Calibration des leviers AFM

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AFM: measurement of cantilever's deflexion Δz

stiffness k

$$F = k\Delta z$$

interaction force F between probe and sample

AFM force measurements requires calibration of cantilever's stiffness

Introduction

1. Theoretical stiffness from Euler Bernoulli model

- theoretical stiffness k_0 of the cantilever
- from k_0 to k : corrections due to position of the probe

2. Calibration using static deformations

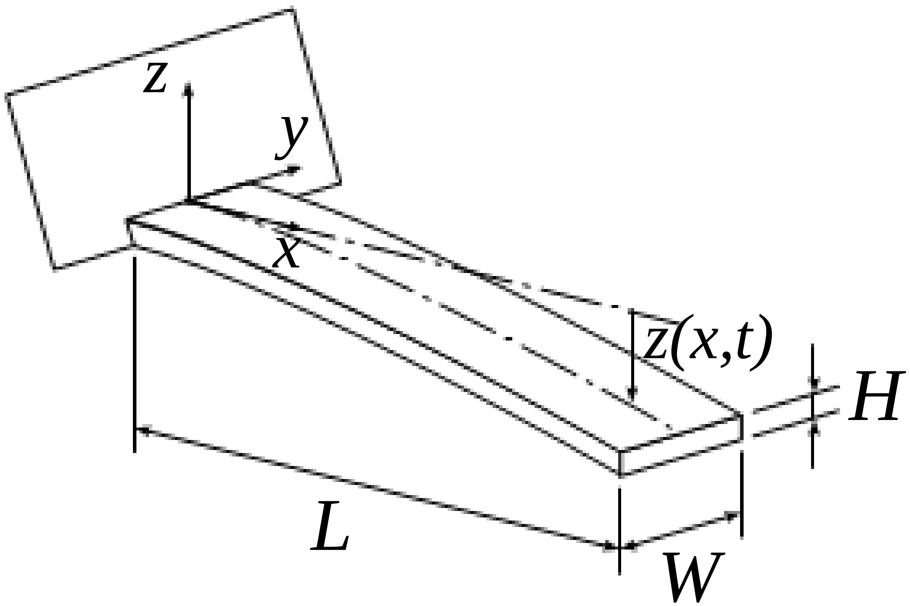
- cantilever against reference cantilever method

3. Calibrations based on modes of oscillation

- simple harmonic oscillator method
- Cleveland's added mass method
- Hydrodynamic Sader method
- Thermal noise calibration

Conclusion

Euler-Bernoulli model for a rectangular cantilever



- long beam: $L >> T, H$
- small deformations : $L << \Delta z = z(L)$
- constant Young modulus E
- constant rectangular section

Flexion only: $z(x,t)$

$$\frac{m_c}{L}\frac{\partial^2 z}{\partial t^2} + E\frac{WH^3}{12}\frac{\partial^4 z}{\partial x^4} = f_{ext}(x)$$

- constant linear mass

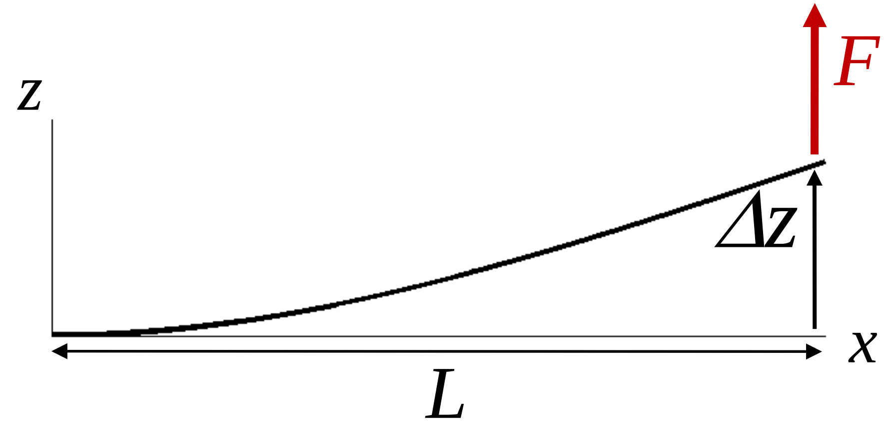
where m_c is the cantilever mass

external force / unit length

Static deformations in Euler-Bernoulli model

Point F force at the end of the cantilever

$$E\frac{WH^3}{12}\frac{\partial^4 z}{\partial x^4} = f_{ext}(x) = 0$$



with boundary conditions

- at $x = 0$: **fixed position and angle**

$$z(x=0) = \left.\frac{\partial z}{\partial x}\right|_{x=0} = 0$$

- at $x = L$: **point force F + no torque**

$$\left.\frac{\partial^3 z}{\partial x^3}\right|_L = -\frac{12}{EWH^3}F; \quad \left.\frac{\partial^2 z}{\partial x^2}\right|_L = 0$$

$$z(x) = \frac{(6Lx^2 - 2x^3)}{EWH^3}F$$

$$\Delta z = \frac{4L^3}{EWH^3}F$$

$1/k_0$

Static deformations in Euler-Bernoulli model

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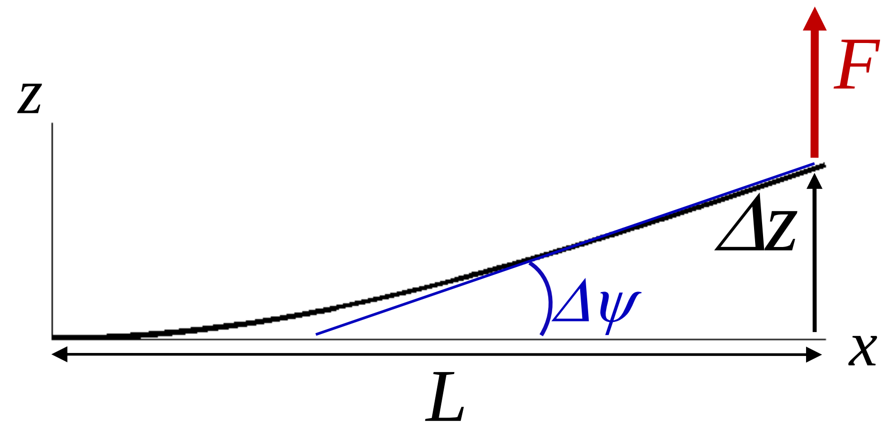

