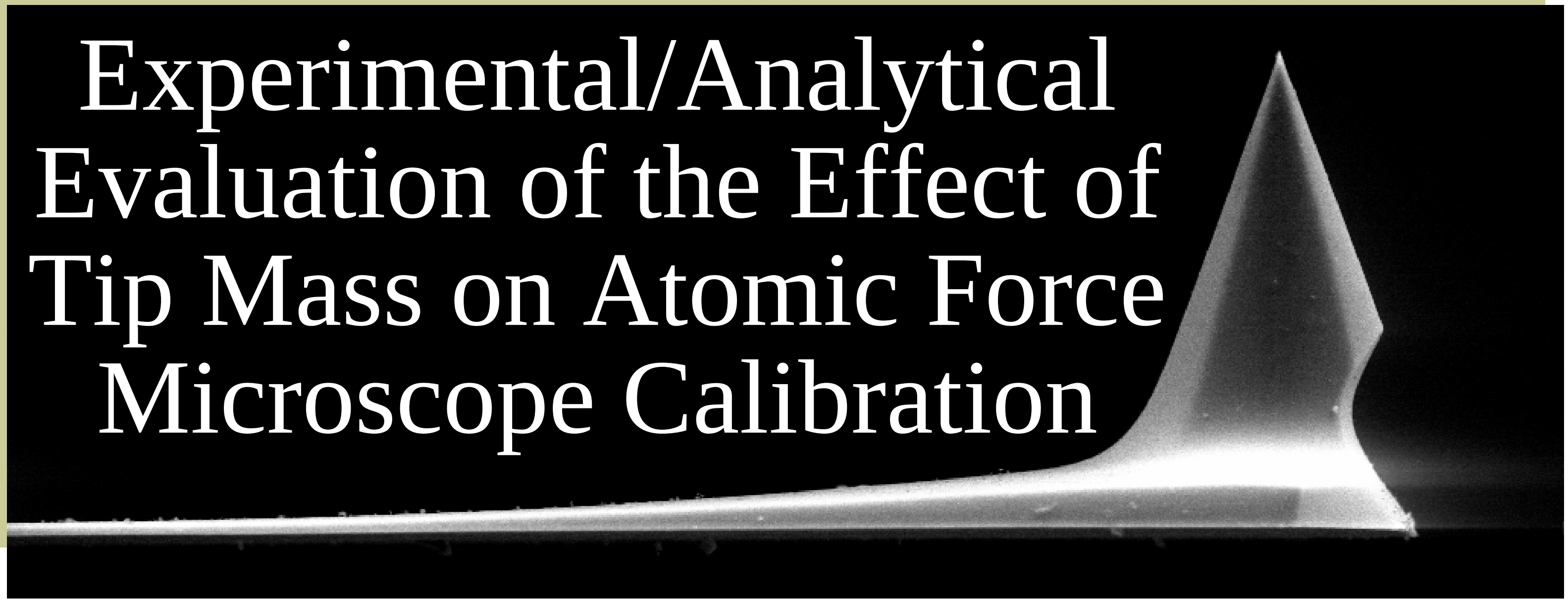
Experimental/Analytical Evaluation of the Effect of Tip Mass on Atomic Force Microscope Calibration



Scanning electron microscope image of AFM cantilever and probe tip

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February, 2008

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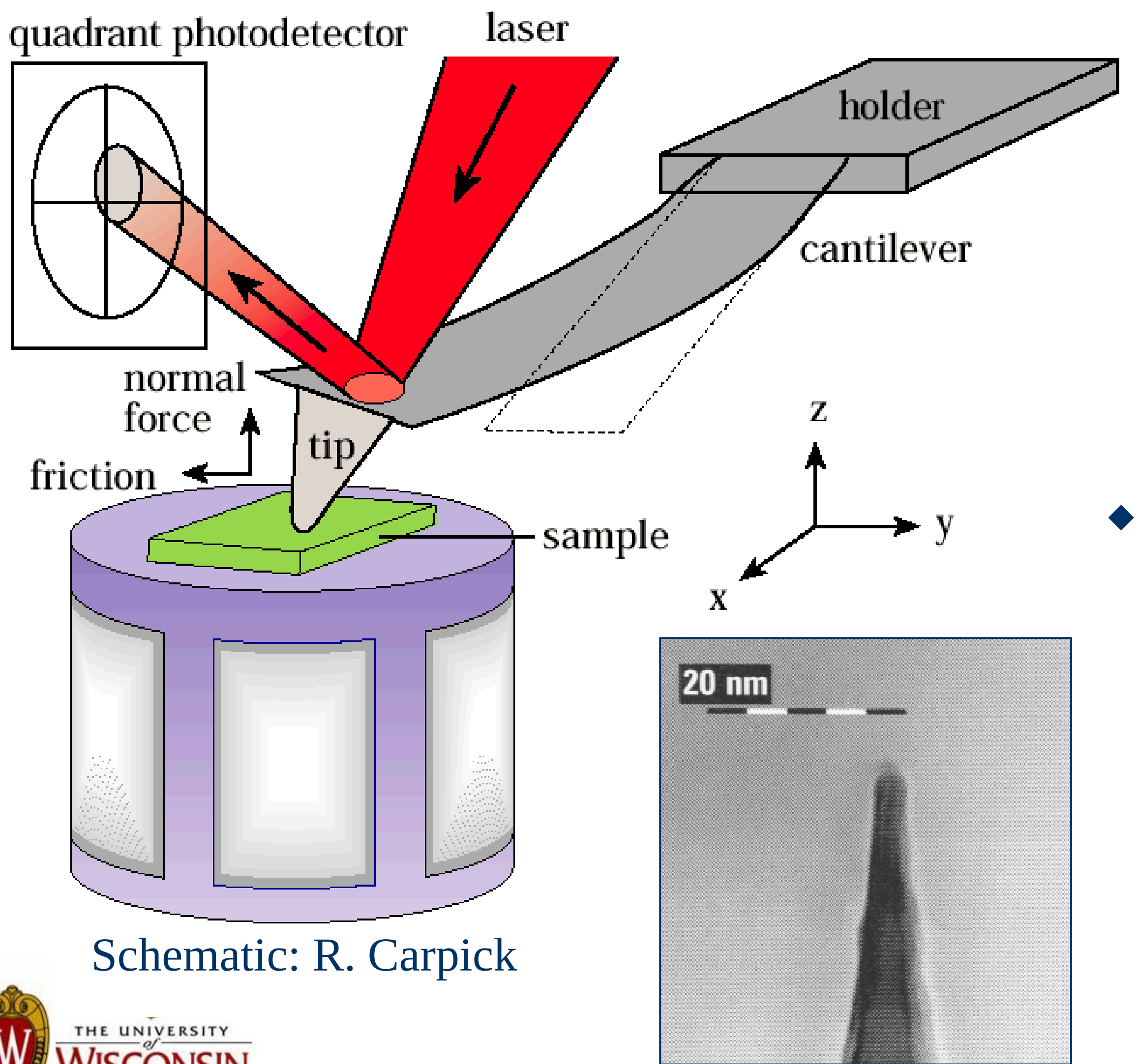
Atomic Force Microscopy

