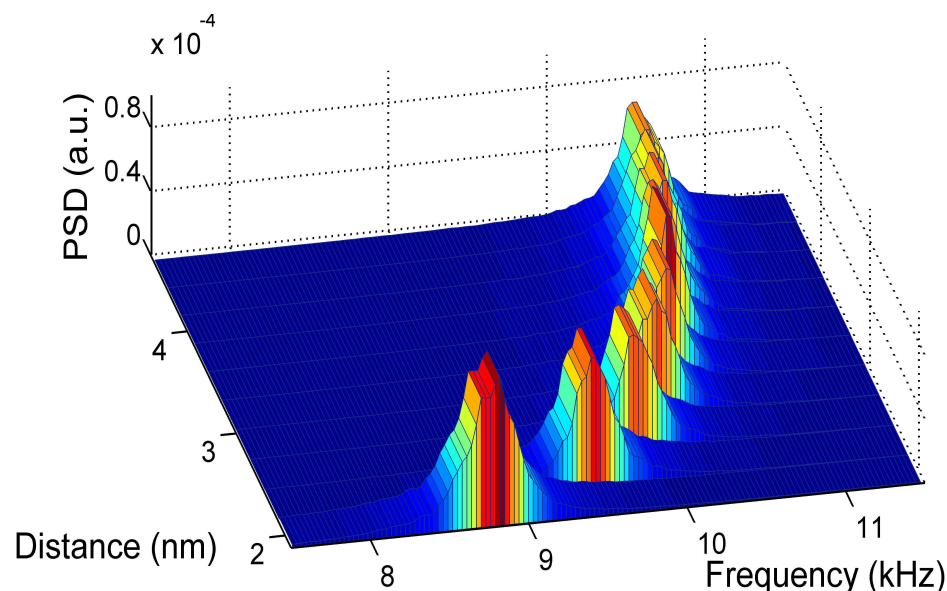




Tip-sample interaction on graphite studied in the thermal oscillations regime

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Dipartimento di Fisica, **Università degli Studi di Milano**, Milano, Italy

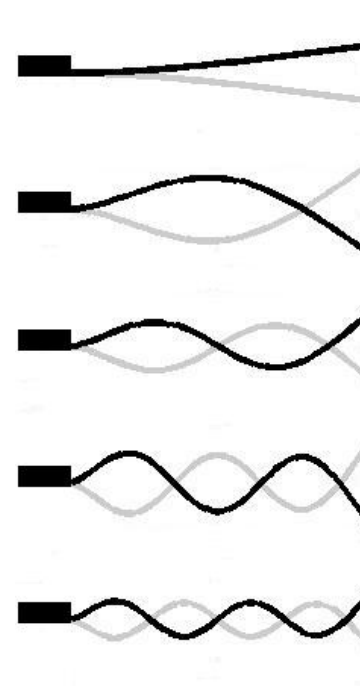


- Introduction
- Probing the tip-sample interaction
 - Frequency shift
 - Potential from Boltzmann distribution
 - Mean-square displacement from power spectral density



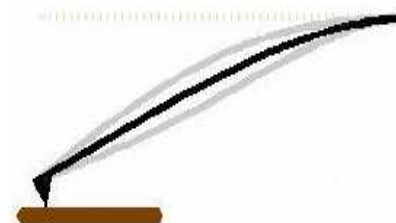
AFM cantilevers display thermal flexural oscillations

- noise source
- calibration of the cantilever spring constant k [*]
- **probing van der Waals tip-sample interaction**



[*] J.E.Sader *et al.*, *Rev. Sci. Ins.* **70**, 3967 (1999)

- The **free** AFM **cantilever** oscillates due to random thermal excitations
- As the tip approaches the sample surface, the **tip-sample interaction** deflects the cantilever and modifies its thermal vibrations → tip-sample force gradient
- The **jump-to-contact** occurs due to liquid meniscus pulling the tip (humidity) [*]



[*] M. Luna *et al.* *J. Phys. Chem B* **103**, 9576 (1999)



Free cantilever thermal motion →
Langevin equation

$$m^* \frac{d^2 u}{dt^2} + \gamma \frac{du}{dt} + ku = F_{rand}$$

- u cantilever displacement
- k spring constant
- m^* effective mass
- γ damping
- F_{rand} thermal stochastic force



$$\langle F_{rand}(t) \rangle = 0$$

$$\langle F_{rand}(t_1), F_{rand}(t_2) \rangle = \delta(t_1 - t_2)$$

V.L.Mironov *Fundamentals of SPM* (2004)

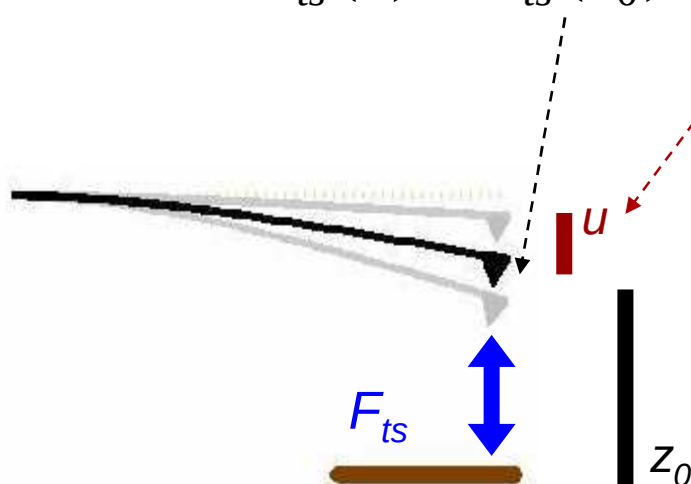
F.J.Giessibl, *Rev. Mod. Phys.* **75**, 949 (2003)

D.T.Gillespie, *Am. J. Phys.* **61**, 1077 (1993)

Cantilever near the surface $\rightarrow F_{ts}$ tip-sample interaction force

$$m^* \frac{d^2 u}{dt^2} + \gamma \frac{du}{dt} + ku = F_{rand} + F_{ts}(z)$$

$$F_{ts}(z) = F_{ts}(z_0) + \frac{\partial F_{ts}(z_0)}{\partial z} u + \dots$$



- The constant force $F_{ts}(z_0)$ only displaces the equilibrium position z_0 (static deflection)
- The force derivative $\partial F_{ts} / \partial z$ influences the cantilever oscillations



Interacting cantilever

Small amplitude oscillations →

force gradient $\partial F_{ts} / \partial z$ constant during the cantilever oscillations

$$m^* \frac{d^2 u}{dt^2} + \gamma \frac{du}{dt} + ku = F_{rand} + F_{ts}(z_0) + \frac{\partial F_{ts}(z_0)}{\partial z} u$$

$$m^* \frac{d^2 u'}{dt^2} + \gamma \frac{du'}{dt} + k^* u' = F_{rand} \quad k^* = k - \frac{\partial F_{ts}}{\partial z}$$

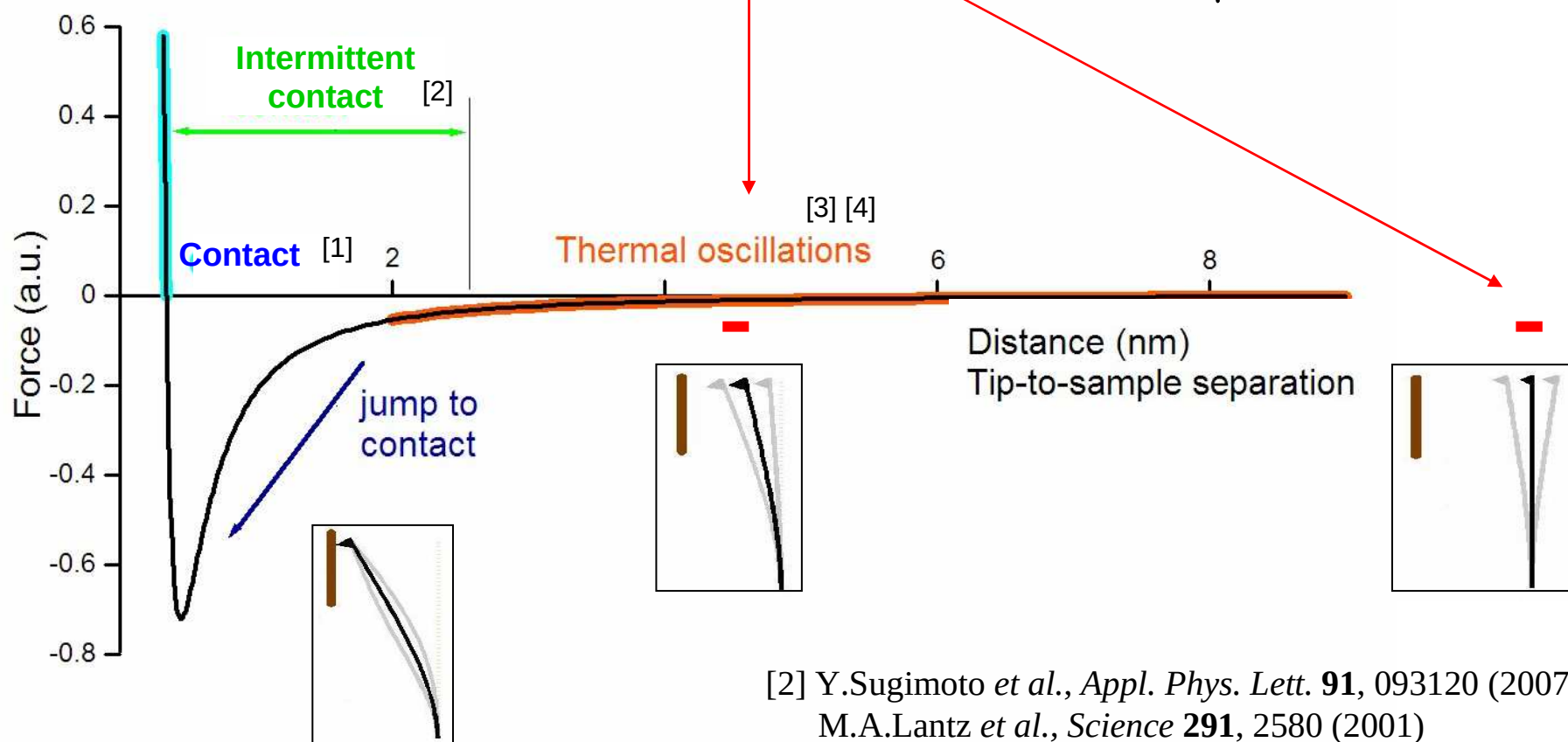
u' cantilever displacement from the new equilibrium position z_0

The tip-sample force gradient is directly related to the effective spring constant k^*



- Introduction
- Probing the tip-sample interaction
 - Frequency shift
 - Potential from Boltzmann distribution
 - Mean-square displacement from power spectral density

Thermal vibrations amplitude $\sqrt{\langle u^2 \rangle} = \sqrt{\frac{k_B T}{k}} < 0.2 \text{ nm}$



- [1] O.Pfeiffer *et al.*, *Phys. Rev. B* **65**, 161403 (2002)
T.Drobek *et al.*, *Phys. Rev. B* **64**, 045401 (2001)

- [2] Y.Sugimoto *et al.*, *Appl. Phys. Lett.* **91**, 093120 (2007)
M.A.Lantz *et al.*, *Science* **291**, 2580 (2001)
H.Hoelscher *et al.*, *Phys. Rev.Lett.* **83**, 4780 (1999)
[3] D.O.Koralek *et al.*, *Appl. Phys. Lett.* **76**, 2952 (2000)
A.Roters *et al.*, *J. Phys. Condens. Matter* **8**, 7561 (1996)
[4] J. Buchoux *et al.*, *Nanotechn.* **20**, 475701 (2009)



Experimental setup

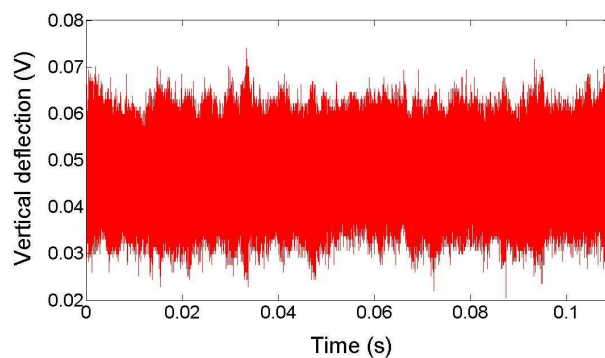
A.P.E. Research
NANOTECHNOLOGY

Acquisition system

- Bandwidth: > 1 MHz
- Buffer memory: 128MS
- Sensitivity: 50-200 nm/V

Time signal

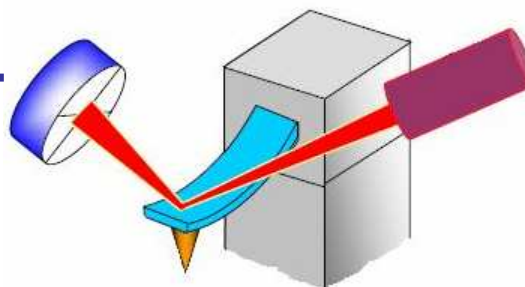
Vertical → flexion
Lateral → torsion



Off-line
analysis

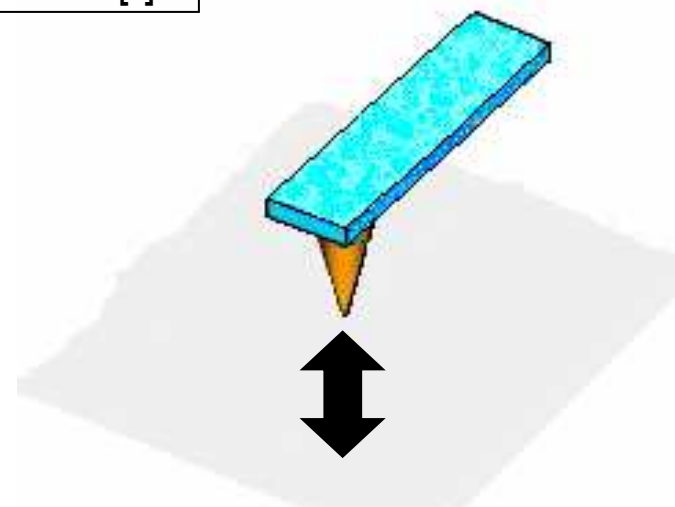
Tip-sample interaction

Quadrant
PhotoDiode



Laser

Cantilever tilt $15^\circ \rightarrow$
Sensitivity correction [*]



[*] J.L.Hutter, *Langmuir* **21**, 2630 (2005)

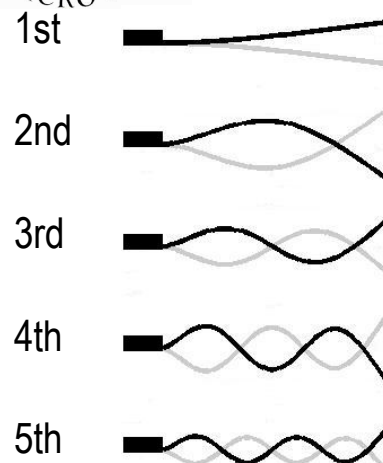


Tip-sample interaction

- One temporal trace of the cantilever thermal motion
- Three different approaches to measure the tip-sample interaction
 - Frequency shift method
 - Potential from Boltzmann distribution
 - Mean-square displacement from power spectral density



- Introduction
- Free cantilever thermal vibrations
- Probing the tip-sample interaction
 - Frequency shift
 - Potential from Boltzmann distribution
 - Mean-square displacement from power spectral density



Boundary conditions

$$f_n^F = \alpha_n^2 \frac{h}{L^2} \sqrt{\frac{E}{12\rho}}$$

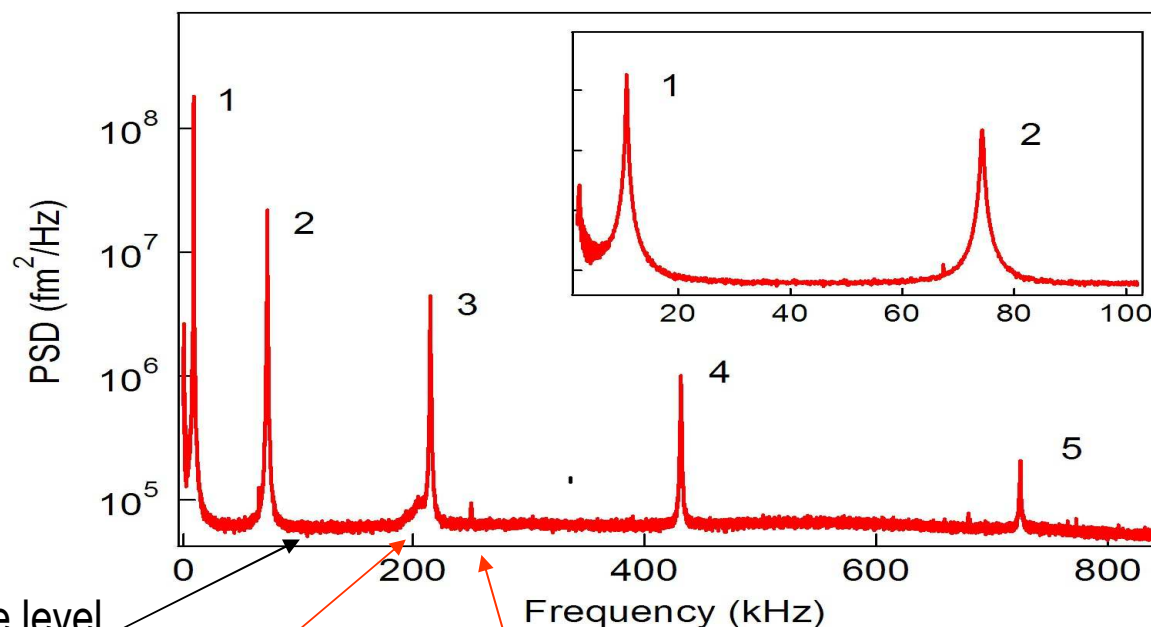
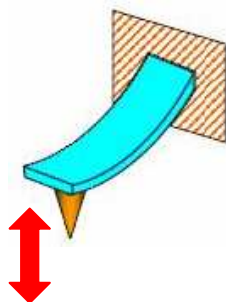
Geometrical
parameters