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$1/k_0$

Angle at $x = L$ for optical lever detection

$$\Delta\psi = \left.\frac{\partial z}{\partial x}\right|_L$$

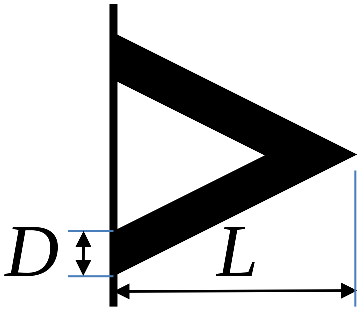
$$\frac{\Delta z}{\Delta\psi} = \frac{2L}{3} = cL$$

Theoretical stiffness from Euler-Bernoulli model

- **Rectangular cantilever**

$$k_0 = \frac{EWH^3}{4L^3}$$

- **V-shaped cantilever**



- no exact analytical expression
- approximate expression:

Sader, Rev. Sci. Instrum. 66, 4583 (1995)

$$k_0 = \frac{EDH^3}{2L^3} \times \text{geometrical correction}$$

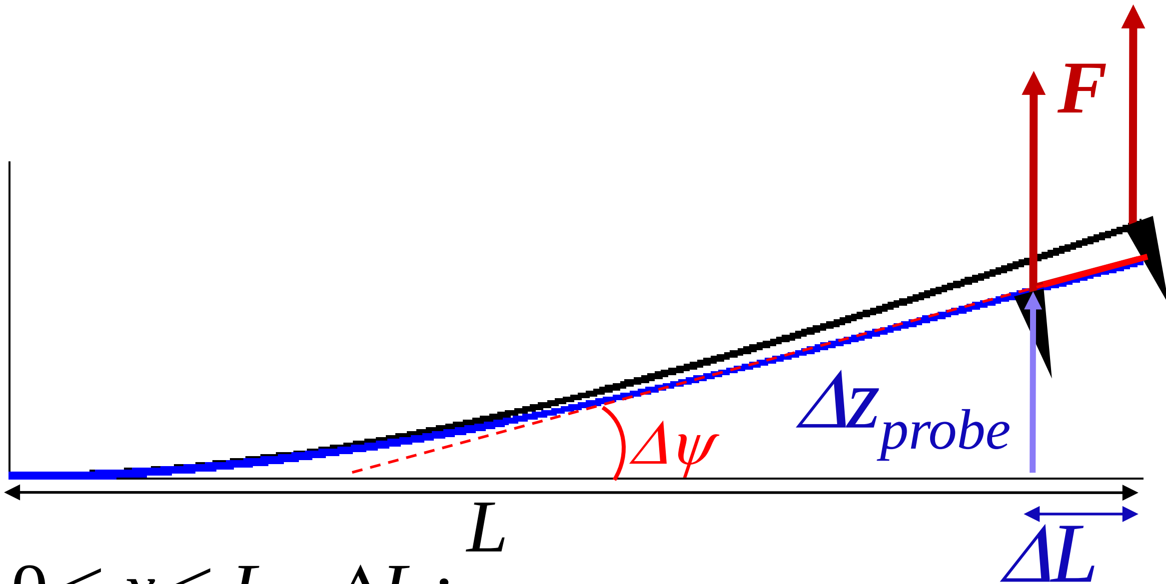
Parallel beams approximation

Expressions used by manufactors for cantilever stiffness estimation

Main error sources: thickness H (to the power 3), and Young modulus E

Theoretical stiffness from Euler-Bernoulli model

Correction when point force acts ΔL away from end



$L - \Delta L \leq x \leq L:$

constant angle part

$$\Delta\psi = \left.\frac{\partial z}{\partial x}\right|_{x \geq L-\Delta L} = \frac{6(L-\Delta L)^2}{EWH^3}F$$

$$\frac{\Delta z_{probe}}{\Delta\psi} = \frac{2L}{3} = cL$$

$0 \leq x \leq L - \Delta L:$

As if beam length was *L-ΔL* instead of *L*

$$z(x) = \frac{(6(L-\Delta L)x^2 - 2x^3)}{EWH^3}F$$

$$\Delta z_{probe} = z(L-\Delta L) = \frac{4(L-\Delta L)^3}{EWH^3}F$$

$1/k_{0\Delta L}$

$$k_{0\Delta L} = k_0\left(\frac{L}{L-\Delta L}\right)^3$$

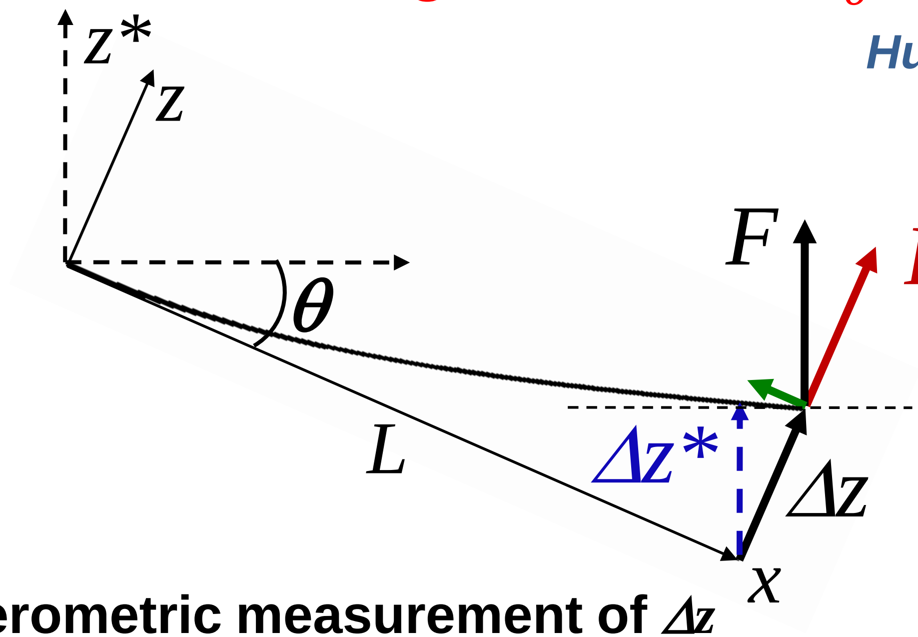
L = 300 µm, *ΔL*=10 µm

→ 10% correction

Theoretical stiffness from Euler-Bernoulli model

Influence of tilt angle θ : from k_0 to k and k^*

Hutter, Langmuir 21, 2630 (2005)