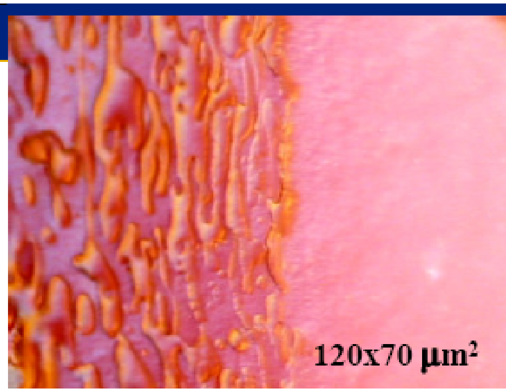


Schematic: R. Carpick

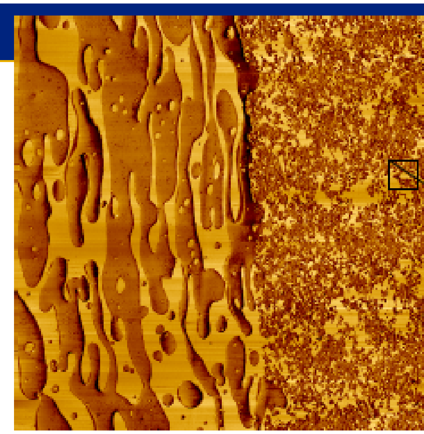
- AFM: A <u>mechanical</u> detection system for studying materials at the nanoscale.
  - Developed in 1986 by Binnig, Quate, and Gerber in a collaboration between IBM and Stanford University
- Laser based detection system:
  - Sub nanometer displacement resolution.
  - Sub nano-Newton force resolution.

Optical & AFM Images

Optical Image

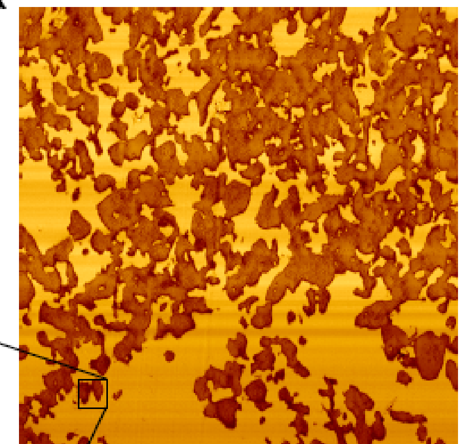


AFM Images

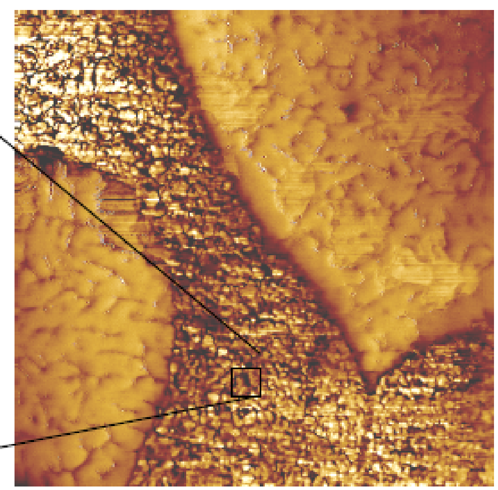


80x80 μm^2

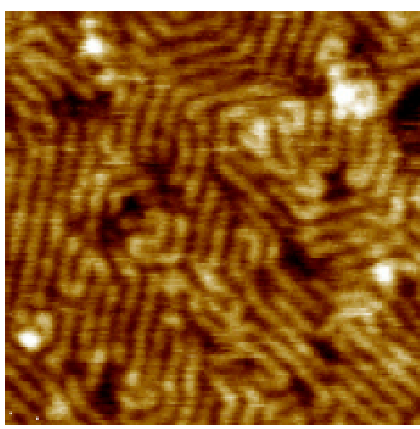
- AFM can produce quantitative topology (x,y,z coordinates)