Material
properties

Mode	1	2	3	4	5
f_n (kHz)	10.8	74.3	215	431	724
f_n / f_1 (exp.)		6.9	20	40	67
f_n / f_1 (th. [*])		6.27	17.55	34.39	56.84



Vertical
signal



White noise level
 $\approx 250 \text{ fmHz}^{-0.5}$

1st Lateral

1st Torsional



Frequency shift method

The resonance frequency of free cantilever is

$$f_0 = \frac{1}{2\pi} \sqrt{\frac{k}{m^*}}$$

k spring constant, m^* effective mass

The tip-sample interaction F_{ts} changes the resonance frequency.
For **small amplitude oscillations**

$$k^* = k - \frac{\partial F_{ts}}{\partial z} \quad f = \frac{1}{2\pi} \sqrt{\frac{k^*}{m^*}} \quad k^* \text{ effective spring constant}$$

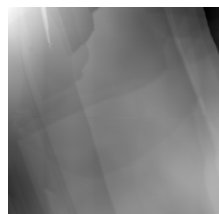
The frequency shift is directly related to the tip-sample force gradient

$$f = f_0 + \Delta f$$

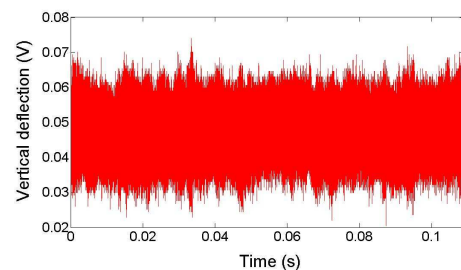
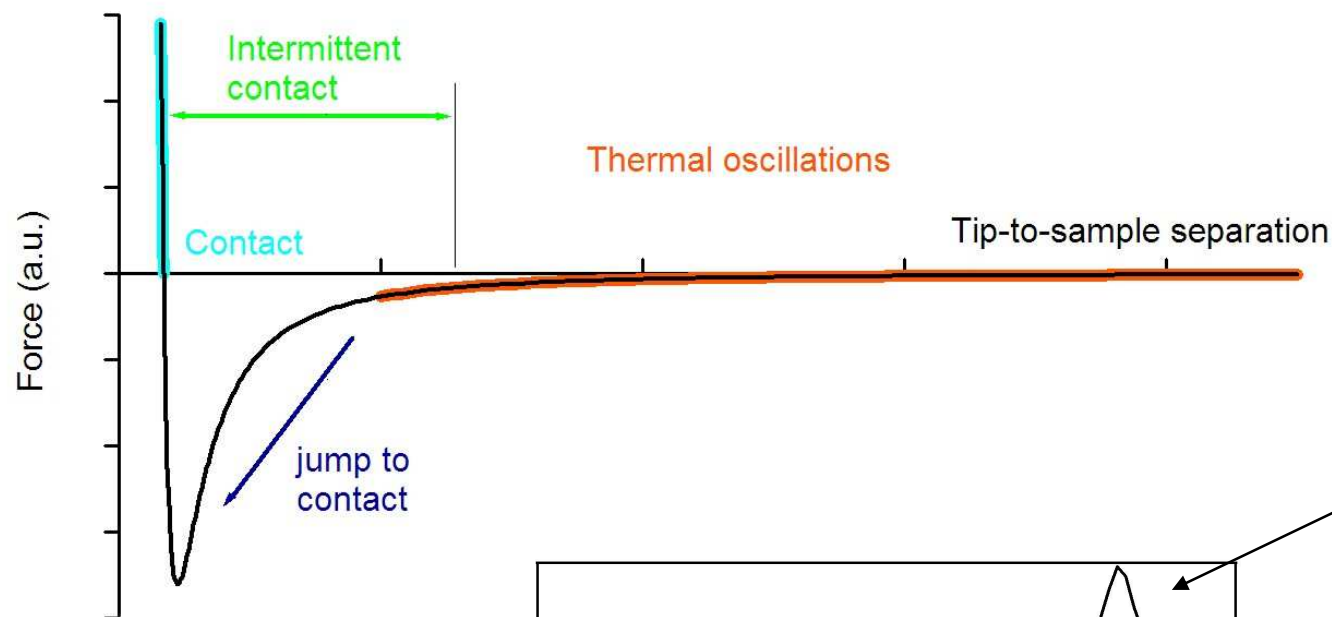
$$\frac{\partial F_{ts}}{\partial z} = -2k \frac{\Delta f}{f_0}$$

Y.Martin *et al.*, *J. Appl. Phys.* **61**, 4723 (1987)

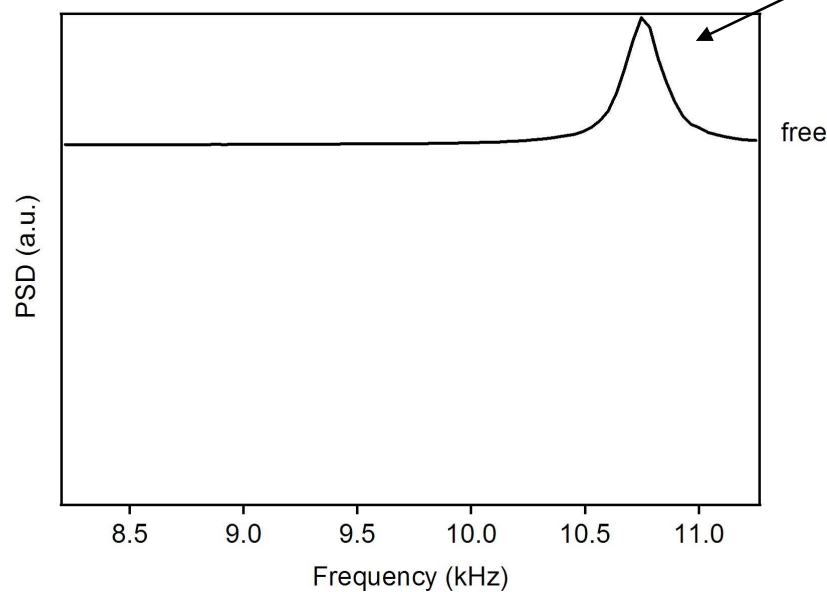
F.J.Giessibl, *Rev. Mod. Phys.* **75**, 949 (2003)

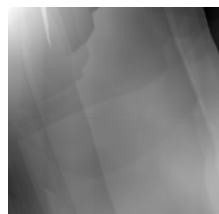


HOPG

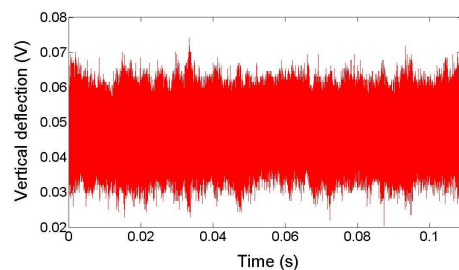
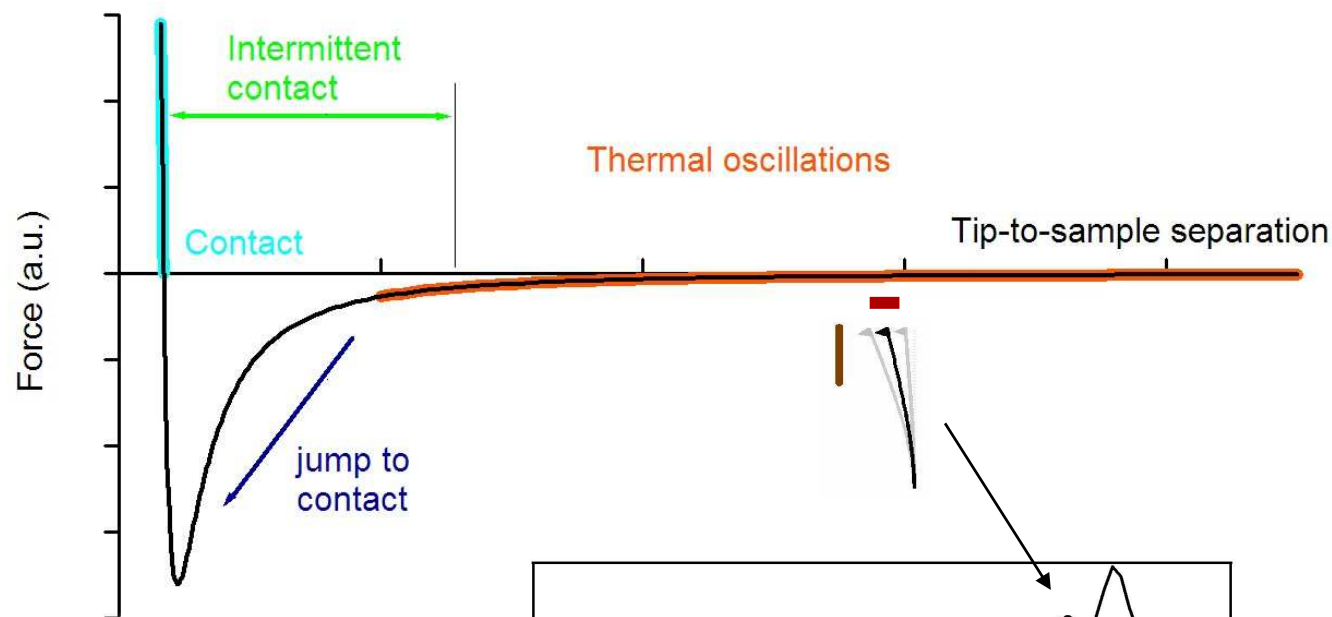


Power
Spectral
Density

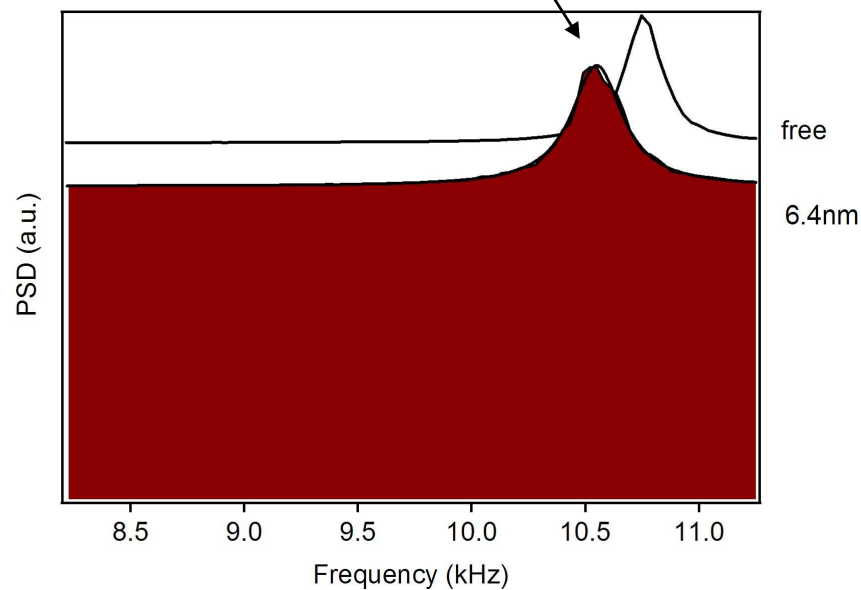


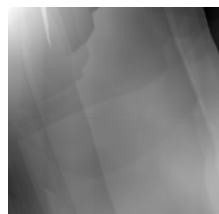


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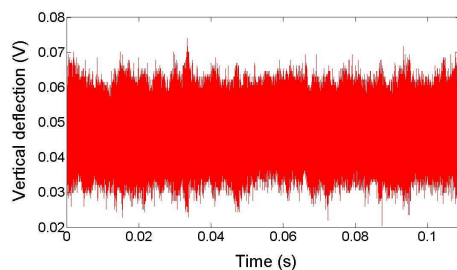
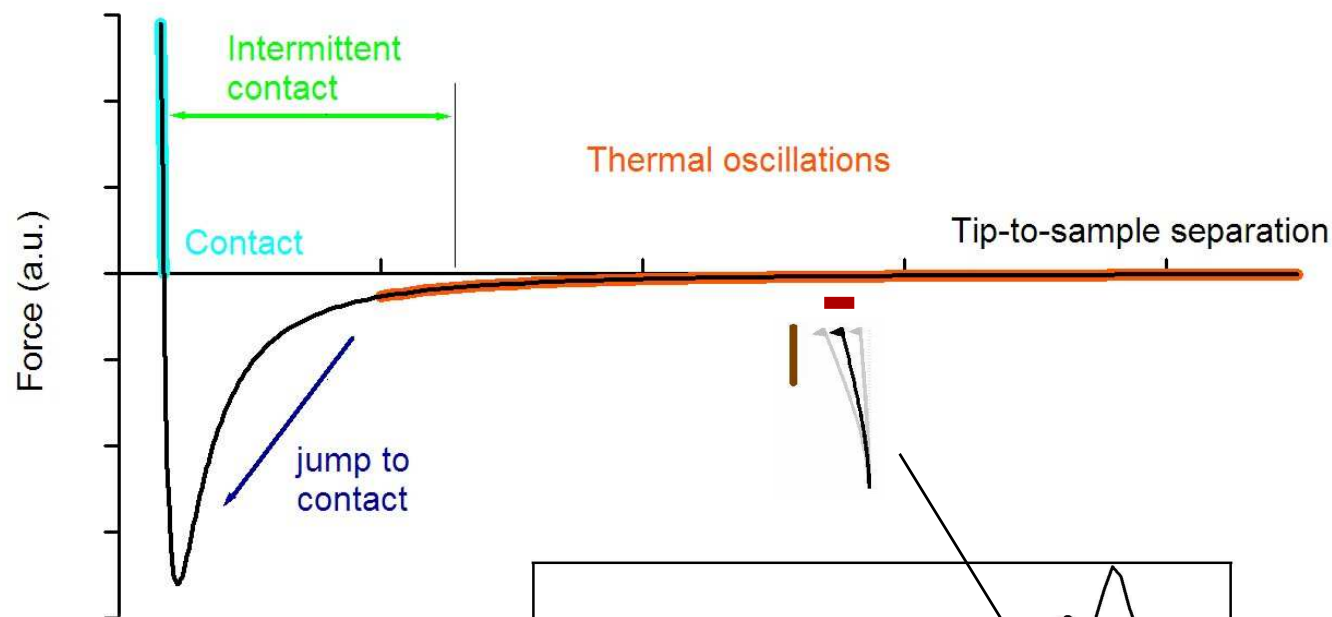


Power
Spectral
Density

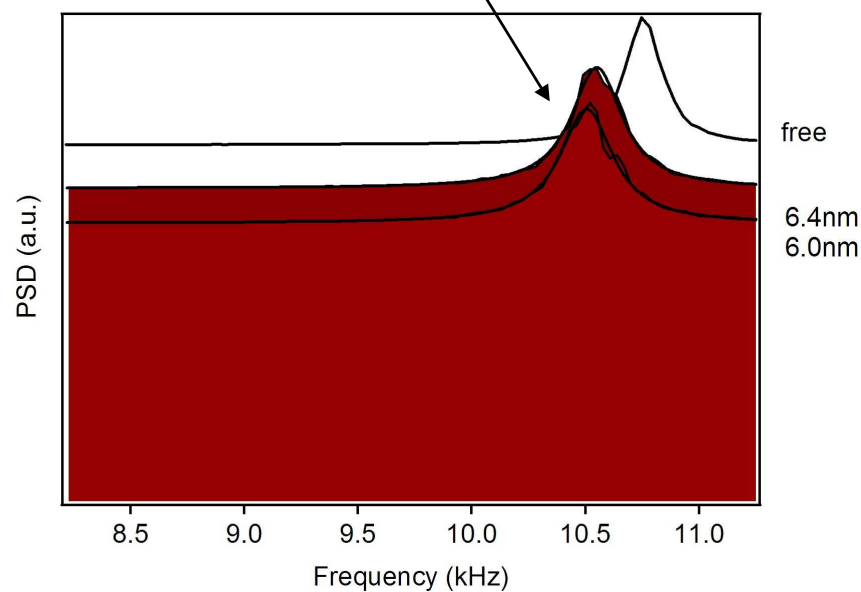


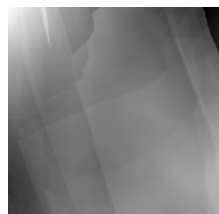


HOPG

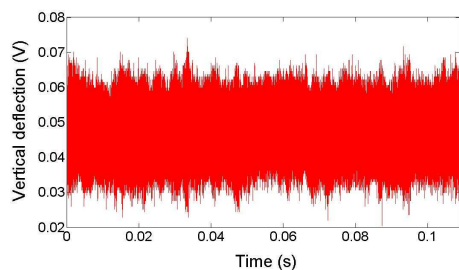
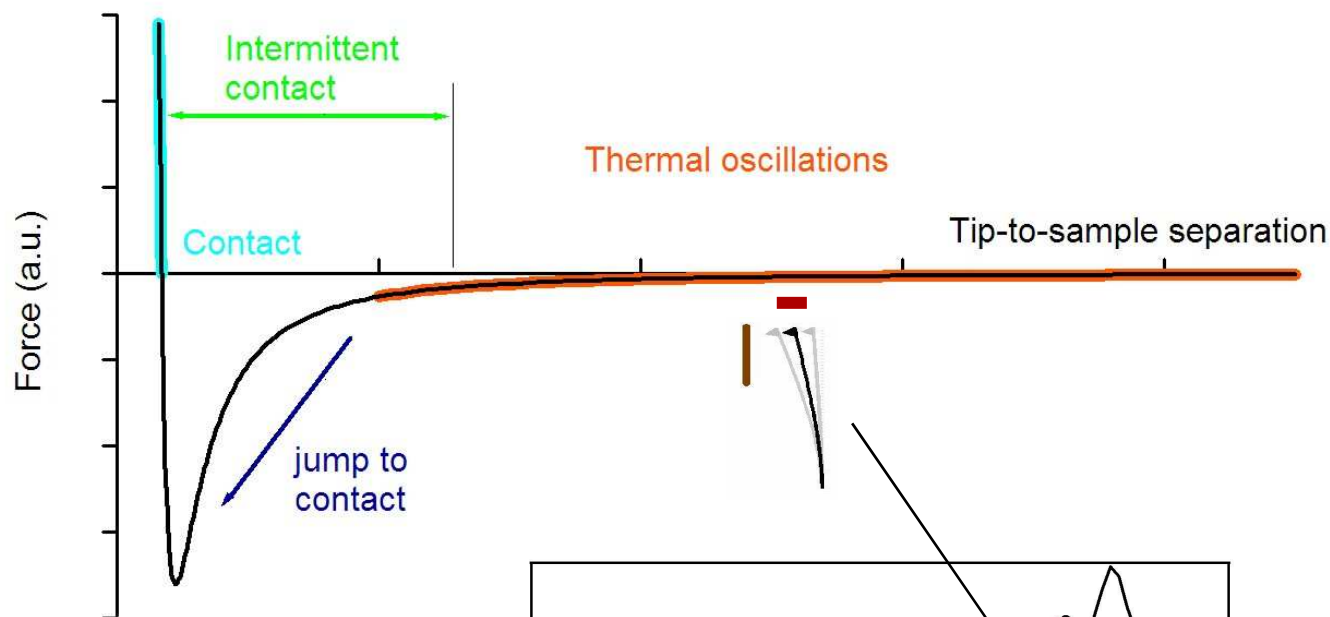


