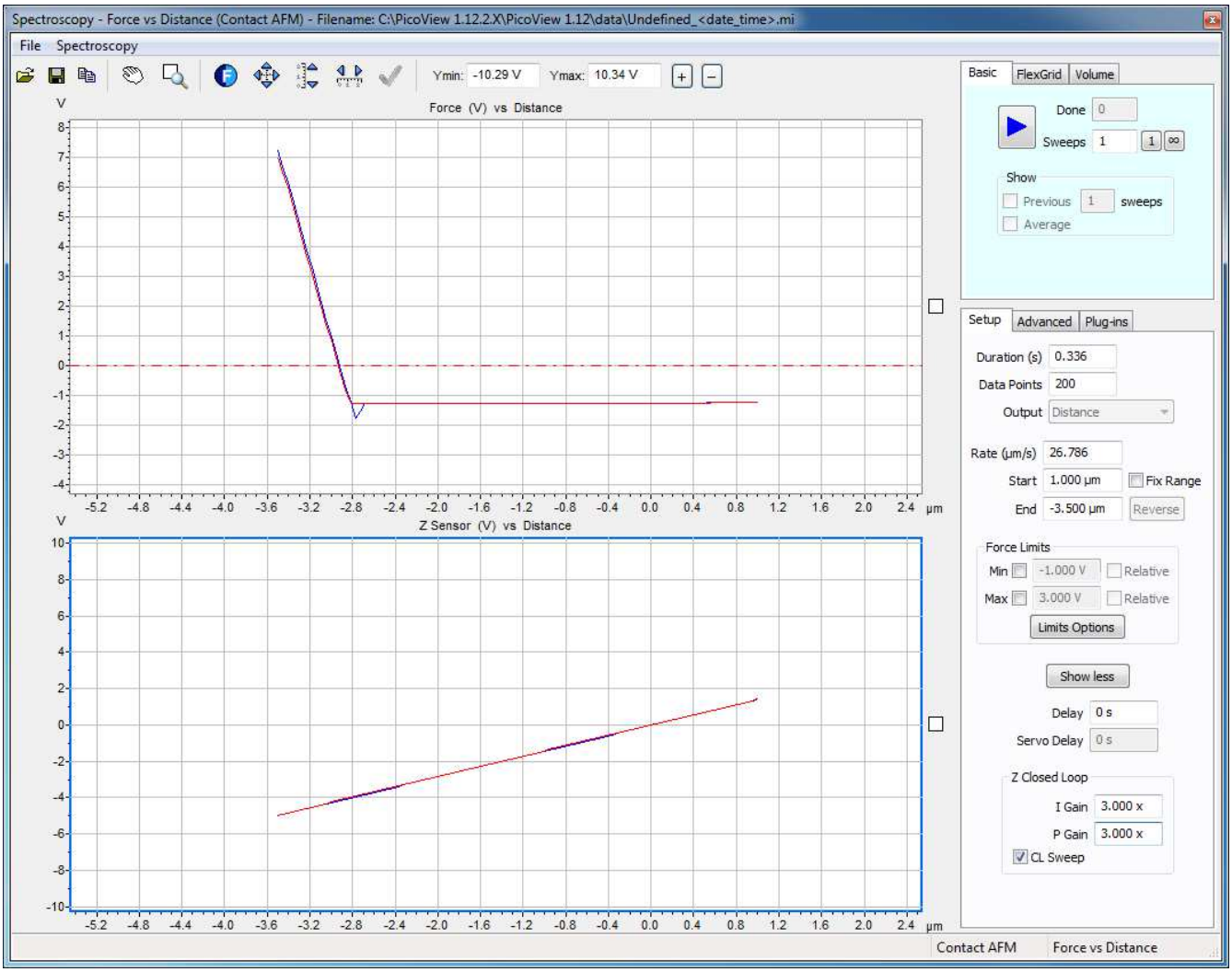

Figure 14-9 Force curve with calibrated measurements