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# SI-traceable verification of a thermal cantilever calibration method for AFMs

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## Abstract

Knowledge of cantilever stiffness (k) is important for many atomic–force microscopy (AFM) applications. We compare a new thermal calibration method that relies on the measurement of the cantilever's resonant frequency, quality factor, and resonant amplitude to two other methods for measuring the stiffnesses of AFM cantilevers. For the geometric calibration method, the AFM cantilever's width, resonant frequency, density, and elastic modulus are used to calculate the stiffness, where only the width, length, and resonant frequency are measured. For the loading calibration method, a cantilever with an unknown stiffness is pushed against a known piezoresistive cantilever (piezolever). The known piezolever is a secondary artifact that has been calibrated by pushing against the primary artifact, an SI-traceable EFB (Electrostatic Force Balance). The loading (secondary artifact transfer), geometric, and thermal methods were found to agree within 10%, demonstrating that traceable force metrology is viable.

## Frequency Response of Thermally Driven Cantilevers

An oscillator's amplitude distribution is routinely determined by recording its deflection in time and Fourier transforming the time domain data. The probability density of the squared amplitude being in the frequency range of $\nu$ to $\nu+\Delta\nu$ is given by the well-known result [1]

$$\frac{P(\nu)}{\Delta\nu}=\frac{2}{\pi\nu_k Q\left\{\left[1-\left(\frac{\nu}{\nu_k}\right)^2\right]^2+\left(\frac{\nu}{\nu_k Q}\right)^2\right\}}, \qquad (1)$$

where: $\Delta\nu$ is the sampling interval (frequency resolution),
$\nu_k$ is the oscillator's kinetic resonant frequency, and
Q is the quality factor.

Therefore, the squared amplitude density distribution is

$$\frac{x^2(\nu)}{\Delta\nu}=\alpha^2\frac{P(\nu)}{\Delta\nu}=\frac{2\alpha^2}{\pi\nu_k Q\left\{\left[1-\left(\frac{\nu}{\nu_k}\right)^2\right]^2+\left(\frac{\nu}{\nu_k Q}\right)^2\right\}}. \qquad (2)$$

The mean-squared amplitude must be normalized to $k_BT/k$ as required by the equipartition theorem,

$$\frac{1}{2}k_BT=\frac{1}{2}k\langle x^2\rangle, \qquad (3)$$

to yield the normalization factor $\alpha^2 = k_BT/k$.

Hence, the mean-squared amplitude distribution becomes

$$\langle x^2(\nu)\rangle=\frac{k_BT}{\pi\nu_k Qk}\cdot\frac{\Delta\nu}{\left\{\left[1-\left(\frac{\nu}{\nu_k}\right)^2\right]^2+\left(\frac{\nu}{\nu_k Q}\right)^2\right\}}. \qquad (4)$$

## Thermal calibration method

Provided that $\nu_k$, Q, and $\langle x^2(\nu_k)\rangle$ can be measured, Equation (4) suggests an easy way to calculate the cantilever stiffness. At kinetic resonance, (4) simply becomes

$$k=\frac{Q}{\pi}\frac{k_BT}{\langle x^2(\nu_k)\rangle}\frac{\Delta\nu}{\nu_k}. \qquad (5)$$

For high-Q oscillators there is almost no distinguishable difference between amplitude and kinetic resonance. Therefore, experimental values at amplitude resonance can be safely substituted in Equation (5) for calculation of the cantilever stiffness.

## Instrument Calibration

### *Detector Calibration*

- Cantilever displacement is monitored by reflecting a laser beam from the backside of a cantilever.
- The movement of the laser beam on the photodiode is calibrated by interferometry (Figures 1 & 2).
- The wavelength of the laser is measured by an optical spectrum analyzer.
- The detector signal (voltage) is calibrated to the cantilever displacement by force curve acquisition yielding the scaling factor $\Delta z_m/\Delta V_m$.

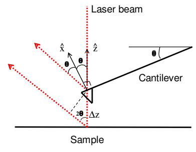

Figure 1: Cantilever Displacement Geometry. The detection laser is placed half on and half off a cantilever. Some of the light is reflected from the cantilever and some of it from a reflective sample surface. The cantilever is translated in the z-direction and an interference pattern is observed at the photodiode (Figure 2).

### *AFM Corrections*

- Cantilever angle (Figure 1)

$$\Delta x=\Delta z/\cos\theta. \qquad (6)$$

- Scanner movement (Figures 1 & 2)

$$\Delta z=\lambda/(1+\cos 2\theta). \qquad (7)$$

- "0.81744" Levy & Maaloum factor [2]

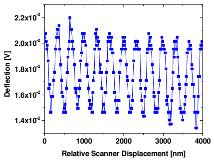

Figure 2: Force Curve Interference. The path difference ($\Delta z_p[1+\cos 2\theta]$) between the cantilever and sample surface must be made equal to the wavelength of the light ($\lambda$). The ratio of the wavelength to the path difference is the correction factor in Equation (8).