15x15 μm^2



2x2μm^2



Molecular structure

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

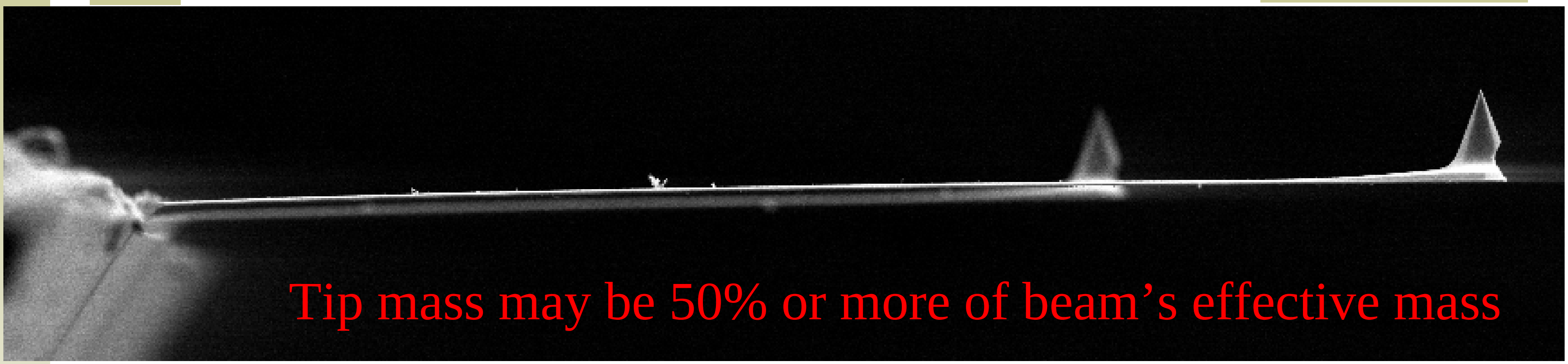
Slide courtesy of VEECO

Calibration

- How does one calibrate the world's smallest force sensor?

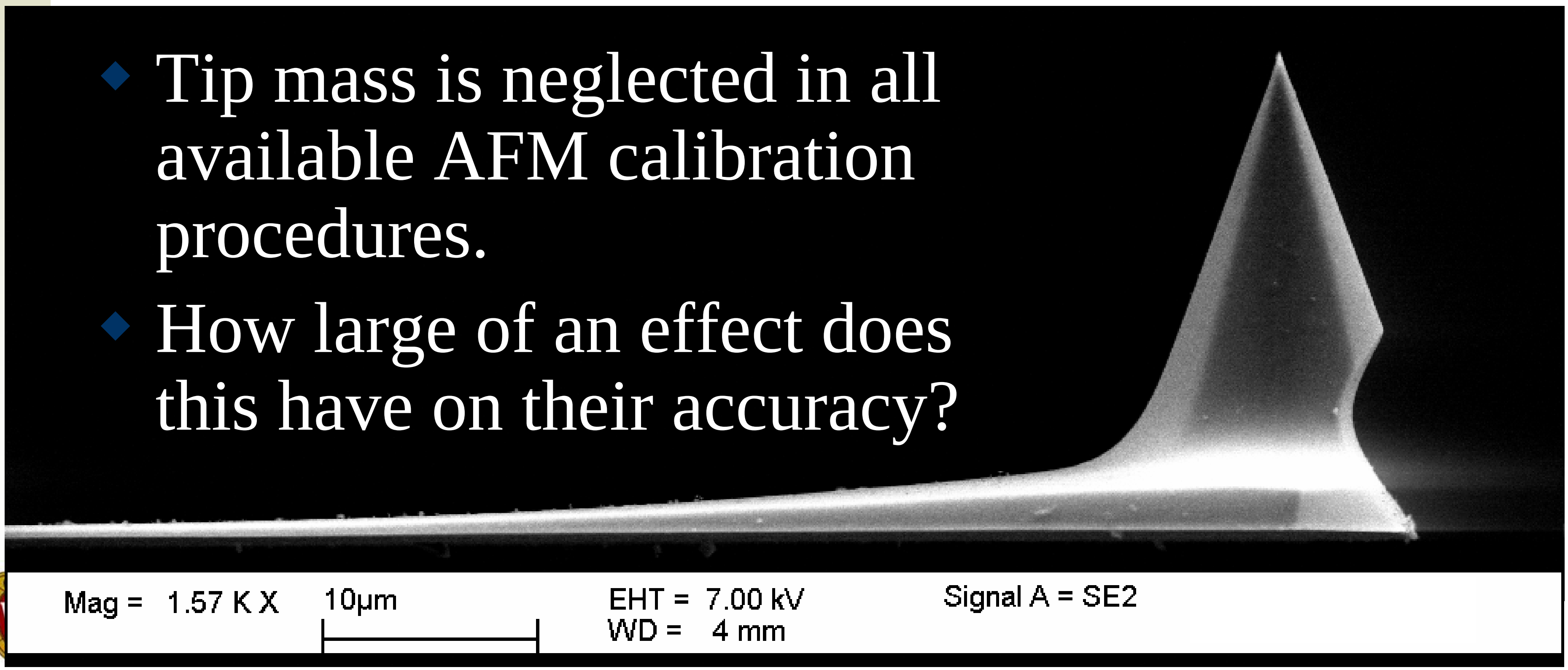
  - Calibration procedures approximate the probe as an Euler Bernoulli beam and find effective mass and stiffness from vibration measurements.