Power
Spectral
Density

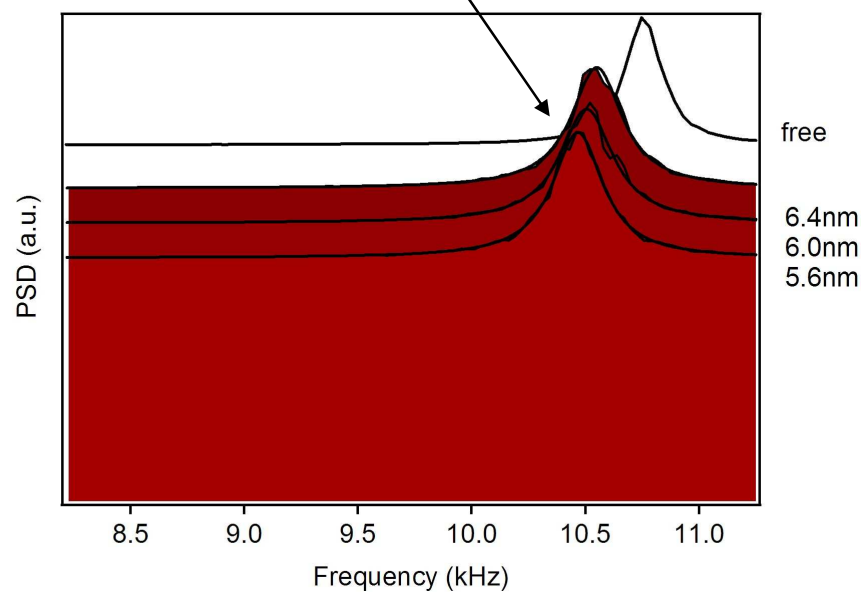


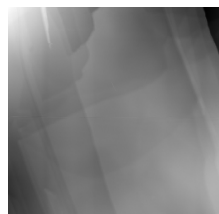


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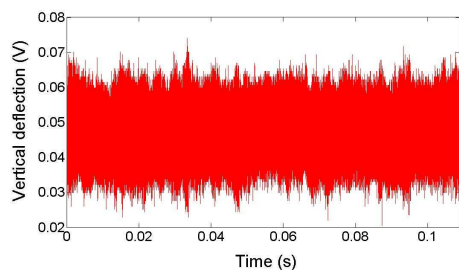
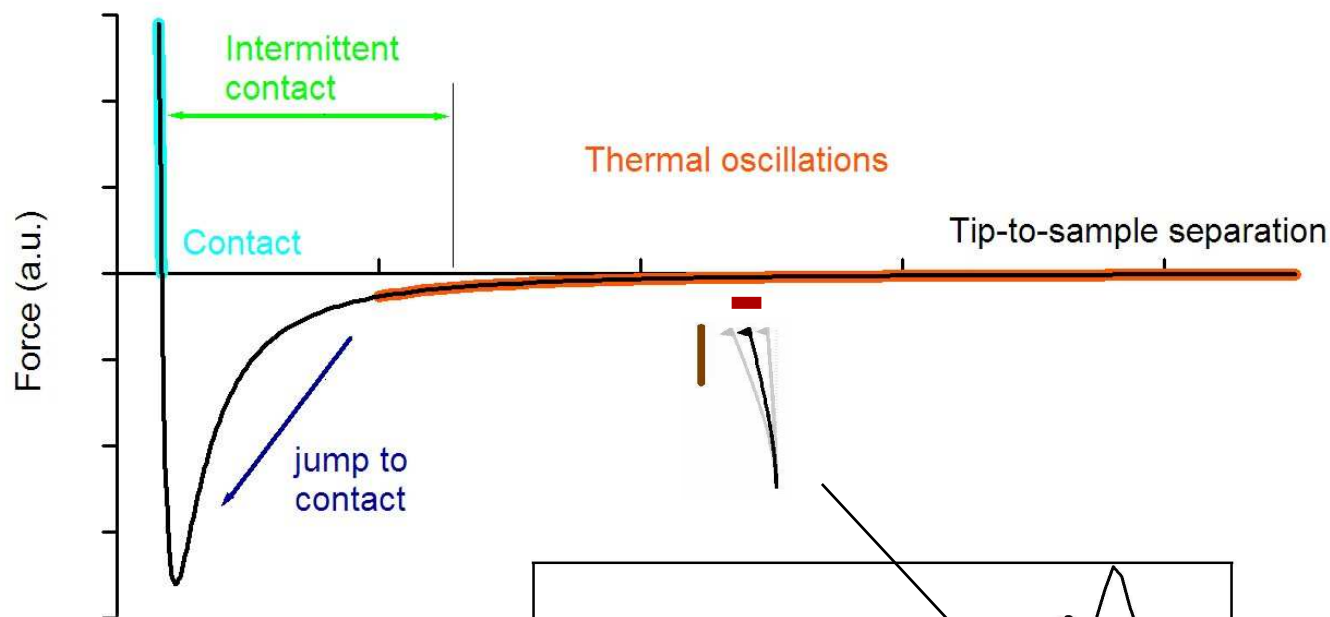


Power
Spectral
Density

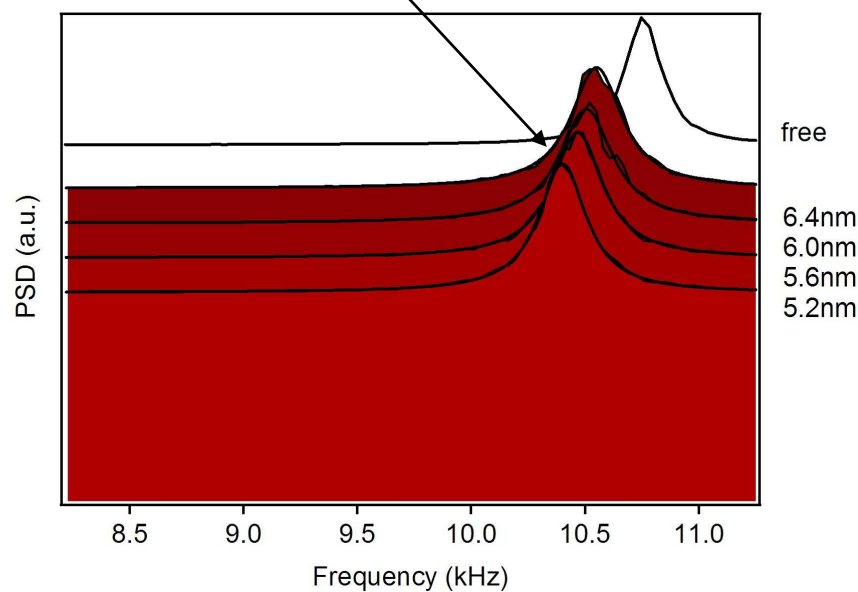


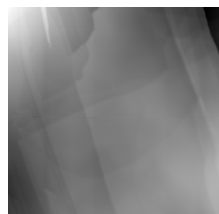


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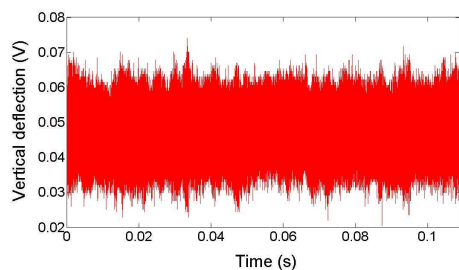
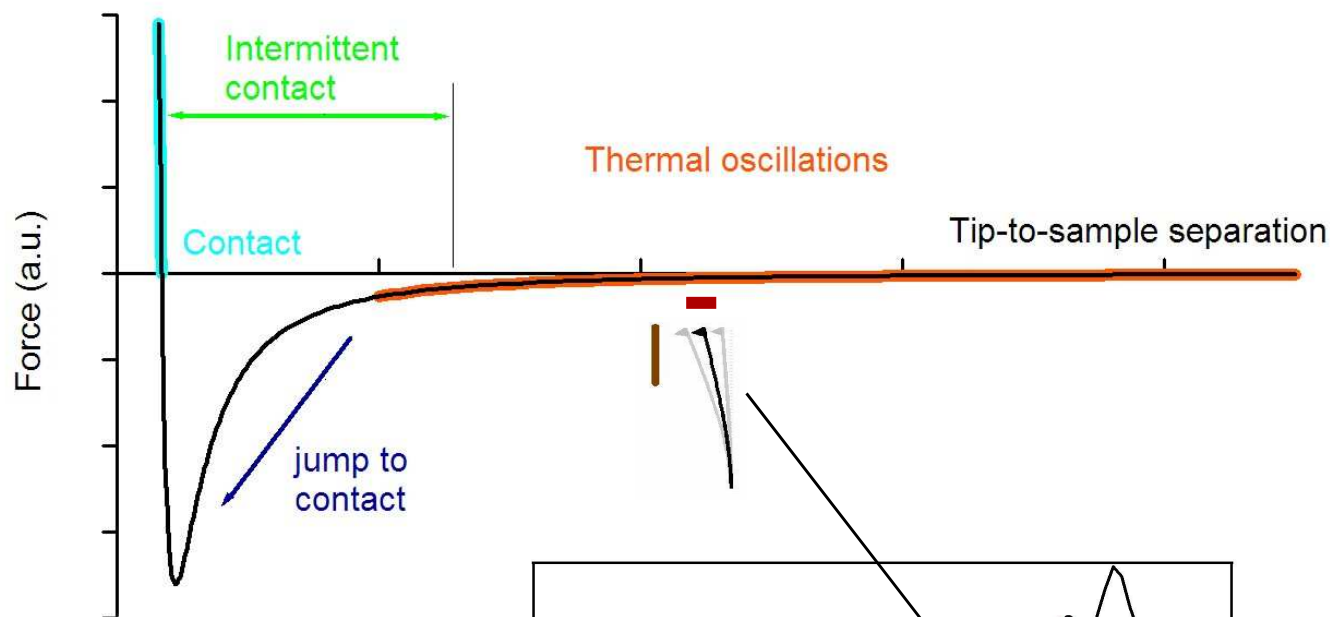


Power
Spectral
Density

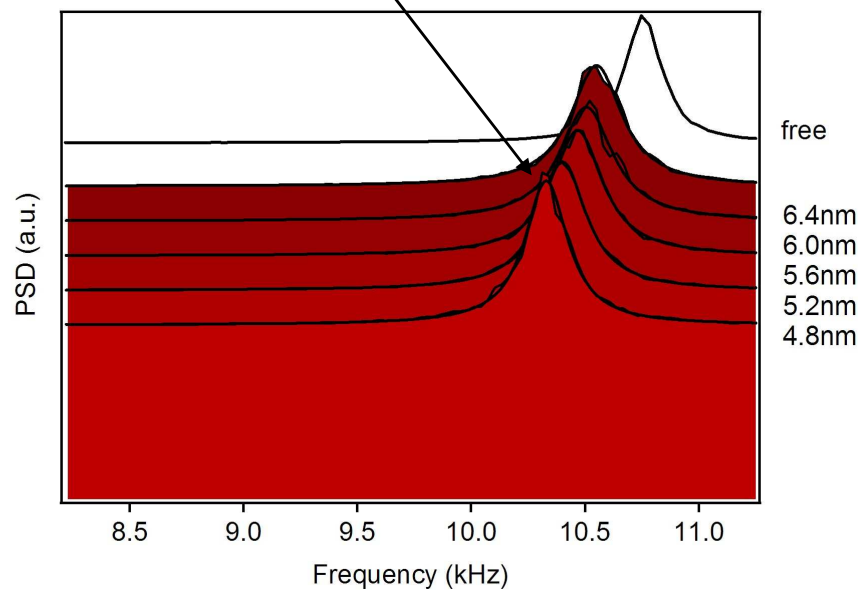


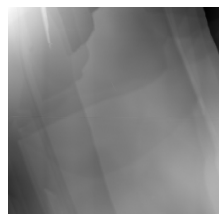


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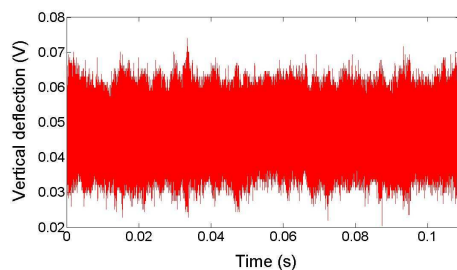
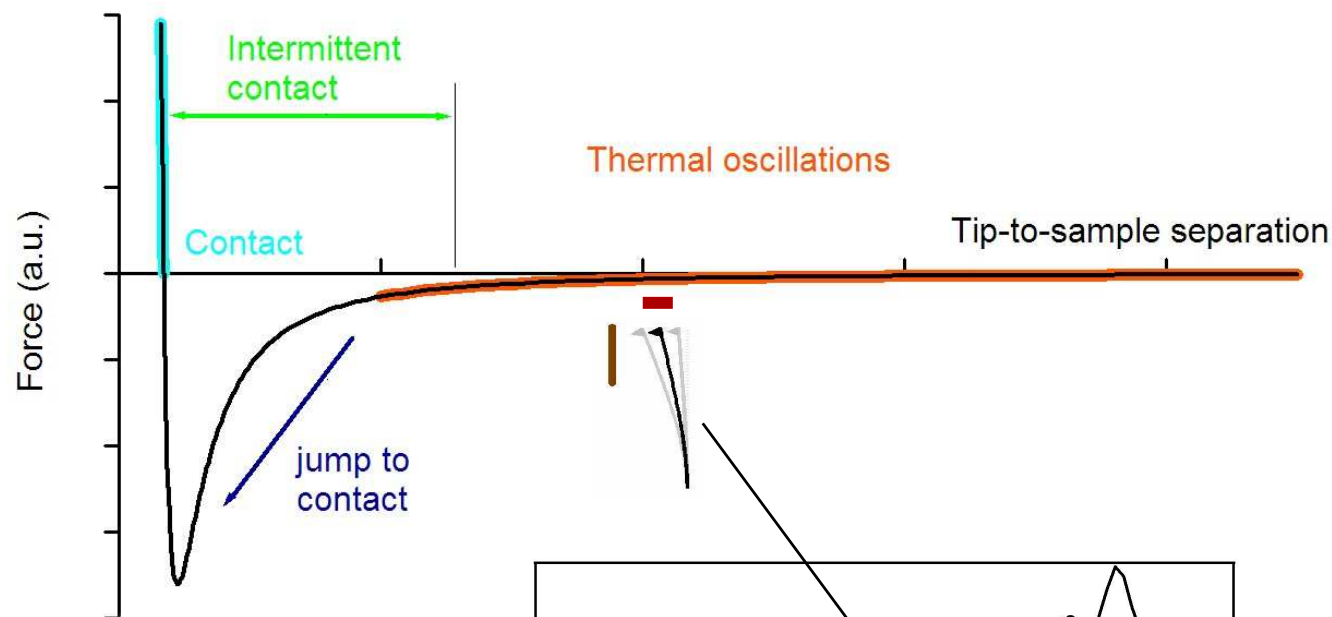