$$F\cos\theta = k_{0\Delta L}\Delta z$$

tilt angle θ = 11

- **Interferometric measurement of Δz**

$$F = k\Delta z$$

$$k = \frac{k_{0\Delta L}}{\cos\theta}$$

2% correction

- **Optical lever with 4-quadrant photodiodes**

$$F = k^* s\Delta V$$

$$s\Delta V = \Delta z^* = \Delta z\cos\theta$$

$$k^* = \frac{k}{\cos\theta} = \frac{k_{0\Delta L}}{\cos^2\theta}$$

4% correction

s **calibrated from slope of ΔV versus Δz^* when pushing on hard surface**

Theoretical stiffness from Euler-Bernoulli model

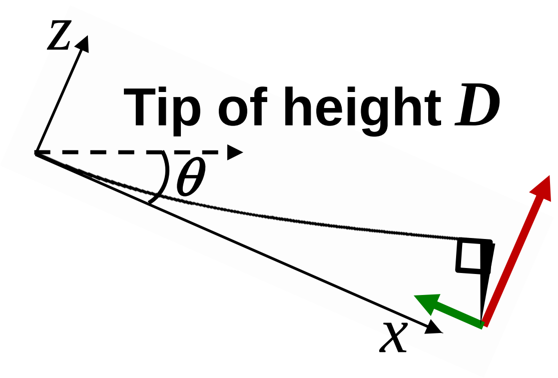
Influence of tilt angle θ: torque correction

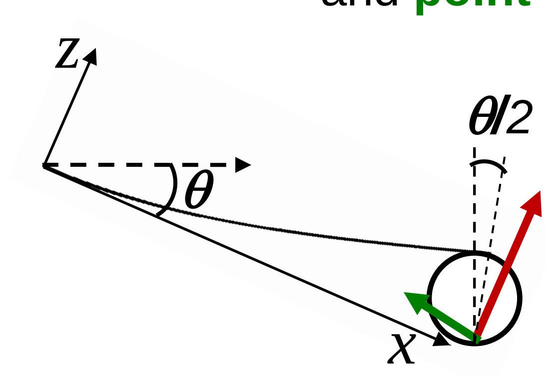
Hutter, Langmuir 21, 2630 (2005); Edwards et al., J. Appl. Phys. 103, 064513 (2008)

Static deformations with boundary conditions

At x=0: clamped $z(x=0) = \left.\frac{\partial z}{\partial x}\right|_{x=0} = 0$; At $x=L$: point force $\left.\frac{\partial^3 z}{\partial x^3}\right|_L = -\frac{3F\cos\theta}{k_0 L^3}$

and **point torque** $\left.\frac{\partial^2 z}{\partial x^2}\right|_L = -\frac{3DF\sin\theta}{k_0 L^3}$





Sphere of radius *R*:
replace *D* by *R*

$$z(x) = \frac{(3L(1-\frac{D}{L}\tan\theta)x^2 - x^3)}{2k_0L^3} F\cos\theta$$

$$F = \frac{k_0}{\cos\theta} \frac{1}{(1-\frac{3D}{2L}\tan\theta)} \Delta z$$

$$\frac{\Delta z}{\Delta\psi} = \frac{1-3D\tan\theta/2L}{1-2D\tan\theta/L}\frac{2L}{3} = cL$$

Torque correction

L **= 300 µm, *D*=10 µm, tilt angle θ = 11**

→ 1% correction on *k*

→ 0.3% correction on $\Delta\varphi/\Delta z$

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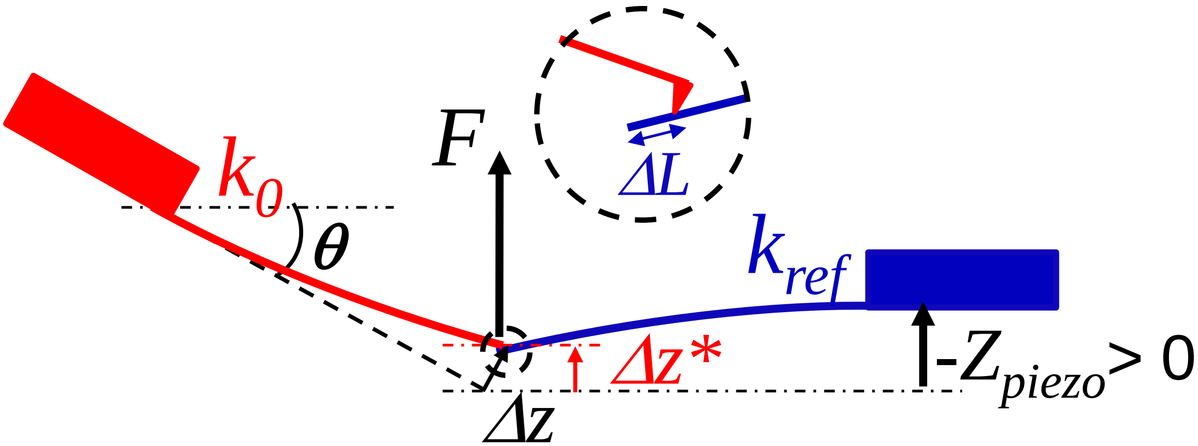
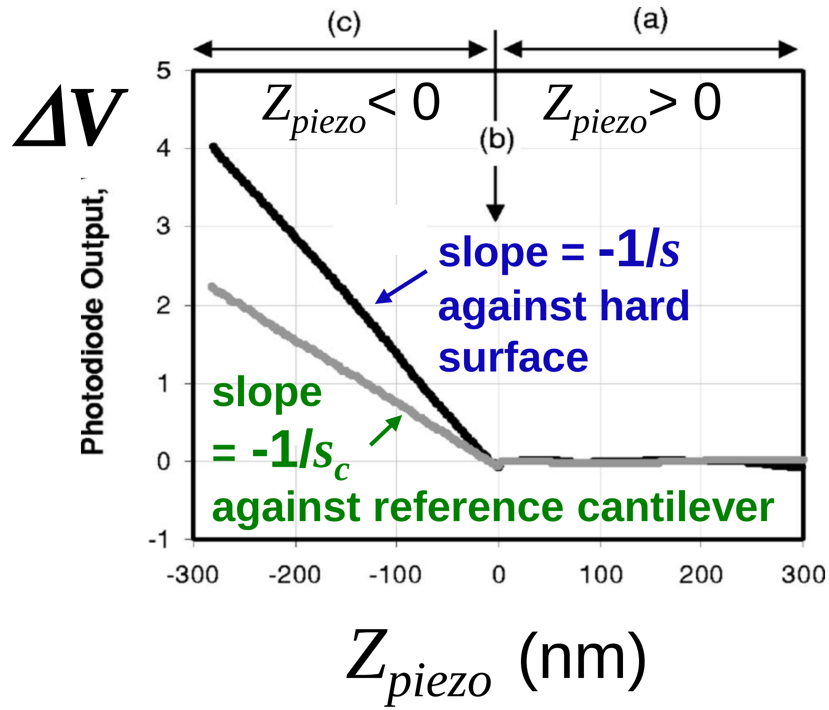
Calibration using static deformations:

Cantilever against reference cantilever method



$$\Delta z^* = \Delta z \cos\theta = s\Delta V$$

Calibration optical lever against hard surface



$$F = k_{ref}\left(\frac{L}{L-\Delta L}\right)^3\left(-Z_{piezo} - \Delta z^*\right) = k\Delta z = k^*\Delta z^*$$

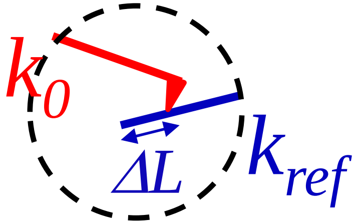
$$k = k_{ref}\left(\frac{L}{L-\Delta L}\right)^3\left(\frac{-Z_{piezo}}{\Delta z} - \cos\theta\right)$$

$$k^* = k_{ref}\left(\frac{L}{L-\Delta L}\right)^3\left(\frac{s_c}{s} - 1\right)$$

Gates et al., Rev. Sci. Instrum, 78, 086101(2007)

Calibration using static deformations:

Cantilever against reference cantilever method



Avantages:

- **works for all type of cantilevers and probes**
- **direct measure of k and k^*, ie stiffness of interest in experimental configuration**

Drawbacks:

- **requires reference cantilever with $k_{ref} \sim k_0$**
- **requires mechanical contact (risk of damaging the probe)**
- **sensitive to position ΔL of the probe on the reference cantilever**

Main error sources: measurement of slope(s) (because of solid friction), and position ΔL

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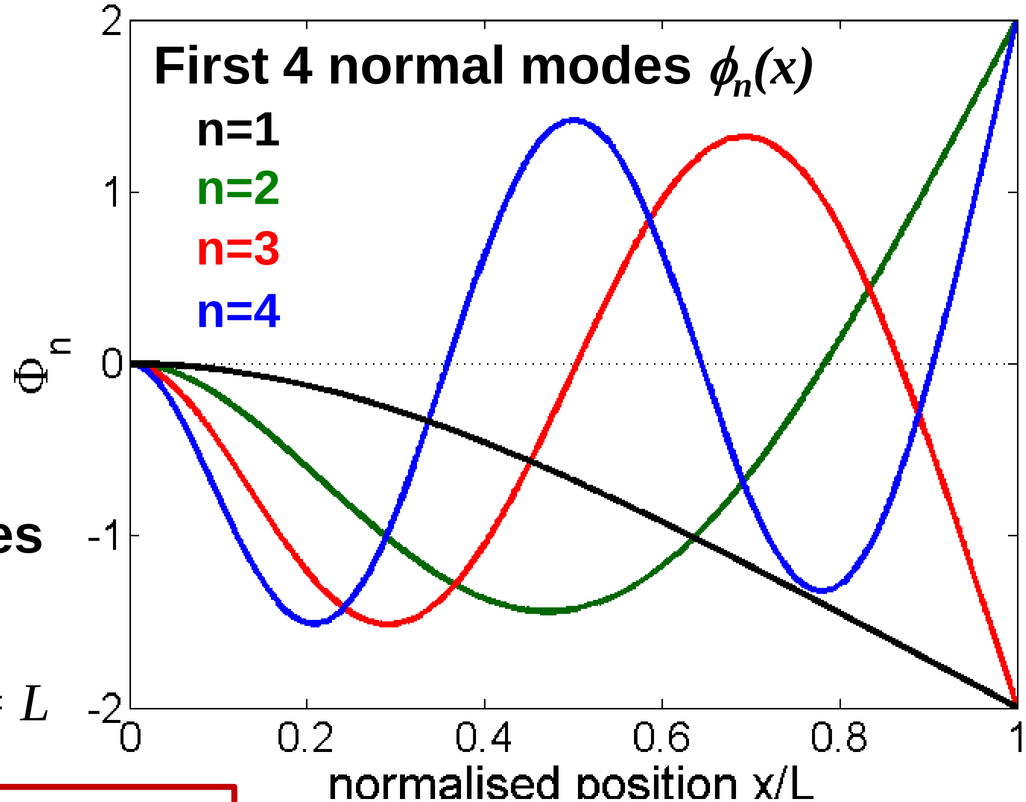
Calibrations based on modes of oscillation: Normal modes in Euler-Bernoulli model

$$\tilde{z}(x,\omega) = \phi(x)e^{i\omega t}$$

$$L^4 \frac{\partial^4 \phi}{\partial x^4} = \boxed{\frac{3m_c\omega^2}{k_0}}\phi(x)$$