Introducing the above corrections, we relate the mean–squared amplitude fluctuations of the cantilever to the mean–squared voltage fluctuations by

$$\langle x^2(\nu)\rangle=0.81744\langle\Delta V^2(\nu)\rangle\left[\frac{\Delta z_m}{\Delta V_m}\right]^2\left[\frac{\lambda}{\Delta z_p(1+\cos 2\theta)}\right]^2\left[\frac{1}{\cos\theta}\right]^2. \qquad (8)$$

## Cantilever Calibration

We fitted the resonant peaks to the response function of a simple harmonic oscillator (SHO) driven by the thermal noise (4), with added white noise and 1/f noise [3],

$$\langle x^2(\nu)\rangle=\frac{A}{\nu}+B+\frac{\langle x^2(\nu_k)\rangle}{Q^2}\cdot\frac{1}{\left\{\left[1-\left(\frac{\nu}{\nu_k}\right)^2\right]^2+\left(\frac{\nu}{\nu_k Q}\right)^2\right\}}. \qquad (9)$$

The five parameters, A, B, $\nu_k$, $\langle x^2(\nu_k)\rangle$, and Q, were obtained by a nonlinear least-squares fit; the last three are used in Equation (5).

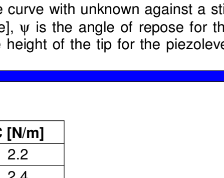

Figure 3: Sample Thermal Spectrum. A force calibration cantilever (uncoated cantilever, Q = 175, k = 2.7 N/m).

## Geometric calibration method

To calibrate using the geometric method, the resonant frequency, length, and width of an AFM cantilever with an unknown stiffness is measured. The method does not account for a reflective coating on the cantilever, which can be significant. Thus the method is most appropriate for tipless, uncoated, uniform rectangular silicon beams.

$$k_g=2w\frac{(\pi\nu_k L)^3}{\sqrt{E}}\left(\frac{33\rho}{35}\right)^{3/2}, \qquad (10)$$

where w is the width of the unknown cantilever, $\nu_k$ the resonant frequency, L the length, $\rho$ the density, and E the elastic modulus of the cantilever.

## Loading calibration method

To calibrate using the loading method, an AFM cantilever with an unknown stiffness is pushed against the secondary artifact (Figure 4). The secondary artifact is a piezolever that was calibrated with an EFB at NIST [6], [7].

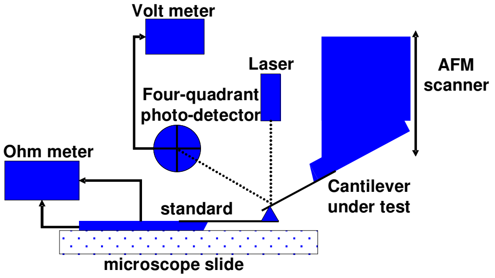

Figure 4: Secondary Artifact Transfer

The stiffness for the unknown can be calculated as follows

$$k_l=\frac{S\Delta\Omega}{\Delta V\left(\frac{\delta z}{\delta V}\right)}\left[\cos\theta\left\{\frac{L\cos\psi\text{-}h\sin\psi}{L\cos\theta+h\sin\theta}\right\}\right]. \qquad (11)$$

S is the sensitivity [nN/Ohm], $\Delta\Omega$ is the change in resistance of the piezolever [Ohm], $\Delta V$ is the change in voltage on the four-quadrant photodetector [V], $\delta z/\delta V$ is the slope of a force curve with unknown against a stiff sample [nm/V], $\theta$ is the angle of repose of the unknown cantilever [Degree], $\psi$ is the angle of repose for the piezolever [Degree], L is the length of the piezolever [Micron], and h is the height of the tip for the piezolever [Micron].

## Results

| **Cantilever** | **A [N/m]** | **B [N/m]** | **C [N/m]** |
|---|---|---|---|
| **$k_t$** | 2.7 | 2.2 | 2.2 |
| **$k_l$** | 2.9 | 2.4 | 2.4 |
| **$k_g$** | 3.2 | 2.5 | 2.3 |
| **Std. Dev.** | 0.3 | 0.1 | 0.1 |
| **Average** | 2.9 | 2.4 | 2.3 |
| **% diff.** | 9.1 | 6.0 | 4.7 |

Table 1: Stiffness Results

The AFM cantilevers in Table 1 were rectangular, tipless, uncoated, uniform, rectangular, and made of silicon.

## Conclusions

- Stiffnesses as high as 77.3 N/m have been measured with this thermal method.
- The precision was found to be 5%. The SI-traceable relative uncertainty was found to be 10%.
- The thermal method works well for uncoated cantilevers, coated cantilevers, cantilevers made of any material, and cantilevers with any type tip.

## References

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One of the authors (E. J. T.) is thankful for the financial support of Analog Devices, Inc. through a graduate fellowship.