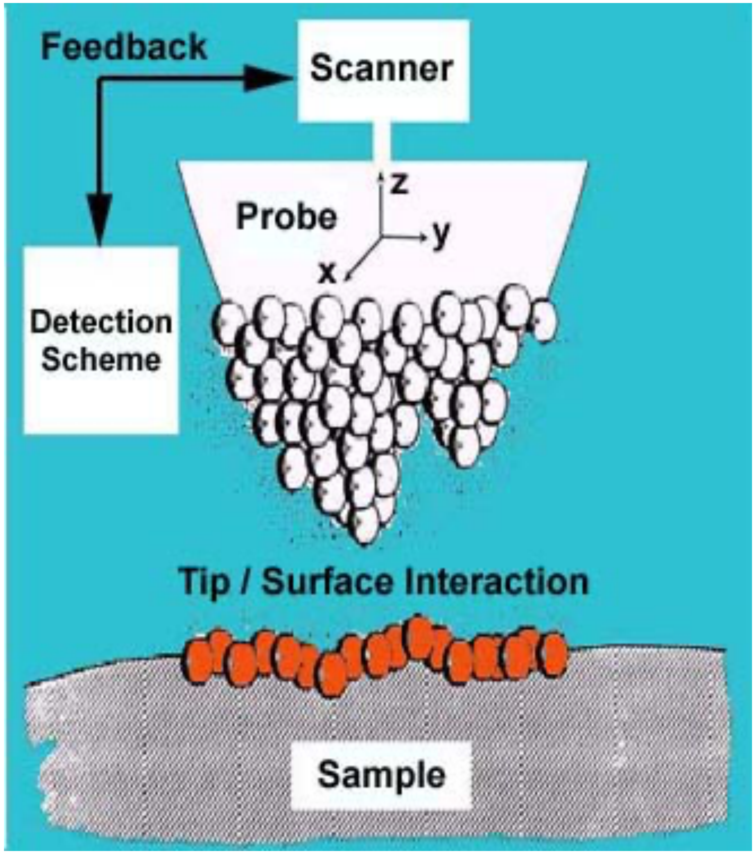
Scanning Probe Microscopy (SPM)



Scanning probe microscope (**SPM**) is a branch of microscopy that forms images of surfaces using a physical probe that scans the specimen. SPM was founded in 1981, with the invention of the scanning tunneling microscope. The key to their success was using a feedback loop to regulate gap distance between the sample and the probe. Many scanning probe microscopes can image several interactions simult-aneously. The manner of using these interactions to obtain an image is generally called a mode.

The resolution varies somewhat from technique to technique, but some probe techniques reach a rather impressive atomic resolution. The other common denominator is that the data are typically obtained as a two-dimensional grid of data points, visualized in false color as a computer image.

Scanning Tunneling Microscopy (STM)

--- G. Binnig, H. Rohrer et al, (1982)

Near-Field Scanning Optical Microscopy (NSOM)

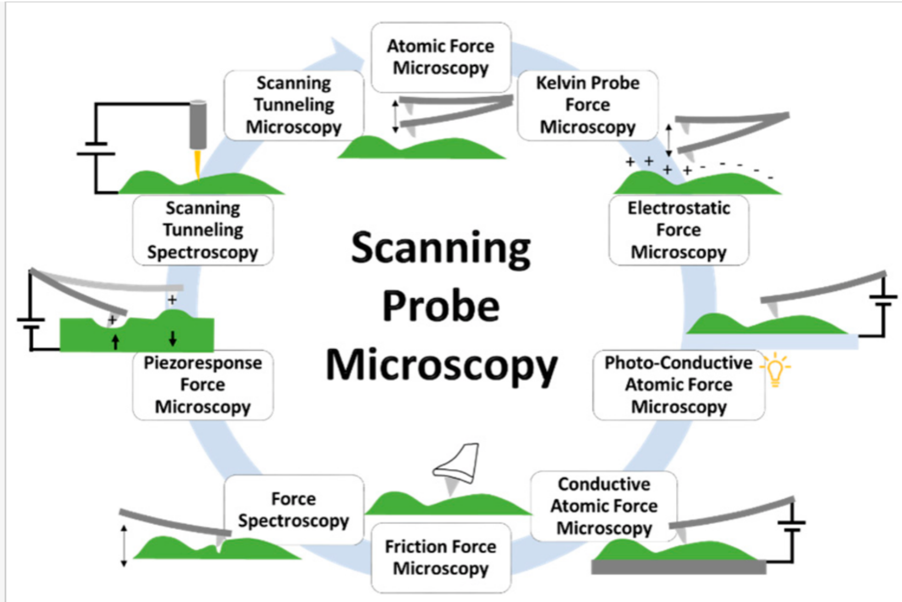
--- D. W. Pohl (1982)