Problem



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

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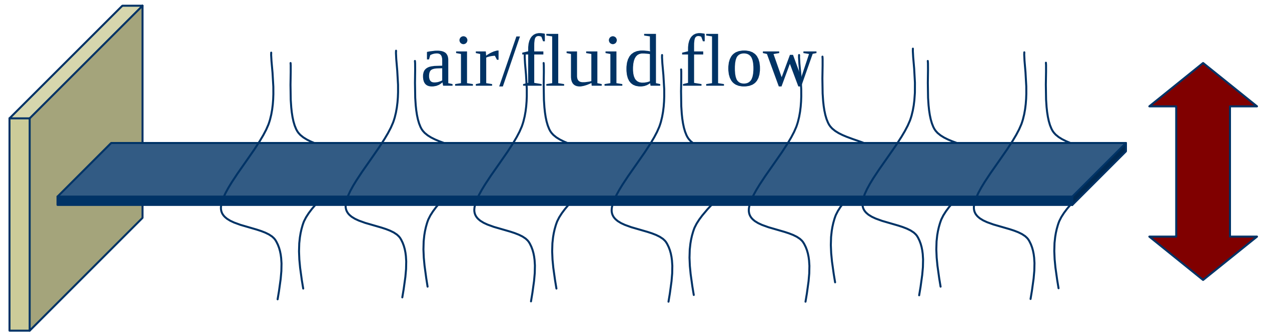
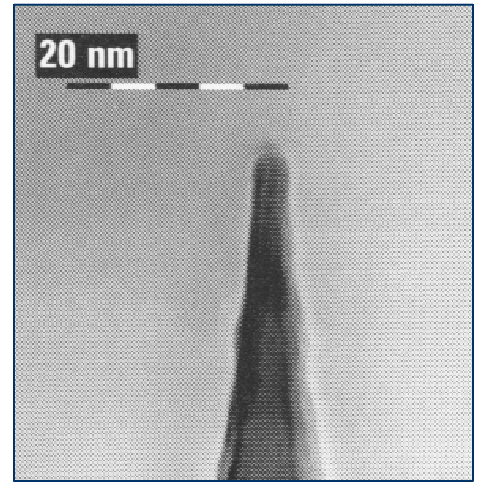
Calibration: Method of Sader

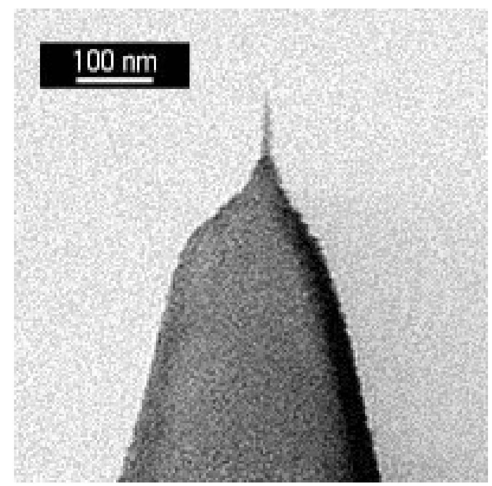


- Measure:
  - **Natural frequency** & **damping ratio**
  - AFM probe's in-plane dimensions (optical image)
  - Density & viscosity of air
- Solve fluid-structure interaction problem to obtain:
  - **area density** & **stiffness** of the AFM probe.
- This is one of the most convenient calibration procedures 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.





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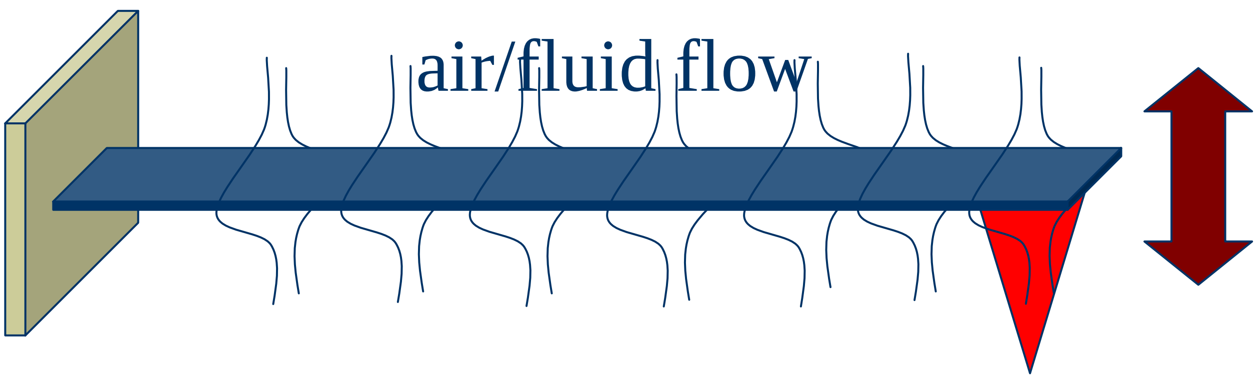
Extensions

- 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:

$$F_{hydro}\left(x,\omega\right)=\frac{\pi}{4}\rho_f\omega^2 b^2\Gamma\left(\omega\right)W\left(x,\omega\right)$$

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

beam mass fluid mass tip mass tip inertia

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

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

fluid damping effect beam stiffness

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

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:

$$m_{11} = \int_0^1 \left(\psi\left(x\right)\right)^2 dx \approx 1.8556$$

$$k_{11} = \int_0^1 \left(\frac{d^2}{dx^2}\psi\left(x\right)\right)^2 dx \approx 22.94$$

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

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

term from Sader

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

Tip mass falls out of expression for k_s!

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Include Tip Mass in Method of Sader (3)

- 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?

