Improving the Accuracy of AFM Force Measurements: *The Thermal Tune Solution to the Cantilever Spring Constant Problem*

By: F. M. Serry

Introduction

The atomic force microscope (AFM) is a sensitive force measurement instrument. But sensitivity and measurement accuracy are two different issues. This application note describes a solution for improving the accuracy of AFM force measurements through more precise calibration of the AFM cantilever spring constant. There are several well-documented methods for calibrating the spring constant, but the one described here offers not only improved precision, but also ease of use and speed, because the procedure is straightforward, and relies only on the AFM's own hardware and software.

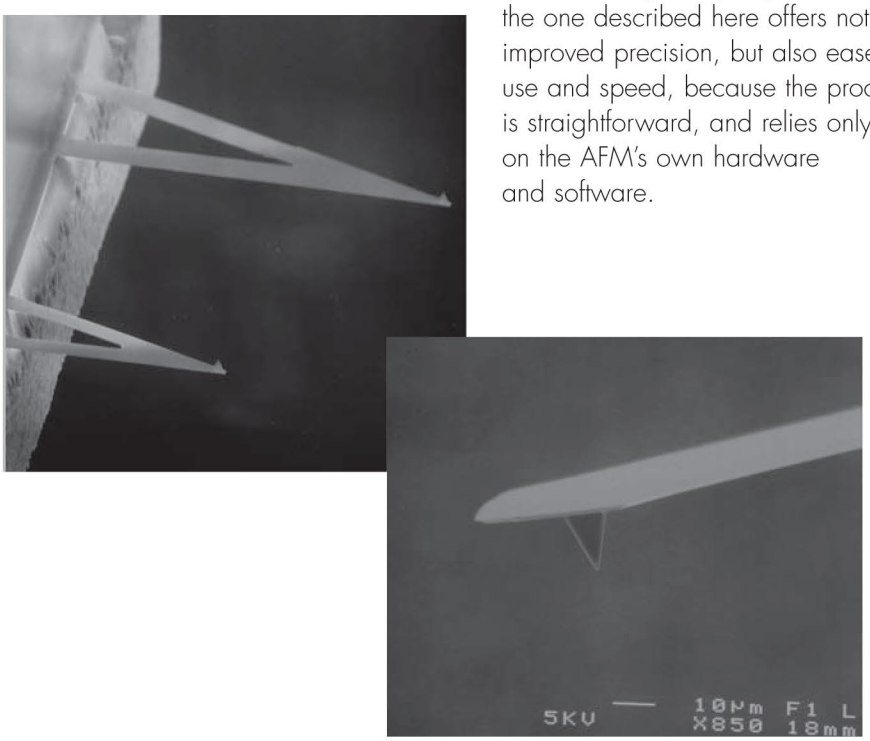


Figure 1. The AFM probe, a micro-fabricated cantilever, is typically made of silicon nitride (top, left) or single-crystal silicon (bottom, right). The sharp tip (with end radius often less than 5nm) is integrated into the free end of the cantilever.

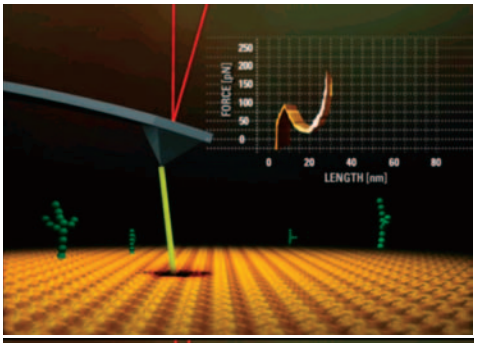
The Sensitivity of the AFM for Force Measurements

The typical Atomic Force Microscope (AFM) probe is a micro-fabricated cantilever with a sharp tip integrated into its free end (Figure 1). The AFM can directly probe a given location on the sample surface with the tip of this probe, which can sense the surface by either touching it, or by detecting long-range forces without coming in direct contact with the surface. Measuring tip-sample forces at a given location on the sample surface is usually called force spectroscopy (see sidebar on next page.)

