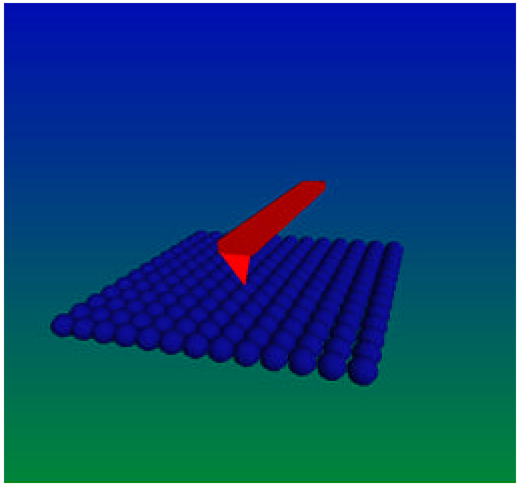
Atomic Force Microscopy (AFM)



Arvind Raman, Associate Professor
Mechanical Engineering
Birck Nanotechnology Center
NASA Institute of Nanoelectronics and Computation (I NAC)

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Further reading

- J. Gomez-Herrero, and R. Reifenberger, “Scanning Probe Microscopy”, to appear in *Encyclopaedia of Condensed Matter Physics*, edited by F. Bassani, J. Leidl, and P. Wyder, Elsevier Science Ltd., 2004.
- D. Sarid, *Scanning Force Microscopy with Applications to Electric, Magnetic and Atomic Forces*, Revised Edition, Oxford University Press, 1994.
- U. Dürig, “Interaction sensing in dynamic force microscopy”, *New Journal of Physics*, Vol. 2, pp. 5.1-5.12, 2000.
- F. Giessibl, “Advances in atomic force microscopy”, *Reviews of Modern Physics*, Vol. 75, pp. 949-983, 2003.
- R. García, R. Pérez, “Dynamic atomic force microscopy methods”, *Surface Science Reports*, Vol. 47, pp. 197-301, 2002.
- B. Cappella, G. Dietler, “Force-distance curves by atomic force microscopy” *Surface Science Reports*, Vol. 34, pp. 1-104, 1999.

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Outline

- History of Atomic Force Microscopy (AFM)
- Instrumentation
- Static force-distance curves and force spectroscopy
- Dynamic AFM and force gradient spectroscopy
- Imaging
- Applications and emerging areas

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The starting point- STM

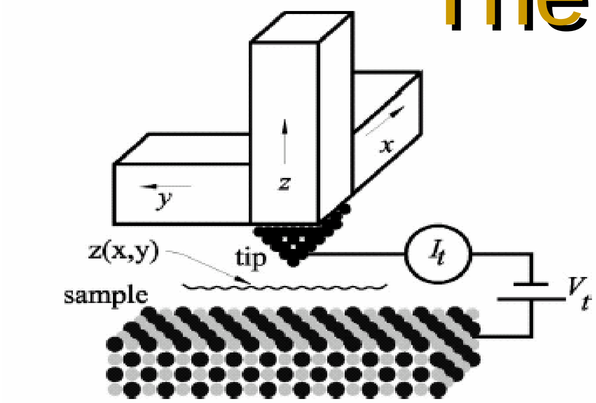

FIG. 2. A scanning tunneling microscope (schematic).

F. Giessibl's Rev. Mod. Phys.

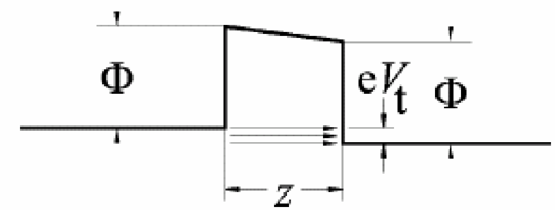

FIG. 3. Energy diagram of an idealized tunneling gap. The image charge effect (see Chen, 1993) is not taken into account here.

- Binnig, Gerber, Rohrer, Wiebel (1982)
- Binnig and Rohrer awarded Nobel Prize in Physics in 1986 for STM
- If $|V_t|$ is small compared to workfunction Φ, and tunneling current is given by $I_t(z) = I_0 e^{-2k_t z}$ where z is the gap I_0 is a function of the applied voltage and the density of states in the tip and the sample, and $k_t = \sqrt{2m\Phi} / \hbar$
- For most metals, $\Phi \sim$ 4eV, so that $\kappa_t = 1 Å^{-1}$
- Most current carried by "front atom"blunt tips , so atomic resolution possible even with relatively blunt tips
- Only electrically conductive samples, restricting its principal use to metals and semi-conductors

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The AFM

G. Binnig, C. F. Quate and Ch. Gerber, PRL 56, 930 (1986)

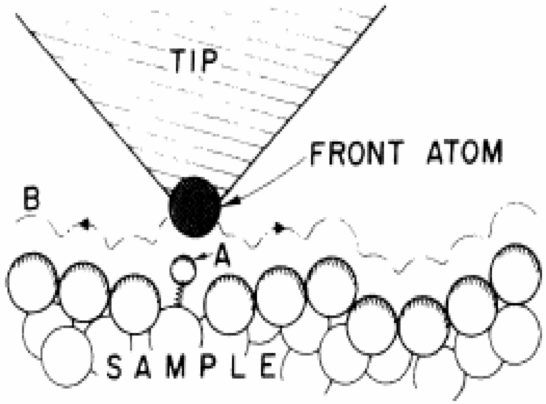

FIG. 1. Description of the principle operation of an STM as well as that of an AFM. The tip follows contour *B*, in one case to keep the tunneling current constant (STM) and in the other to maintain constant force between tip and sample (AFM, sample, and tip either insulating or conducting). The STM itself may probe forces when a periodic force on the adatom *A* varies its position in the gap and modulates the tunneling current in the STM. The force can come from an ac voltage on the tip, or from an externally applied magnetic field for adatoms with a magnetic moment.

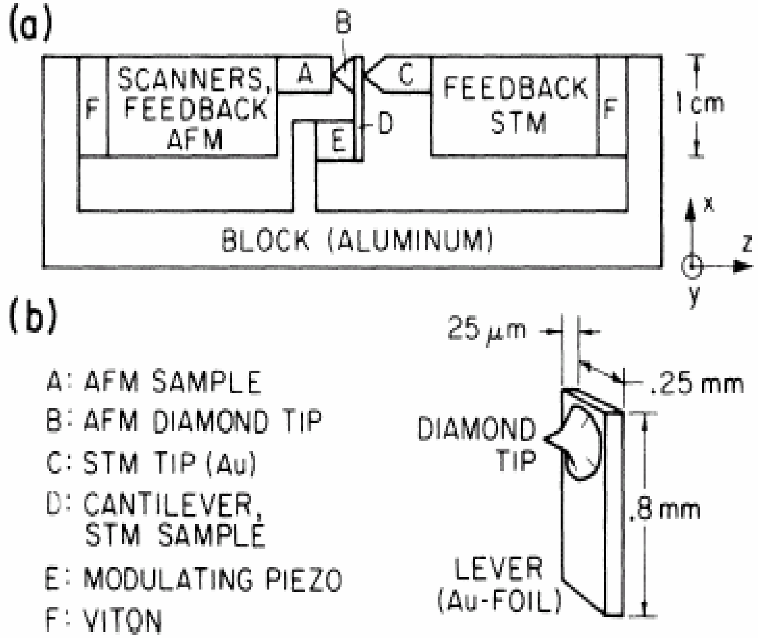

FIG. 2. Experimental setup. The lever is not to scale in (a). Its dimensions are given in (b). The STM and AFM piezoelectric drives are facing each other, sandwiching the diamond tip that is glued to the lever.

- Binnig invented the AFM in 1986, and while Binnig and Gerber were on a Sabbatical in IBM Almaden they collaborated with Cal Quate (Stanford) to produce the first working prototype in 1986

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Early AFM Images

G. Binnig, C. F. Quate and Ch. Gerber, PRL 56, 930 (1986)

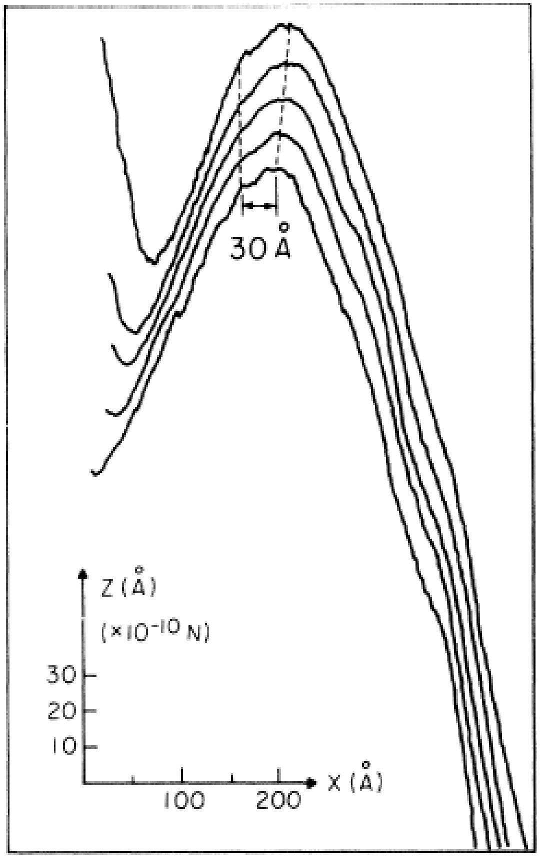

FIG. 3. The AFM traces on a ceramic (Al_2O_3) sa
The vertical scale translates to a force between sampl
tip of 10^{-10} N/Å. For the lower trace the force is
3×10^{-8} N. The stability of the regulated force is bette
10^{-10} N. The successive traces are displaced by a smal
along the *y* axis.

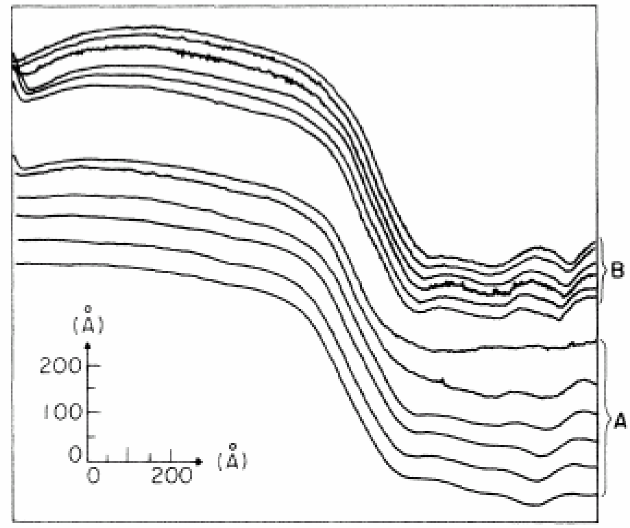

FIG. 4. The AFM traces for another area of the ceramic sample. The curves grouped under *A* were recorded with additional low-pass filtering. For this set the stabilizing force, f_0, was reduced by thermal drifts as we moved from the lowest to the highest traces of set *A*. The force f_0 is near 10^{-8} N for the highest curve. We note that the structure vanishes on the traces when the sample-to-tip force is reduced below this level. The force f_0 was reset to a higher value near 5×10^{-8} N for the traces marked *B*.