Power Spectral Density

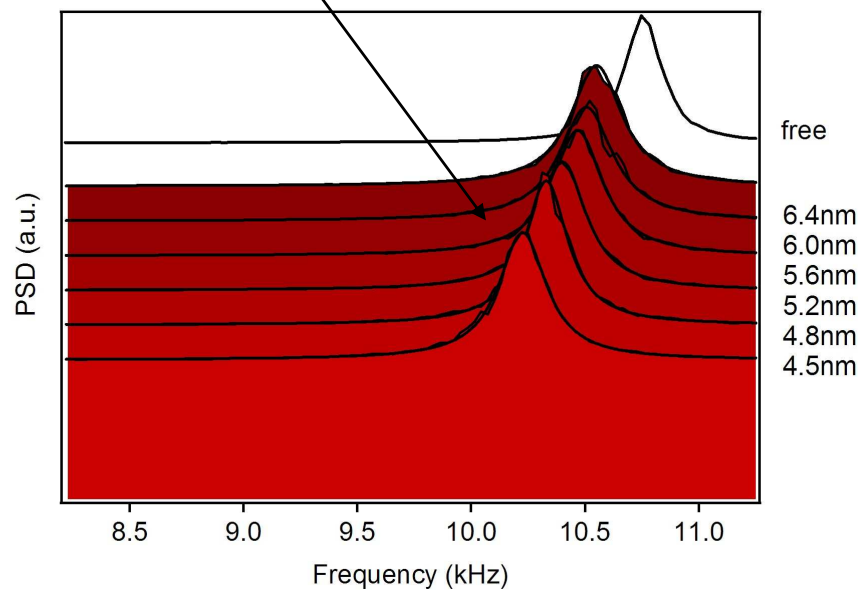


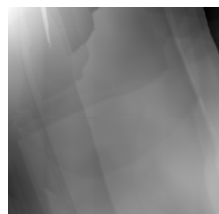


HOPG

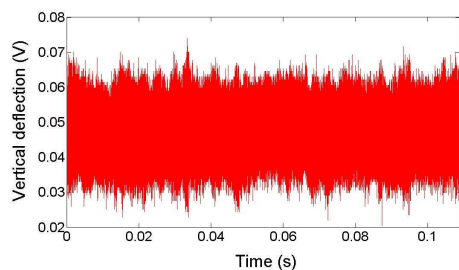
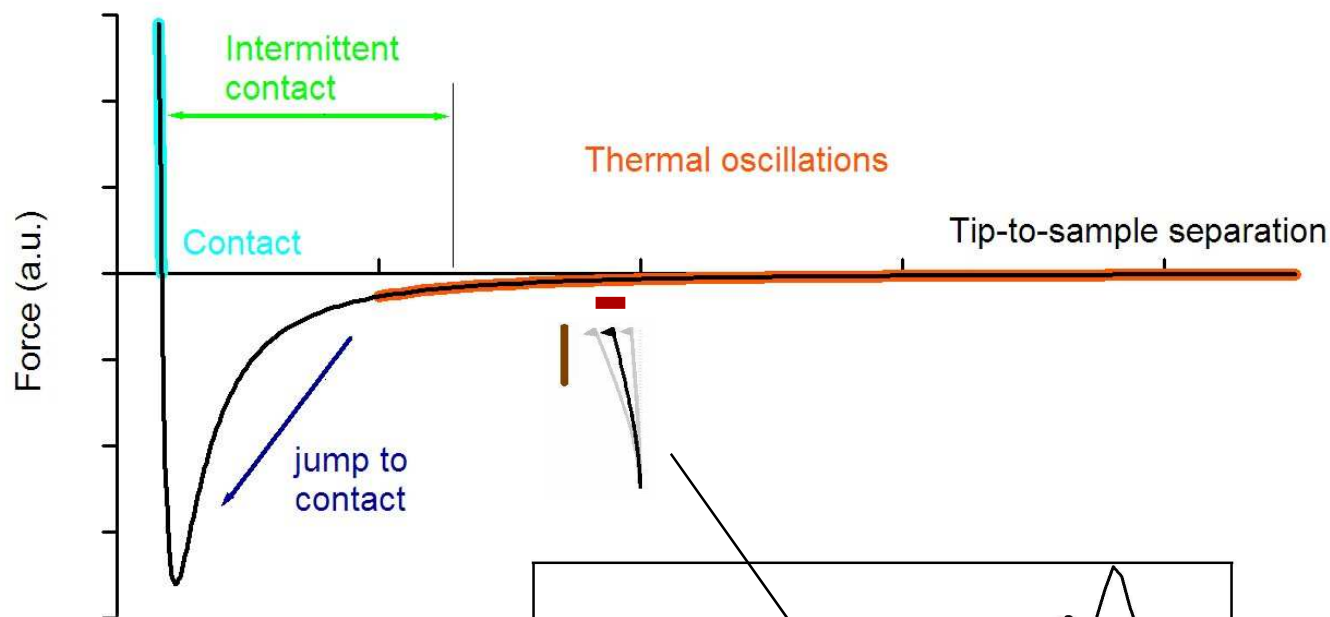


Power
Spectral
Density

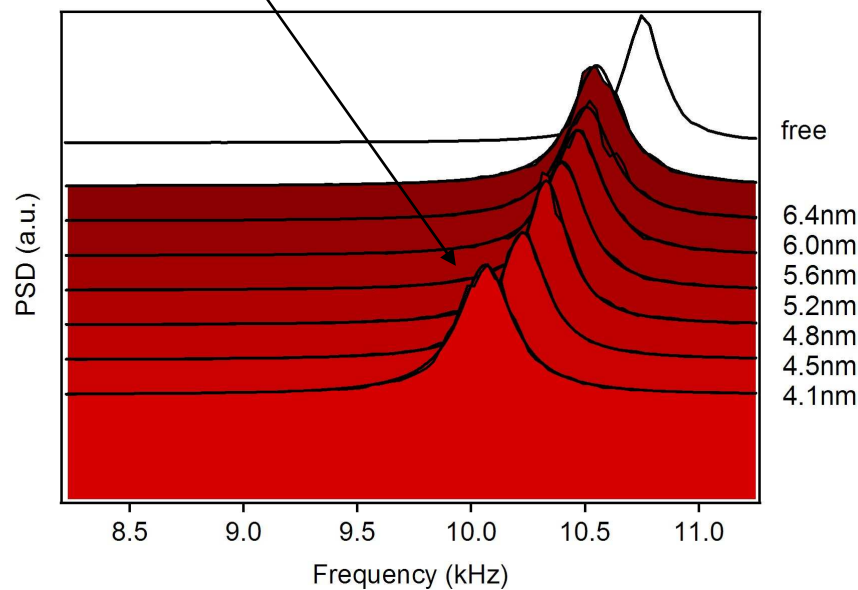


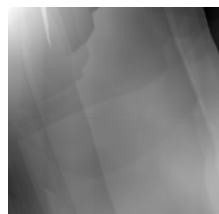


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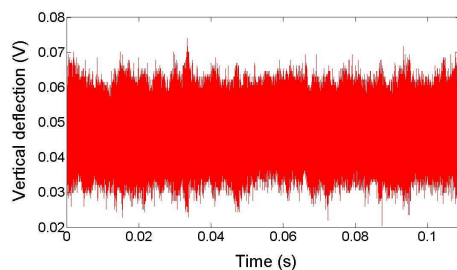
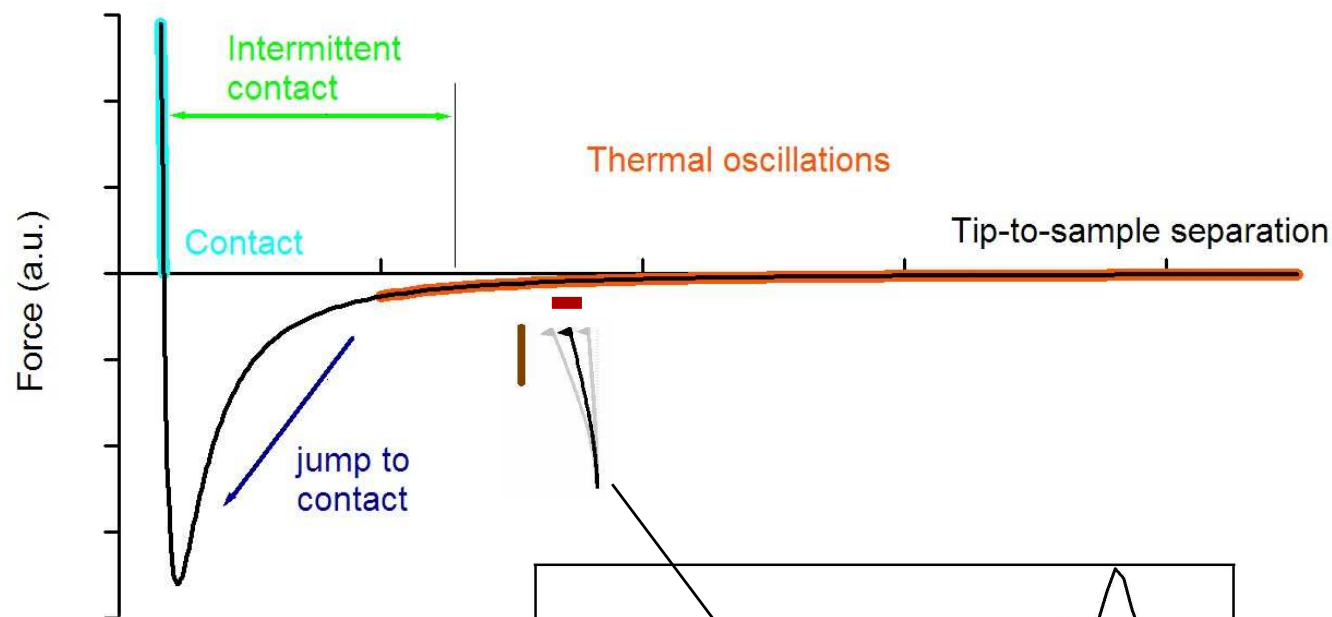


Power
Spectral
Density

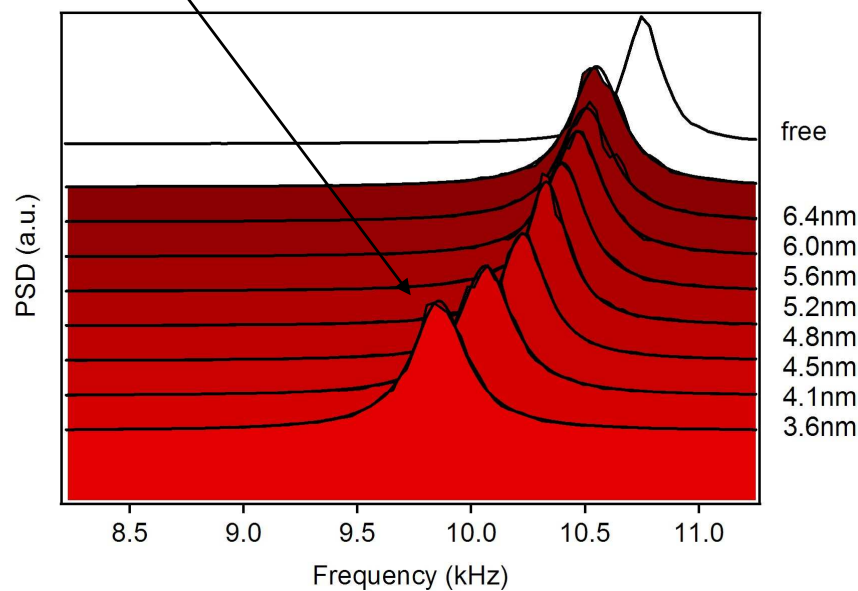


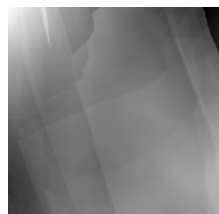


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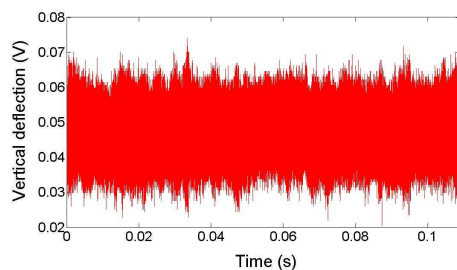
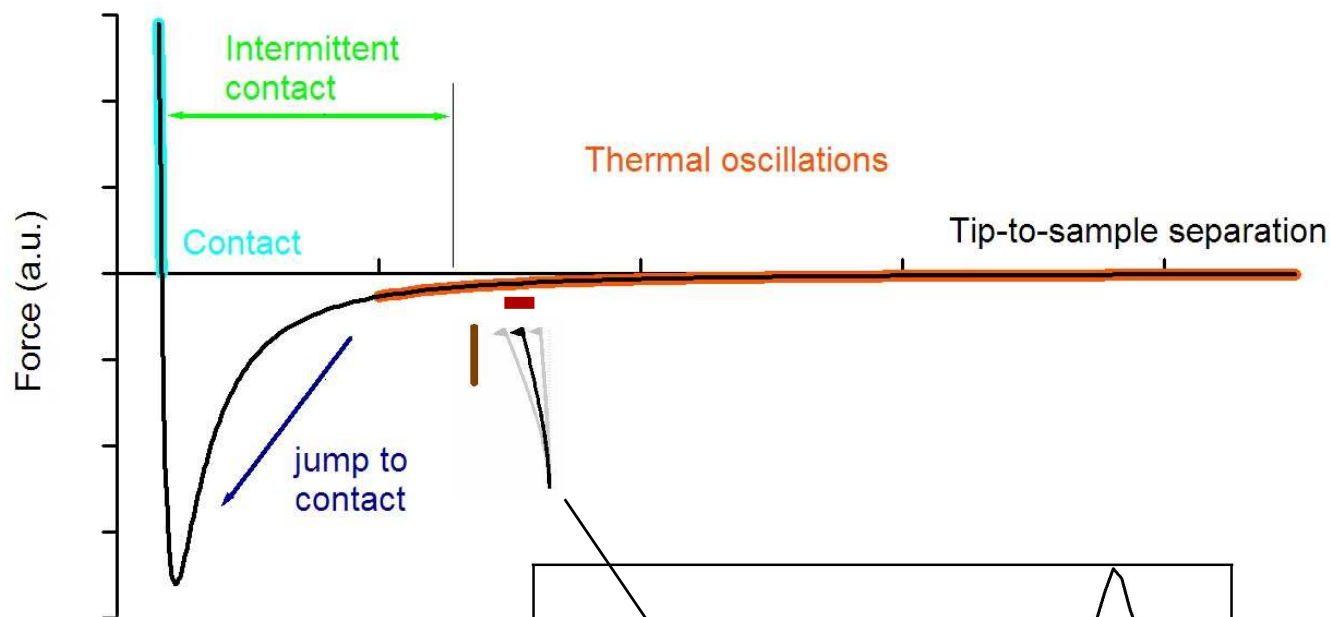


Power
Spectral
Density

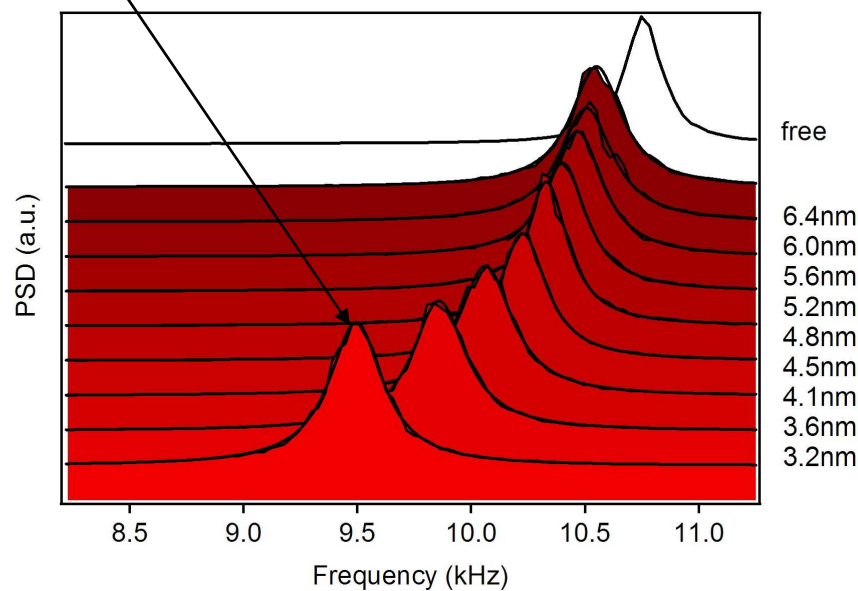


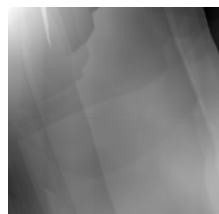


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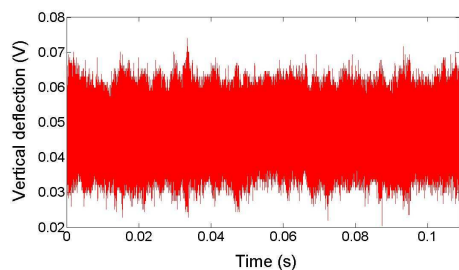
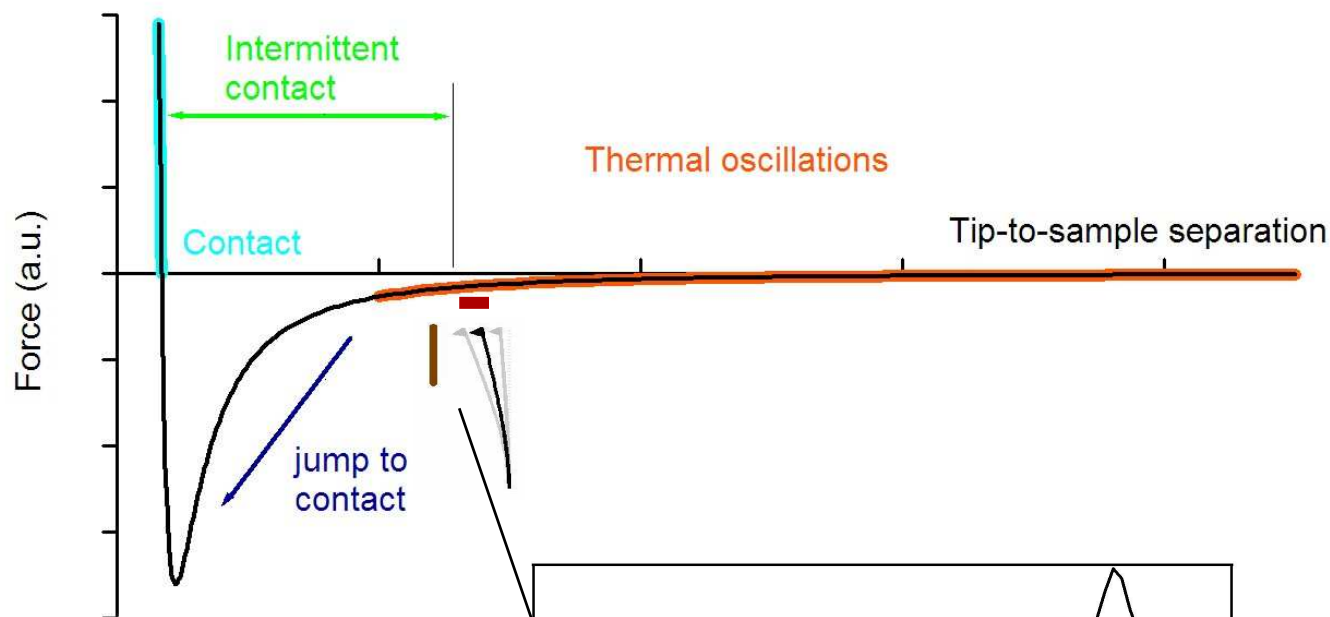