The force sensitivity of the AFM probe is in part related to the cantilever's stiffness, which is commonly quantified in the parameter called the spring constant, k. As the sample and the tip interact, the force F on the cantilever, at the position of the tip, is related to the distance z that the cantilever's free end bends up or down from its equilibrium position. In the context of the well-known *Hooke's* law, we have

$$F = - k.z.$$

This is a simple description that, if we consider only the cantilever geometry, is most accurately descriptive of the real situation when the cantilever deflection is small compared with the cantilever thickness, and that thickness is small compared with the cantilever's length.



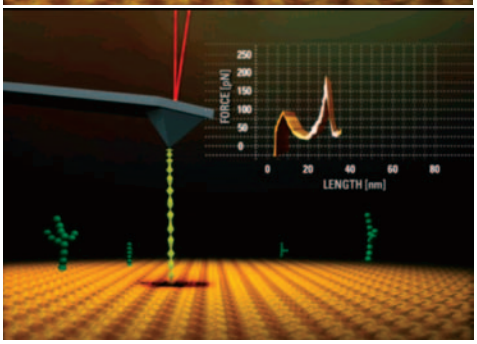


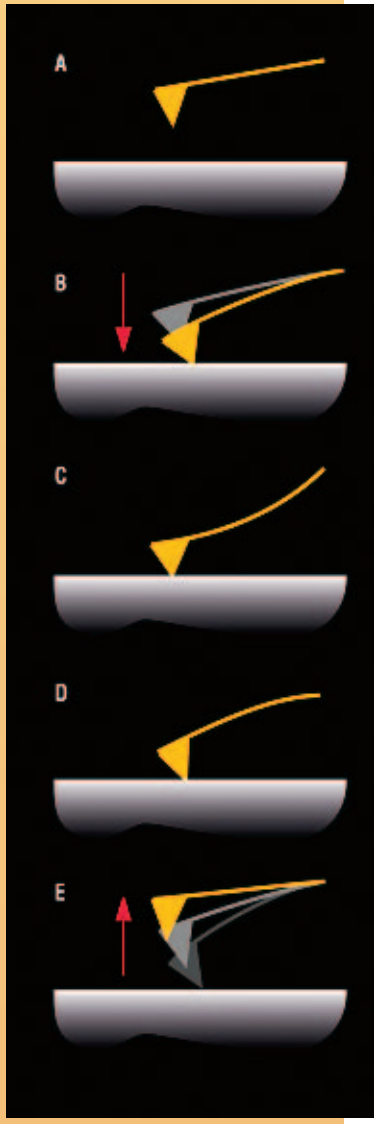
Figure 2. AFM force spectroscopy on a macromolecule. Pico-Newton range measurements are relevant to intermolecular and intra-molecular forces. These measurements are made more accurate with a better estimate of the value of cantilever spring constant.

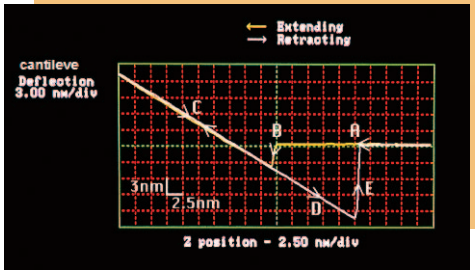
With advances in nanofabrication, it is now possible to make AFM cantilevers with extremely small spring constants. Cantilevers with spring constants smaller than 10^{-4} N/m have been made and used for AFM-based force measurements in the attonewton regime (10^{-18} N) in research laboratories.[1,2] Cantilevers with spring constants as low as 10^{-2} N/m are readily available from numerous commercial manufacturers. This, together with the AFM's capability to measure sub-nanometer cantilever deflection, enables force sensitivities on the order of 100pN (10^{-10} N) routine on many commercially available AFMs. Piconewton sensitivity (10^{-12} N) is available on more advanced AFMs such as the MultiMode PicoForce and the NanoMan II system from Veeco Instruments.