Atomic Force Microscopy (AFM)

--- G. Binnig, C. F. Quate, C. Gerber (1986)

Scanning Thermal Microscopy (SThM)

--- C. C. Williams, H. Wickramasinghe (1986))



Magnetic Force Microscopy (MFM)

--- Y. Martin, H. K. Wickramasinghe (1987)

Friction Force Microscopy (FFM or LFM)

--- C. M. Mate et al (1987)

Electrostatic Force Microscopy (EFM)

--- Y. Martin, D. W. Abraham et al (1988)

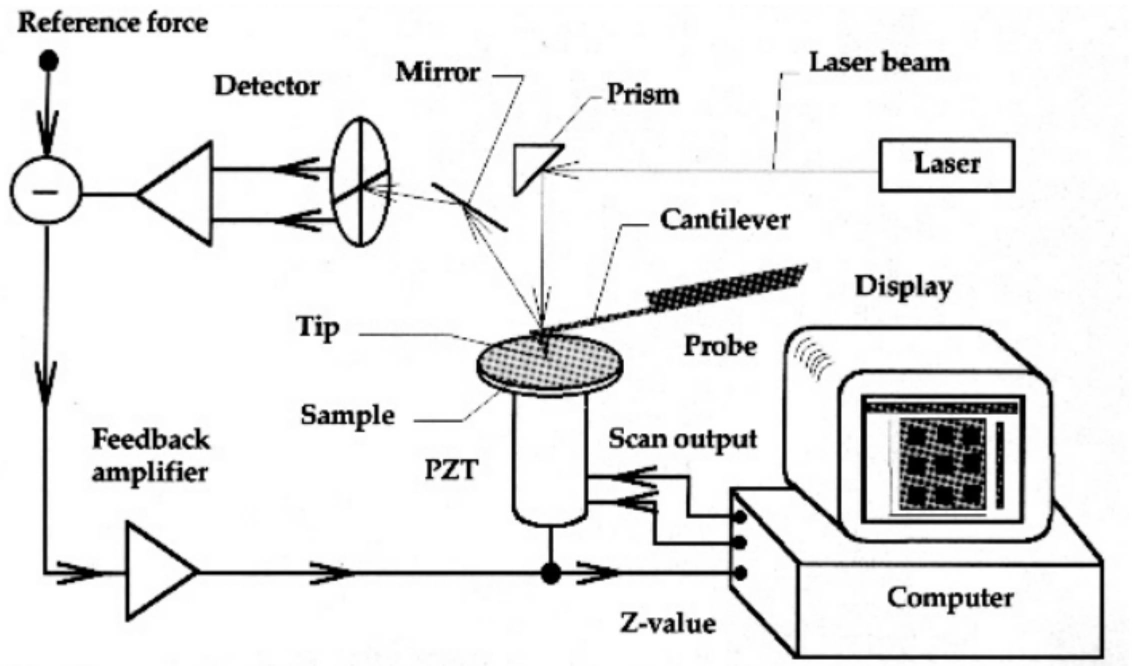
Scanning Capacitance Microscopy (SCM)

--- C. C. Williams, J. Slinkman et al (1989)

Force Modulation Microscopy (FMM)

--- P. Maivald et al (1991)

Atomic Force Microscopy (AFM)