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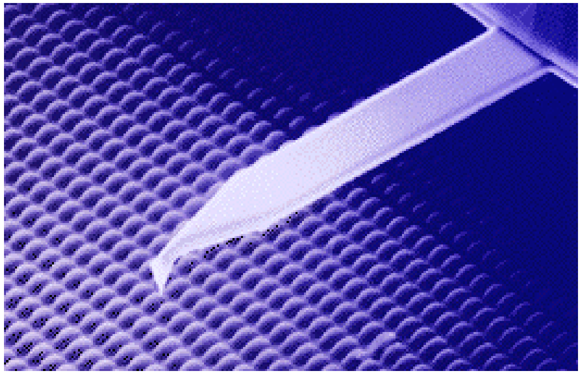
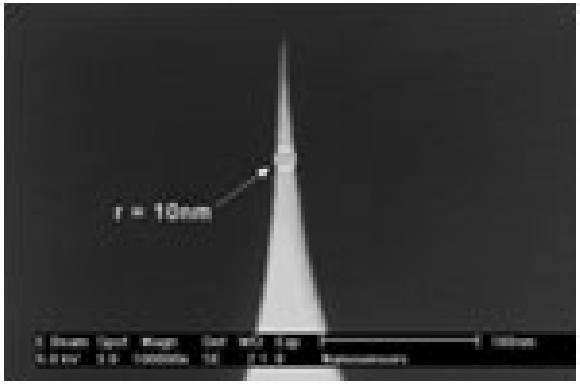
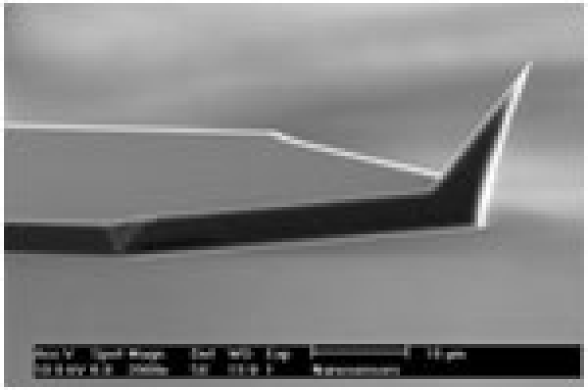
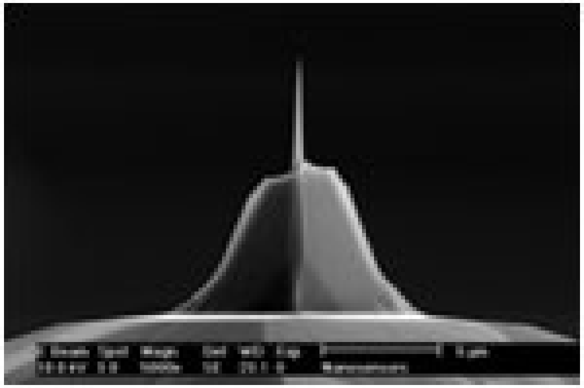
The microcantilever – the force sensor



www.olympus.co.jp







www.nanosensors.com

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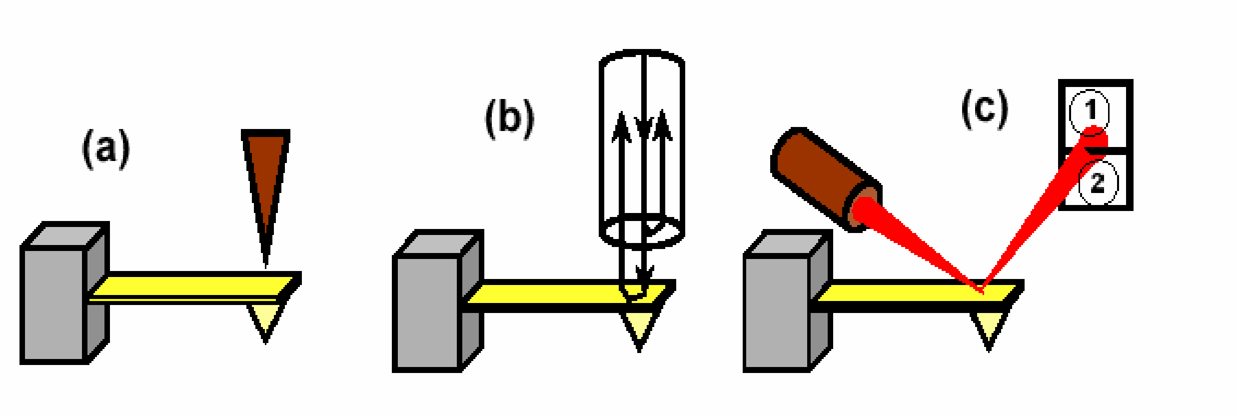
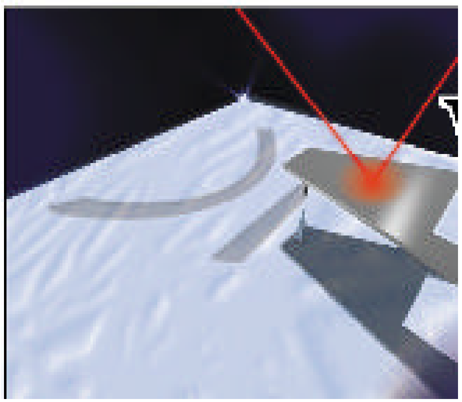
Detecting deflection



- STM tip
- Capacitance/laser interferometry
- **Beam deflection**



Courtesy - J. Gomez, UAM, Spain

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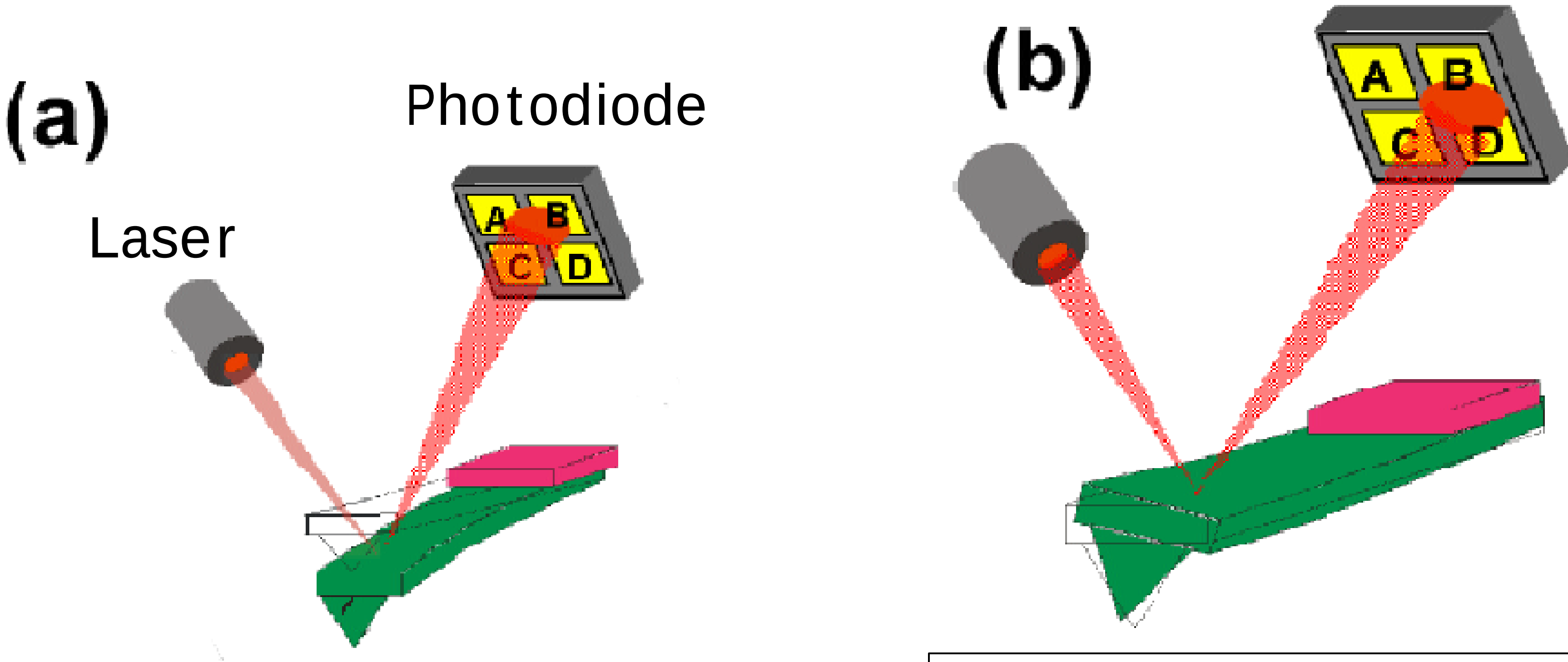
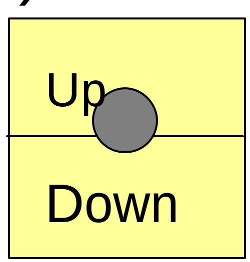
The beam deflection method



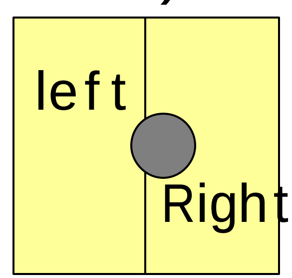
a) Normal force



A+B= UP

C+D=DOWN

b)Lateral Force



A+C= LEFT

B+D=Right

Courtesy - J. Gomez-Herrero, UAM, Spain

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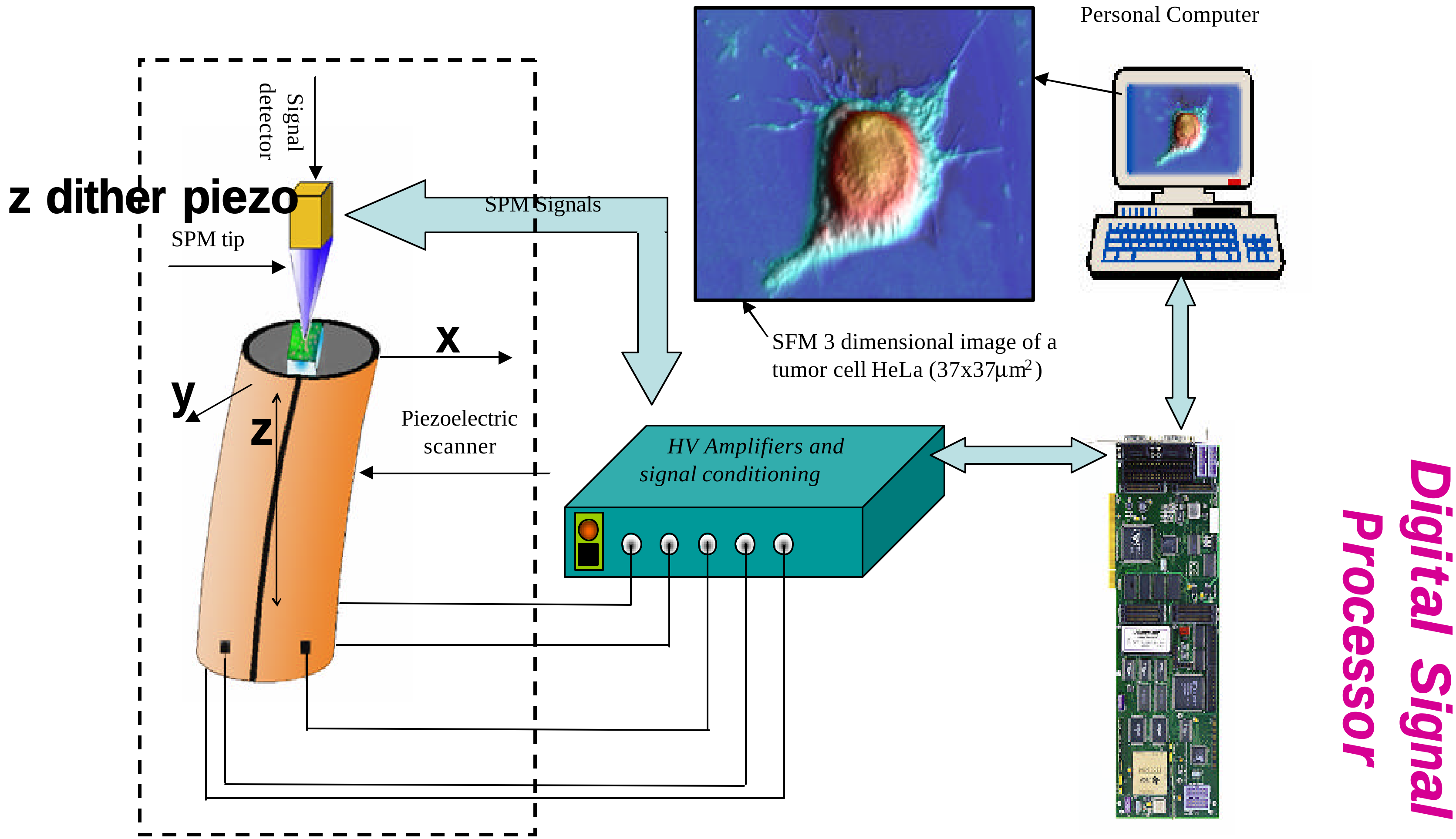
AFM Block Diagram