Power
Spectral
Density

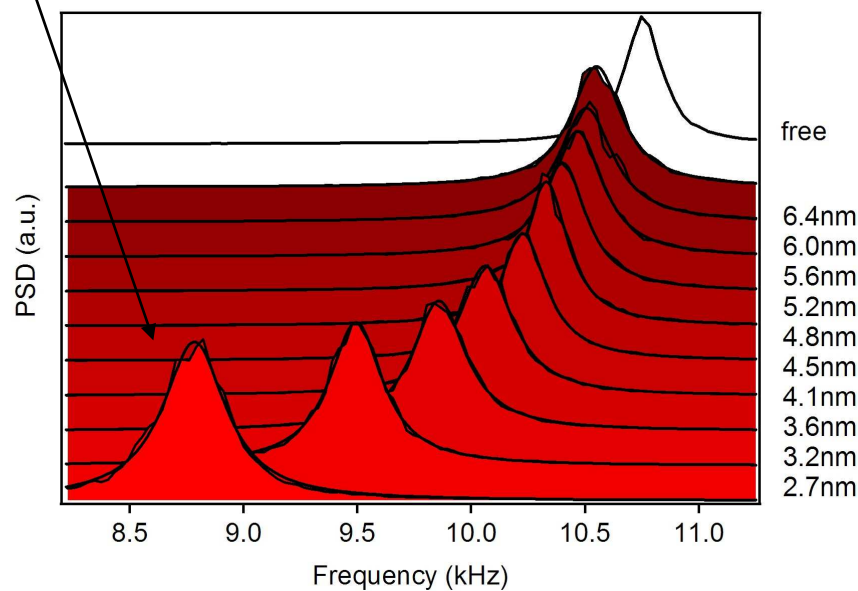


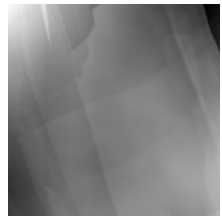


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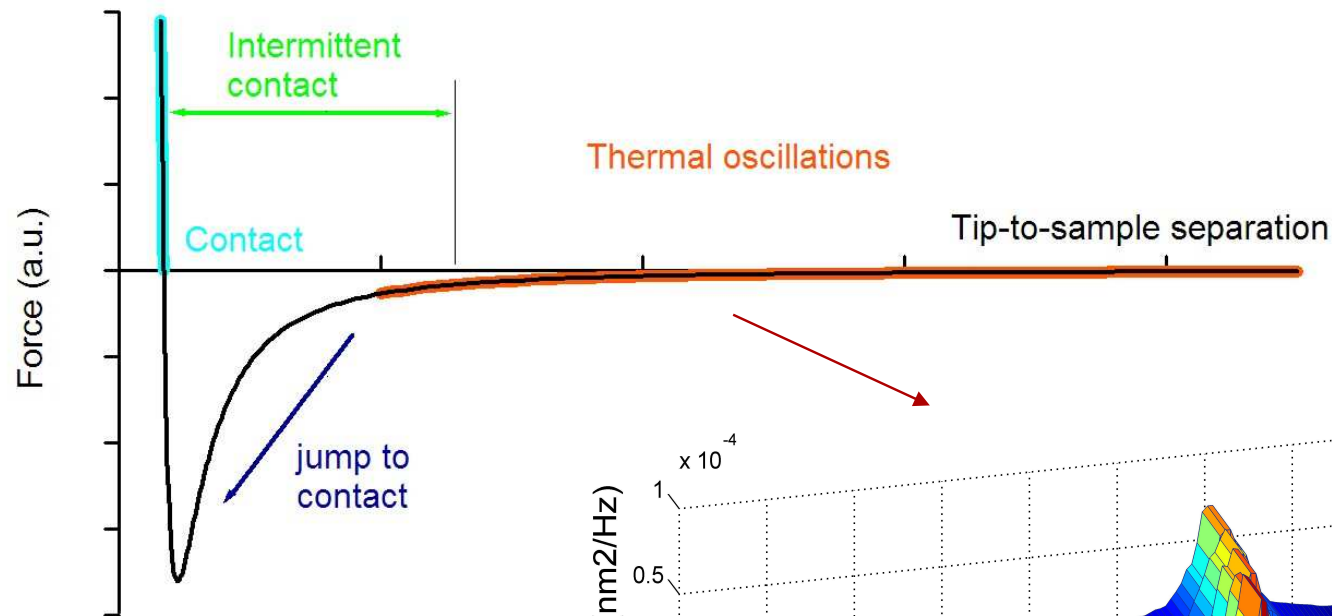


Power Spectral Density





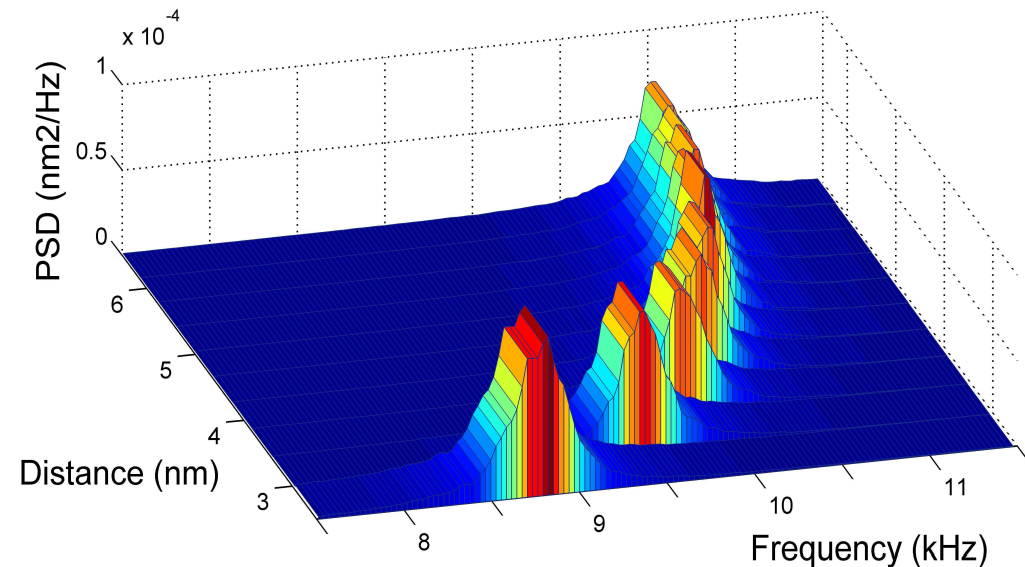
HOPG



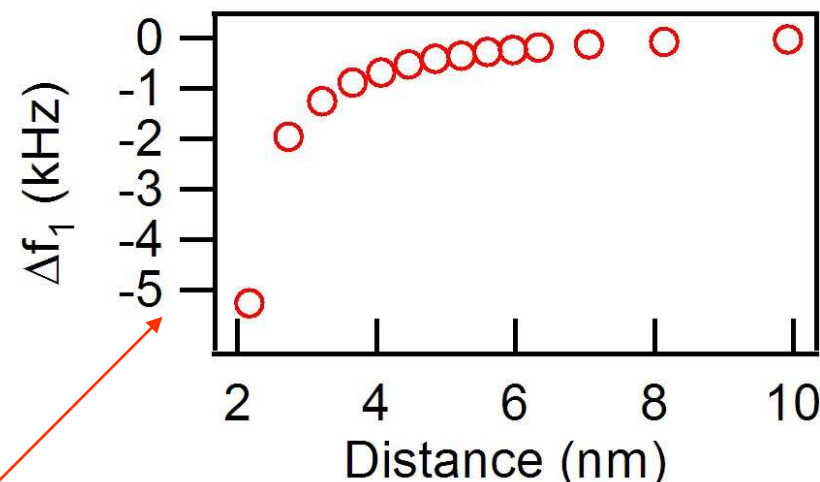
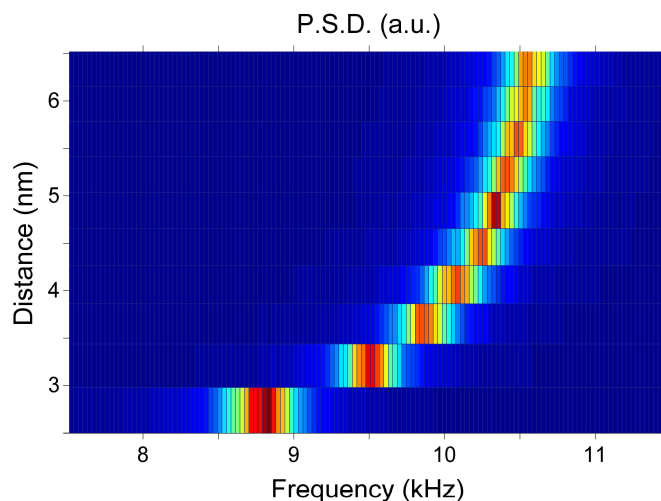
The jump-to-contact occurs when

$$\left. \frac{\partial^2 V_{ts}}{\partial z^2} \right|_{\max} = - \left. \frac{\partial F_{ts}}{\partial z} \right|_{\max} > k$$

Only long-range forces measurements (Hamaker constant) are possible



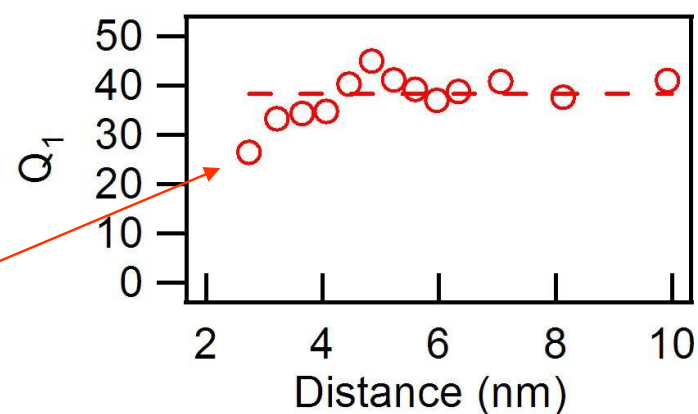
F.J.Giessibl, *Rev. Mod. Phys.* 75, 949 (2003)



Resonance frequency decreases →
Attractive tip-sample force

Q-factor constant →
Non-dissipative interaction

- Tip-sample not in contact
- Dissipation mechanism probably due to some interaction of the very end of the tip with the surface

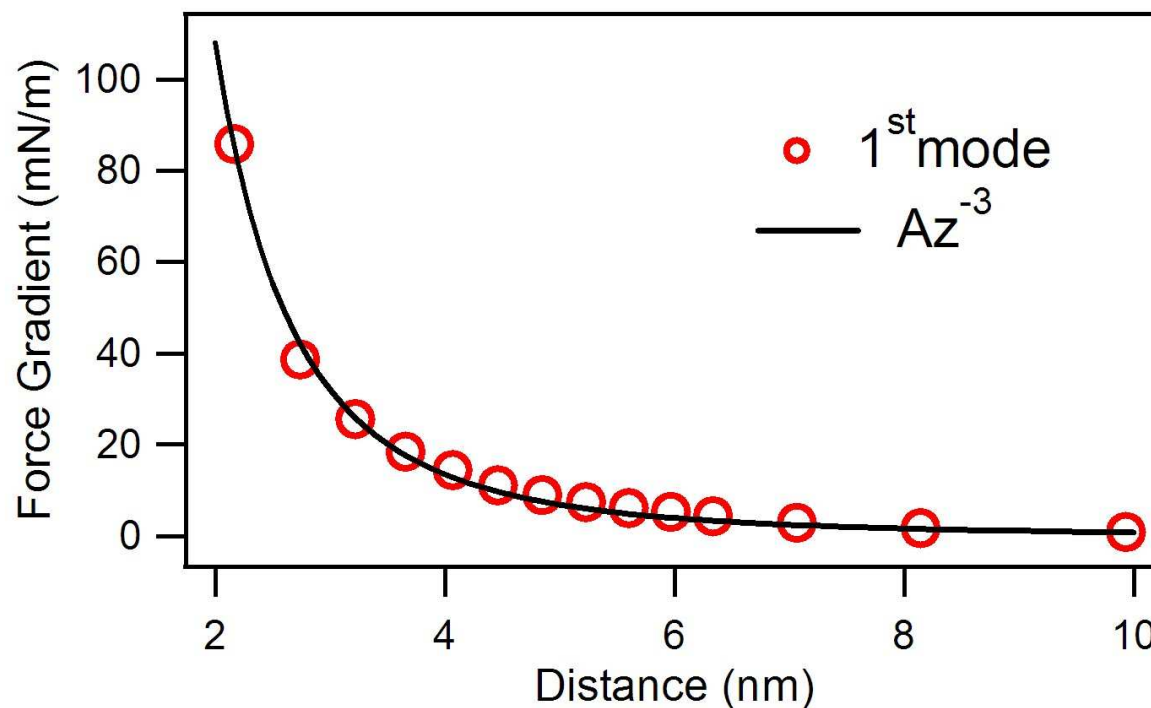




Non retarded van der Waals forces



$$\frac{\partial F_{ts}}{\partial z} = -2k \frac{\Delta f}{f_0}$$



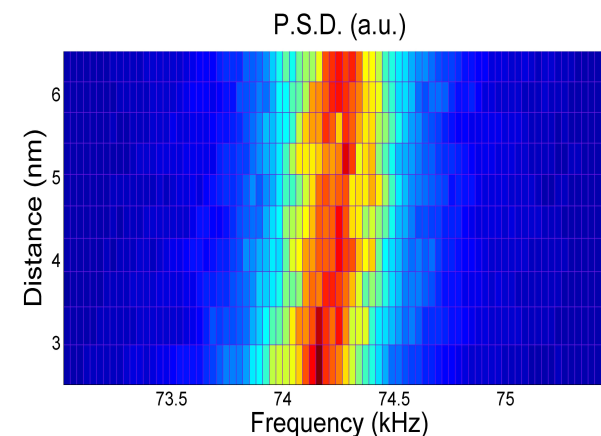
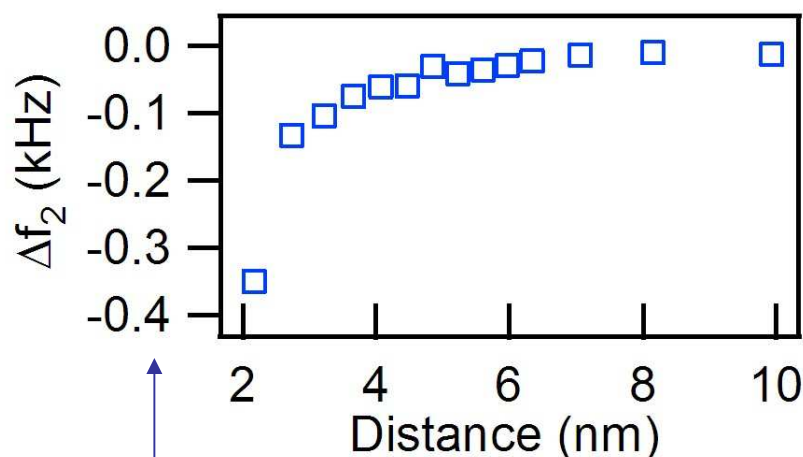
Non retarded van
der Waals force

$$\frac{\partial F_{ts}}{\partial z} = \frac{HR}{3(z - z_0)^3}$$

$$HR = 3 \cdot 10^{-27} \text{ J m}$$

z_0 surface position

H Hamaker constant, R tip radius (≈ 30 nm), $z - z_0$ tip-sample distance (piezo-tube position, z_0 surface position and cantilever static deflection $\Delta z = F_{ts} / k$)



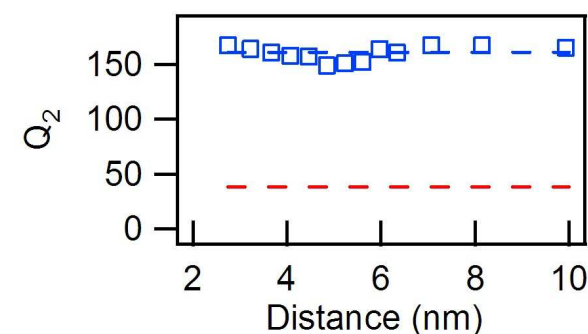
Second mode's stiffness higher than first mode → lower resonance frequency decreases

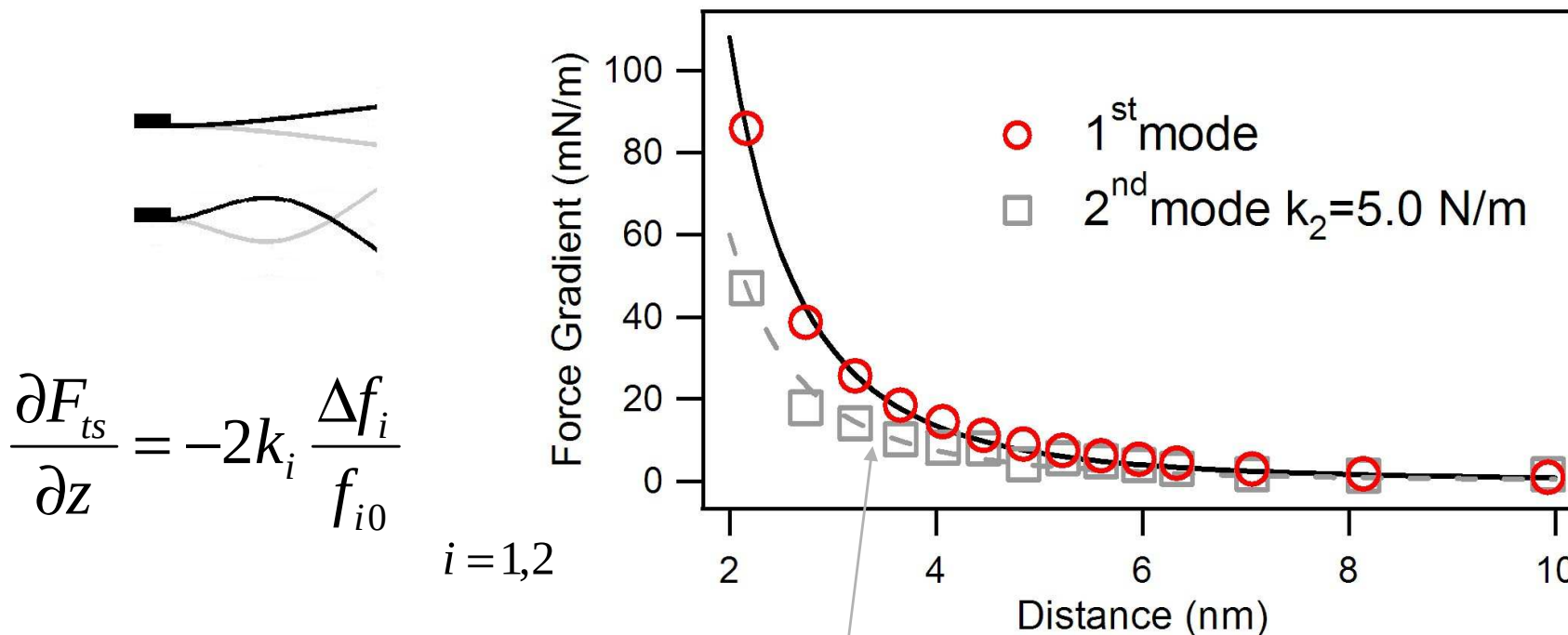
Flexural mode	1	2	3
k_n (N/m) [1]	0.12	5.0	40
k_n / k_1 (exp.)		43	350
k_n / k_1 (th. [2])		39	300