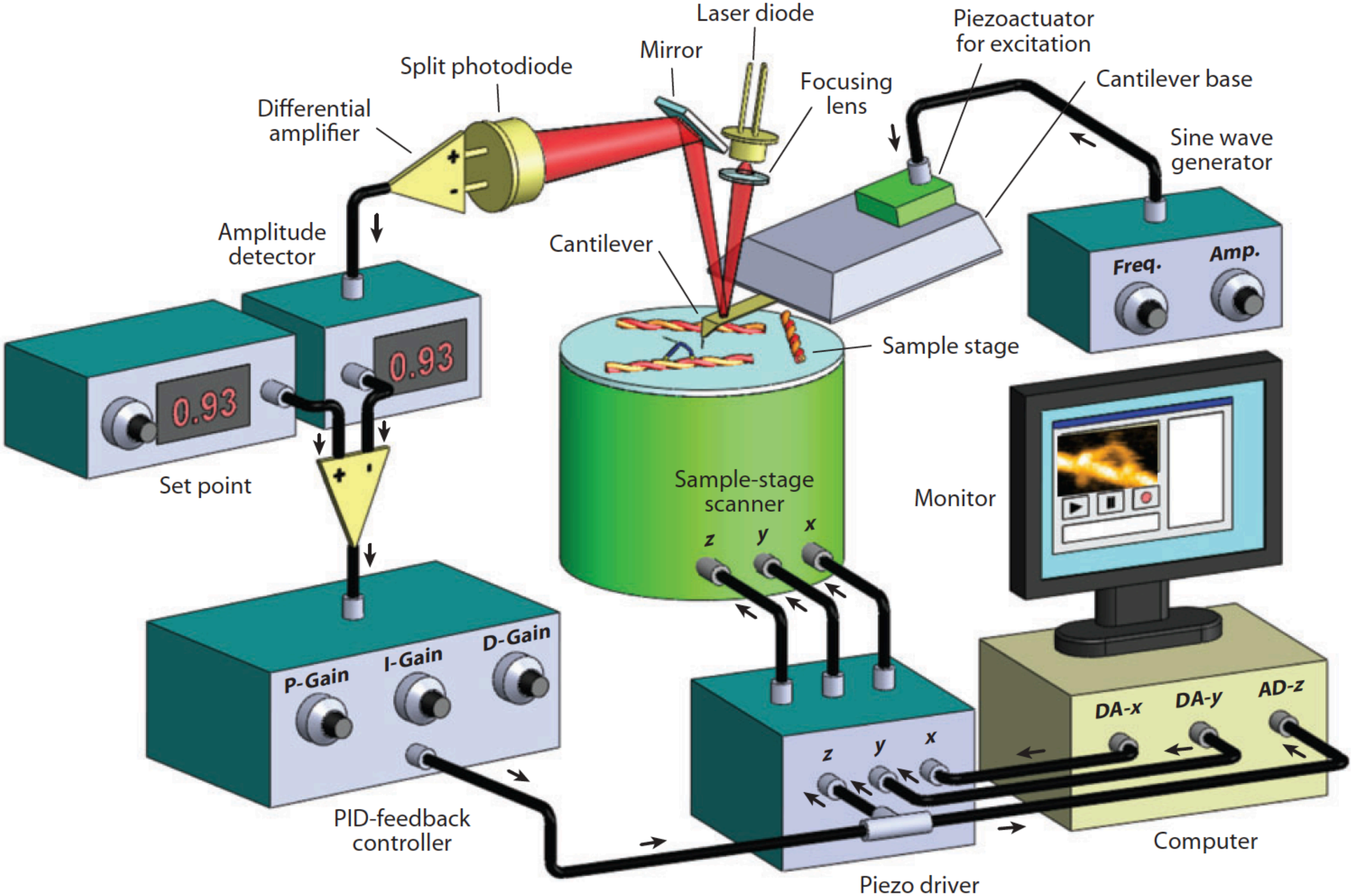
$$F = k\Delta z$$

$$F = 10^{-9} – 10^{-6}\ N$$

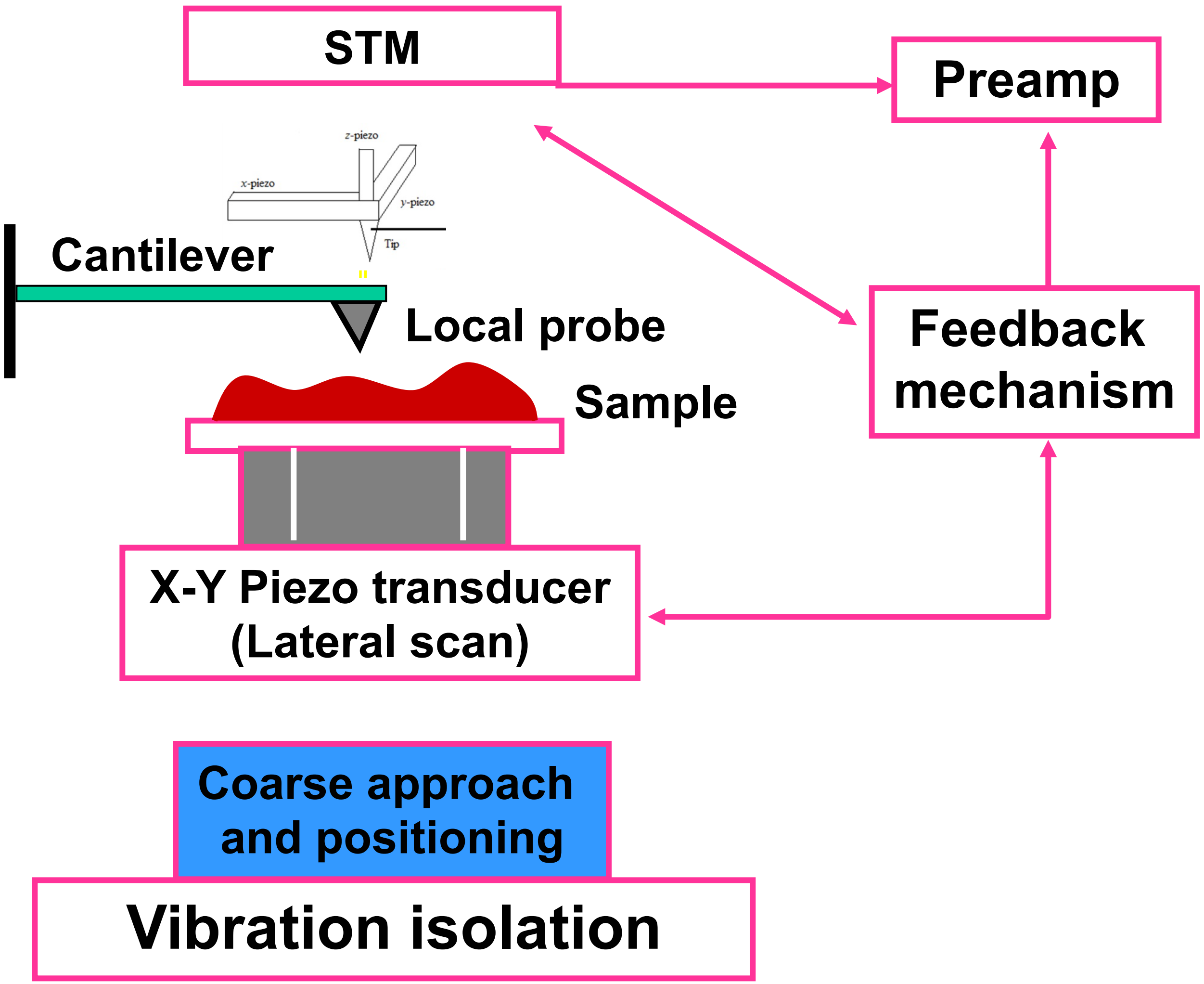
$$k = 0.1 – 1\ N/m$$

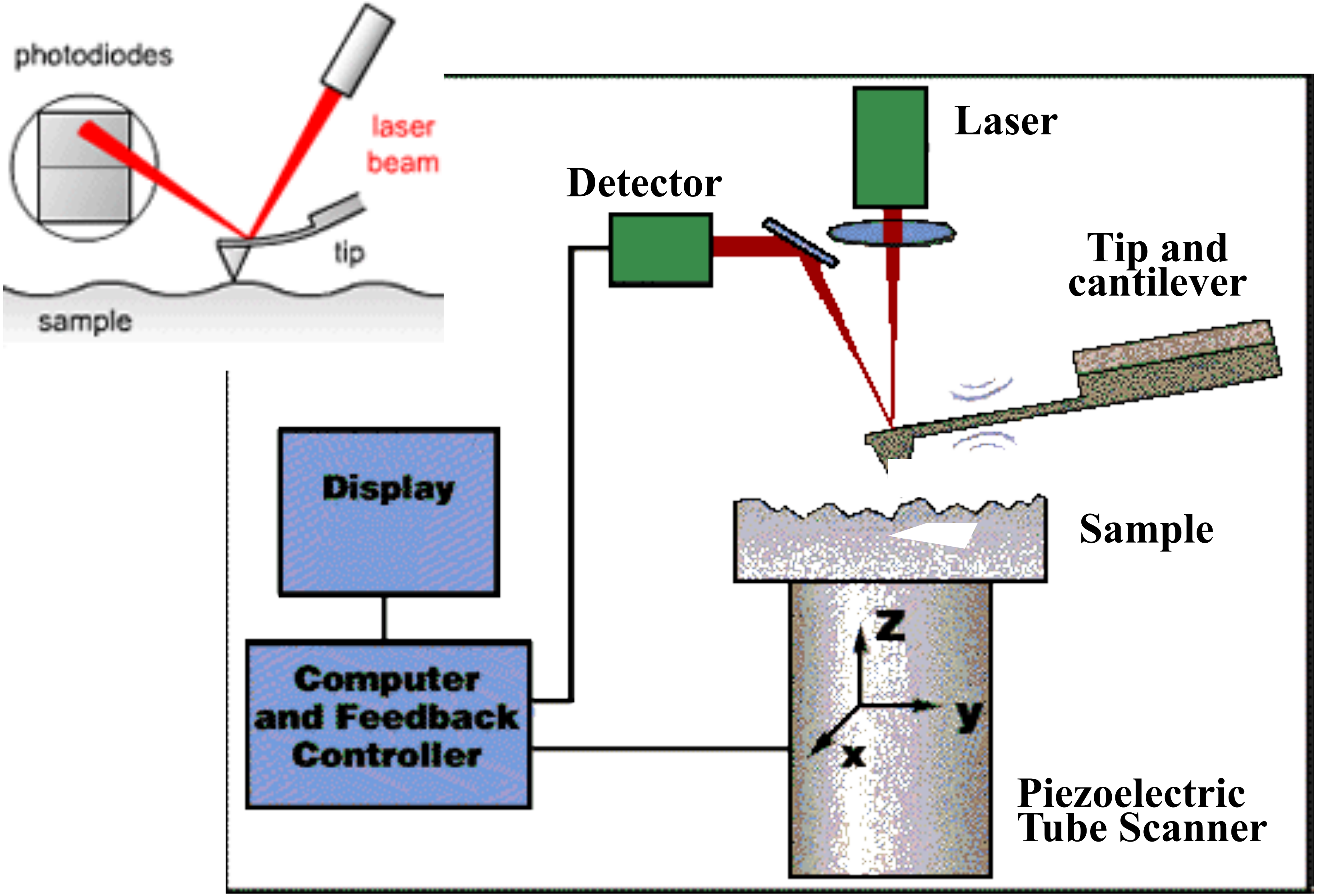
References:

- G. Binnig, C. F. Quate, and C. Gerber, Phys. Rev. Lett. 56, 930 (1986).
- C. Bustamante and D. Keller, Physics Today, 32, December (1995).
- R. Wiesendanger and H.J. Güntherodt, *Scanning Tunneling Microscopy II* , Springer-Verlag, (1992).


Laser diode
Piezoactuator for excitation
Mirror
Split photodiode
Focusing lens
Cantilever base
Differential amplifier
Sine wave generator
Amplitude detector
Cantilever
Freq.
Amp.
Sample stage
0.93
0.93
Set point
Sample-stage scanner
Monitor
z
y
x
P-Gain
I-Gain
D-Gain
DA-x
DA-y
AD-z
PID-feedback controller
Computer
Piezo driver