Courtesy - J. Gomez - Herrero, UAM, Spain

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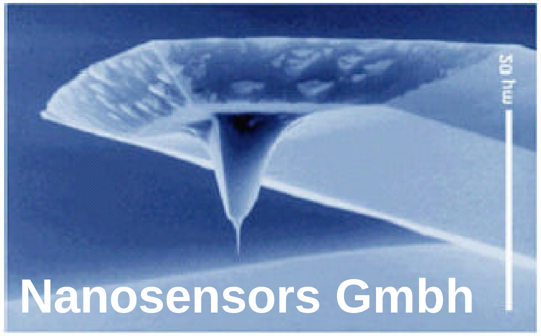
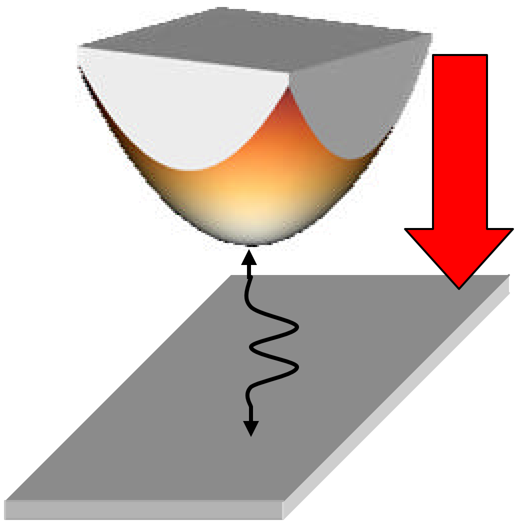
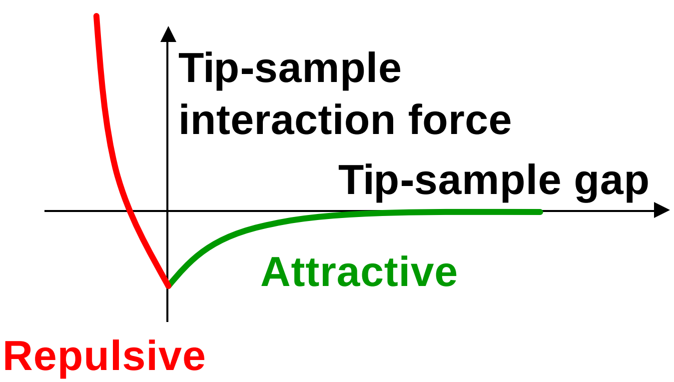
Tip-sample interaction forces in AFM







- Long-range electrostatic and magnetic forces (upto 100 nm)
- Capillary forces (few nm)
- Van der Waals forces (few nm) that are fundamentally quantum mechanical (electrodynamic) in nature
- Casimir forces
- Short-range chemical forces (fraction of nm)
- Contact forces
- Electrostastic double-layer forces
- Solvation forces
- Nonconservative forces (Dürig (2003))

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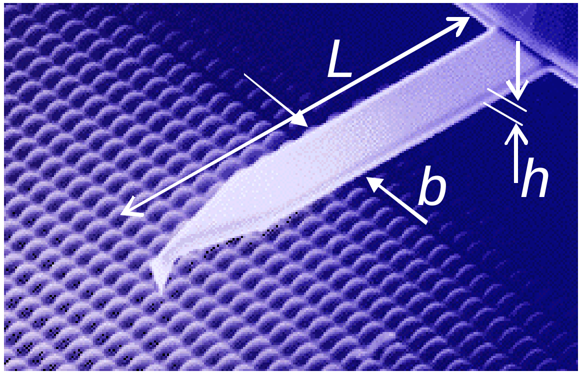
The microcantilever – the force sensor



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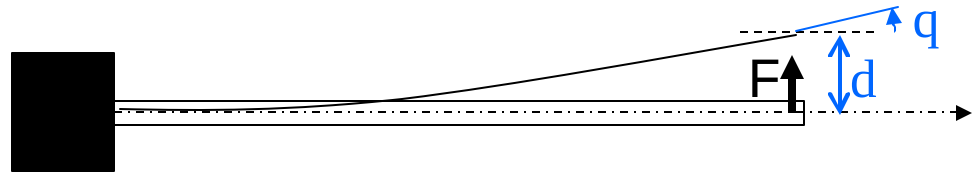

- From elementary beam theory, if E=Young's modulus, $I=bh^3/12$ then
- $\delta=w(L)=F\,L^3/(3EI)$, and $\theta=dw(L)/dx=FL^2/(2EI)$
- Deflection and slope linearly proportional to force sensed at the tip
- $k=3EI/L^3$ is called the bending stiffness of the cantilever

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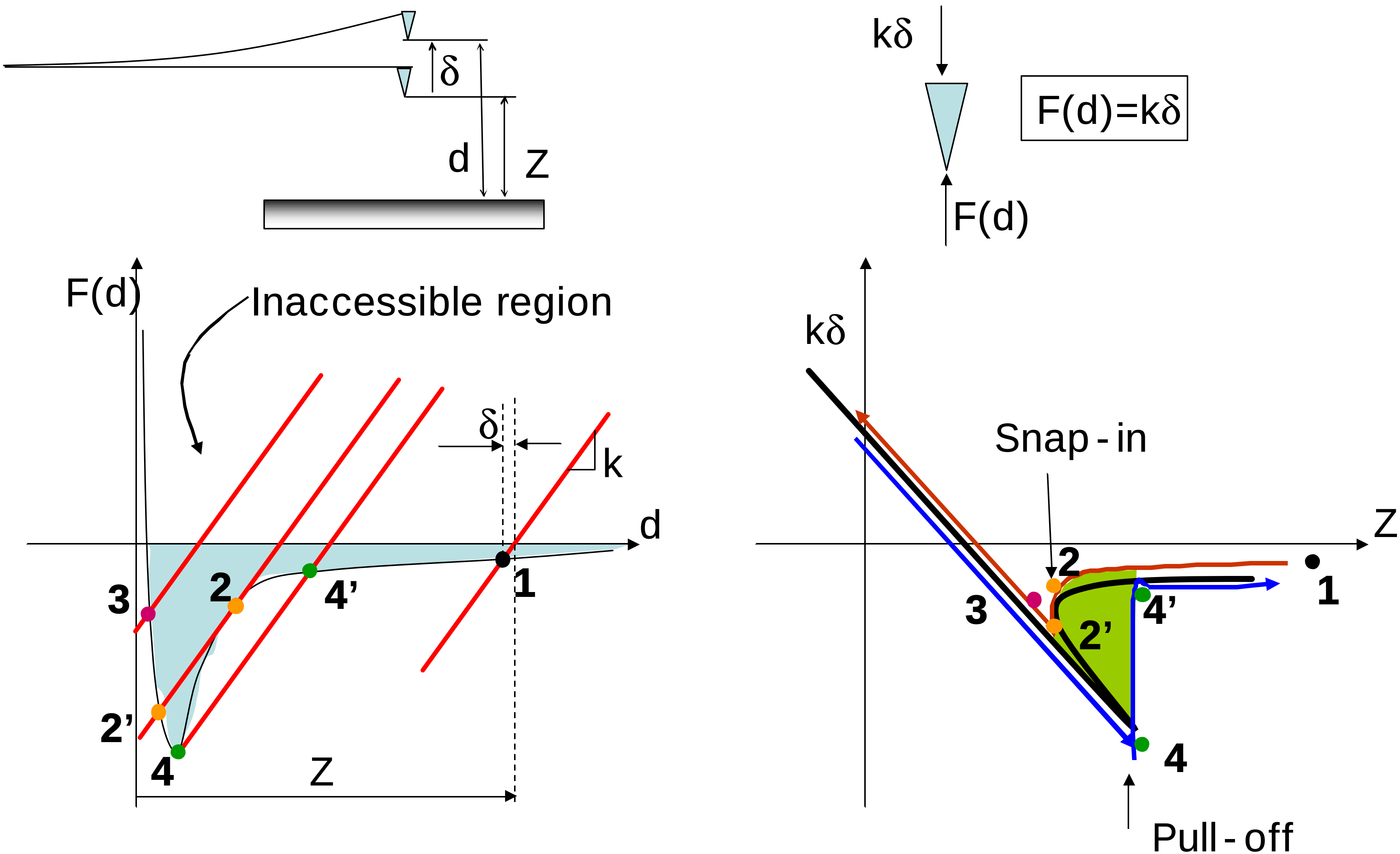
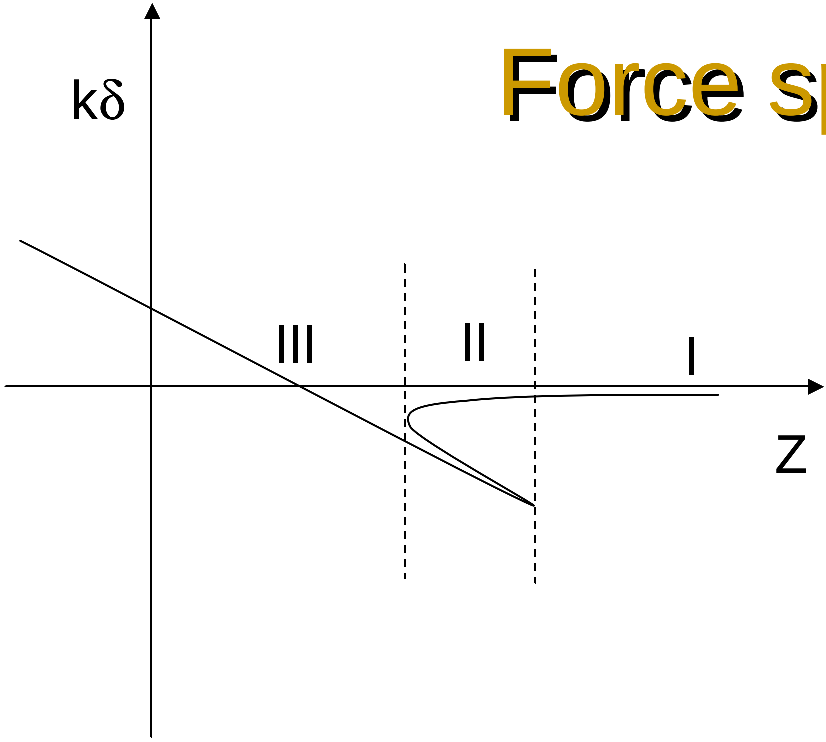
Force-displacement curves

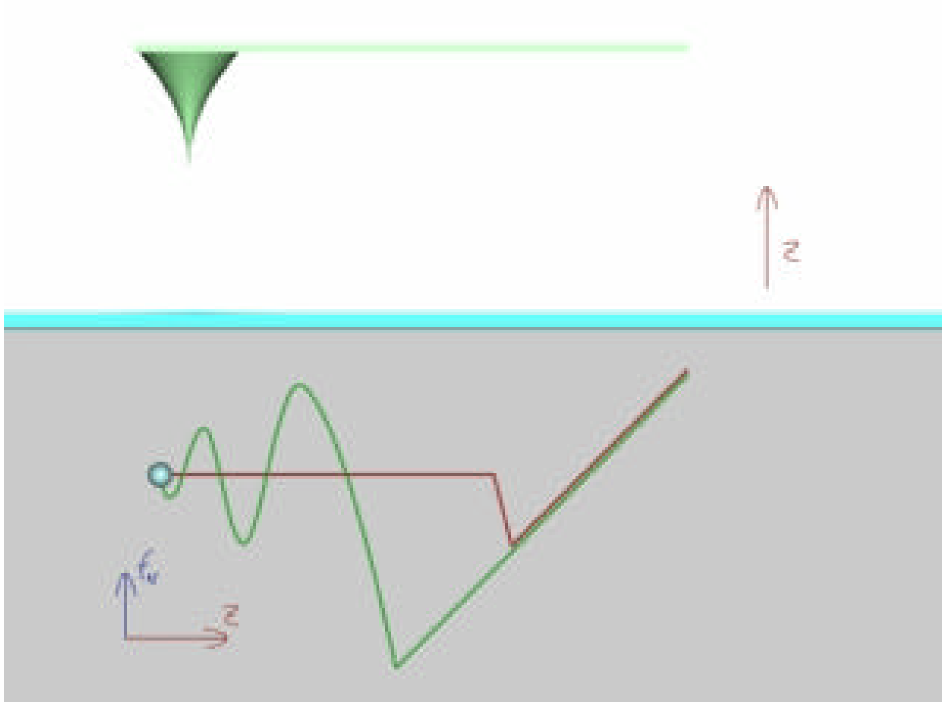


$W_{Adhesion}$=blue shaded area above

$W_{Cantilever}$=shaded area above

Force spectroscopy





- Three distinct regions
- If k is known then from the static-force distance curve, F(d) can be calculated for all d except for inaccesible range near snap-in
- It can be shown that $W_{Cantilever}$ is related to the $W_{Adhesion}$
- Slope in III is good measure of repulsive forces (local elasticity)

