Lecture 11
Three important callibrations

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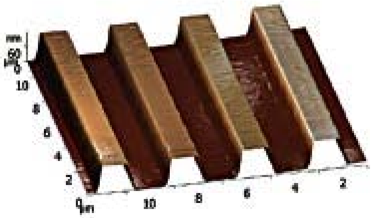
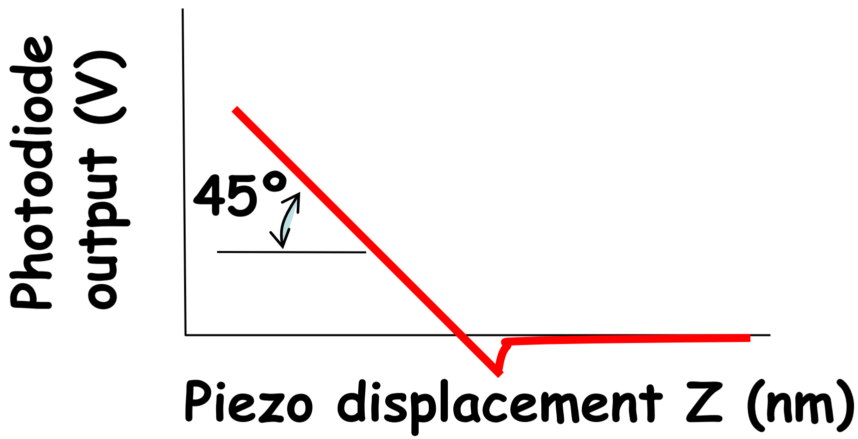
Three important calibrations

- Ultimately one only applies a Voltage to the Z piezo and measures voltages of cantilever deflection
- Z-piezo calibration: By scanning a sample of known height (calibration grating) in contact mode

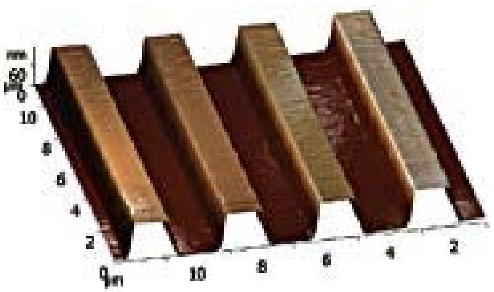
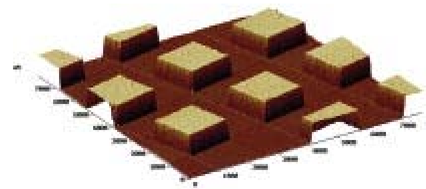


- Cantilever deflection calibration: δ-Z curve on hard sample



- Cantilever stiffness, k, calibration

Z piezo calibration


Gratings from Ted Pella Inc.

- Standard gratings of known dimensions (with uncertainties traced to wavelength of light) are available commercially
- Scan in contact mode plotting X,Y,Z in volts and set measured dimensions in volts to known dimensions (nm) to calibrate X‹Y‹Z axis piezos
- Closed loop piezo stages don't need this but it is still recommended to do this test often

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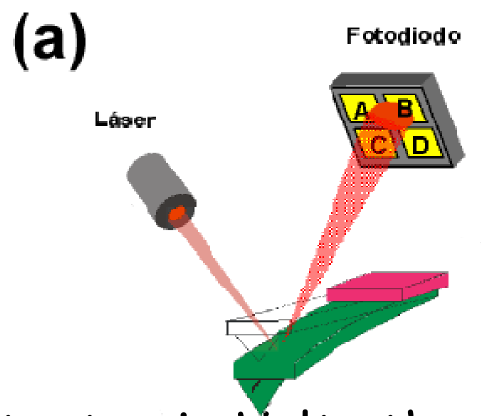
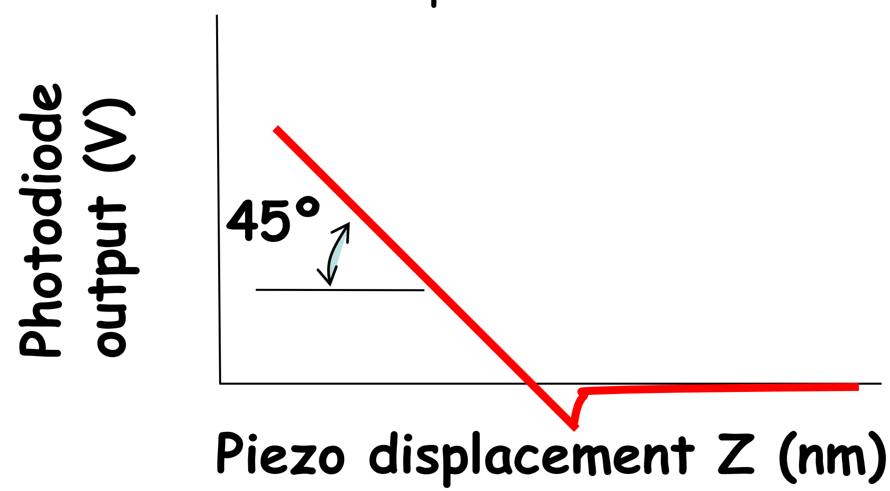
Cantilever deflection calibration