Force Spectroscopy with AFM

The distance is changed between the AFM probe and the sample, while the cantilever's position (deflection in z) is detected and recorded. The deflection is a measure of the force between the sample surface and the probe's sharp tip. From the deflection data, we can extract the force data. This force may be a result of direct contact between sample surface and tip, or it may be mediated through mechanical contact of a molecule or other object that is attached at one end to the sample surface and at the other end to the probe tip. It may also be mediated through a force field, e.g., electric, magnetic. The operational details of the AFM force measurement are largely the same across these applications, and this is one reason that makes the AFM a uniquely useful tool for force measurements. The force data provides information about the sample, and sometimes about the probe itself.

*(Right) The AFM changes the separation between the probe and sample surface, either by "Extending" the sample towards the probe, or by "Extending" the probe towards the samples at the position of the base of the cantilever. At the same time, the AFM detects and plots the motion (usually at or near the free end) of the cantilever. In the graph shown here (below), the cantilever's vertical (or up down) deflection is plotted ("Deflection"). As the tip nears the sample surface but is too far to sense any force from the sample surface, the cantilever remains undeflected from its equilibrium position. This corresponds to "A" in the plot and the drawings. The tip jumps into contact with the sample surface when it is near enough to feel attractive forces, bending the cantilever slightly **down** in the process (B). The cantilever bends **up** as the AFM continues to move the sample (or the cantilever) and pushes the tip further against the sample surface (C), and bends **down** again as the AFM pulls the cantilever and the sample apart ("Retracting") but the tip is adhering to the sample (D). Finally the tip snaps free of the surface (E) and the cantilever returns to its equilibrium position (A). The cycle may repeat.*





The Force Measurement Accuracy Problem

The accuracy of force measurements depends in part on the sensitivity of the AFM, but also in part on the accuracy of the knowledge we have of the spring constant. The value of the spring constant is most sensitive to the variation of the cantilever thickness, which is also the most difficult to control in fabrication. The spring constant of an AFM cantilever is usually estimated by the manufacturer, and noted on the documentation that accompanies the probe. This estimate is usually quite gross (though good enough for many applications).

Increasingly, however, the AFM is being used in force spectroscopy applications that demand more *accurate* measurements. These are measurements of the strength and polarity (attractive or repulsive) of the force that the cantilever bears interacting with the sample surface, in a gas, or in a liquid. The force may be related, for example, to the binding strength between different parts of a macromolecule on the sample surface (Figure 2), or between a molecule *and* the sample surface, or between a cell receptor molecule and a target molecule. The force may also be related to the mechanical properties (e.g., compliance) of a hard or soft sample, or of a thin film deposited on a substrate (for example in nano-indentation experiments). More accurate force spectroscopy requires better estimates of the spring constant.

The Thermal Tune Solution

One method to arrive at a more accurate estimate of the spring constant involves measuring the cantilever's mechanical response to thermal noise. This is the cantilever's motion in response to thermal agitations, including from the Brownian motion of the molecules of the encompassing fluid. That fluid is usually ambient air or water. This method offers a combination of speed and ease-of-use, and is now widely accepted and adopted by AFM users worldwide.