Animation courtesy J. Gomez - Herrero, UAM, Spain

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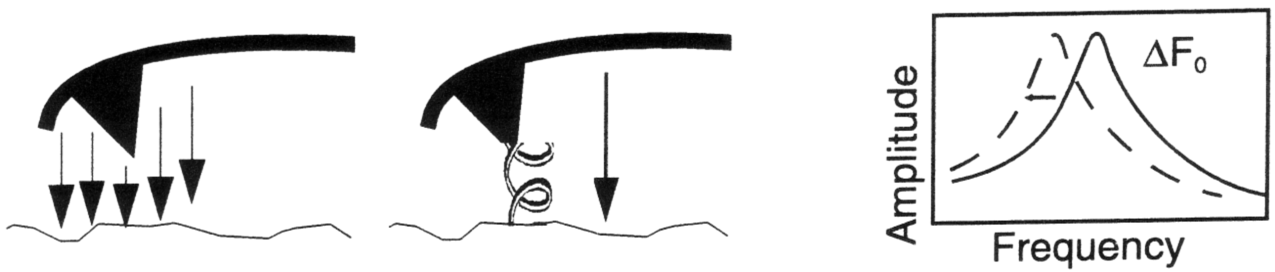
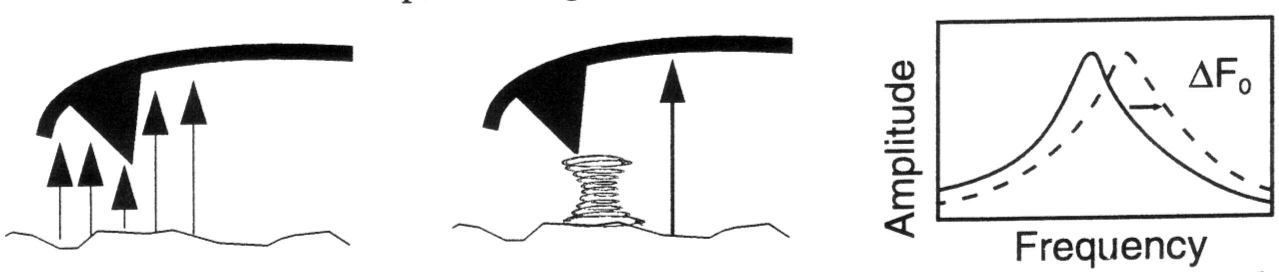
Dynamic AFM

- Cantilever driven near resonance
- Non-contact AFM, Tapping mode AFM, Amplitude Modulated AFM, Frequency Modulated AFM are all dynamic AFM
- The cantilever's resonant frequency, phase and amplitude are affected by short-scale force gradients



Attractive gradient equivalent to additional spring in tension attached to tip, reducing the cantilever resonance frequency.



Repulsive gradient equivalent to additional spring in compression attached to tip, increasing the cantilever resonance frequency.

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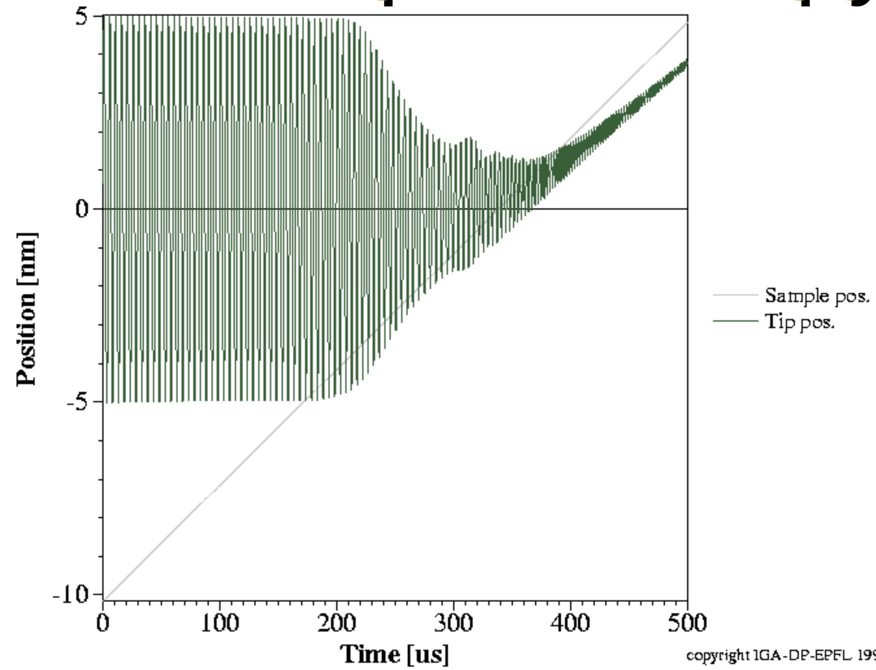
Dynamic AFM & force gradient spectroscopy

- Variation of amplitude, resonance frequency, and phase measured as Z is decreased
- From this it is possible to reproduce the Force gradients between the tip and the sample
- Even non-conservative interactions can be resolved
- Offers many advantages over static-force distance curve based force spectroscopy
- Quantitative information is hard to come by because the forces are nonlinear



Simulation results
DMT contact mechanics
v. d. Waals surface force

Input parameters

Din = -10.0 [nm]
PPA tip = 10.0 [nm]
Fex = .2 [MHz]
V = 3.000000000000000E-02 [nm/us]
Q = 100.0
Fre = .2 [MHz]
kc = 10.0 [N/m]
R = 10.0 [nm]
K = 10.0 [GPa]
rat = 1.0
Deq = .165 [nm]
w = 7.199999999999999E-02 [J/m^2]

Computed data
----
m* = 6.332573975 99238 [pkg]
lambda = .6847861733899854
bc = 6.283028234257542E-03 [1/us]
bi = 6.283028234257542E-03 [1/us]
D0 = .0 [nm]
Dm = .0 [nm]
Dnorm = .1723144028935211 [nm]
Pnorm = .4178408698922936 [GPa]

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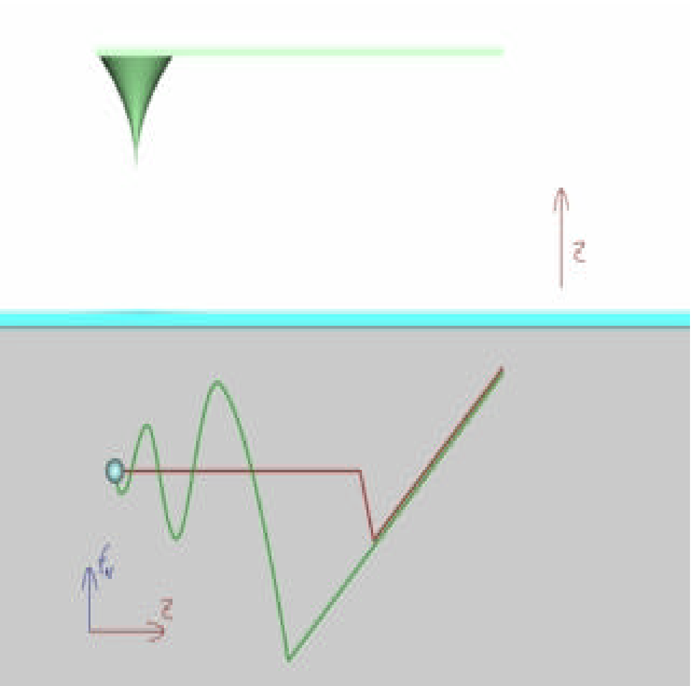
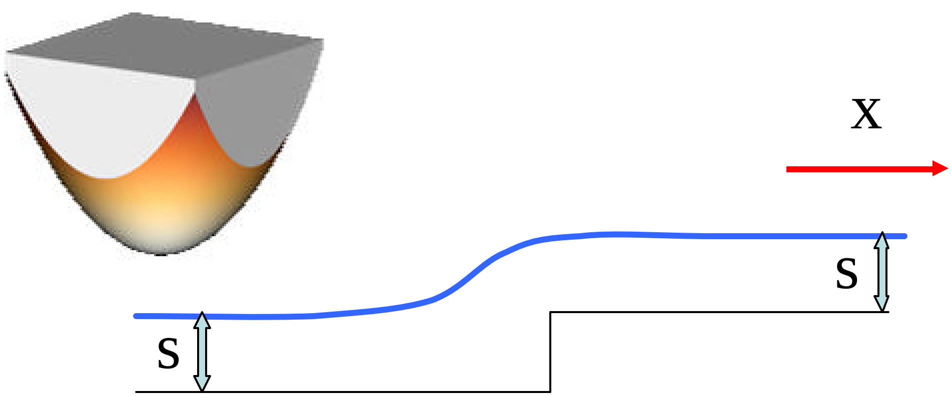
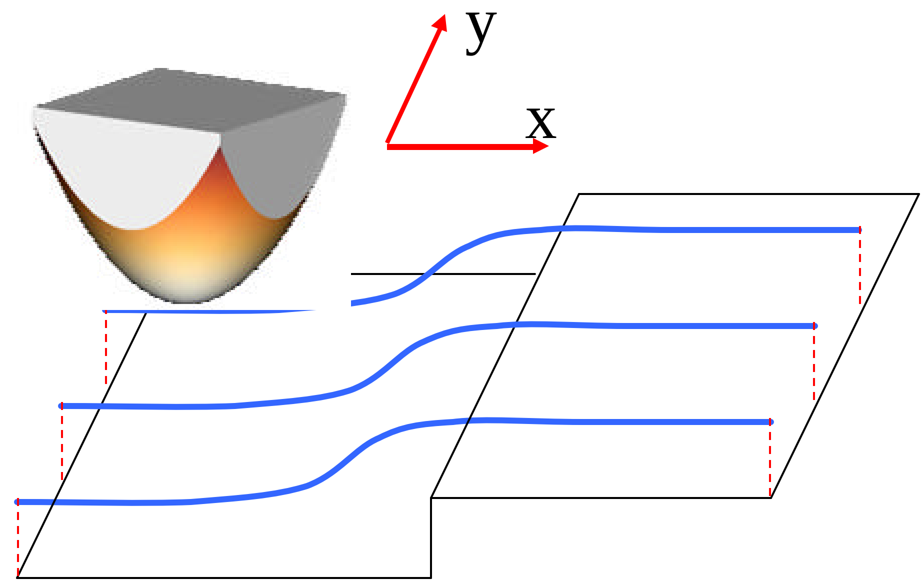
Contact Mode Imaging







First tip contacts surface with some setpoint normal force which is kept constant during the scan

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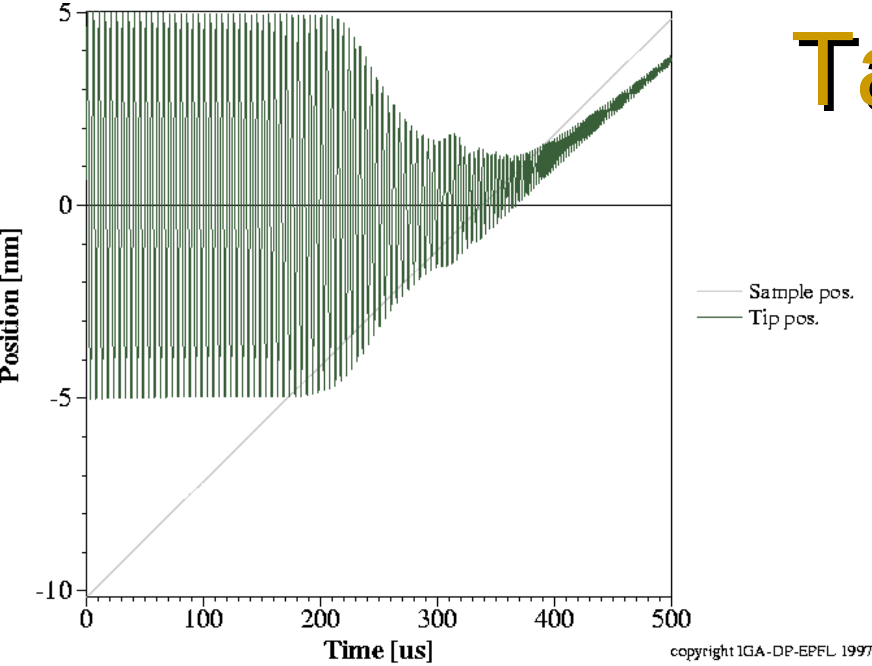
Tapping Mode