α^4



Free cantilever: no external forces

Boundary conditions

➢ clamped at $x = 0$; free end at $x = L$

➡ **α must obey : cos(α) + cosh(α) = -1**

Roots α_n (α_1=1.875; α_2=4.694…)

Normal spatial modes ϕ_n (x, α_n) form a basis set of fonctions in [0,L]

Butt et al., Nanotechnol. 6, 1 (1995)

Calibrations based on modes of oscillation: :

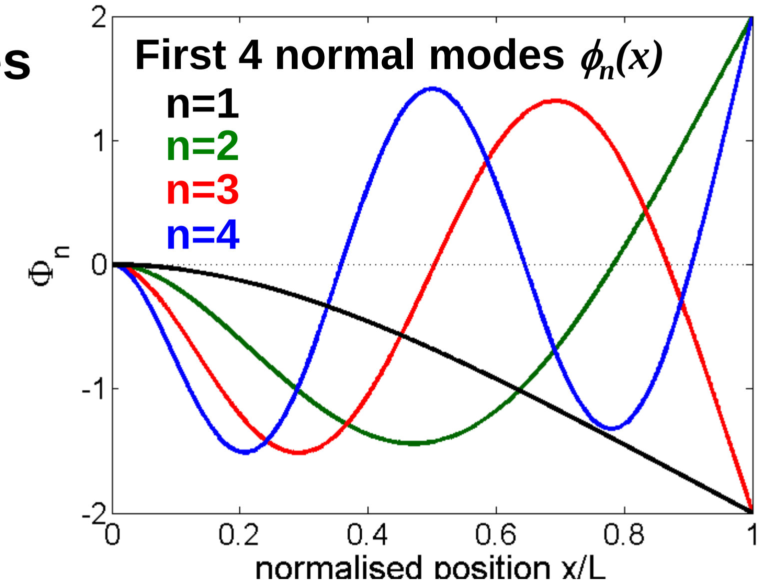
From normal modes to simple harmonic oscillators

Decomposition on basis of normal modes

$$\tilde{z}(x,\omega) = \sum_n \tilde{Z}_n(\omega)\phi_n(x)$$

$$\tilde{f}_{ext}(x,\omega) = \frac{1}{L}\sum_n \tilde{\beta}_{ext,n}(\omega)\phi_n(x)$$

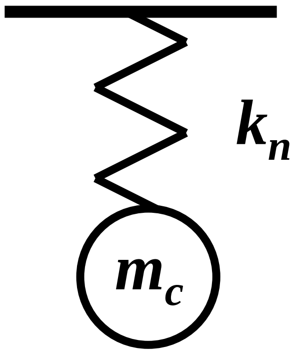
$$\left(\frac{\alpha_n^4 k_0}{3} - \omega^2 m_c\right)\tilde{Z}_n(\omega) = \tilde{\beta}_{ext,n}(\omega)$$



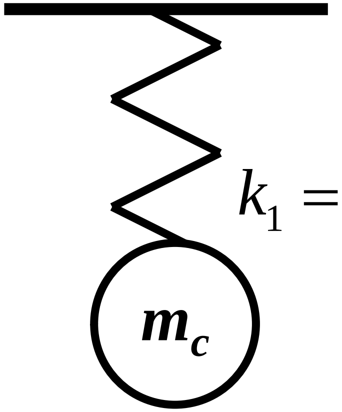
Each mode of oscillation behaves as a simple harmonic oscillator (SHO) with

stiffness $k_n = \frac{\alpha_n^4 k_0}{3}$

mass m_c



Calibrations based on modes of oscillation:
Simple harmonic oscillator (SHO) method



$$k_1 = \frac{\alpha_1^4 k_0}{3}$$

- **Measure angular frequency ω_1 of 1st resonance**

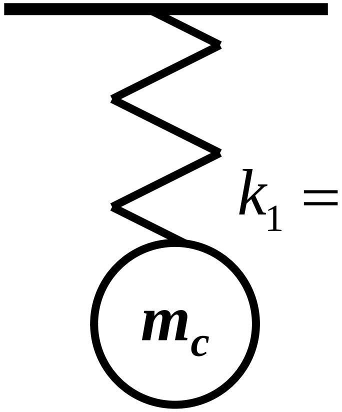
For a rectangular cantilever, in vacuum:

$$\omega_1 = \sqrt{\frac{\alpha_1^4 k_0}{3m_c}}$$

$$k_0 = \frac{3m_c}{\alpha_1^4}\omega_1^2$$

Sader et al., Rev. Sci. Instrum. 66, 3789 (1995)

Calibrations based on modes of oscillation: Simple harmonic oscillator (SHO) method



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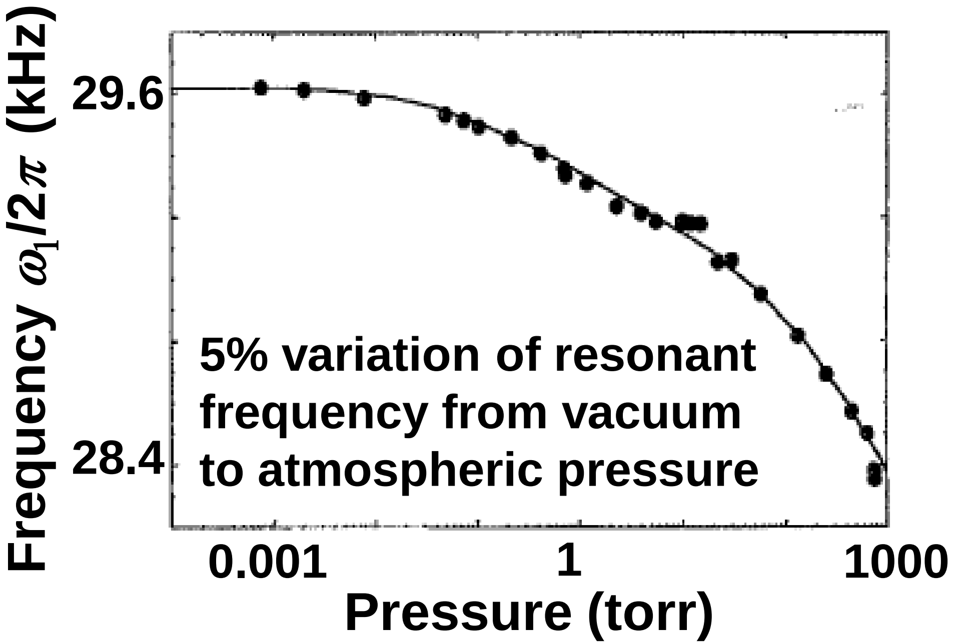
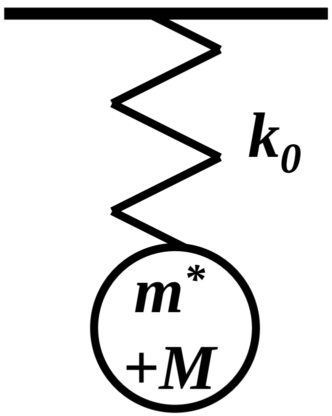
Main error sources:

- **mass m_c**
- **sensitivity of resonant frequency ω_1 to fluid loading**



Sader et al., Rev. Sci. Instrum. 66, 3789 (1995)

Calibrations based on modes of oscillation: Cleveland's added mass method



- **Attach spheres of mass M at the end of the cantilever**
- **Measure angular frequency ω_1 of 1st résonance**

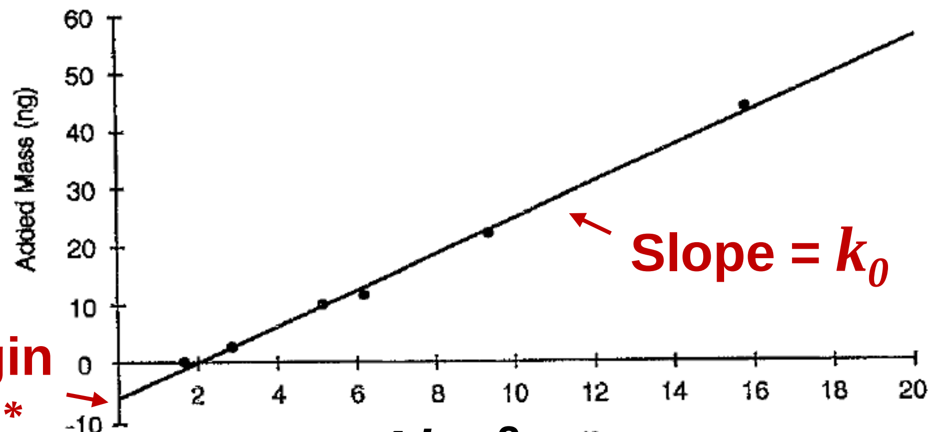
$$\omega_1 = \sqrt{\frac{k_0}{m^* + M}}$$

For a rectangular cantilever:

$$m^* = \frac{3m_c}{\alpha_1^4} = 0.24 m_c$$

- **Plot M as a function of $1/\omega_1^2$**

Added mass M



origin = m^*

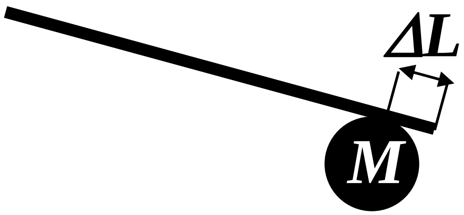
$1/\omega_1^2$ (10^{-10} s^2)

Cleveland et al., Rev. Sci. Instrum. 64, 403 (1993)

Calibrations based on modes of oscillation: Cleveland's added mass method

Cleveland et al., Rev. Sci. Instrum. 64, 403 (1993)

Evaluated in: *Sader et al., Rev. Sci. Instrum. 66, 3789 (1995)*



$$\omega_1 = \sqrt{\frac{k_{0\Delta L}}{m^* + M}}$$

Measures $k_{0\Delta L}$ rather than k_0

- **Plot** $M\left(\frac{L-\Delta L}{L}\right)^3$ **versus** $1/\omega_1^2$ **to get k_0 slope**

Avantages:

- **works for all type of cantilevers and probes**
- **independant of type and calibration of detection**
- **insensitive to fluid-loading effects when $M > 10\, m_c$**

Drawbacks:

- **requires micromanipulations**
- **post-mortem due to risk of damaging the probe**
- **sensitive to position ΔL of the added mass**

Main error sources: mass M, and position ΔL

Calibrations based on modes of oscillation:

Sader model for hydrodynamic load

When a cantilever is immersed in a fluid (viscosity η_f and density ρ_f)

- **broadening of resonance due to viscous damping**
- **diminution of resonant frequency due to added mass by the fluid**

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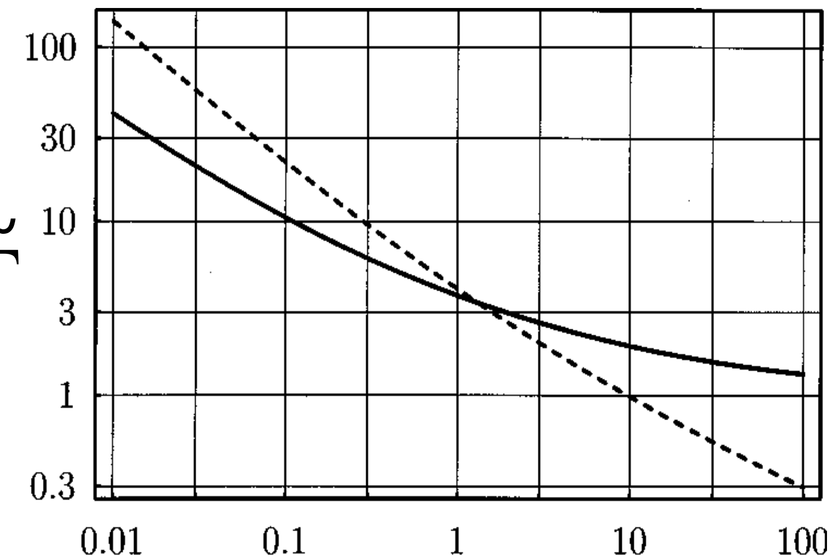
Hydrodynamic load

$$\tilde{f}_{hydro}(x,\omega) = \frac{\pi}{4}\rho_f \omega^2 W^2 \tilde{\Gamma}\left(\frac{\rho_f W^2 \omega}{4\eta_f}\right)\tilde{z}(\omega, x)$$