[1] J.E.Sader *et al.*, *Rev. Sci. Ins.* **70**, 3967 (1999)

[2] J.Melcher *et al.*, *Appl. Phys. Lett.* **91**, 053101 (2007)

Non-dissipative interaction



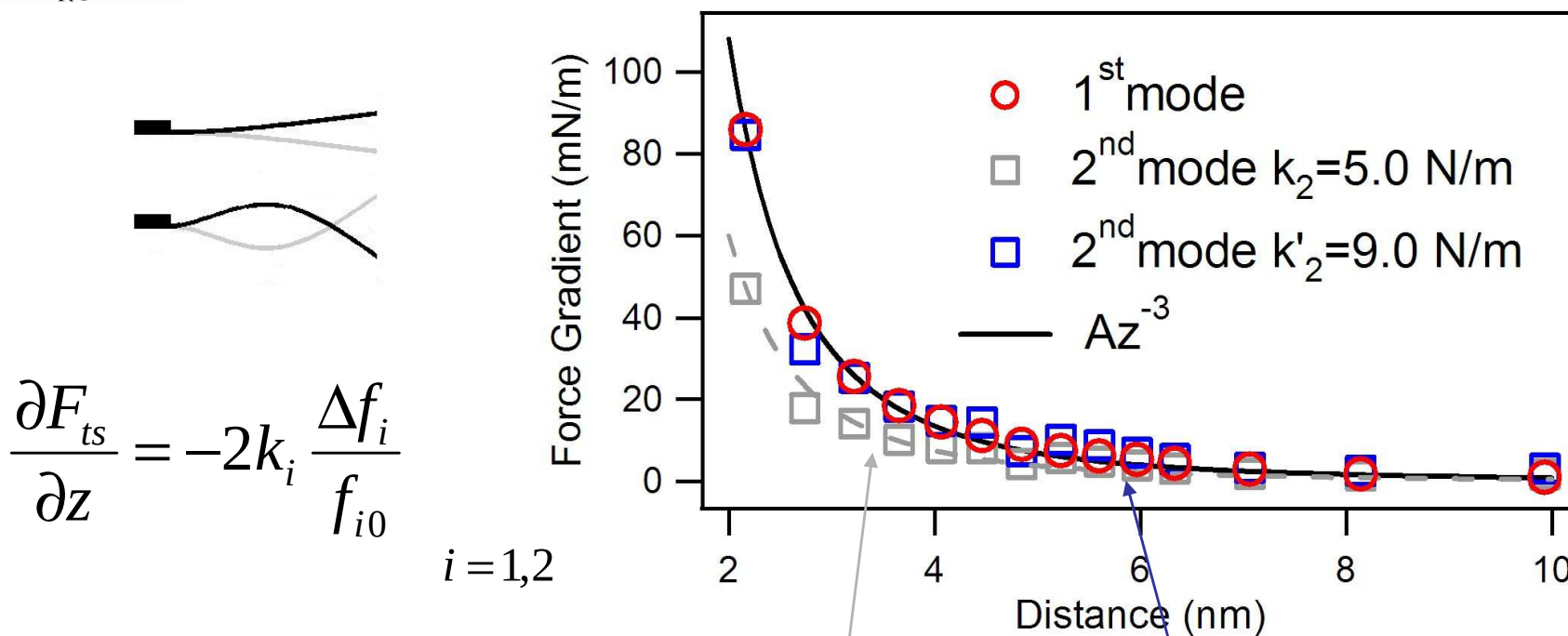


$$\frac{\partial F_{ts}}{\partial z} = -2k_i \frac{\Delta f_i}{f_{i0}}$$

$i = 1, 2$

Second mode's spring constant k_2

- by Sader method → No matching



$$\frac{\partial F_{ts}}{\partial z} = -2k_i \frac{\Delta f_i}{f_{i0}}$$

$i = 1, 2$

Second mode's spring constant k_2

- by Sader method → No matching
- Stiffer spring constant (tip mass effect) → Good matching

Tip mass loading



1st: no significant effect (1%)



2nd: the node shifts toward the free end
the cantilever effective length reduces
the equivalent stiffness increases

- A **tip mass** that is 10% of the cantilever mass nearly doubles the **second mode** equivalent stiffness. [1]
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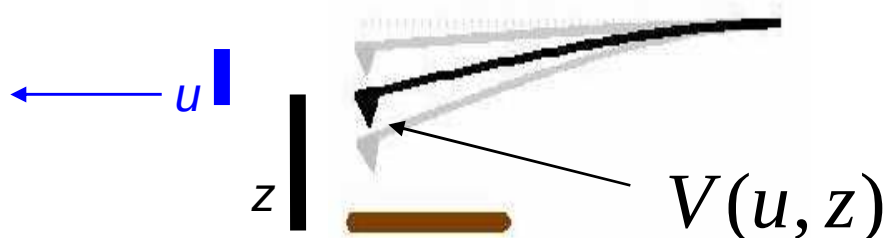
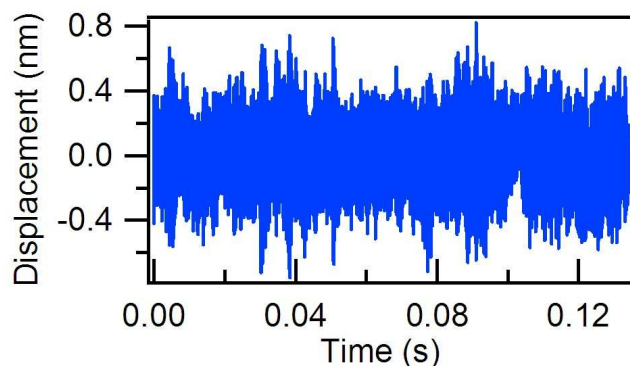
$k = 0.12 \text{ N/m}$ [2]	k_2 Sader meth.	k'_2 fit to k_{ts}	Th. no tip [1]	Th. tip 10% CL [1]
k_2 (N/m)	5.0	9.0		
k_2/k	42	75	40.2	74.9

[1] J.Melcher *et al.*, *Appl. Phys. Lett.* **91**, 053101 (2007)
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[2] J.E.Sader *et al.*, *Rev. Sci. Ins.* **70**, 3967 (1999)



- Introduction
- Free cantilever thermal vibrations
- Probing the tip-sample interaction
 - Frequency shift
 - Potential from Boltzmann distribution
 - Mean-square displacement from power spectral density

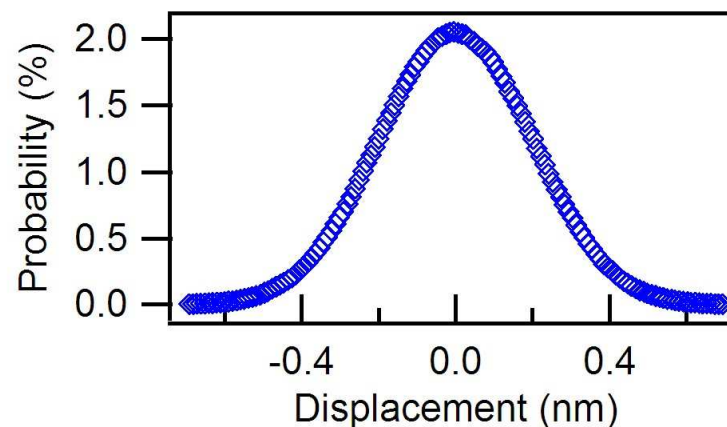


- $p(u, z)$ probability of observing the tip at a deflection u from the equilibrium position/tip-surface distance $z \rightarrow$
- number of count at the deflection u divided by the total number of count

Boltzmann probability distribution

$$p(u, z) = p_0 e^{-\frac{V(u, z)}{K_B T}}$$

$V(u, z)$ position dependent total potential



W.F. Heinz *et al.*, *J. Phys. Chem. B* **104**, 622 (2000)

D.O.Koralek *et al.*, *Appl. Phys. Lett.* **76**, 2952 (2000)



Harmonic potential

$$V(u, z) = -K_B T \ln \frac{p(u, z)}{p_0}$$

$$V(u, z) = V_C(u, z) + V_{ts}(u, z)$$

V_C cantilever
potential

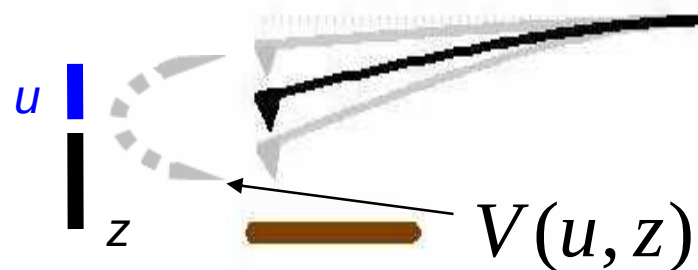
V_{ts} tip-sample interaction potential

$$V = \frac{1}{2} k u^2 - \frac{1}{2} \frac{\partial F_{ts}}{\partial z} u^2 = \frac{1}{2} k^* u^2 \quad k^* = k - \frac{\partial F_{ts}}{\partial z}$$

Small oscillation amplitude u ($\partial F_{ts} / \partial z$ constant) → harmonic potential

Tip-sample force gradient

$$\frac{\partial F_{ts}}{\partial z} = - \frac{\partial^2 V_{ts}}{\partial s^2}$$

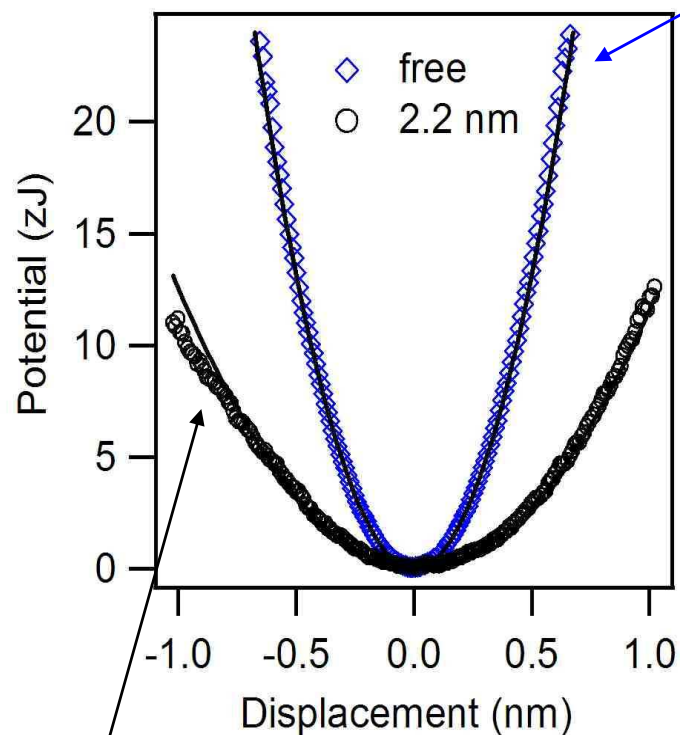


W.F. Heinz *et al.*, *J. Phys. Chem. B* **104**, 622 (2000)

D.O.Koralek *et al.*, *Appl. Phys. Lett.* **76**, 2952 (2000)

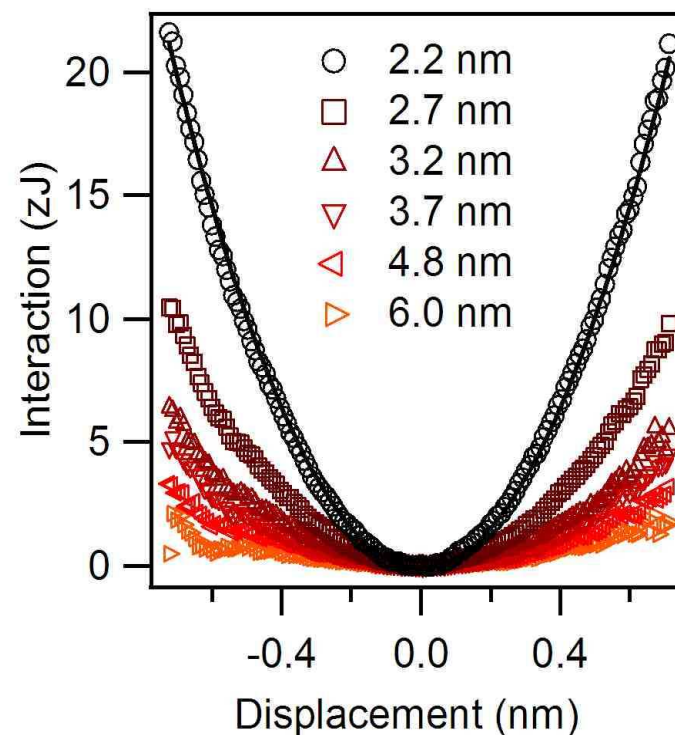
$$V_{ts} = V - V_C$$

$V(u,z)$ total potential



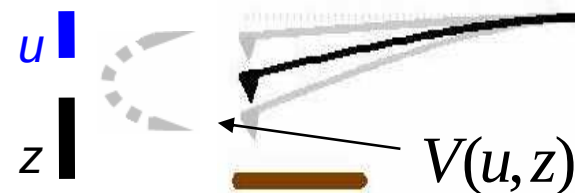
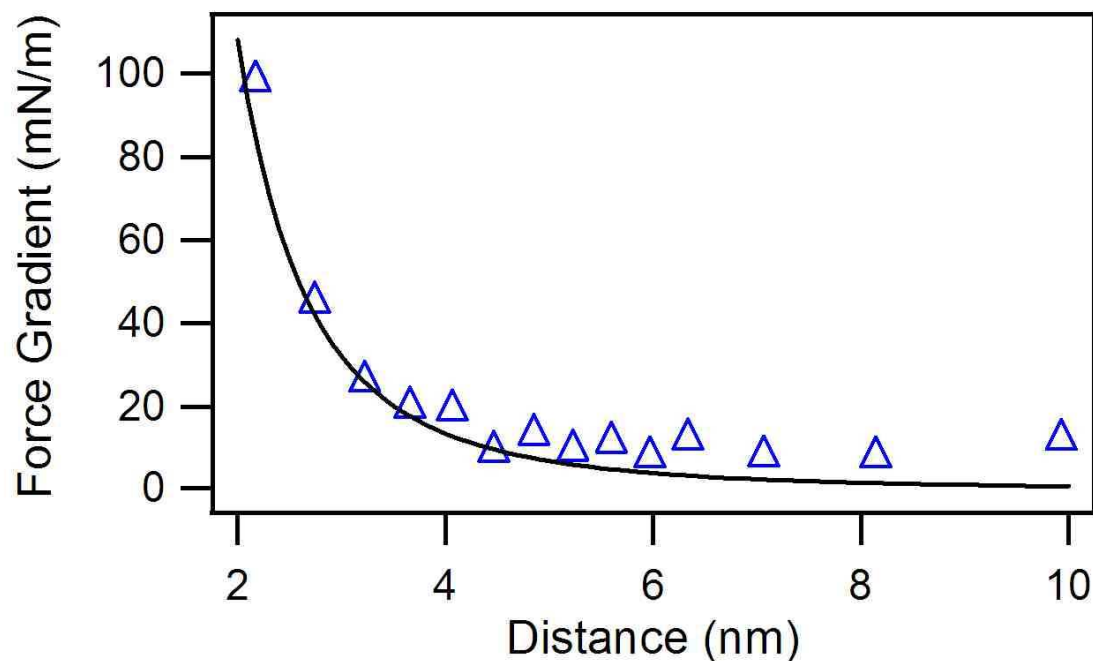
Anharmonic contribution
near the surface

$V_{ts}(u,z)$ tip-sample
interaction potential



Interaction potential **parabolic** →
mass-spring approximation correct

Tip-sample forces gradient



Boltzmann probability distribution

$$\frac{\partial F_{ts}}{\partial z} = -\frac{\partial^2 V_{ts}}{\partial s^2}$$

Frequency shift method

$$\frac{\partial F_{ts}}{\partial z} = \frac{HR}{3(z - z_0)^3}$$

$HR = 3 \cdot 10^{-27} \text{ J m}$
from the frequency shift data fitting



- Introduction
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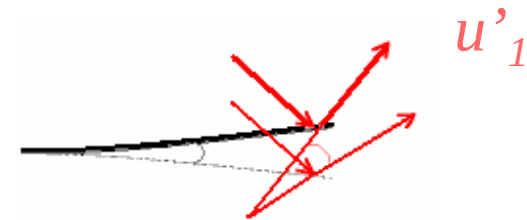
Mean-square displacement from PSD

$$\frac{1}{2}K_B T = \frac{1}{2}k \langle u^2 \rangle$$

Equipartition Theorem

k free cantilever spring constant

$\langle u^2 \rangle$ cantilever mean square deflection



The first mode mean square virtual deflection $\langle u_1'^2 \rangle$ and then the effective spring constant k^* are evaluated at different tip-sample distances z

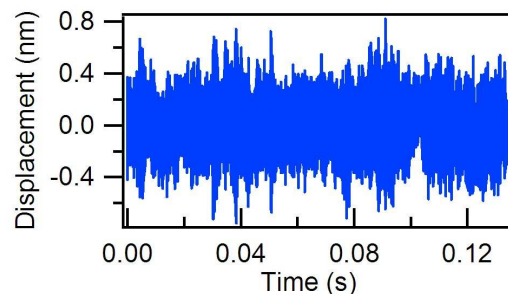
$$k^* = 0.82 \frac{K_B T}{\langle u_1'^2 \rangle}$$

$$\frac{\partial F_{ts}}{\partial z} = k - k^*$$

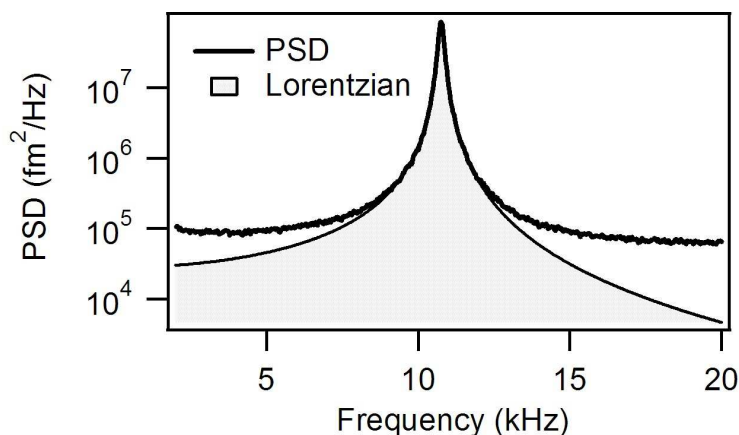
H.J.Butt *et al.*, *Nanotechnology* **6**, 1 (1995)

R.Lévy *et al.*, *Nanotechnology* **13**, 33 (2002)

Time domain → electronic noise adds to the cantilever virtual deflection



Frequency domain → thermal oscillations isolated from white noise background



$\langle u_1'^2 \rangle$ equals the integral of the power spectrum of the thermal fluctuation alone (Lorentzian function) → fit minus white-noise background



Mean-square
displacement from P.S.D.