Key components of AFM





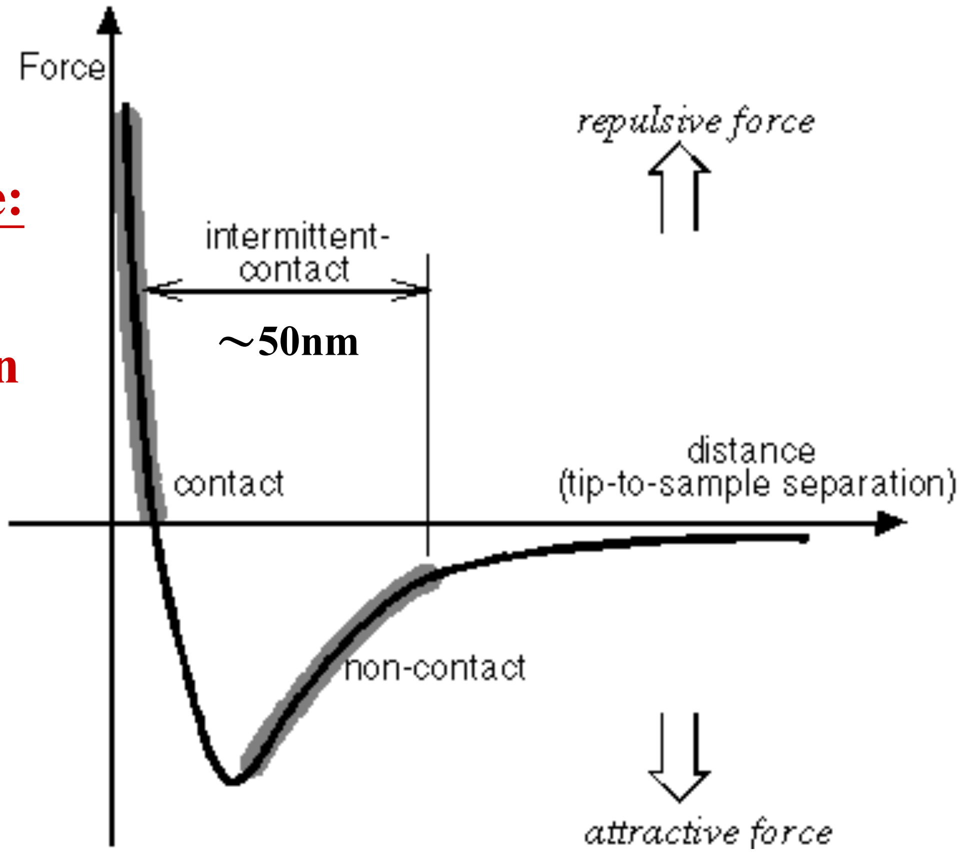
photodiodes
laser
beam
tip
sample
Laser
Detector
Tip and
cantilever
Display
Computer
and Feedback
Controller
Sample
Z
y
x
Piezoelectric
Tube Scanner

Interaction between the probe and sample

Short-range:

1) Bonding

2) Repulsion



Long-range:

1) Van der Waal

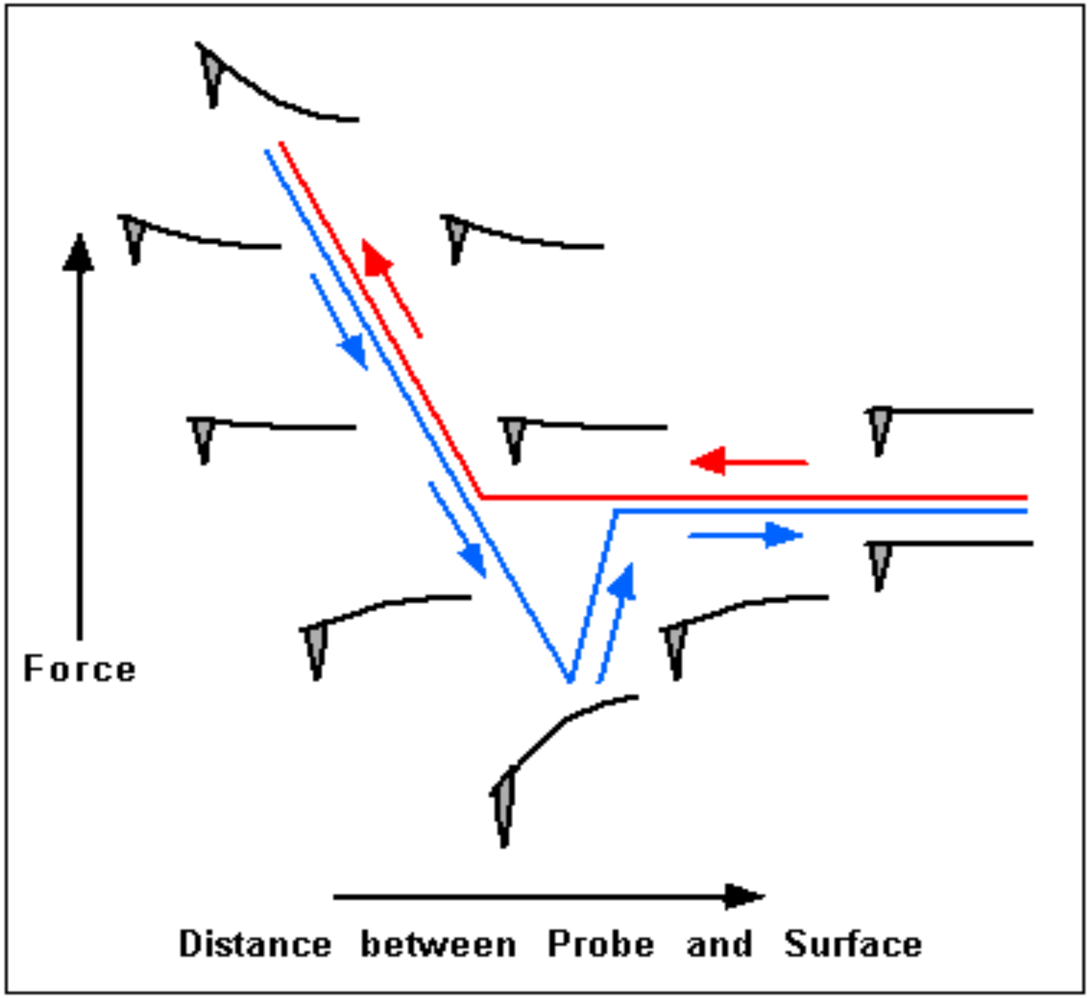
2) Capillary

3) Magnetic

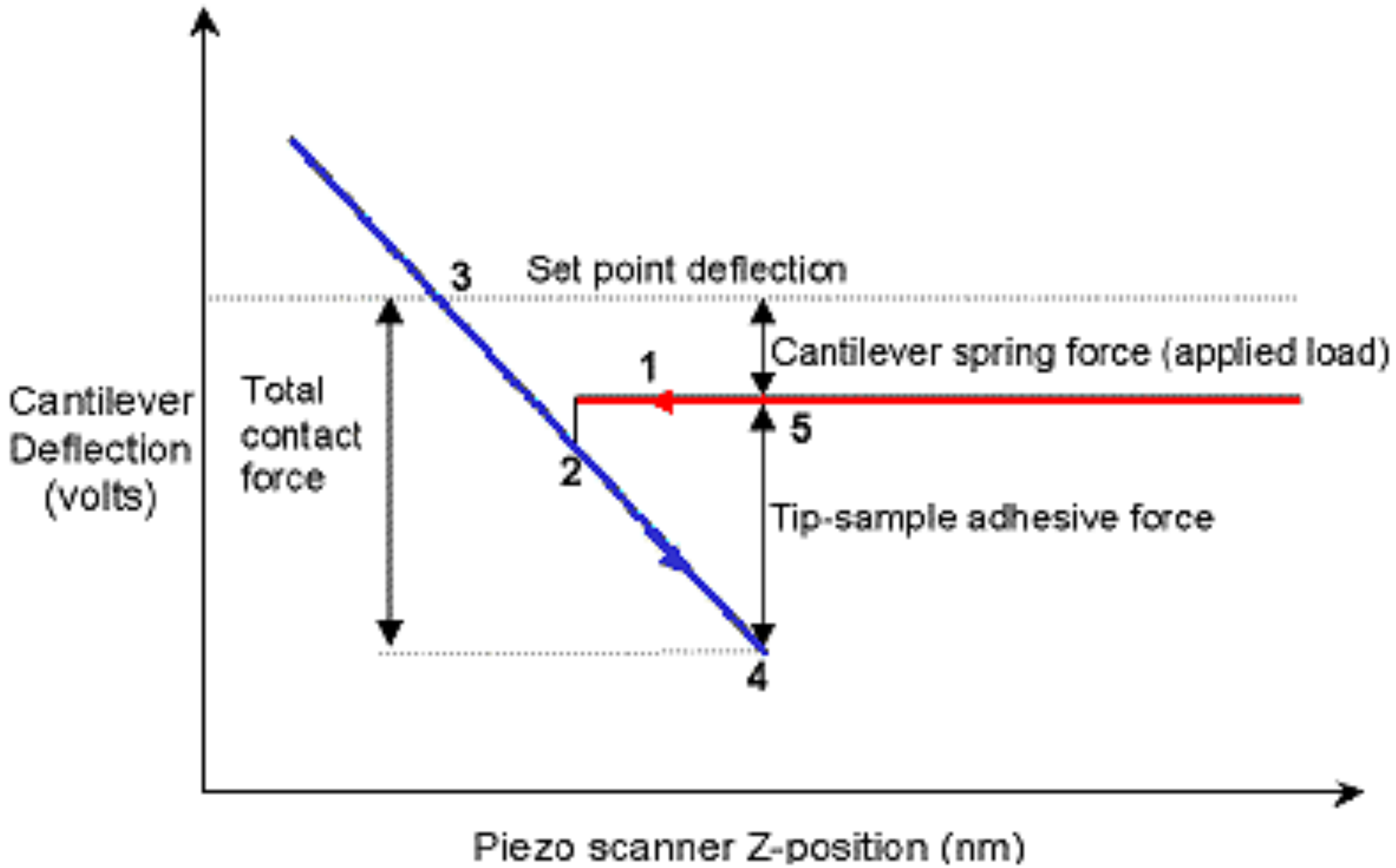
4) Electrostatic

Lennard-Jones potential $\phi(r) = - A/r^6 + B/r^{12}$

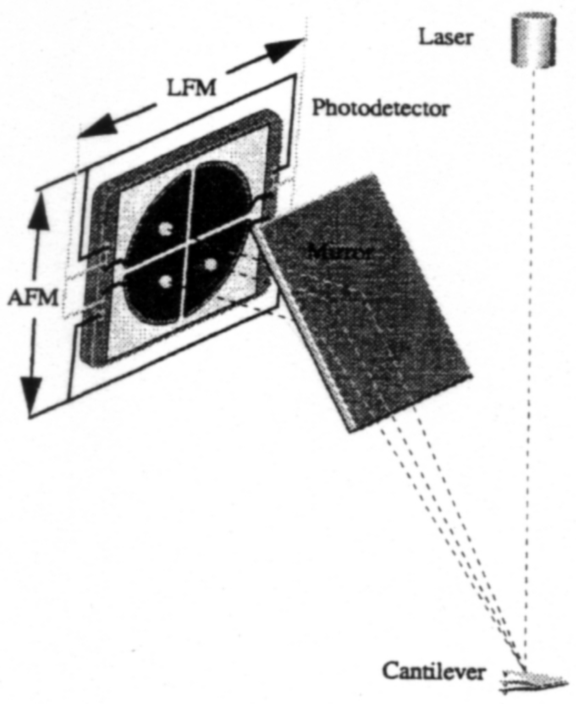