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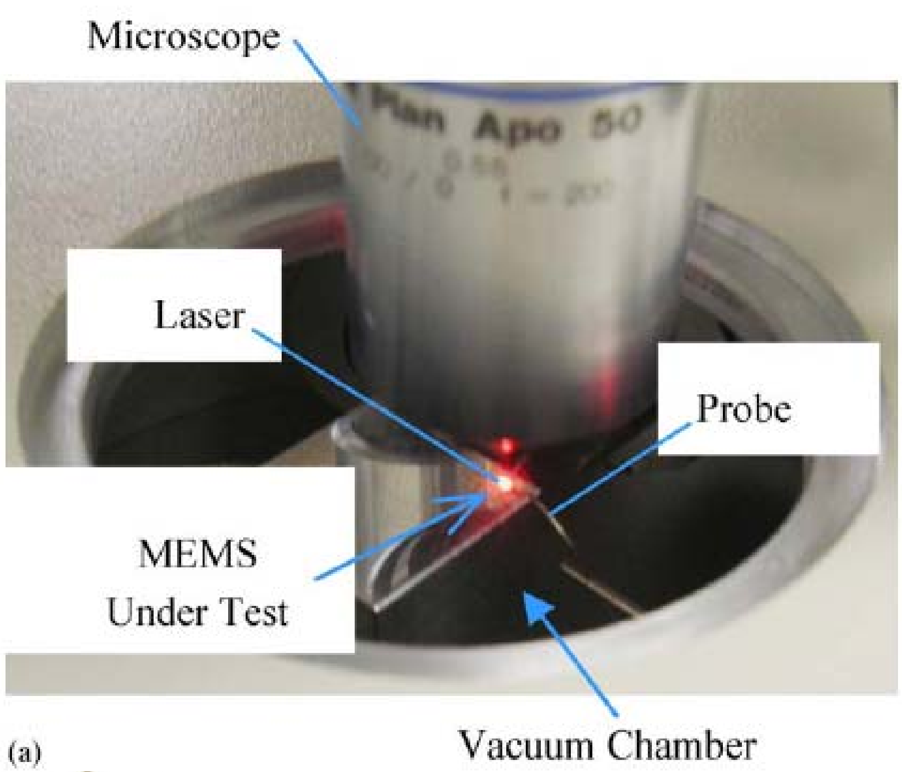
Experimental Procedure

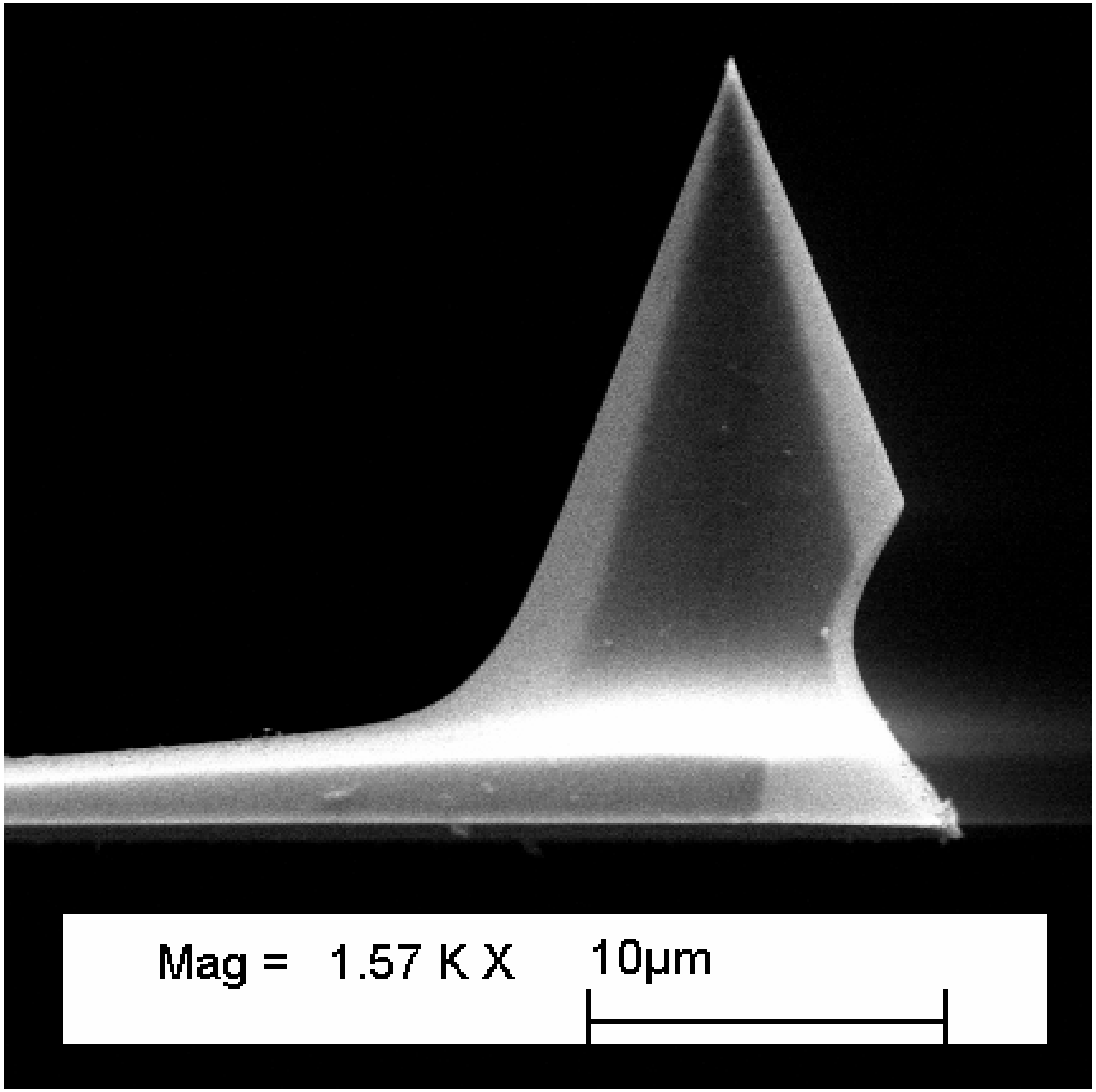
- Operating deflection shapes of cantilever probes measured using Polytec Micro Systems Analyzer (Laser Vibrometer) at Sandia National Labs.