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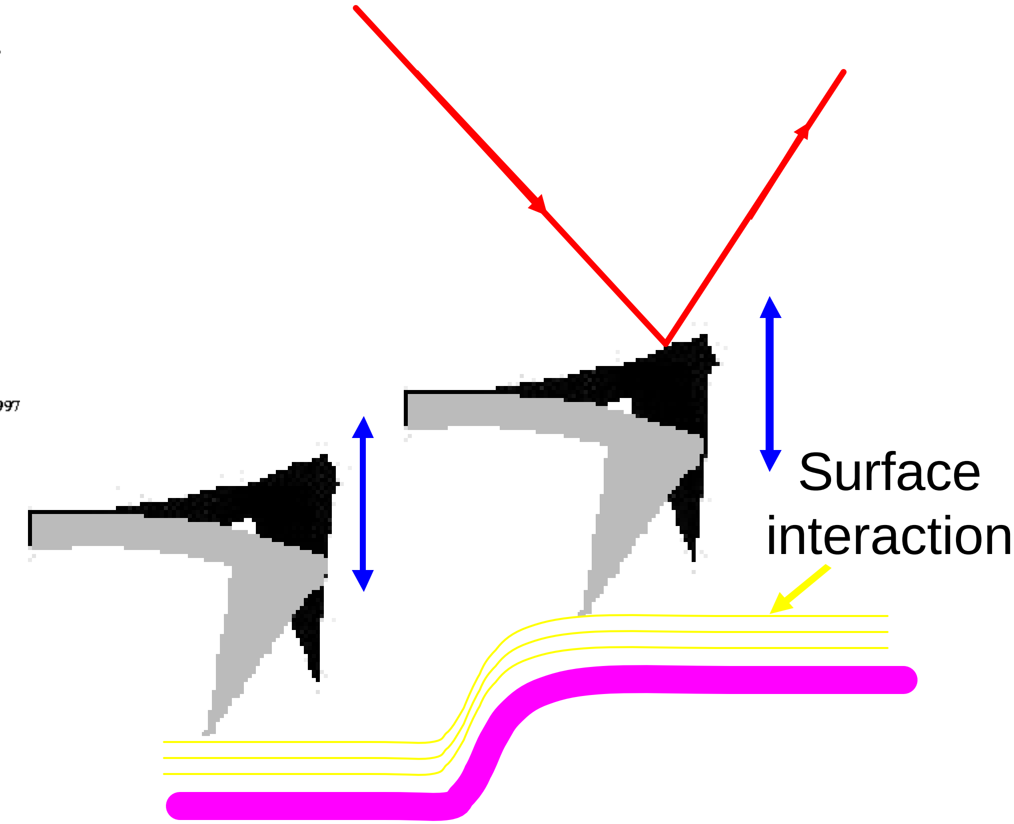

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Computed data

----

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lambda = .684786173389985 4
bc = 6.283028234257542E-03 [1/us]
bi = 6.283028234257542E-03 [1/us]
D0 = .0 [nm]
Dm = .0 [nm]
Dnorm = .1723144028935211 [nm]
Pnorm = .4178408698922936 [GPa]

In Tapping mode the tip is oscillated at the resonance frequency and the amplitude of oscillation is kept constant while the tip intermittently enters the repulsive regime

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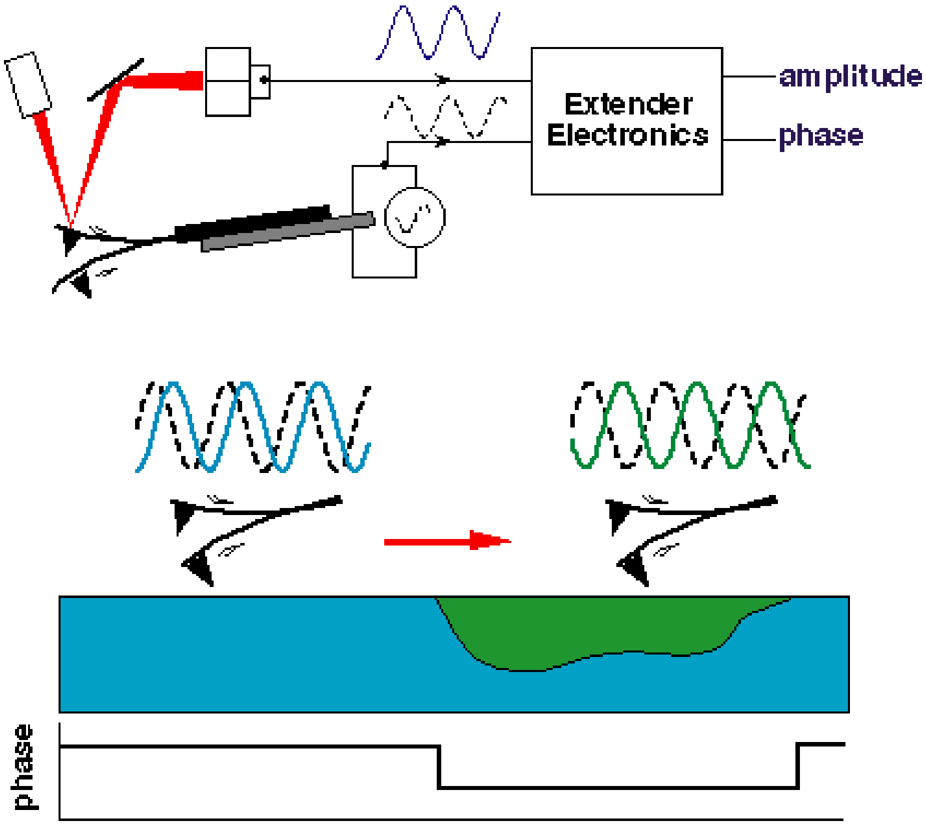
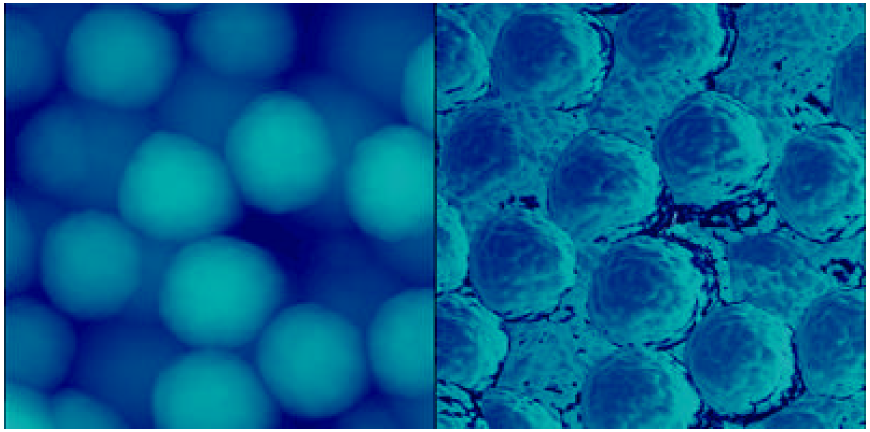
Phase Imaging





AFM height (left) and phase (right) images of poly(methylmethacrylate)

(Digital Instruments, Inc.)

- Regular tapping mode implemented but signal phase monitored
- Phase contrasts are related to differences in local dissipation

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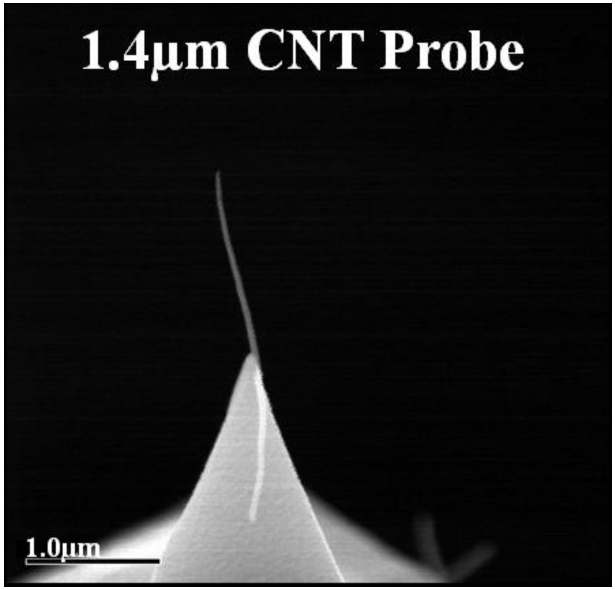
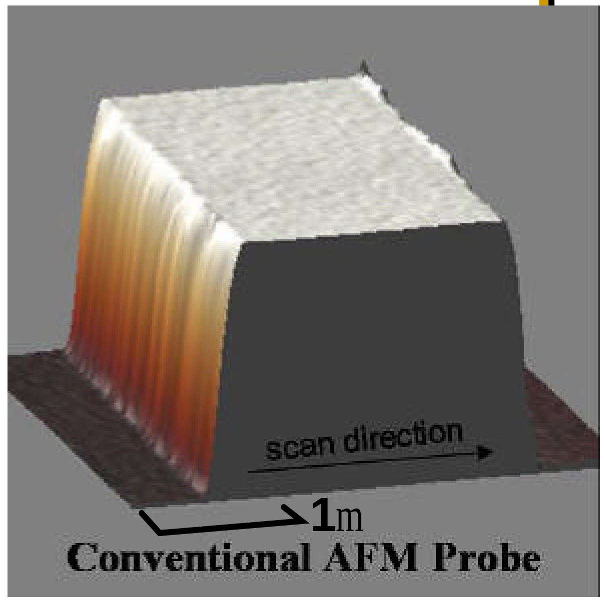
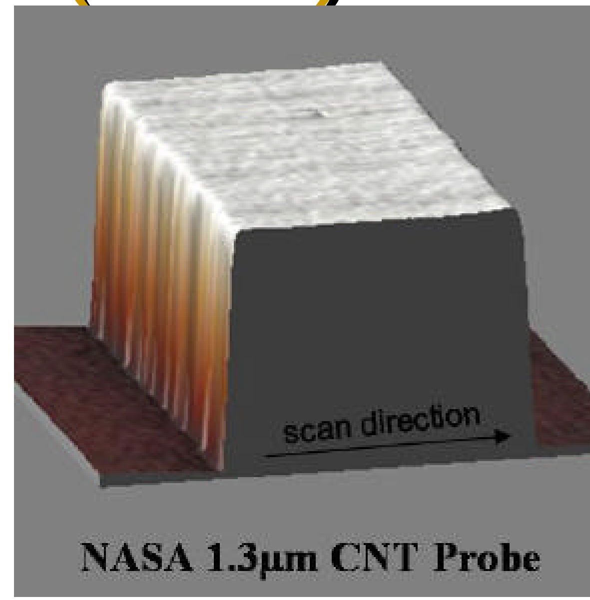
Carbon nanotube tips (CNT)





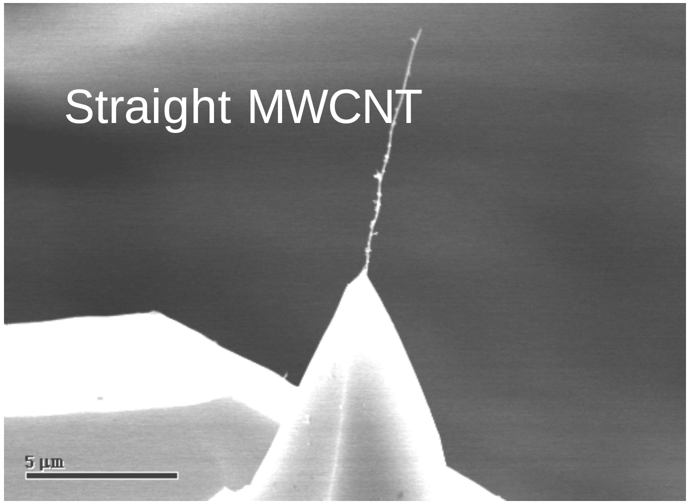


Dynamic AFM images of a **100 nm** trench on Si using conventional silicon probe (left) and a MWCNT probe (right)

