Experimental/Analytical Evaluation of the Effect of Tip Mass on Atomic Force Microscope Calibration

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February, 2008
Atomic Force Microscopy

- **AFM**: A mechanical detection system for studying materials at the nanoscale.
  - Developed in 1986 by Binnig, Quate, and Gerber in a collaboration between IBM and Stanford University

- **Laser based detection system**:
  - Sub nanometer displacement resolution.
  - Sub nano-Newton force resolution.

Schematic: R. Carpick
AFM can produce quantitative topology (x,y,z coordinates)

Versatile: Images of topography, material stiffness and viscoelasticity, etc…

Slide courtesy of VEECO
How does one calibrate the world’s smallest force sensor?

Calibration procedures approximate the probe as an Euler Bernoulli beam and find effective mass and stiffness from vibration measurements.
Tip mass may be 50% or more of beam’s effective mass

- Tip mass is neglected in all available AFM calibration procedures.
- How large of an effect does this have on their accuracy?
Outline

- Calibration Procedures
  - Method of Sader
  - Thermal Tune (Hutter and Beechoefer)
- Modifications to account for tip mass
- Experimental Application
  - Tip mass estimated from SEM images
  - Experimentally procedure to measure mode shapes and frequencies
  - Comparison with analytical models
- Conclusions
Calibration: Method of Sader

- Measure:
  - Natural frequency & **damping ratio**
  - AFM probe’s in-plane dimensions (optical image)
  - Density & viscosity of air

- Solve fluid-structure interaction problem to obtain:
  - **area density** & **stiffness** of the AFM probe.

- This is one of the most convenient calibration procedures available and is widely used by AFM users and probe manufacturers.

- Sader’s method assumes beam with rectangular cross section and constant properties along the length of the beam. Tip is not included!
Calibration: Thermal Tune

- Initially presented by Hutter and Bechhoefer (1993)

- **Measure:**
  - Power spectrum of cantilever oscillating freely under the influence of thermal excitation
  - **Temperature** of probe
  - **displacement sensitivity** of photodetector

- Equipartition theorem relates the RMS amplitude of vibration of each mode with the temperature.

- Derivation assumes beam with constant cross section and neglects the effect of the tip.
 Extensions

- Can one modify either of these methods to account for the tip mass?
- … YES!
Include Tip Mass in Method of Sader

Solution of fluid dynamic equations gives the force applied to the beam as a function of frequency:

\[ F_{\text{hydro}}(x, \omega) = \frac{\pi}{4} \rho_f \omega^2 b^2 \Gamma(\omega) W(x, \omega) \]

Include hydrodynamic force and tip-mass in single-term Ritz model for cantilever beam.

\[-\omega^2 \left( \rho_c h b L + \frac{\pi}{4} \rho_f b^2 L \Gamma_r(\omega) \right) m_{11} + m_t (\psi(x_m))^2 + \frac{I_t}{L^2} \left( \frac{d}{dx} \psi(x_m) \right)^2 \right] Y + \]

\[ i\omega \left[ \frac{\pi}{4} \rho_f \omega b^2 L \Gamma_i(\omega) m_{11} \right] Y + \left[ \frac{k_s}{3} \ k_{11} \right] Y = 0 \]

Fluid damping effect \quad Beam stiffness

\[ \psi(x) = \sin(\alpha_1 x) - \sinh(\alpha_1 x) + R_1 \left[ \cos(\alpha_1 x) - \cosh(\alpha_1 x) \right] \]

Basis function: mode function for cantilever beam
Include Tip Mass in Method of Sader (2)

- Using mode shapes of an ideal cantilever beam as basis functions:
  \[ m_{11} = \int_0^1 (\psi(x))^2 \, dx \approx 1.8556 \]
  \[ k_{11} = \int_0^1 \left( \frac{d^2}{dx^2} \psi(x) \right)^2 \, dx \approx 22.94 \]

- Invert the procedure to solve for the area density and spring constant from \( f_n \) and \( Q = 1/(2\zeta) \)

\[
\rho_c h = \frac{\pi}{4} \rho_f b \left( \frac{1}{2\zeta} \Gamma_i(\omega) - \Gamma_r(\omega) \right) - \frac{m_t}{b L m_{11}} (\psi(x_m))^2 - \frac{I_t}{b L^3 m_{11}} \left( \frac{d}{dx} \psi(x_m) \right)^2
\]

Term from Sader

\[ k_s = \frac{3\pi \rho_f b^2 L m_{11} \Gamma_i(\omega)}{4k_{11}} Q \omega_n^2 \]

Tip mass falls out of expression for \( k_s \)!
Conclusions:

- Sader’s method accurately estimates the stiffness of AFM cantilever probes even when the tip mass is ignored, so long as the mode function is accurate!
- Sader’s method overestimates the area density of the AFM probe when the tip mass is neglected.