- 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 μm^3
- Nominal beam volume:
  - $350\mu m \times 35\ \mu m \times 1\mu m = 12250\ \mu m^3$
- 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 $0.25*m_{beam}$)



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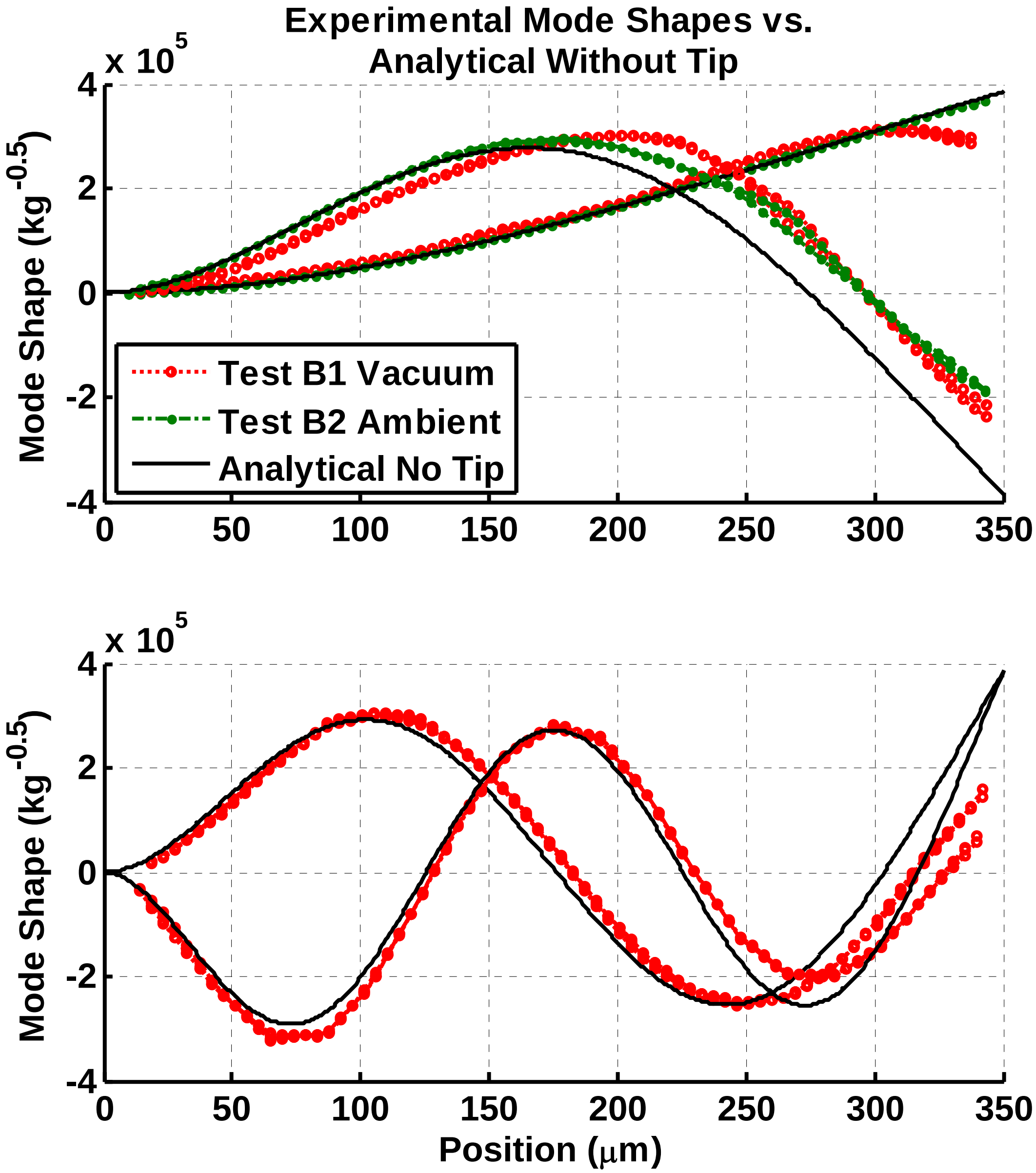
Experimental Mode Shapes