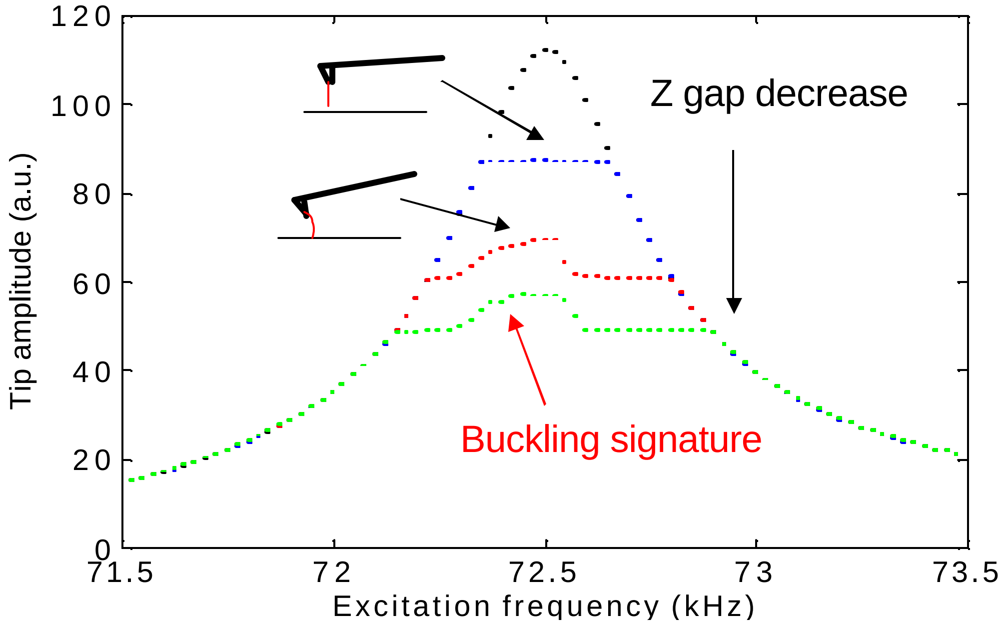
- Provide high resolution
- Show little evidence of wear
- Promising technology for critical dimension metrology of semiconductors, and nanobiological investigations
- Buckling, friction and stiction of CNT become important

Raman et al, Ultramicroscopy (2003), Nanotechnology (2003, 2005)

CNT tips – tapping mode



(SEM images: NASA & Purdue, 2002)



- CNT attached strongly to Force modulation etched Si probe (Ni evaporation)
- Straight MWCNT, diameter 10 nm, length 7.5µm, Frequency 72.5 kHz
- Repulsive and attractive states do not appear to co-exist for long CNT tips

Raman et al, Ultramicroscopy (2003), Nanotechnology (2003)

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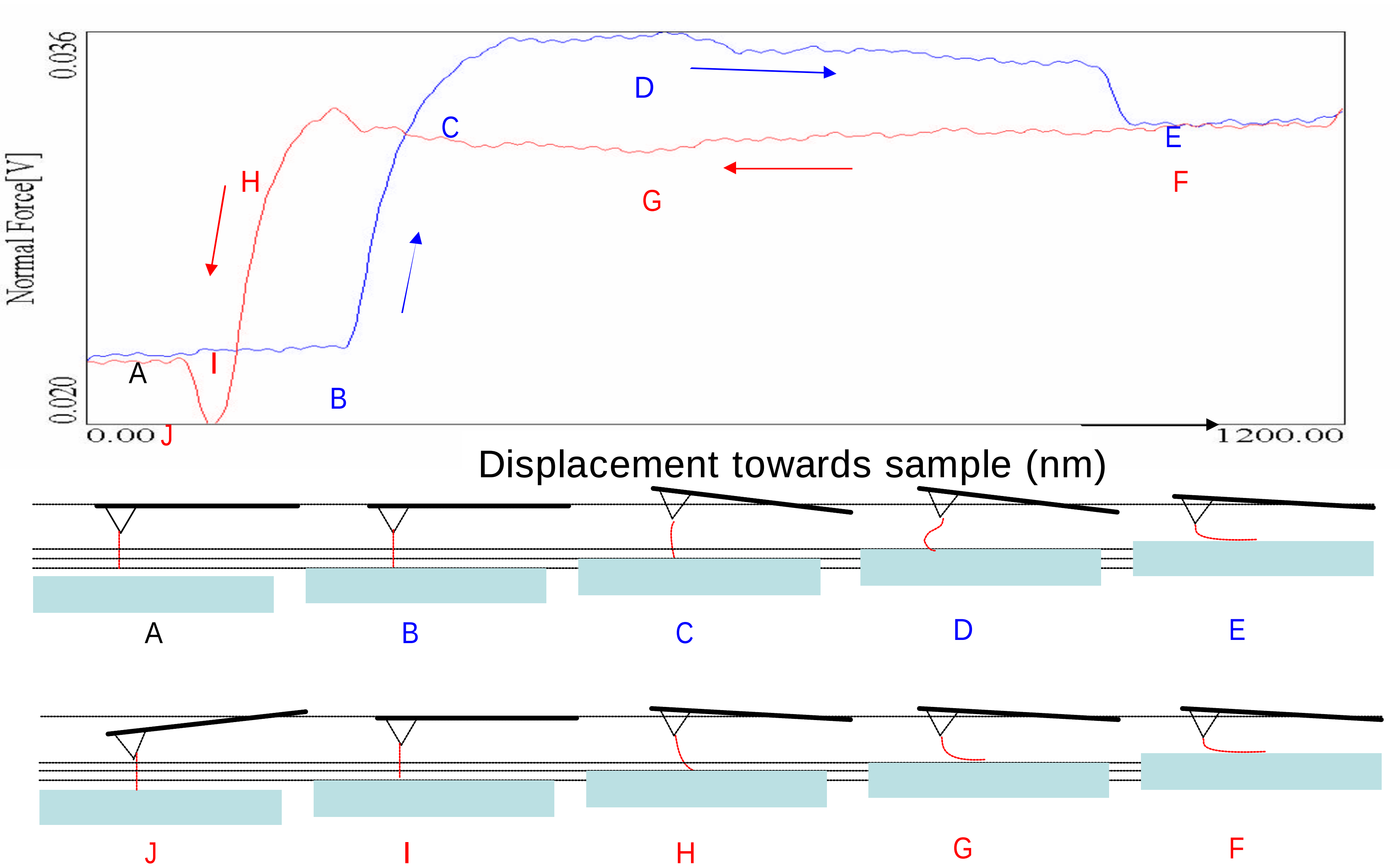
Static force-distance curves



- CNT buckles, slips, and slides
- High adhesion on the CNT sidewalls

Raman et al, Nanotechnology (200

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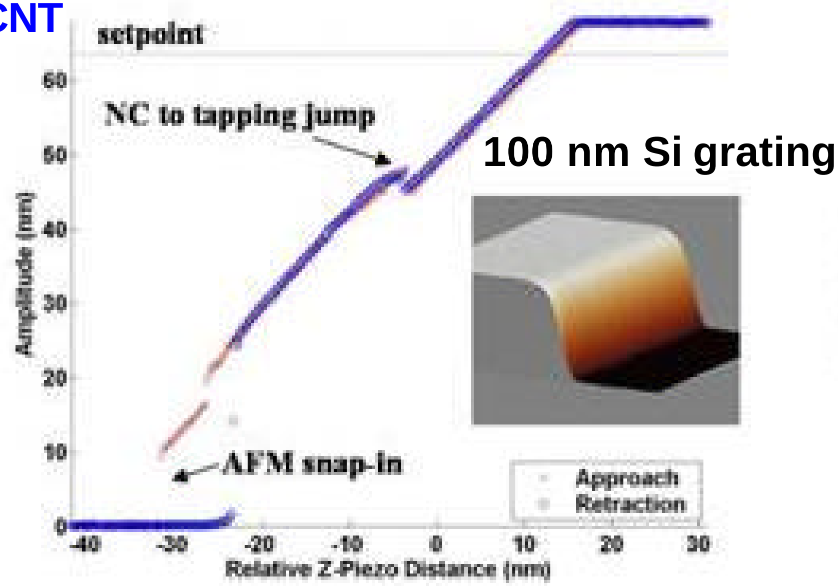
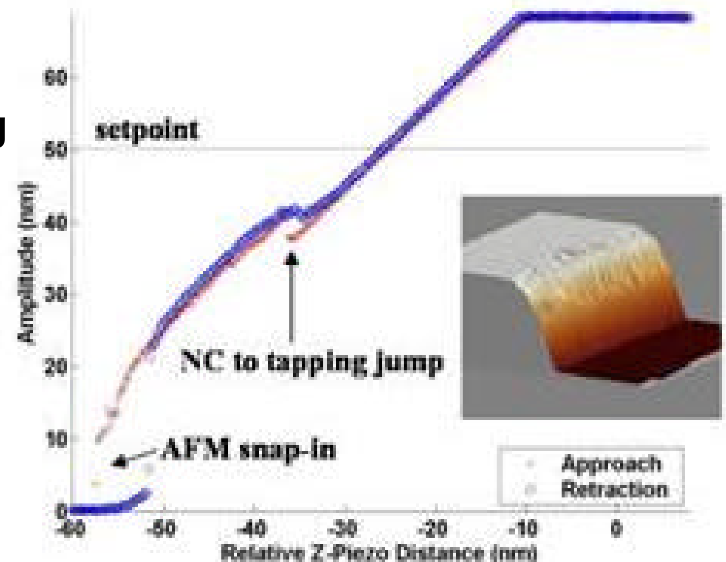
Shorter CNT tips- noncontact mode

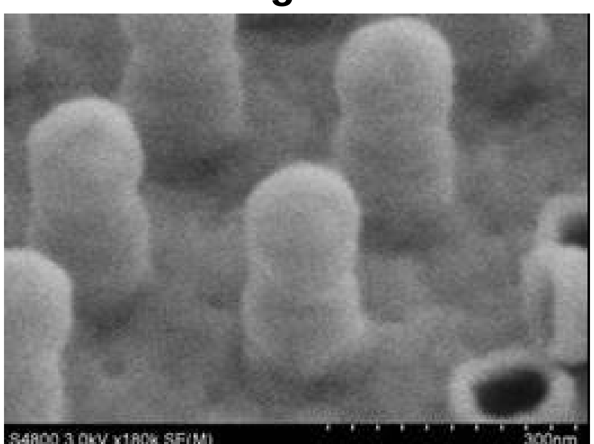
0.4 m CNT

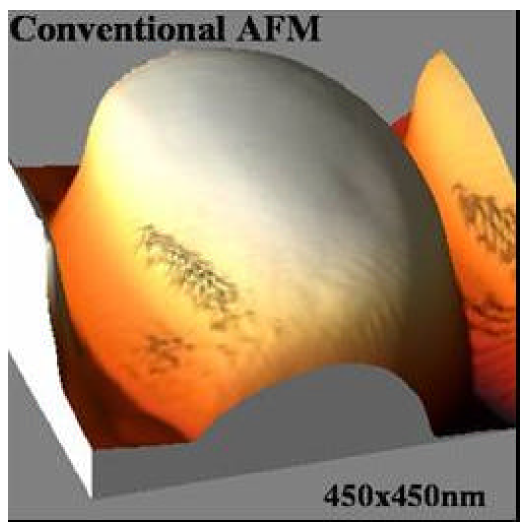


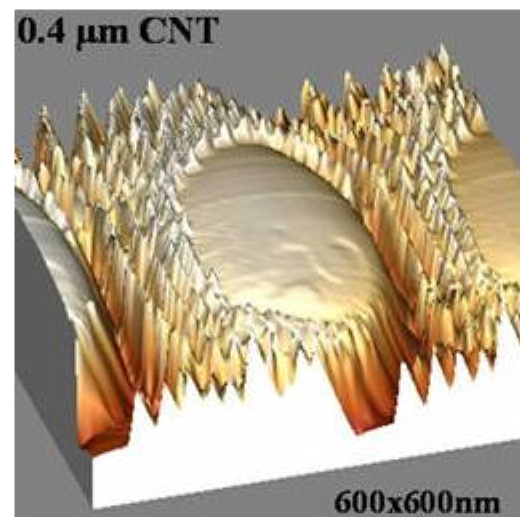


- Divot artifacts associated with switching between attractive (noncontact) and repulsive (tapping states)
- Ringing artifacts associated with CNT adhesion and stiction to sidewalls