- Recall photodiode output is in Volts that are proportional to bending angl e which in turn is proportional to tip deflection
- How to convert cantilever deflection in Volts to nm?
- Perform F-Z curve on a hard sample (mica/silicon/sapphire)
- Slope of delfection (V) vs Z (nm) should be 1:1 ! This provides the calibration (also called sensitivity)



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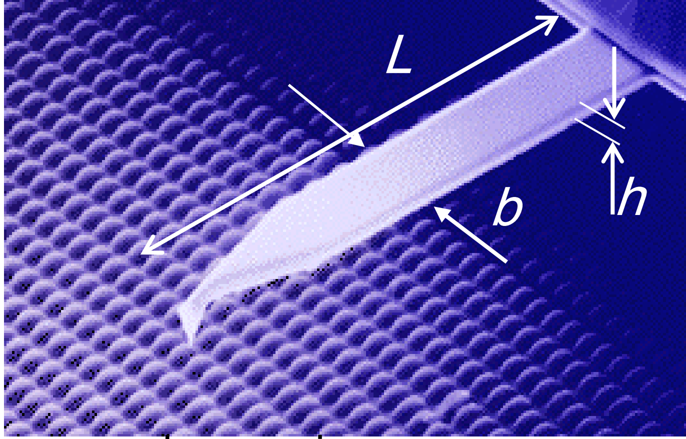
What is k?

- In principle $k_c = 3EI/L^3$ but

$I = bh^3/12$ and

'h' is rarely known accurately



www.olympus.co.jp

- Never trust manufacturer supplied value of k_c, it could be 100% off
- So how to know k_c ?
- Direct measurement requires application of known force and measurement of cantilever deflection. But how to apply known force?
- Static k_c is different from k_i of each eigenmode i=1,2,3.....

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Why do we need to know kc, k_1, etc.?

- In contact mode its important to know what force you are applying while imaging
- For static F-Z curve based force spectroscopy (local elasticity/adhesion)
- In dynamic mode measurements to know what the imaging force is
- In tapping mode to convert phase into energy dissipation

In essence, without accurate knowledge of k_c quantitative AFM is not possible

Existing methods

- Most common methods
  - *Geometric methods*

  (Cleveland and Sader methods)
  - *Thermal methods*

  (Hutter and Bechhoefer and Butt and Jaschke)

Nice review article - Burnham et al., Nanotechnology, **14**, 2003. Also the review article of Butt, Cappella, Kappl (Reader)

Table 1. A summary of the different techniques for obtaining spring constants.