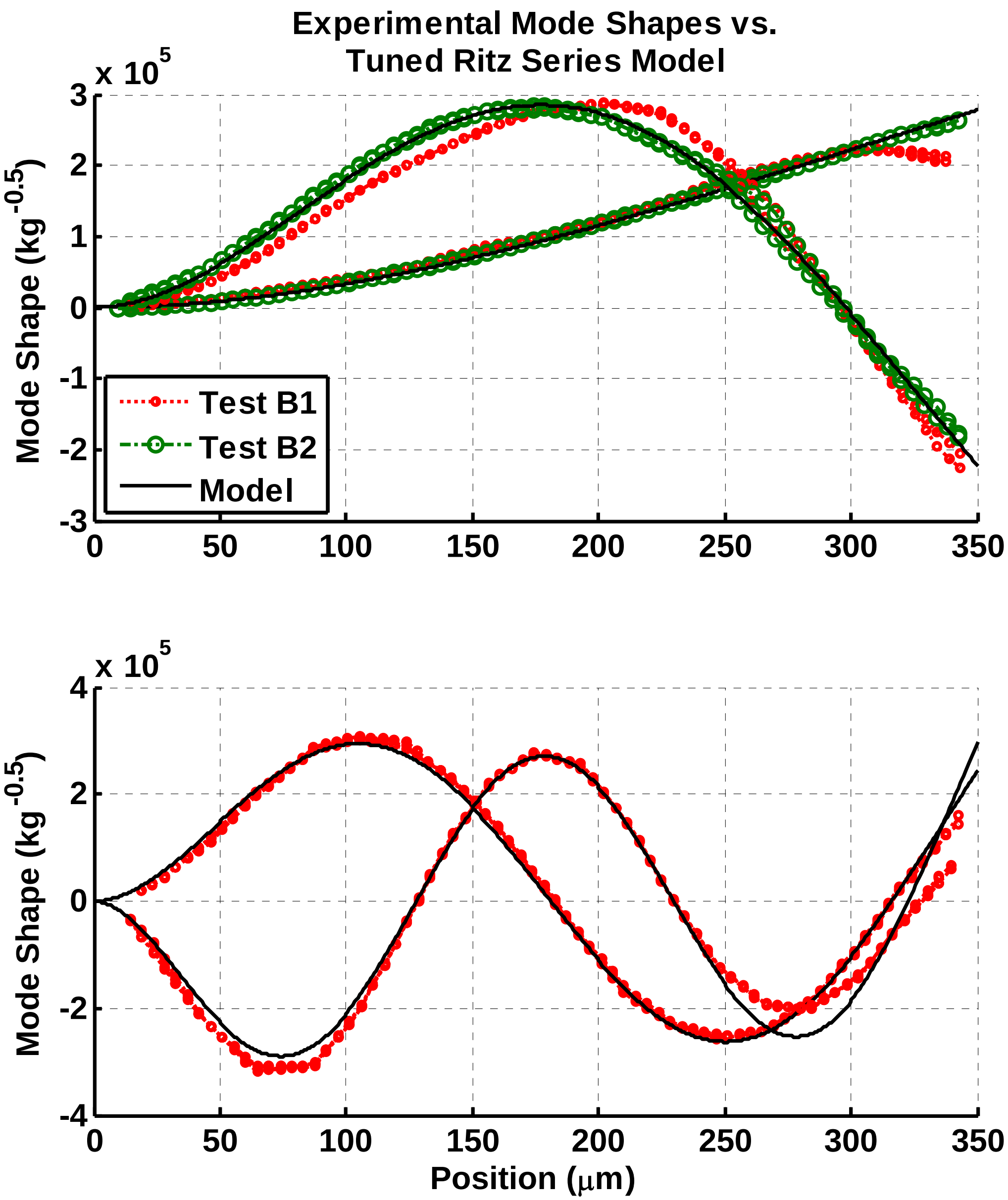
- 1st experimental mode is almost identical to analytical shape for a cantilever without a tip mass.
- Experimentally measured mode shapes are significantly different from the analytical shapes for modes 2-4.
- Tip motion is reduced as one would expect due to the added mass.



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.

Mode #	Exp. (kHz)	Model (kHz)
1	9.07	9.07
2	70.8	70.5
3	213.8	210.3
4	439.8	419.8



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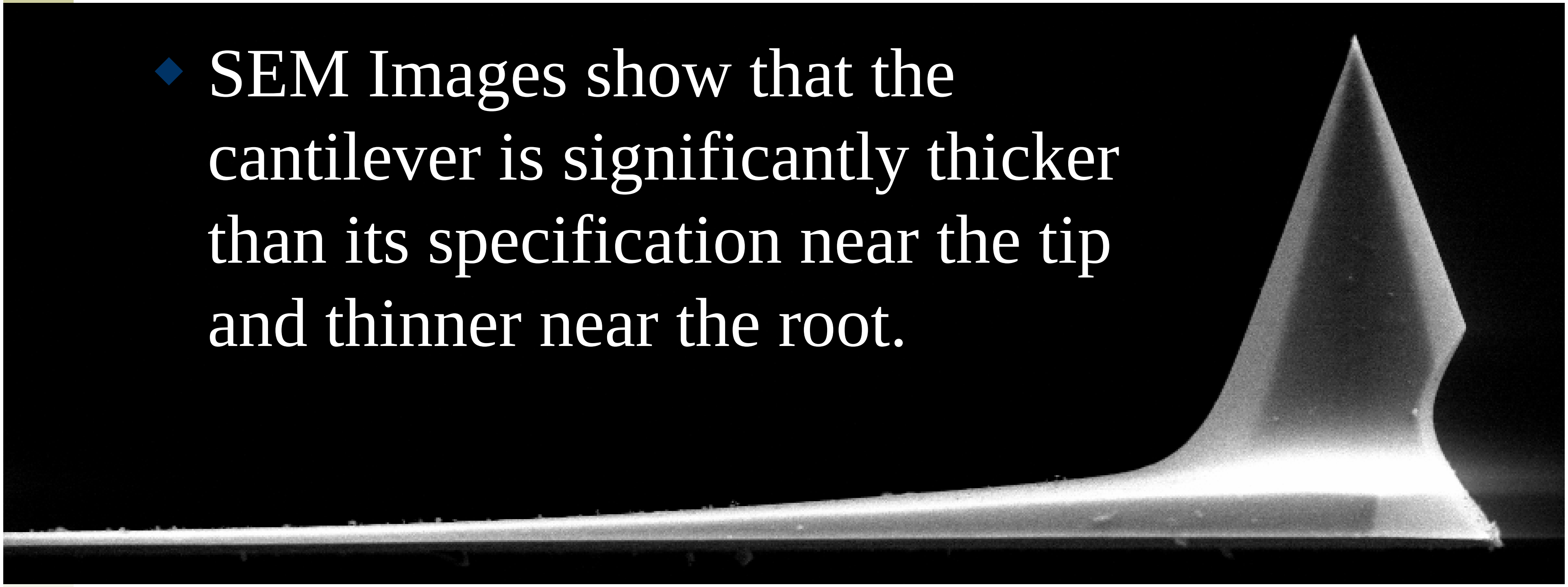
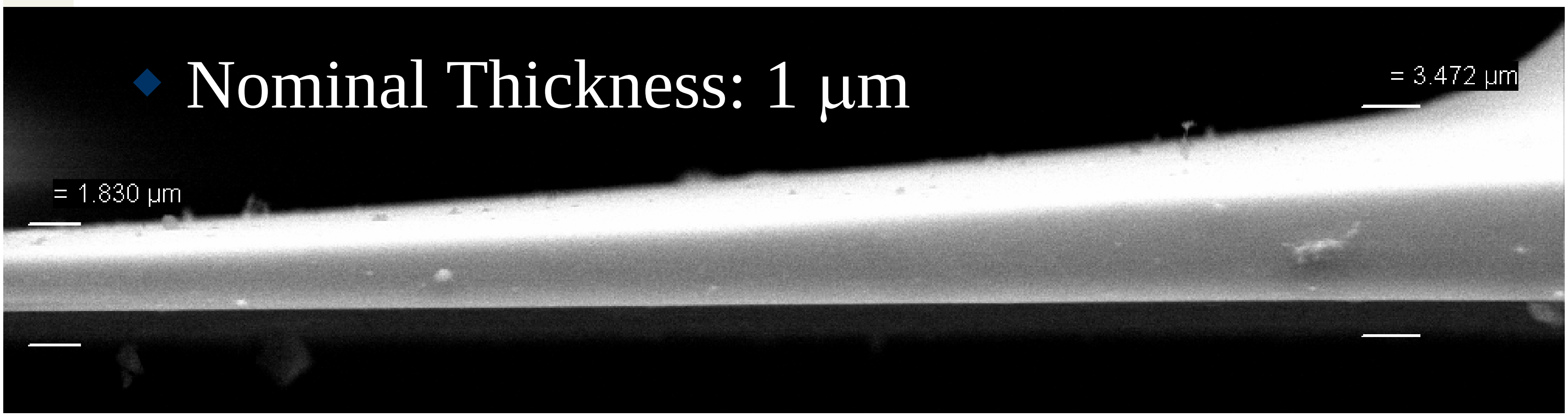
An Observation