300 nm Tungsten nanorods







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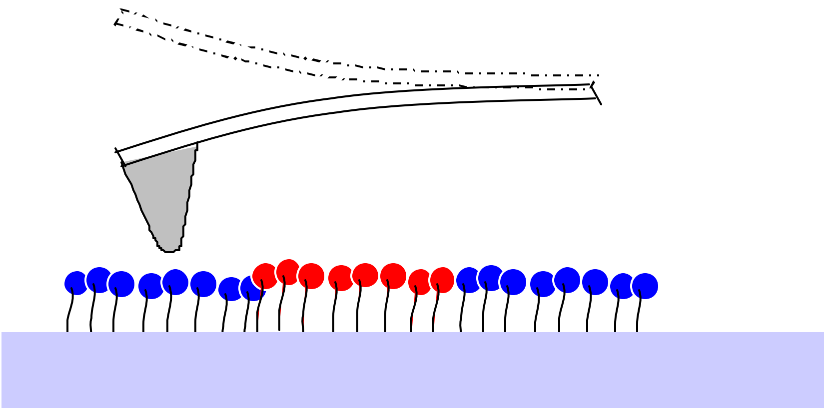
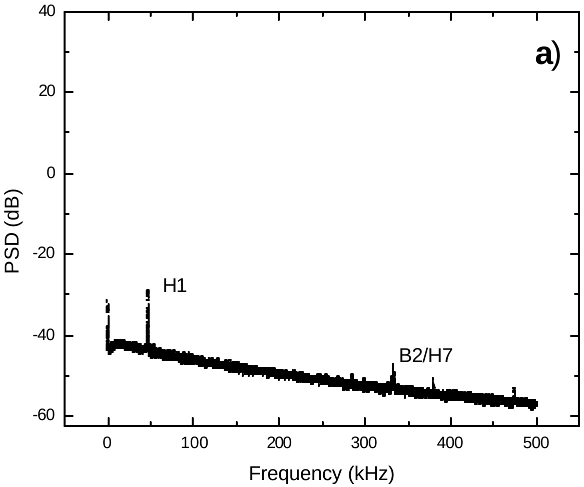
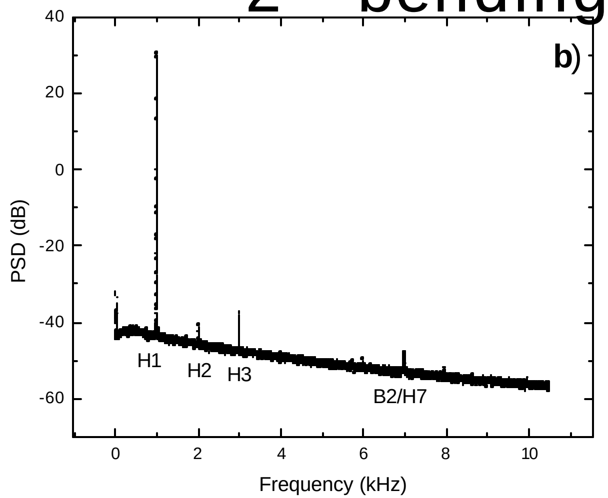
Exploiting anharmonic oscillations



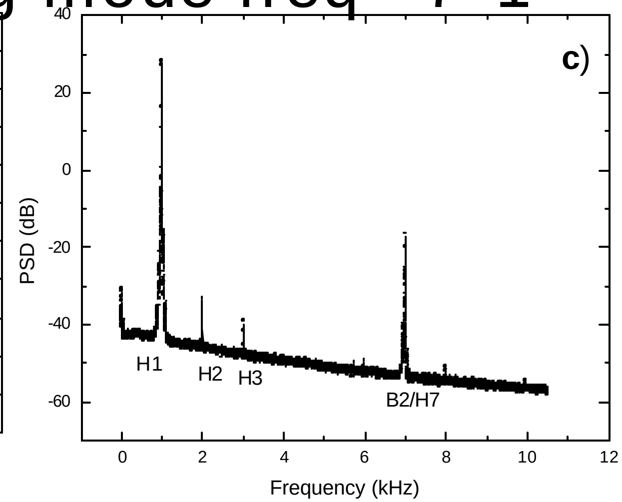
- NC vibration spectrum depends on local adhesion properties
- Experiments performed using 47 kHz microcantilever on wild and mutant bacteriorhodopsin membrane
- 2^{nd} bending mode freq ~$7*1^{st}$



Thermal vibration



Driven in air



On mica (50 % setpoint)

"Probing Van der Waals forces at the nanoscale using higher harmonic dynamic force microscopy", Crittenden, Raman, Reifenberger (in press PRB)

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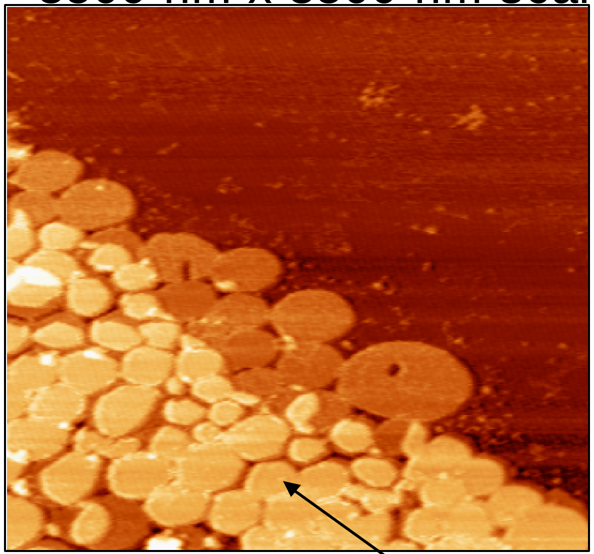
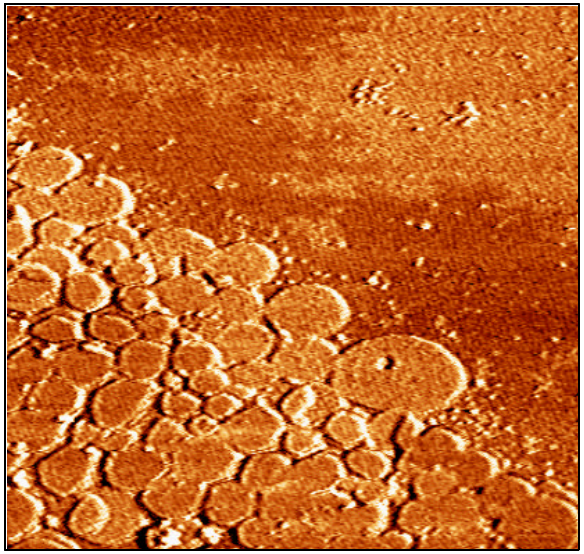
Application to local adhesion estimation

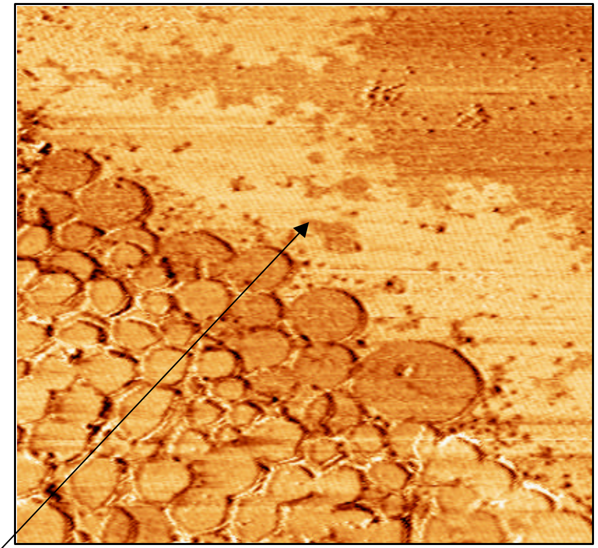
3500 nm x 3500 nm scans



Topography



Second harmonic image



Seventh harmonic image

proteins

Lipid deposits

- Clear distinction between lipids and proteins
- Presence of internal resonance critical in the method
- The method shows promise for the measurement of local attractive forces of soft biomolecules
- Can be extended to electrostatic force microscopy or capacitance microscopy for dopant profiling

"Probing Van der Waals forces at the nanoscale using higher harmonic dynamic force microscopy"
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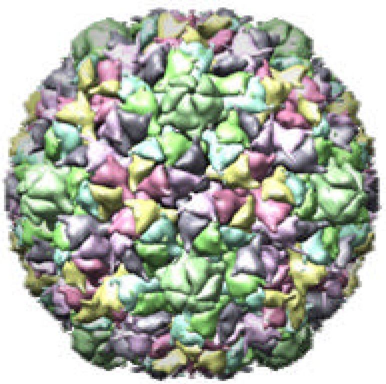
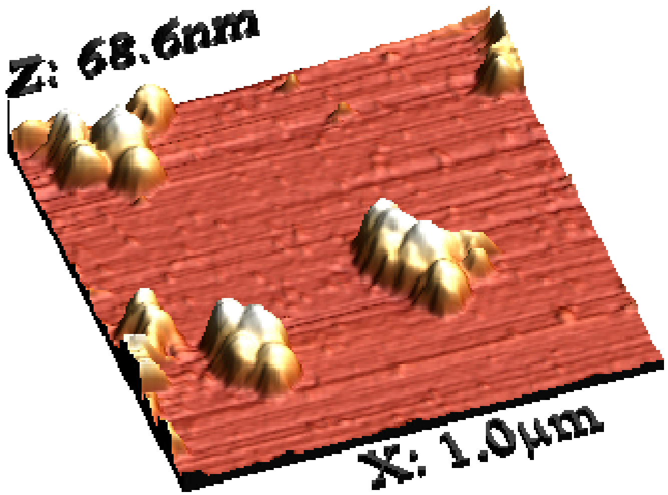
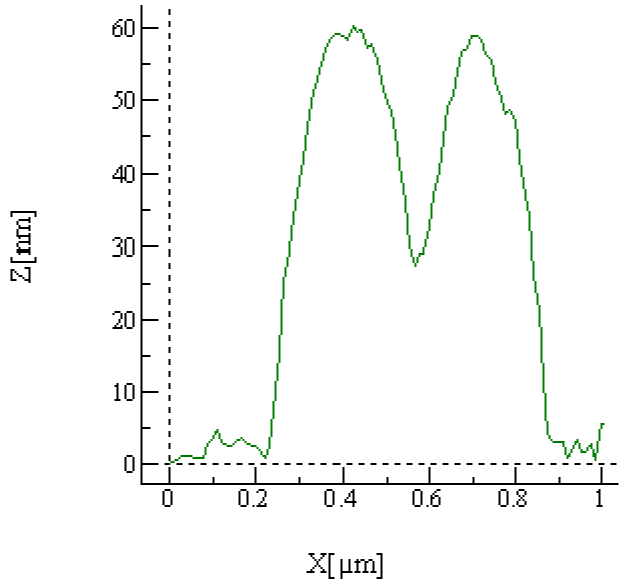
Virus capsid mechanics studied using AFM



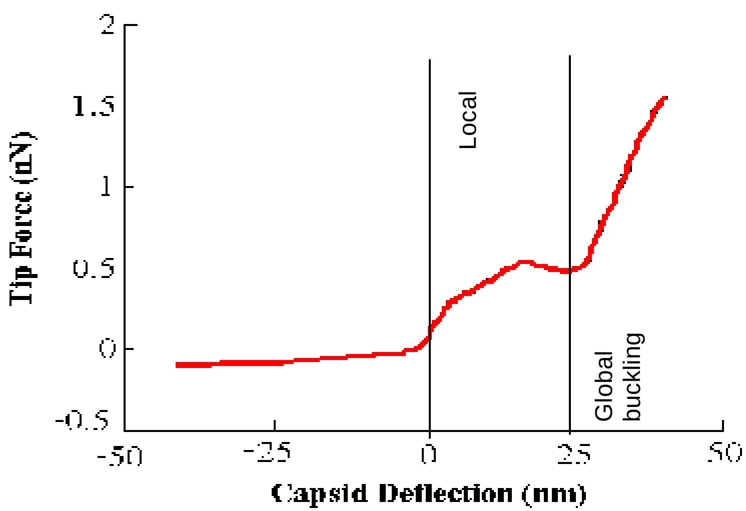
A computer model of the prohead structure of the P22 and HK97 virus cap sids. (T. Ferrin, UCS F Computer Grap hics Lab)