Technique	Uncertainty	Advantages	Disadvantages
Forced oscillation/FEA [2]	10% compared with [1] for V-shaped levers	No masses need to be added, therefore non-destructive. Just depends on unloaded resonant frequency.	Technique relies on accurate values of cantilever density and thickness.
Forced oscillation/FEA [4]	5% for rectangular cantilevers compared with manufacturers values, and 10% compared with [1].	Plan view dimensions only required. Tested over a wide range of spring constants.	Requires knowledge of the Reynolds number for the fluid.
Forced oscillation [1]	10% for V-shaped cantilevers.	Absolute deflections measured giving direct measure of cantilever stiffness.	Difficult and risks damaging cantilever. Requires cantilever density and elastic modulus.
Thermal oscillations [6]	20% as obtained by [22] comparing with a static loading technique. 5% as determined by [6].	Simple and quick to use.	Requires cantilever to be pressed against hard surface for calibration. Ignores damping effects.
Static loading using pendulum [13]	50% as quoted in paper	Measures cantilever stiffness directly.	Requires calibration of pendulum.
Static loading by inverting loaded cantilever [14]	15% compared with manufacturer's figures.	Just one particle required to be added to the tip.	Cantilever movement calibration required. Potentially destructive.
Static loading using two probes [16]	10–30% depending on ratio of stiffnesses of two probes used.	Once one of the probes is calibrated it can be used to calibrate many different probes accurately.	Best for two probes of similar stiffness. Requires accurate probe positioning.
FEA of statically loaded triangular cantilevers [8]	No attempt made at comparing with any other technique.	Computation allows both normal and lateral spring constants to be determined.	Spring constant calculation is complex. V-shaped cantilevers simulated with end loading only.
FEA of oscillating composite V-shaped cantilevers [9]	6 and 25% compared with two different parallel beam approximations.	'Real' V-shaped cantilever geometry used.	Accuracy depends on uncertainty in material properties and type of parallel beam approximation used.
FEA of oscillating composite V-shaped cantilevers [10]	10% for full FEA solution.	Simple formula suggested relating the cube of the resonant frequency to the spring constant.	Applies to limited resonant frequency range. Gold coating thickness dependent.
FEA of oscillating composite V-shaped cantilevers [11]	Up to 40% for simple formula. Full FEA solution regarded as correct.	Full FEA gives very precise values for spring constant. Real V-shaped geometry used.	Gold coating thickness significantly affects outcome of FEA.

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Geometric methods

- Cleveland method (add known masses and measure frequency shift) Cleveland et al, Rev. Sci. Inst. **64**, 1993

$$\omega_1 = \sqrt{\frac{k_1}{m_1}} \quad \omega_1' = \sqrt{\frac{k_1}{m_1 + M}} \quad k_1 = \frac{M}{1/\omega_1'^2 - 1/\omega_1^2}$$

Quite accurate but not very convenient, also added mass difficult to know accurately

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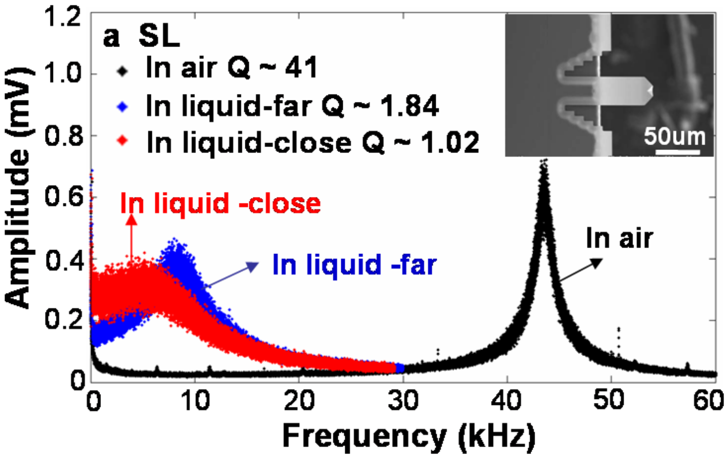
Geometric methods

Sader method (measure Q and ω_f and use hydrodynamic theory to estimate k)



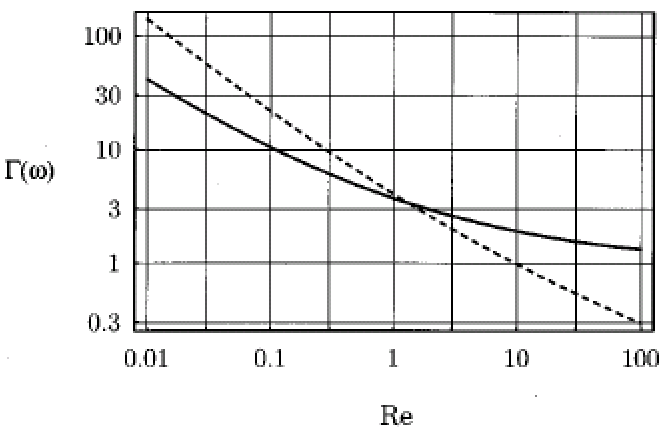


FIG. 1. Plot of the real and imaginary components of the hydrodynamic function $\Gamma(\omega)$ as a function of the Reynolds number $\mathrm{Re}=\rho_f \omega b^2/(4\eta)$. The real component Γ_r is shown by the solid line; the imaginary component Γ_i is shown by the dashed line.