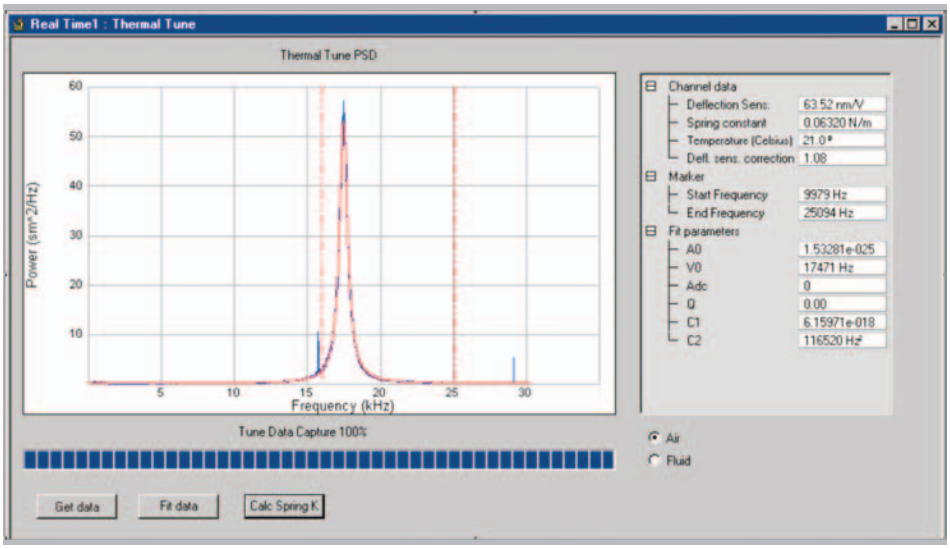


Figure 3. Thermal tune data (power spectral density) extracted from time-series measurements and plotted (blue) for an AFM cantilever in air, and a Lorentzian line shape fit (red). Veeco's NanoScope software includes numerous options and user-adjustable parameters, for example for temperature, for model-based corrections (e.g., deflection sensitivity correction), and for excluding spurious data from the fit.

In the *thermal tune method*, as it is commonly called, the AFM hardware measures the cantilever's fluctuations as a function of time, and from the time-domain measurement, it extracts the frequency spectrum of the cantilever's mechanical response (proportional to the power spectral density, PSD). By fitting the frequency spectrum to a Lorentzian line shape (Figure 3), the AFM software arrives at an estimate of the cantilever's spring constant.

The method assumes the cantilever has a single degree of freedom, and makes use of the energy equipartition theorem. This theorem relates the temperature to the cantilever's average fluctuation energy, which is found through integrating the Lorentzian fit.[3,4]

With the cantilever's spring constant estimated more accurately using the thermal tune method, plots of cantilever deflection (see sidebar page 2) are converted to equally more accurate force spectroscopy plots, which in turn improve the reliability of the force estimates. Not only that, but this conversion is done in real-time; that is, the data is now displayed, and from the outset recorded, as a plot of force-versus-distance (as opposed to cantilever deflection-versus-distance.)

The thermal tune solution is available to work with several Veeco Instruments NanoScope controllers, including NanoScope IIIa, IV, and IVa, and several SPMs, including the MultiMode PicoForce, the NanoMan II nano-manipulation and nanolithography system, and the BioScope II. The thermal tune package includes numerous software options, including provisions for distinguishing between air and liquid, for changing the temperature value used in the computations, and for improving the accuracy of the computations even further by accounting for correction factors related to assumptions that go

into the theory.[5] Much of the computation is automated, and the measurement and computation times are quite short: less than a minute per cycle of real-time measurement and spring constant calibration.

Summary

Thermal tuning of AFM cantilevers is a technique for determining the value of the cantilever's spring constant with better accuracy than is usually provided from the manufacturer. Once this value is better known, it can be used to estimate more accurately the forces between the sample and the AFM tip. The thermal tune method works in gases and in liquids, and enables more accurate and better repeatable high-resolution force spectroscopy experiments, such as in molecular pulling and nano-indentation.

[1] H. J. Mamin and D Ruger, "Sub-attonewton force detection at millikelvin temperatures," in *Applied Physics Letters*, Vol. 79, No. 20, 12 Nov 2001, pp 3358-3360.

[2] Seppe Kuehn, Sean Garner, John Marohn, "Attonewton force detection near a surface," in the Proceedings of the American Physical Society 2005 March Meeting, Session J20: Surfaces: Novel Instrumentation and Techniques.

[3] For more on the theory, please see the article by Jeffrey L. Hutter and John Bechhoefer: "Calibration of Atomic Force Microscope Tips" in *Review of Scientific Instruments*, 64(7), July 1993, page 1868.

[4] For more detail on the implementation, please see Veeco Instruments Support Notes 398 "Atomic Force Microscope Thermal Tune Adapter" and Veeco Instruments "MultiMode PicoForce Manual."

[5] One such correction accounts for the difference between a cantilever deflecting under a static force and a cantilever executing random motion at a broad spectrum of frequencies in response to thermal noise. For more information on this subject, please see the book by D. Sarid, Scanning Force Microscopy, With Applications to Electric, Magnetic, and Atomic Forces" 2nd Edition, Oxford University Press, New York 1994, pp5-6 and pp 10-11.