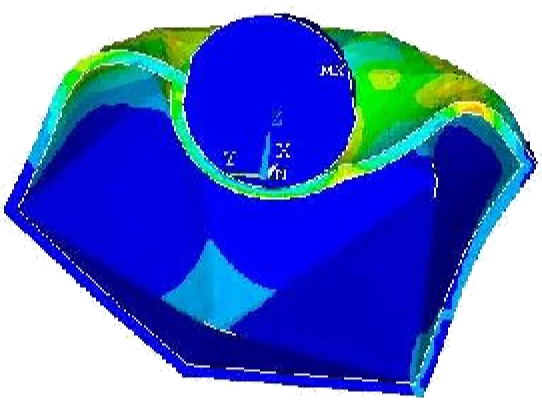


Bacteriophage P22 (in buffer)



Experimental force-indentation





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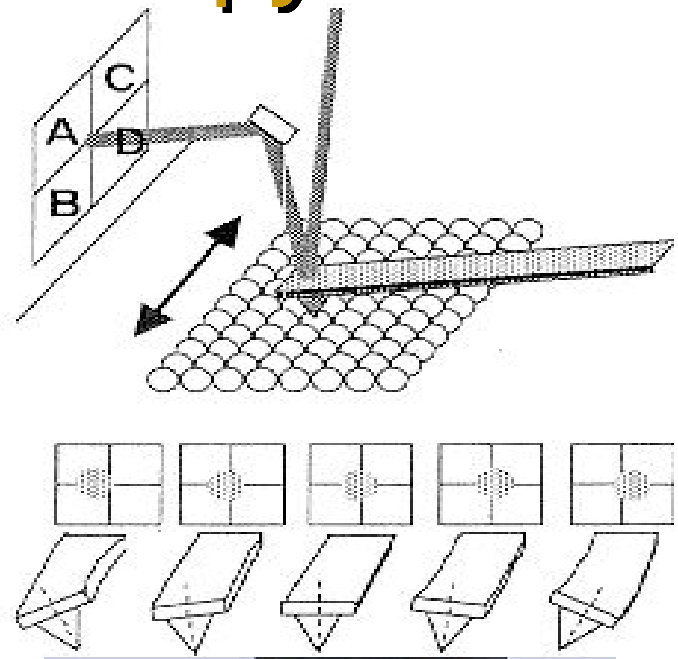
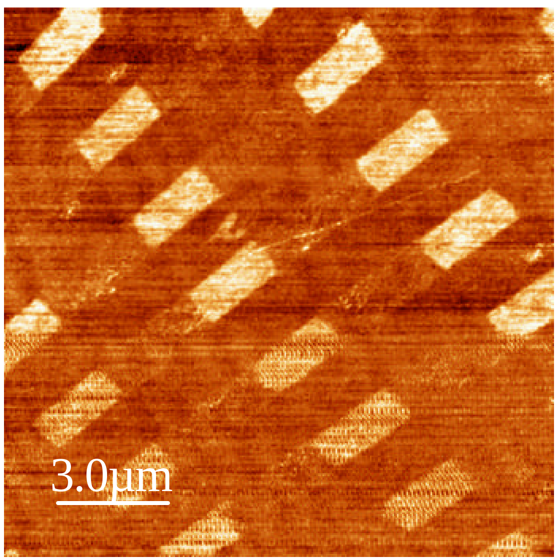
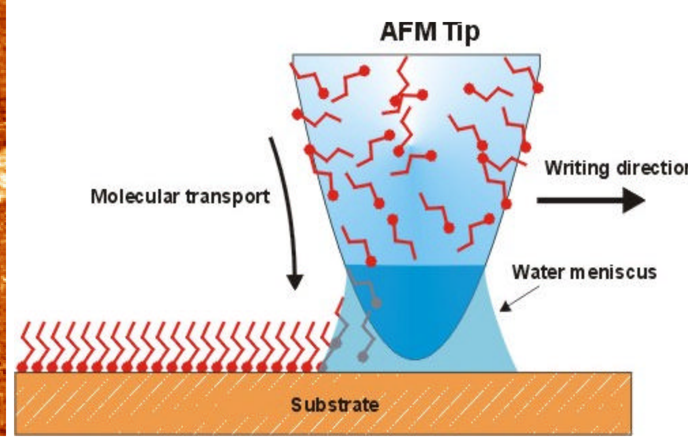
Friction Force Microscopy

- Torsional vibrations due to atomic and molecular friction
- Lateral forces are specific
- Applications to nanotribology, probe based lithography

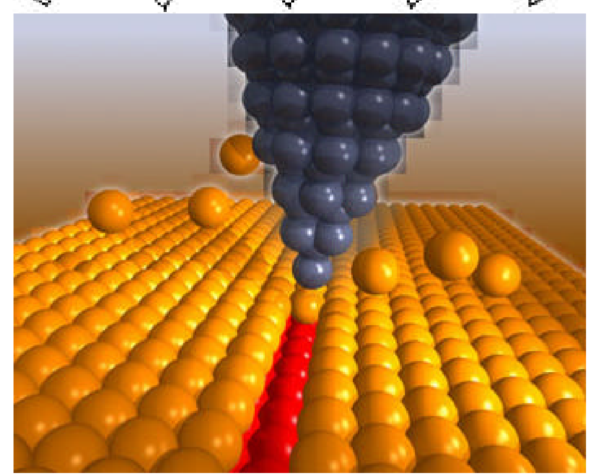




Friction force image of a self assembled monolayer (Riefenberger Group)



www.chem.nwu.edu/~mkngrp/
Dip-pen lithography



Contact mode oxidation lithography

Conley, Raman, Krousgrill, submitted J

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Acknowledgements

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  - Sebastian Rützel (Darmstadt)
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  - Amy McGough (Biology, Purdue)
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  - NASA
  - DoE
  - Purdue Research Foundation

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Questions & Answers

Application to local adhesion estimation

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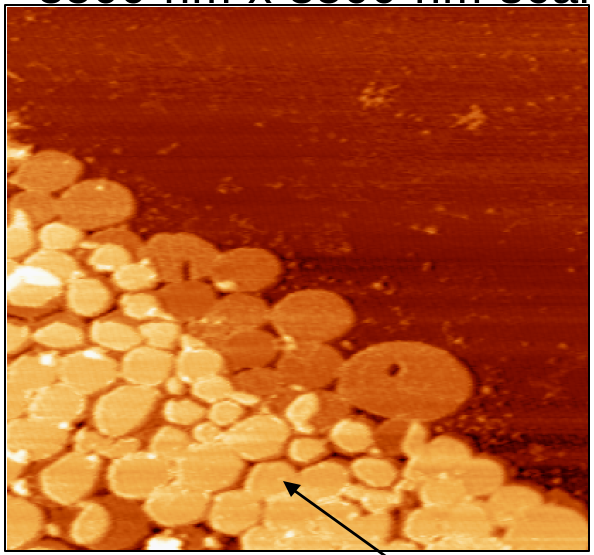
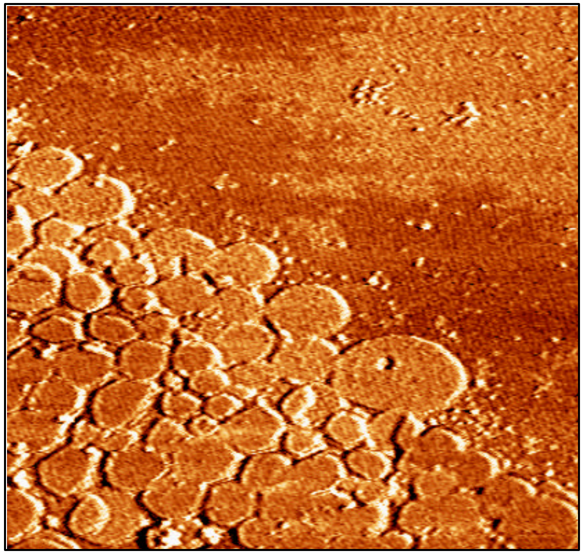
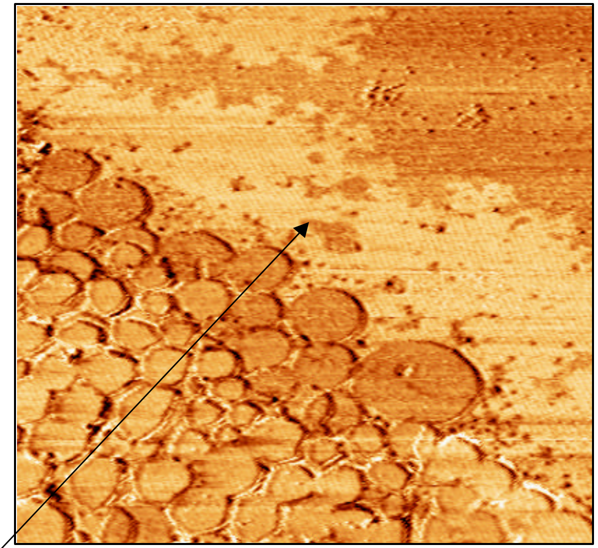


Topography



Second harmonic ima
ge



Seventh harmonic imag
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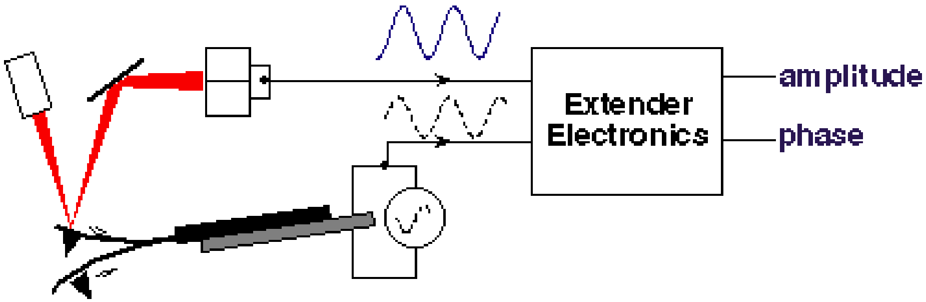
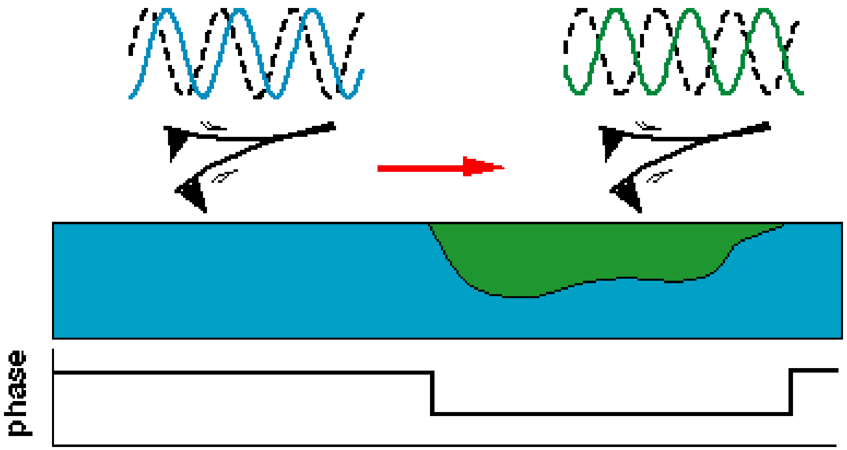
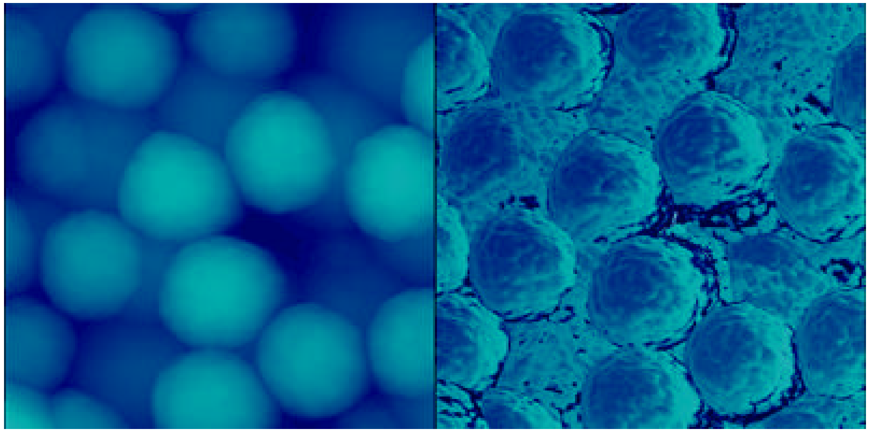
Phase Imaging







AFM height (left) and phase (right) images of poly(methylmethacrylate)

(Digital Instruments, Inc.)

- Regular tapping mode implemented but signal phase monitored
- Phase contrasts are related to differences in local dissipation