- 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

	Sader (no tip)	Modified Sader (nominal tip)	Percent Difference (nominal tip)	Modified Sader (tuned tip)	Percent Difference (tuned tip)
k_s	0.0424 N/m	0.0424 N/m	0 %	0.0424 N/m	0 %
$\rho_c h$	4.39 g/m^2	3.28 g/m^2	34%	1.90 g/m^2	130%

- 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 1st, 2nd & 3rd Modes respectively.

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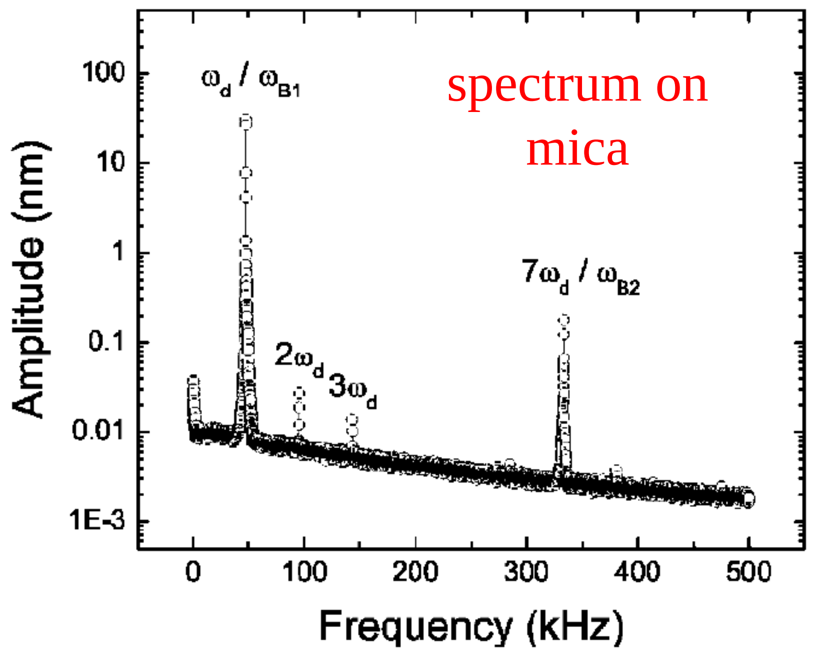
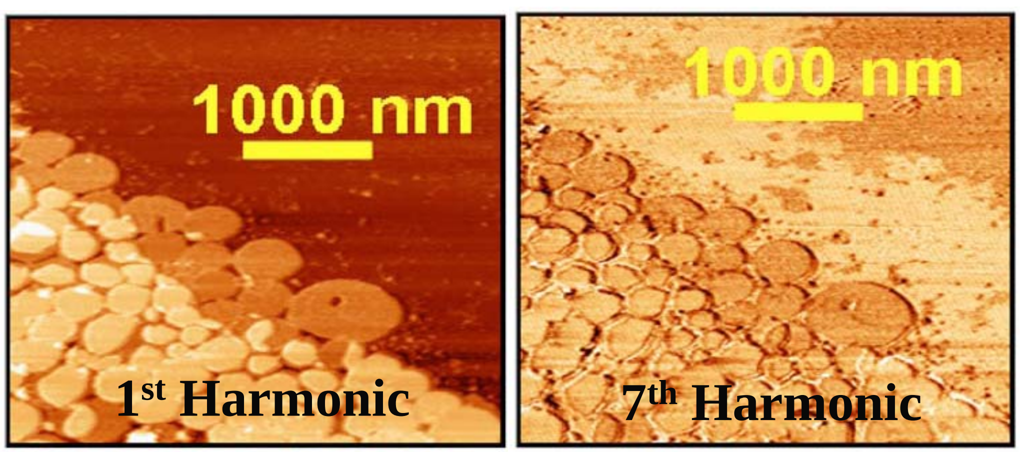
Other Implications





- 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!

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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 2nd and higher modes resulting in significant calibration errors if these modes are utilized.
  - 1st 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.

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