Hydrodynamic function $\tilde{\Gamma}$ computed for low-order modes of oscillation of rectangular cantilever with $T \ll W \ll L$

$\tilde{z}(x,\omega)$



$\tilde{\Gamma}$

$$\text{Reynolds number} = \frac{\rho_f W^2 \omega}{4\eta_f}$$

Sader, J. Appl. Phys 84, 64 (1998)

Sader et al., J. Appl. Phys 97, 124903 (2005)

Calibrations based on modes of oscillation:

Sader model for hydrodynamic load

Decomposition on basis of normal modes of oscillation

$$\left(k_n - \omega^2 \underbrace{m_c\left(1+\tilde{\tau}_r(\omega)\right)}_{m_{eff}(\omega)} - i\omega \underbrace{\left(m_c \omega \tilde{\tau}_i(\omega)\right)}_{\gamma_{eff}(\omega)}\right)\tilde{Z}_n(\omega) = \tilde{\beta}_{ext,n}(\omega)$$

with $\tilde{\tau}_r(\omega) + i\tilde{\tau}_i(\omega) = \frac{\pi}{4}\frac{\rho_f L W^2}{m_c}\tilde{\Gamma}\left(\frac{\rho_f \omega}{4\eta_f}\right)$

Low damping limit (quality factor $Q \geq 10$):

$m_{eff}(\omega)$ and $\gamma_{eff}(\omega)$ ~ constant arount resonance peak

➡ **Each mode of oscillation behaves as a damped SHO with:**

stiffness $k_n = \frac{\alpha_n^4 k_0}{3}$

mass $m_{eff,n} = m_c\,(1+\tau_r(\omega_n))$

damping $\gamma_{eff,n} = m_c \omega_n\, \tau_i(\omega_n)$

$$\omega_n = \sqrt{\frac{k_n}{m_{eff,n}}}$$

$$Q_n = \frac{k_n}{\omega_n \gamma_{eff,n}} = \frac{1+\tau_r(\omega_n)}{\tau_i(\omega_n)}$$

Calibrations based on modes of oscillation:

Hydrodynamic Sader method

Rectangular cantilever, low damping limit (quality factor $Q_1 \geq 10$)**:**

$$k_0 = \underbrace{\frac{3\pi}{4\alpha_1^4}}_{0.1906} \omega_1^2 \rho_f L W^2 Q_1 \underbrace{\Gamma_i\left(\frac{\rho_f W^2 \omega_1}{4\eta_f}\right)}_{\text{Imaginary part of the hydrodynamic function}}$$

Sader et al., Rev. Sci. Instrum. 70, 3967 (1999)

- η_f **and** ρ_f **tabulated**
- L **and** W **measured by optical microscopy**
- ω_1 **and** Q_1 **measured from damped SHO fit of 1st resonance peak**

Calibrations based on modes of oscillation:

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- **η_f and ρ_f tabulated**
- **L and W measured by optical microscopy**
- **ω_1 and Q_1 measured from damped SHO fit of 1st resonance peak**

Avantages:

- **fast, precise, easy -- online calibration on:** http://www.ampc.ms.unimelb.edu.au/afm/theory.html
- **independant of type and calibration of detection**

Underestimates k_0 if presence of colloidal probe of diameter > $W/2$

Main error sources: width W, and quality factor Q_1

Calibrations based on modes of oscillation:
Equipartition of energy and thermal noise PSD

Small dissipation (high Q):

➡ **Modes of oscillation Z_n are independant degrees of freedom**

$$\frac{1}{2}k_n\langle Z_n^2\rangle = \frac{1}{2}k_BT$$

Total thermal noise of the cantilever

$$\langle \Delta z^2\rangle = \sum_n \langle Z_n^2\rangle \underbrace{|\phi_n(x=L)|^2}_{\to 4} = \frac{k_BT}{k_0}\underbrace{\sum_n \frac{12}{\alpha_n^4}}_{\to 1} = \frac{k_BT}{k_0}$$

Link with thermal noise PSD of the cantilever:

$$\langle \Delta z^2\rangle = \int_0^\infty S_{thermal}^{\Delta z}(f)df = \int_0^\infty (S_{measured}^{\Delta z} - S_{background}^{\Delta z})df$$

Butt et al., Nanotechnol. 6, 1 (1995)

Calibrations based on modes of oscillation:

Equipartition of energy and thermal noise PSD

For detection with optical lever: total thermal noise of ΔV

$$\left\langle \Delta V^2 \right\rangle = \left(cL \frac{\cos\theta}{s} \right)^2 \left\langle \Delta\psi^2 \right\rangle = \left(cL \frac{\cos\theta}{s} \right)^2 \sum_n \left\langle Z_n^2 \right\rangle \left| \frac{\partial \Phi_n}{\partial x} \right|_L^2 = 12c^2 \frac{\cos^2\theta}{s^2} \frac{k_B T}{k_0} \sum_n \frac{1}{\alpha_n^2} \left(\frac{\sin\alpha_n \sinh\alpha_n}{\sin\alpha_n + \sinh\alpha_n} \right)^2$$

$\left\langle \Delta\psi^2 \right\rangle$: thermal noise of angular deflexion

$c \approx 2/3$

$\sum_n \frac{1}{\alpha_n^2} \left(\frac{\sin\alpha_n \sinh\alpha_n}{\sin\alpha_n + \sinh\alpha_n} \right)^2 \to 1/4$

$$\left\langle \Delta V^2 \right\rangle \approx \frac{4}{3} \frac{\cos^2\theta}{s^2} \frac{k_B T}{k_0}$$

Butt et al., Nanotechnol. 6, 1 (1995)

Hutter, Langmuir 21, 2630 (2005)

Link with thermal noise PSD of signal ΔV :

$$\left\langle \Delta V^2 \right\rangle = \int_0^\infty S_{thermal}^{\Delta V}(f)df = \int_0^\infty (S_{measured}^{\Delta V} - S_{background}^{\Delta V})df$$

Calibrations based on modes of oscillation:

Thermal noise calibration

Hutter et al., Rev. Sci. Instrum. 64, 1868 (1993)

Idea:

measure k_0 from integration of thermal noise PSD

but in reality, finite integration range:

thermal noise PSD only available around resonance because of background noise

So no direct access to mean square amplitudes $\left\langle \Delta V^2 \right\rangle$ and $\left\langle \Delta z^2 \right\rangle$

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Contribution of mode n to total thermal noise ?