Hydrodynamic force per unit length of cantilever

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

$$k_1=m_1\omega_{vac}^2 \qquad m_1=0.2427\rho_c bhL \qquad (1)$$

$$\omega_{vac}=\omega_f\left(1+\frac{\pi\rho_f b}{4\rho_c h}\Gamma_r(\omega_f)\right)^{1/2} \qquad (2)$$

$$\rho_c h=\frac{\pi\rho_f b}{4}\left[Q_f\Gamma_i(\omega_f)-\Gamma_r(\omega_f)\right] \qquad (3)$$

Sub (3),(2) *in* (1)

$$k_1=0.1906\rho_f b^2 L Q_f \Gamma_i(\mathrm{Re})\omega_f^2$$

$$\mathrm{Re}=\frac{\rho_f\omega_f b^2}{4\eta}$$

- Elegant, easy to use
- Needs to be modified for tip mass/higher eigenmodes

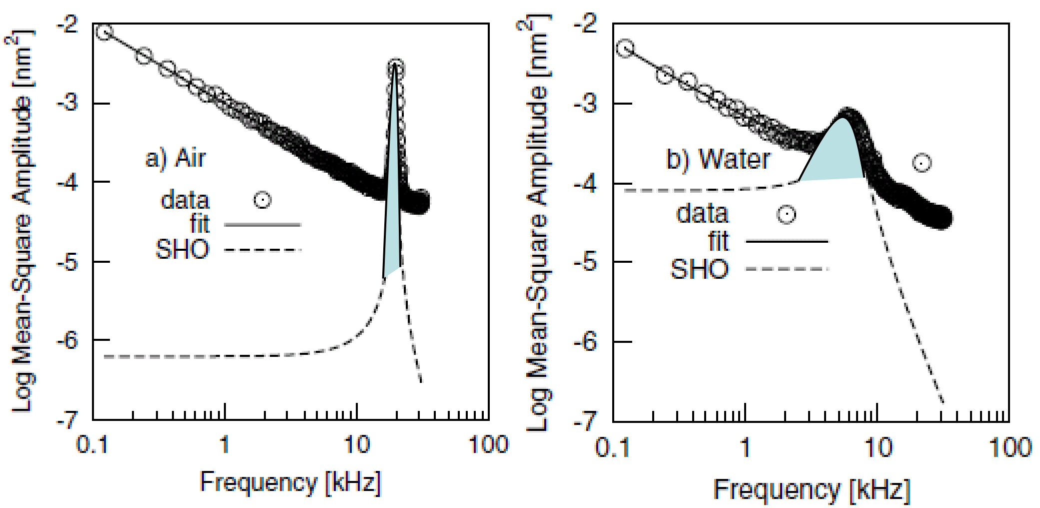
Sader et al., Rev. Sci. Inst., **70**(10), 1999

Thermal methods

- Hutter and Bechhoeffer Rev. Sci. Inst. 1993
  - Equipartition of energy

$$\frac{1}{2}k_c\left\langle x(t)^2\right\rangle = \frac{1}{2}k_BT$$

- Butt and Jaschke (Nanotechnology, 1995)
  - Made it mode specific $\frac{1}{2}k_1\left\langle x_1(t)^2\right\rangle = \frac{1}{2}k_BT$
  - Acquire x(t) in thermal bath, perform power spectral density and consider area under the peak



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Thermal methods - challenges

- Hutter and Bechhoeffer ignore contributions from multiple eigenmodes
- Power spectrum of x(t) contains many peaks some spurious, which one to choose for Butt and Jaschke's approach?
- What is the standard for calculating area?
- Requires accurate cantilever deflection calibration-not easy for higher eigenmodes!
- Spot location is critical! Spot needs to be located where the tip is located!
- Large spot size causes errors

Next time

- Ryan Wagner on experimental uncertainties in extracting elastic moduli and adhesion etc.