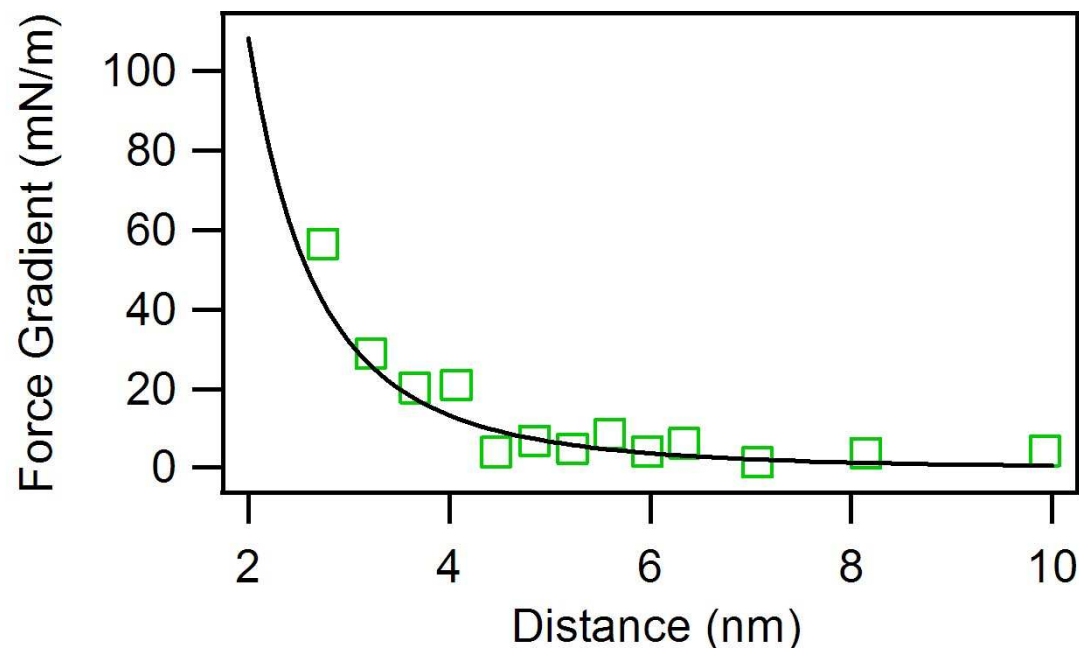
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$$\frac{\partial F_{ts}}{\partial z} = k - k^*$$

Frequency shift method

$$\frac{\partial F_{ts}}{\partial z} = \frac{HR}{3(z - z_0)^3}$$

Tip-sample forces gradient

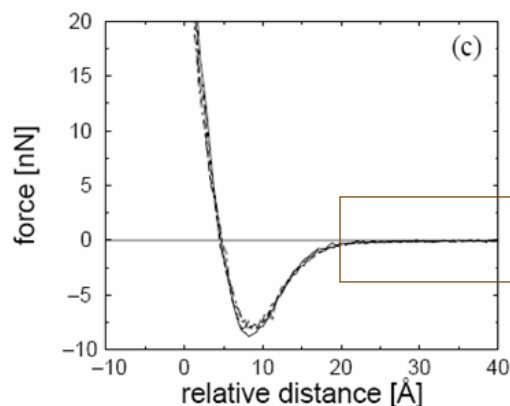


$$HR = 3 \cdot 10^{-27} \text{ J m}$$

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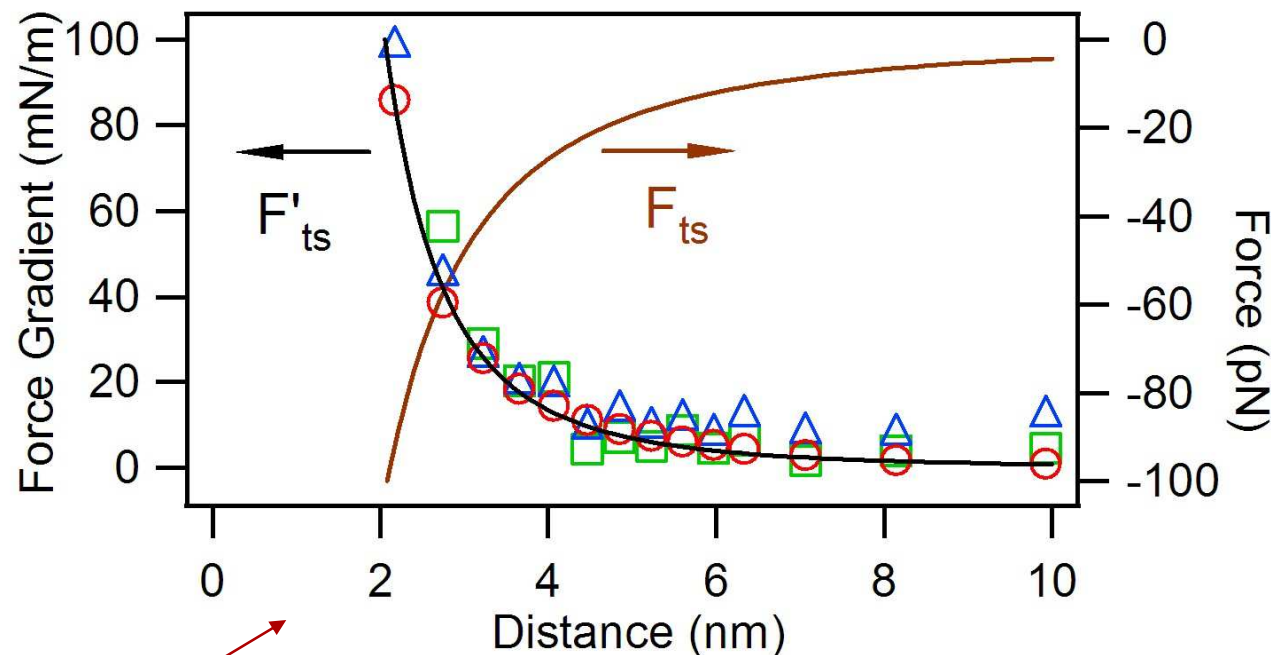
$$F_{ts} = -\frac{HR}{6(z-z_0)^2}$$

$$HR = 3 \cdot 10^{-27} \text{ J m}$$



Intermittent contact →
Short-range forces

H.Hoelscher *et al.*, *Phys. Rev.Lett.* **83**, 4780 (1999)



From nN to
pN range

1. **Frequency shift** ○
2. **Potential** △
3. **Mean square displacement** □

Long-range interaction forces of
the order of tens of piconewton

G.Malegori *et al.*, *J. Vac. Sci. Techn.* in press (2010)

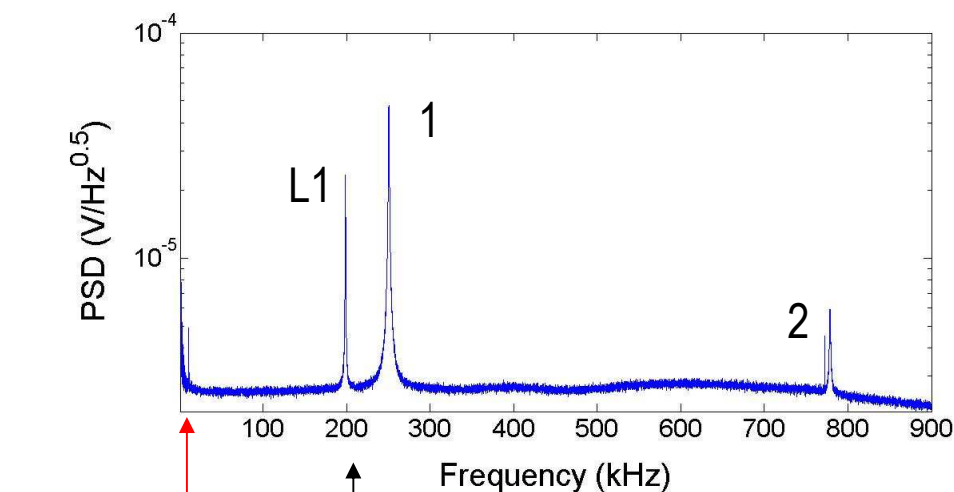
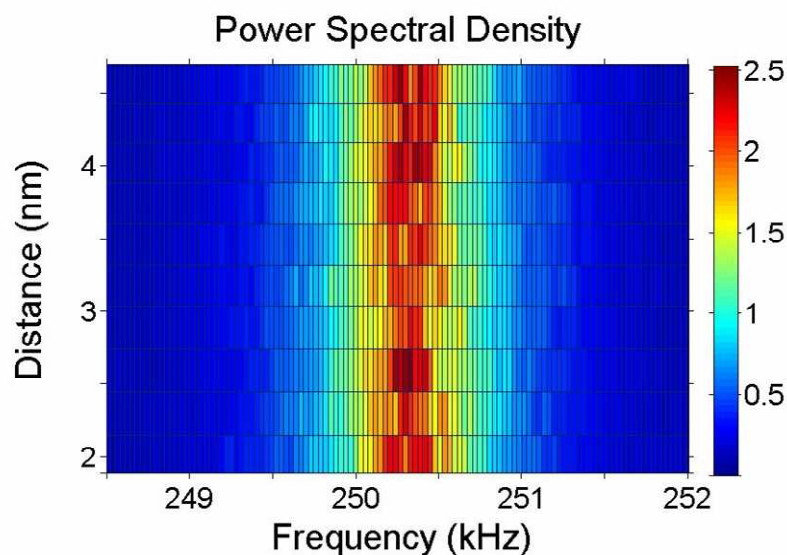
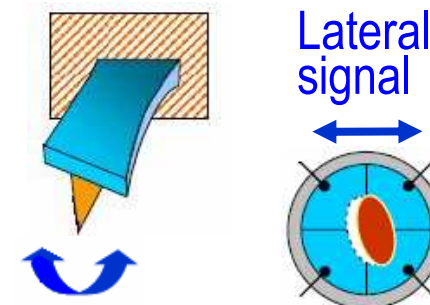


Conclusions

Thermal fluctuations of the AFM cantilever can be exploited to probe the tip-sample interaction

- **One temporal** trace of the cantilever thermal motion
Three different approaches simultaneously used to measure the tip-sample interaction
- The distance dependence and the magnitude of the force observed are in good agreement with a non retarded van der Waals interaction
- Frequency shift method: mass loading effects on the second eigenmode
- Drawback: due to jump to contact only long-range forces are analyzed

- Investigation of other surfaces
- Measurements in liquids
- Automates the spectroscopy
- Torsional modes analysis



Lateral bending mode (as flexural interchanging cantilever width and thickness) [*]

[*] Espinoza-Beltran *et al.*, *New J. Phys.* **11**, 083034 (2009)



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- Vittorio Spreafico, Stefano Prato
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<http://www.aperesearch.com>
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Via Musei 41, Brescia, Italy
gabriele@dmf.unicatt.it
<http://www.dmf.unicatt.it/elphos>
- Fulvio Parmigiani
Sincrotrone Trieste
Università degli Studi di Trieste
Dipartimento di Fisica, Basovizza, Trieste, Italy



Frequency shift method

The resonance frequency of free cantilever is

$$f_0 = \frac{1}{2\pi} \sqrt{\frac{k}{m^*}}$$

k spring constant, m^* effective mass

The tip-sample interaction F_{ts} changes the resonance frequency.
For **small amplitude oscillations**

$$k^* = k - \frac{\partial F_{ts}}{\partial z} \quad f = \frac{1}{2\pi} \sqrt{\frac{k^*}{m^*}} \quad k^* \text{ effective spring constant}$$

The frequency shift is directly related to the tip-sample force gradient
(exact solution)

$$f = f_0 + \Delta f$$

$$\frac{\partial F_{ts}}{\partial z} = k - k^* = k \left(1 - \frac{f^2}{f_0^2} \right)$$

Y.Martin *et al.*, *J. Appl. Phys.* **61**, 4723 (1987)

F.J.Giessibl, *Rev. Mod. Phys.* **75**, 949 (2003)



Free cantilever's torsional modes

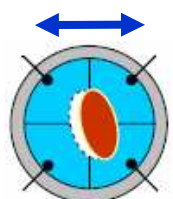
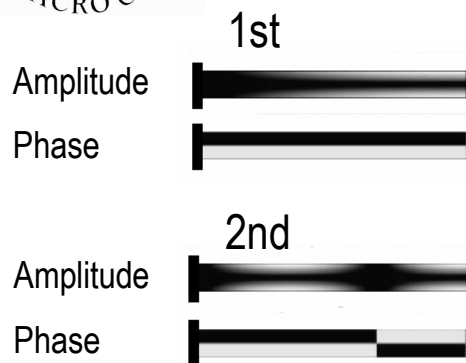
Material
properties

Geometrical
parameters

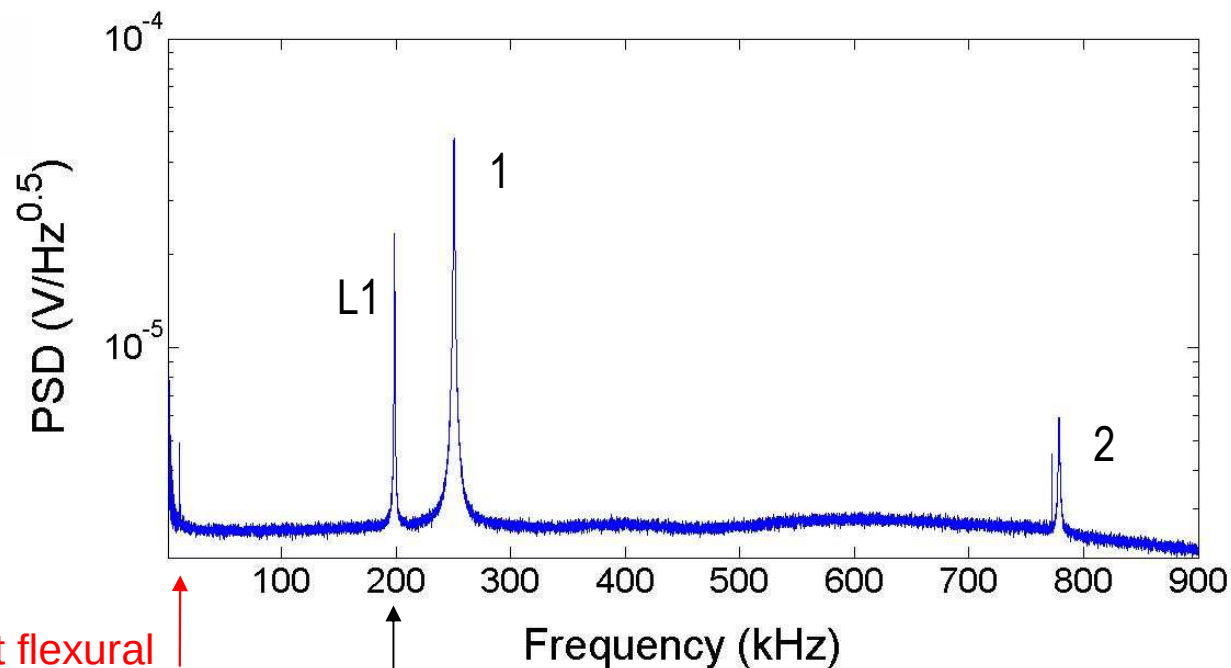
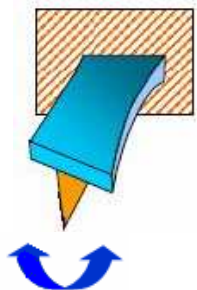
$$\frac{f_n^T}{f^L} = \frac{(2n-1)\pi\sqrt{6}}{\alpha_1^2} \sqrt{\frac{1}{1+\nu} \frac{L}{w}}$$