Fluctuation dissipation theorem on amplitude of normal mode n

$$\frac{\left\langle \left|\tilde{Z}_n(\omega)\right|^2 \right\rangle}{\Delta f} = -\frac{4k_B T}{\omega}\,\mathrm{Im}\left(\frac{\tilde{Z}_n(\omega)}{\tilde{\beta}_{ext,n}(\omega)}\right) = \frac{k_B T}{k_n}\,\frac{4/\omega_n Q_n}{\left(1-\left(\frac{\omega}{\omega_n}\right)^2\right)^2 + \frac{1}{Q_n^2}\left(\frac{\omega}{\omega_n}\right)^2}$$

$$\omega_n = \sqrt{\frac{k_n}{m_{eff,n}}}$$

$$Q_n = \frac{k_n}{\omega_n \gamma_{eff,n}}$$

Calibrations based on modes of oscillation:

Thermal noise calibration

SHO fit of resonance peak in thermal noise PSD of free cantilever:

$$S^{\Delta z}_{thermal,n}(f) = \frac{\left\langle \left| \tilde{z}_n(\omega, L) \right|^2 \right\rangle}{\Delta f} = 4\frac{\left\langle \left| \tilde{Z}_n(\omega) \right|^2 \right\rangle}{\Delta f} = \boxed{\frac{12}{\alpha_n^4}} \frac{k_B T}{k_0} \frac{4/\omega_n Q_n}{\left(1-\left(\frac{\omega}{\omega_n}\right)^2\right)^2 + \frac{1}{Q_n^2}\left(\frac{\omega}{\omega_n}\right)^2}$$

≈ 0.97 pour n=1

$$\int_0^\infty \frac{4/\omega_n Q_n}{\left(1-\left(\frac{\omega}{\omega_n}\right)^2\right)^2 + \frac{1}{Q_n^2}\left(\frac{\omega}{\omega_n}\right)^2} = 1$$

$$S^{\Delta V}_{thermal,n}(f) = \boxed{\frac{12c^2}{\alpha_n^2}\left(\frac{\sin\alpha_n \sinh\alpha_n}{\sin\alpha_n + \sinh\alpha_n}\right)^2} \left(\frac{\cos\theta}{s}\right)^2 \frac{k_B T}{k_0} \frac{4/\omega_n Q_n}{\left(1-\left(\frac{\omega}{\omega_n}\right)^2\right)^2 + \frac{1}{Q_n^2}\left(\frac{\omega}{\omega_n}\right)^2}$$

≈ 0.817 pour n=1

- **k_0 , ω_n and Q_n**

- **fast, precise, easy**
- **depends of calibration of detection**

Butt et al., Nanotechnol. 6, 1 (1995)

Hutter, Langmuir 21, 2630 (2005)

Error sources: error on fit parameters
+ error on s if optical lever technique

Conclusion

Comparison of calibration methods for cantilever's bending stiffness k_0

	Theoretical	SHO	Added mass	Hydro Sader	Thermal noise	Cant. against reference cant.
requires	L, W, H, E	m_c ω_1	M, ΔL ω_1	η_f, ρ_f L, W, ω_1, Q_1	T, **thermal noise PSD** **+ s, θ for optical lever**	k_{ref}, ΔL, θ **+ slopes** $\Delta signal / Z_{piezo}$ **in contact**
Main error sources	H, E	m_c ω_1 **in fluid**	M, ΔL	W, Q_1	**SHO fit,** s	k_{ref}, ΔL **+ slopes** $\Delta signal / Z_{piezo}$ **in contact**
Typical uncertainty	**30-50 %**	**20-40 %**	**10-30 %**	**5-10 %**	**5 -10 %**	**10-30 %**
	can be reduced by MEB pictures to measure H					

Conclusion: to go further…

- **Other calibration methods for cantilever's bending stiffness k_0 and variants…**

  For example:

  - ***mapping of spatial modes along the cantilever from thermal noise***

    Paolino et al., J. Appl. Phys. 106, 094313 (2009) and HDR of L. Bellon

  - ***calibration using electrostatic forces on metal-coated colloidal probes***

    Chung et al., Rev. Sci. Instrum. 80, 065107 (2009)

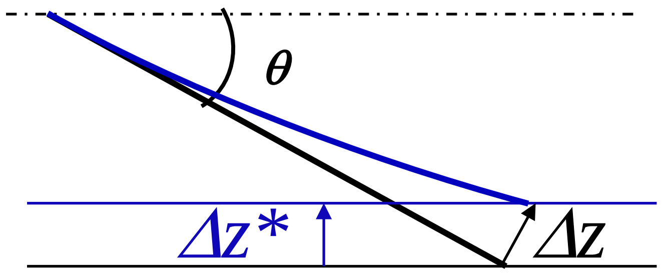
- **Influence of the position of the laser beam on the cantilever on the effective stiffness k and k^* : mode dependent correction factor**

In this course, laser beam assumed focused on point of application of the force

- **Calibration of torsional spring constant for lateral force measurement**

  For example: ***Green et al., Rev. Sci. Instrum. 75, 1988 (2004)***

$$\Delta z^* = \Delta z \cos\theta = s \Delta V$$



$$\left.\frac{\partial z}{\partial x}\right|_L = \frac{3}{2L}\Delta z$$

$$\left|\frac{\partial \Phi_n}{\partial x}\right|_L^2 = \frac{4\alpha_n^2}{L^2}\left(\frac{\sin\alpha_n \sinh\alpha_n}{\sin\alpha_n + \sinh\alpha_n}\right)^2$$