Does the AFM probe’s tip mass alter the mode shapes of the probe significantly?
Experimental Procedure

- Operating deflection shapes of cantilever probes measured using Polytec Micro Systems Analyzer (Laser Vibrometer) at Sandia National Labs.
  - Base excited by a piezoelectric wafer.
  - Pseudo-random excitation used, centered on each mode sequentially.
  - Mode shapes measured both in vacuum and at ambient pressure.
Tip Mass Estimation

- Tip volume estimated from SEM images:
  - 1633 μm³
- Nominal beam volume:
  - 350μm × 35 μm × 1μm = 12250 μm³
- Significant? If the densities are the same:
  - Tip mass is 13% of beam mass.
  - Tip mass is 54% of the effective mass of the beam! (Effective mass of beam is 0.25*m_{beam})
**Experimental Mode Shapes**

- 1st experimental mode is almost identical to analytical shape for a cantilever without a tip mass.
- Experimentally measured mode shapes are significantly different from the analytical shapes for modes 2-4.
- Tip motion is reduced as one would expect due to the added mass.
**Tuned Analytical Model**

- Ten-term Ritz series model created of AFM cantilever including tip mass.
- Tip mass adjusted until the first three freqs measured in vacuum agreed closely.

<table>
<thead>
<tr>
<th>Mode #</th>
<th>Exp. (kHz)</th>
<th>Model (kHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.07</td>
<td>9.07</td>
</tr>
<tr>
<td>2</td>
<td>70.8</td>
<td>70.5</td>
</tr>
<tr>
<td>3</td>
<td>213.8</td>
<td>210.3</td>
</tr>
<tr>
<td>4</td>
<td>439.8</td>
<td>419.8</td>
</tr>
</tbody>
</table>
An Observation

- SEM Images show that the cantilever is significantly thicker than its specification near the tip and thinner near the root.

- Nominal Thickness: 1 μm
### Estimated Calibration Errors

<table>
<thead>
<tr>
<th></th>
<th>Sader (no tip)</th>
<th>Modified Sader (nominal tip)</th>
<th>Percent Difference (nominal tip)</th>
<th>Modified Sader (tuned tip)</th>
<th>Percent Difference (tuned tip)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$k_s$</td>
<td>0.0424 N/m</td>
<td>0.0424 N/m</td>
<td>0 %</td>
<td>0.0424 N/m</td>
<td>0 %</td>
</tr>
<tr>
<td>$\rho_c h$</td>
<td>4.39 g/m²</td>
<td>3.28 g/m²</td>
<td>34%</td>
<td>1.90 g/m²</td>
<td>130%</td>
</tr>
</tbody>
</table>

- Method of Sader overestimates area density significantly
- $k_s$ above assumes mode shapes are unchanged
- Based on tuned analytical model, error in stiffness calibration due to mismatch in the mode shapes is:
  - 0.4%, 13% & 6% for the 1$^{st}$, 2$^{nd}$ & 3$^{rd}$ Modes respectively.
Other Implications

- Higher modes of vibration can cause internal resonance when scanning, which may distort the results.
- This has also been exploited (Crittenden, Raman, Reifenberger) to improve image contrast.
- Yamanaka et al. image with higher harmonics directly to obtain deeper penetration into the sample.
- In either case tip mass should not be neglected!
Conclusions

- Tip mass is a significant portion of the total effective mass of some common commercial AFM probes.
  - Tip changes the mode shapes and frequencies of the 2\textsuperscript{nd} and higher modes resulting in significant calibration errors if these modes are utilized.
  - 1\textsuperscript{st} mode is almost unaffected, so the cantilever stiffness can be accurately estimated using this mode with either the Thermal Tune method or the Method of Sader.
  - Area density is not accurately estimated unless the tip mass is accounted for.