Boundary conditions

Torsional mode	1	2
f_n (kHz)	250	779
f_n / f_1 (exp.)	23	72
f_n / f_1 (th. [1])	24.71	74.13



Lateral
signal

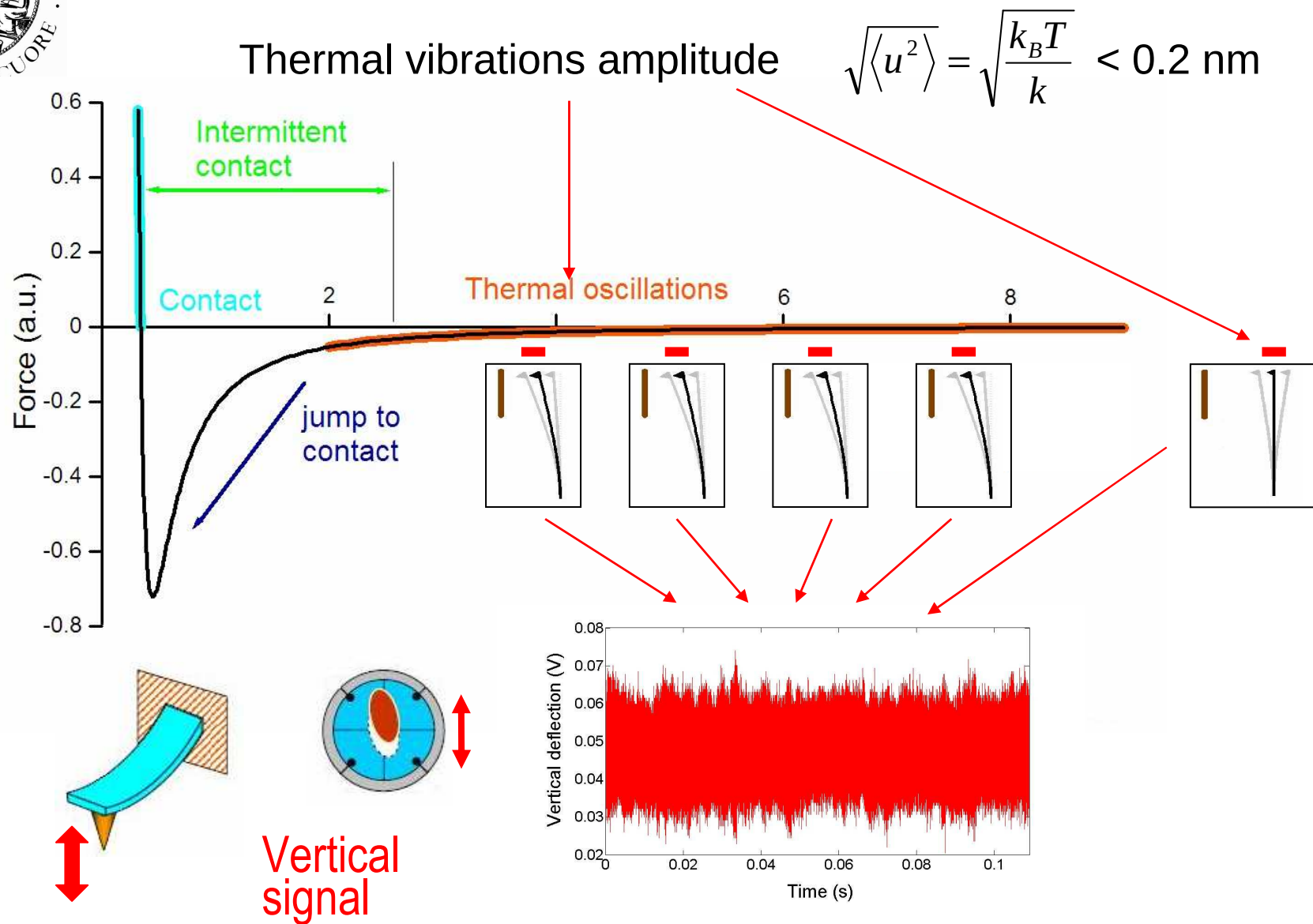


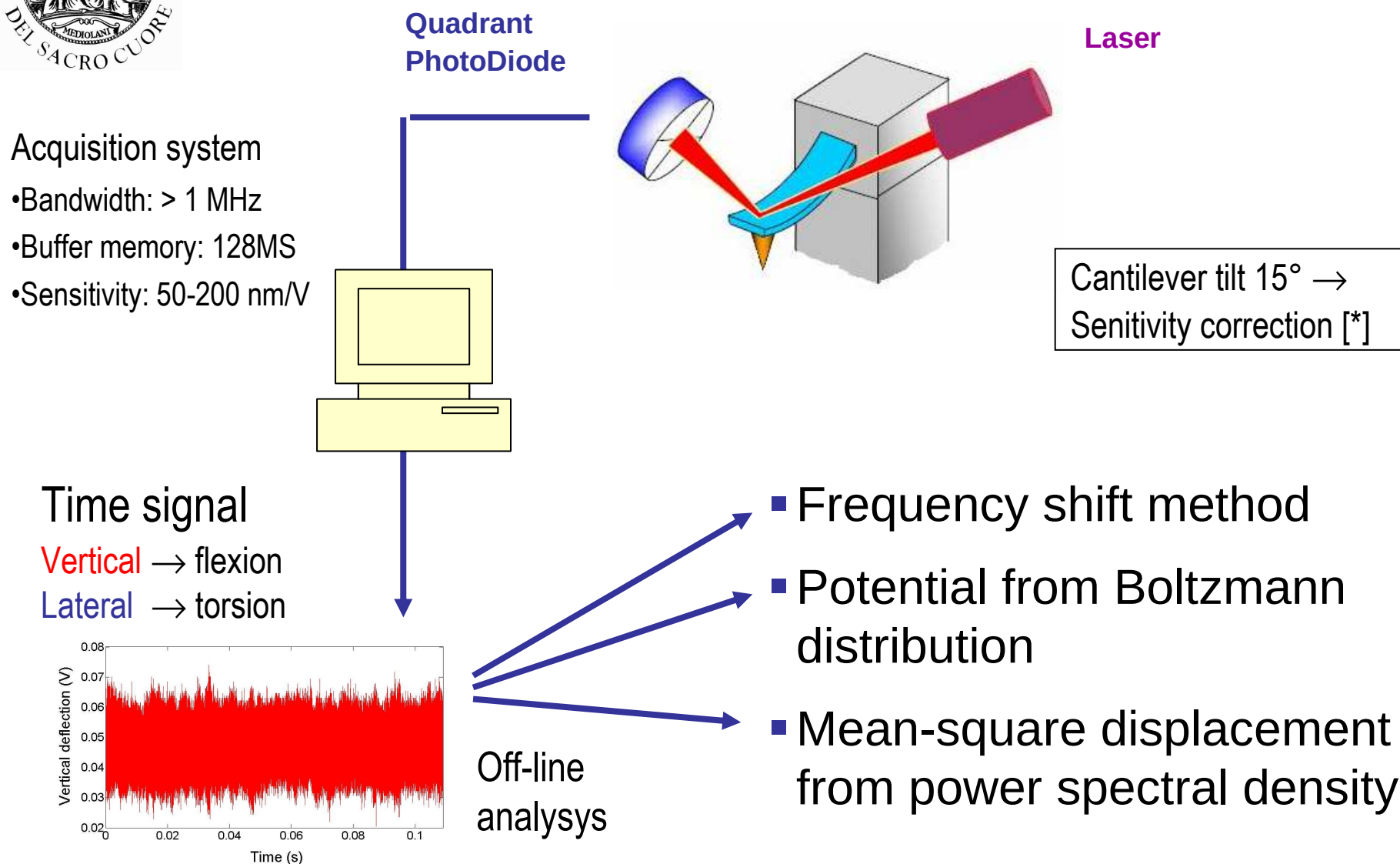
1st flexural

Lateral bending mode (as flexural interchanging
cantilever width and thickness) [2]

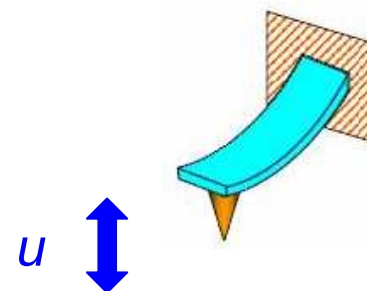
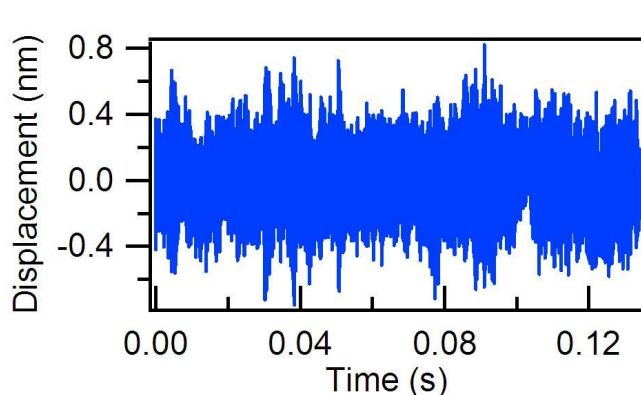
[1] H.J. Van Eysden *et al.*, *J. Appl. Phys.* **101**, 044908 (2007)

[2] Espinoza-Beltran *et al.*, *New J. Phys.* **11**, 083034 (2009)





[*] J.L.Hutter, *Langmuir* **21**, 2630 (2005)

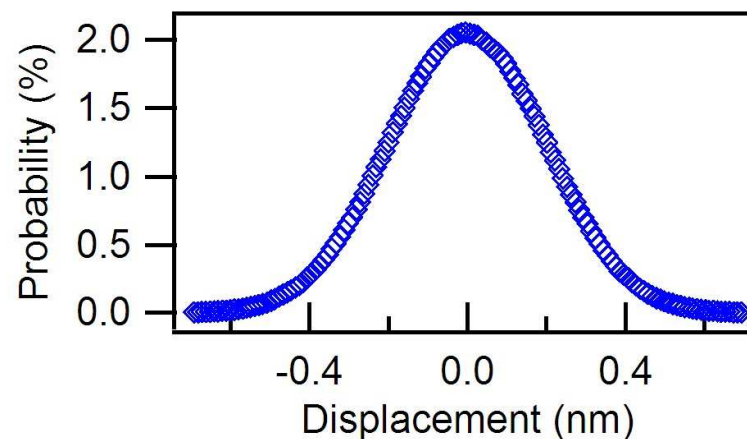


- $p(u, z)$ probability of observing the tip at a deflection u from the equilibrium position/tip-surface distance $z \rightarrow$
- number of count at the deflection u divided by the total number of count

Boltzmann probability distribution

$$p(u, z) = p_0 e^{-\frac{V(u, z)}{K_B T}}$$

$V(u, z)$ position dependent total potential



W.F. Heinz *et al.*, *J. Phys. Chem. B* **104**, 622 (2000)

D.O.Koralek *et al.*, *Appl. Phys. Lett.* **76**, 2952 (2000)



Boltzmann probability distribution

$$p(u) = p_0 e^{-\frac{V(u)}{K_B T}}$$

- p_0 normalization constant
- $V(u)$ position dependent potential
- $p(u)$ probability of observing the tip at a deflection u from the equilibrium position/tip-surface distance z

Inverting the relation gives the total potential $V(u)$

$$V(u) = -K_B T \ln \frac{p(u)}{p_0}$$

$$V(u, z) = V_C(u, z) + V_{ts}(u, z)$$

- V_C free cantilever potential
- V_{ts} tip-sample interaction potential

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Harmonic potential

$$V(u, z) = V_C(u, z) + V_{ts}(u, z)$$

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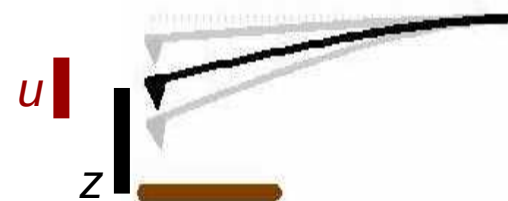
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Small oscillation amplitude u ($\partial F_{ts} / \partial z$ constant) $\rightarrow$ harmonic potential

$$V = \frac{1}{2} k^* u^2 \quad k^* = k - \frac{\partial F_{ts}}{\partial z}$$

Tip-sample force gradient

$$\frac{\partial F_{ts}}{\partial z} = - \frac{\partial^2 V_{ts}}{\partial s^2}$$



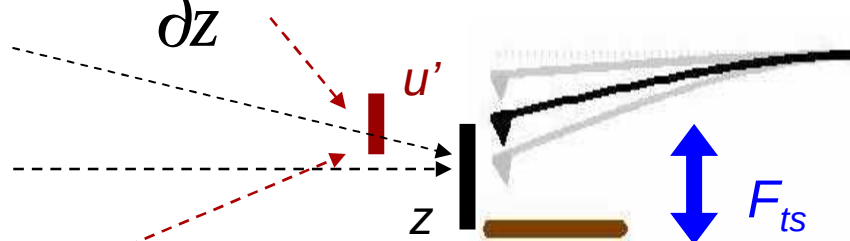
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D.O.Koralek *et al.*, *Appl. Phys. Lett.* **76**, 2952 (2000)

Cantilever near the surface $\rightarrow F_{ts}$ interacting force

$$m^* \ddot{u} + \gamma \dot{u} + ku = F_{ts}(z) = F_{ts}(z_0) + \frac{\partial F_{ts}(z_0)}{\partial z} u$$

- The constant force $F_{ts}(z_0)$ only displaces the equilibrium position z_0
- The force derivative $\partial F_{ts} / \partial z$ influences the cantilever oscillations



Small amplitude oscillations $\rightarrow$ the force gradient $\partial F_{ts} / \partial z$ is constant during the cantilever oscillations u' around the new equilibrium position

$$m^* \ddot{u}' + \gamma \dot{u}' + k^* u' = 0 \quad k^* = k - \frac{\partial F_{ts}}{\partial z}$$

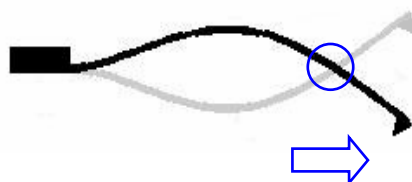
The tip-sample force gradient is directly related to the effective spring constant k^*

V.L.Mironov *Fundamentals of SPM* (2004)

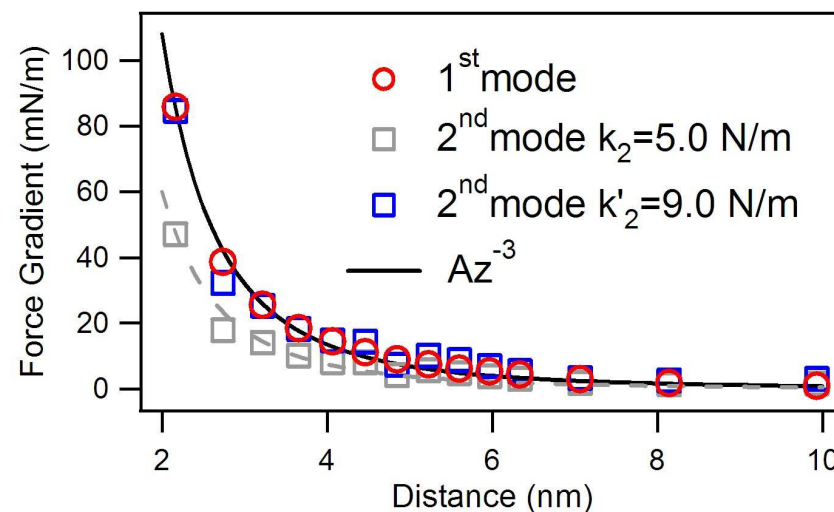
F.J.Giessibl, *Rev. Mod. Phys.* 75, 949 (2003)



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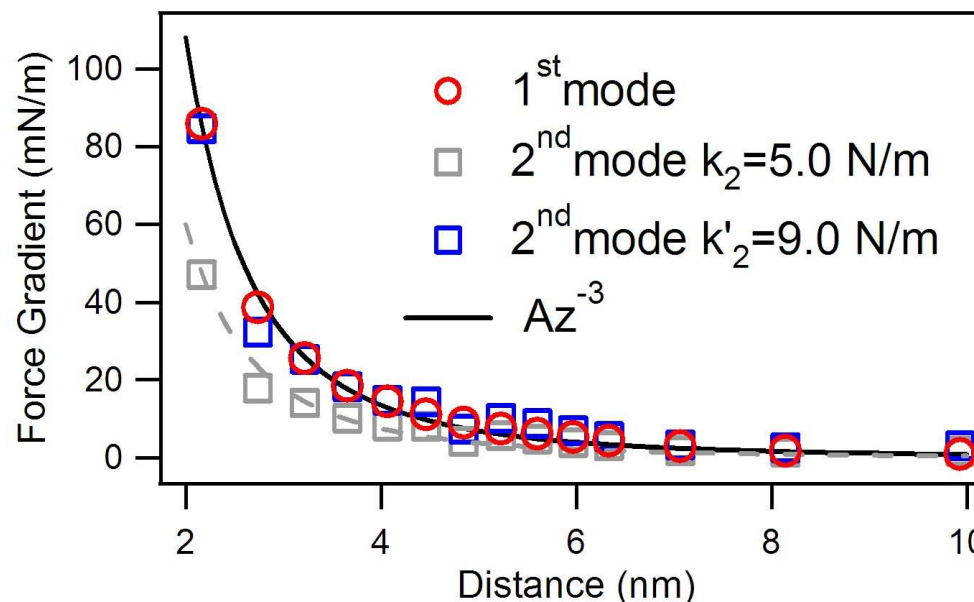
[2] J.E.Sader *et al.*, *Rev. Sci. Ins.* **70**, 3967 (1999)

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Tip mass loading

- The tip mass doesn't influence significantly the equivalent stiffness of the first eigenmode but can have dramatic effect on the equivalent stiffness of the higher eigenmodes.
- A **tip mass** that is 10% of the cantilever mass nearly doubles the **second mode's** equivalent stiffness. [1]
- Sader method [2] is accurate only for the lower modes.



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