Calibration Method for Atomic-Force Microscopy Cantilevers

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Abstract

Knowledge of cantilever stiffness (k) is important for most atomic–force microscopy (AFM) applications. We propose a new thermal calibration method that relies on the measurement of the cantilever's resonant frequency, quality factor, and resonant amplitude. Our method can be applied to all thermally driven harmonic oscillators and does not depend on the instrument or detection scheme used to acquire the data. We evaluate this method for low resonant frequency cantilevers (up to 30 KHz) using the *noise image acquisition* routine. We also extend it to higher resonant frequency cantilevers (up to 200 KHz) using an alternative *oscilloscope based* method to acquire the corresponding noise spectrum.

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 ν to $\nu+\Delta\nu$ is given by the well known result [1]

$$\frac{P(\nu)}{\Delta\nu} = \frac{4}{\omega_k Q\left\{\left[1-\left(\frac{\nu}{\nu_k}\right)^2\right]^2 + \left(\frac{\nu}{\nu_k Q}\right)^2\right\}}, \qquad (1)$$

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

Therefore, the *squared amplitude* density distribution is

$$\frac{x^2(\nu)}{\Delta\nu} = \alpha^2\frac{P(\nu)}{\Delta\nu} = \frac{4\alpha^2}{\omega_k Q\left\{\left[1-\left(\frac{\nu}{\nu_k}\right)^2\right]^2 + \left(\frac{\nu}{\nu_k Q}\right)^2\right\}}. \qquad (2)$$

The *mean-square 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-square amplitude* distribution becomes

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

Cantilever Stiffness

Provided that ν_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

$$k = \frac{1}{\pi Q}\frac{k_BT}{\langle x^2(\nu_k)\rangle}\frac{\Delta\nu}{\nu_k}. \qquad (5)$$

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 detector signal (voltage) is calibrated to the cantilever displacement by force curve acquisition yielding the scaling factor $\Delta z_m/\Delta v_m$.

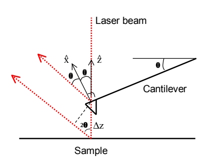

Fig.1: Cantilever Displacement Geometry

Calibration Corrections

- Cantilever angle (Fig.1)

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

- Scanner movement (Figs.1 & 2)

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

- "3/4" Butt & Jaschke factor [2]

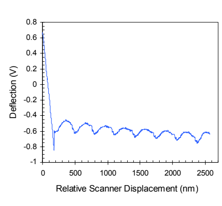

Fig.2: Force Curve Interference

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

$$\langle x^2(\nu)\rangle = \frac{3}{4}\langle \Delta V^2(\nu)\rangle\left[\frac{\Delta z_m}{\Delta V_m}\right]^2\left[\frac{\lambda}{\Delta z_p(1+\cos 2\theta)}\right]^2\left[\frac{1}{\cos\theta}\right]^2. \qquad (8)$$

Data Acquisition and Processing

Digital Instruments Multimode AFM

- Error–signal images acquired in "contact mode" with zero scan range and zero gains
- Thermal noise spectrum obtained by FFT algorithm
  - No. of points: 256
  - Resolution: 122 Hz
  - Bandwidth: 31 KHz - limited by the scan speed

TMMicroscopes AutoProbe M5 AFM

- A-B signal directly monitored with a digitizing oscilloscope
- Thermal noise spectrum obtained by FFT algorithm
  - No. of points: 4096
  - Resolution: 49 Hz
  - Bandwidth: 200 KHz - limited by M5 cutoff frequency (Fig.3)

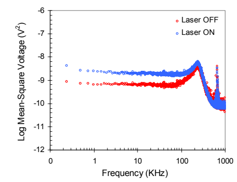

Fig.3: M5 Electronics Noise Spectrum

Cantilever Calibration

We fitted the resonant peaks to the response function of a simple harmonic oscillator (SHO) driven by the thermal noise (4), with an added white floor 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, ν_k, $\langle x^2(\nu_k)\rangle$, and Q, were obtained by a nonlinear least-squares fit, the last three being used in equation (5). Figs.4–6 show the spectra and fits for various cantilevers in different media.

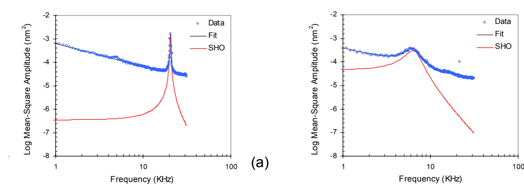

Fig.4: Uncoated FC2 cantilever
(a) air spectrum
Q = 66.6, k=0.3282 N/m
(b) water spectrum
Q = 2.5, k = 0.1806 N/m
(Multimode AFM)

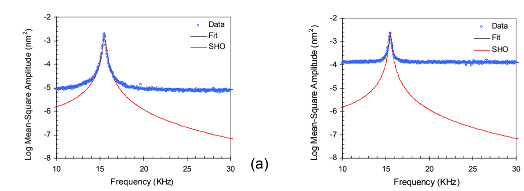

Fig.5: Coated NSC12E cantilever
(a) coated side spectrum
Q = 63.4, k = 0.1253 N/m
(b) uncoated side spectrum
Q = 63.8, k = 0.1231 N/m
(AutoProbe M5 AFM)

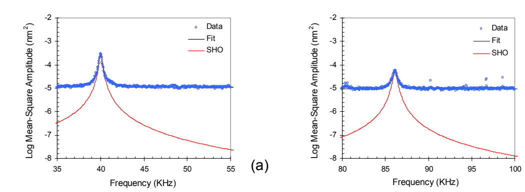

Fig.6: (a) Uncoated NSC12F cantilever
Q = 123.3, k = 0.7015 N/m
(b) Uncoated FC4 cantilever
Q = 174.8, k = 2.7378 N/m
(AutoProbe M5 AFM)

Conclusions

- We have demonstrated a new thermal method of calibration based on SHO theory that relies on the measurement of the cantilever's resonant frequency, quality factor, and resonant amplitude .
- For well defined rectangular Si cantilevers (Figs.4 & 6), our method agreed within 2% of Hutter & Bechhoefer's thermal approach [4] and 5% of Cleveland's geometric method [5].
- For Si_3N_4 or coated Si cantilevers, the agreement between the new method and Cleveland's method is worse, most likely due to the latter's dependence on cantilever geometry and material properties.
- Our measured stiffness for coated and uncoated sides of the same cantilever (Fig.5) agreed within 5%, independent of the coating reflectivity.
- Unlike Hutter & Bechhoefer's method which requires the square amplitude be summed over a large frequency range, our approach can be applied to resonant frequencies occurring near the edge of the bandwidth.
- When resonant peaks are well above the noise our method can be equally applied to low and high resonant frequency cantilevers without any noise subtraction.

References

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[4] J.L. Hutter and J. Bechhoefer. *Rev. Sci. Instrum.*, **64**, 1868-73, 1993
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