Reaction of the probe to the force

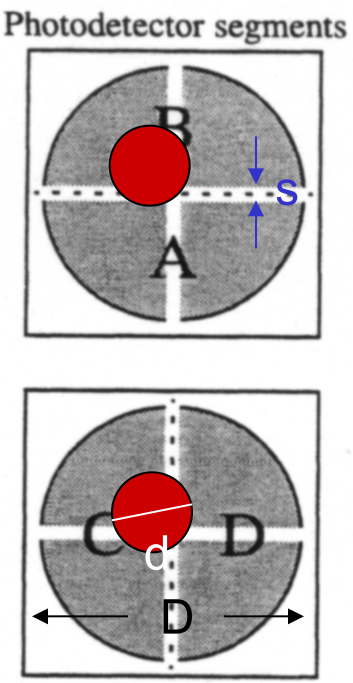


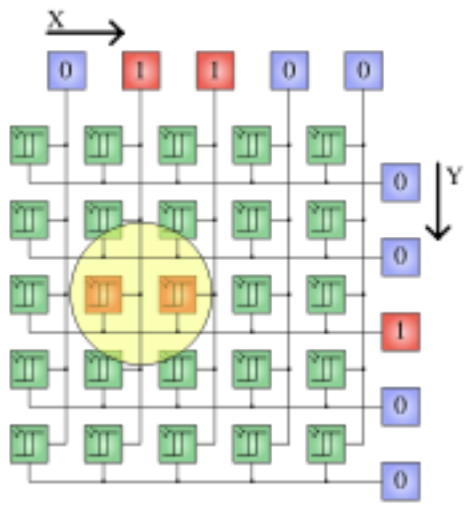
Deflection of Cantilever vs Piezo displacement



Position-sensitive Photo Diode (PSPD)

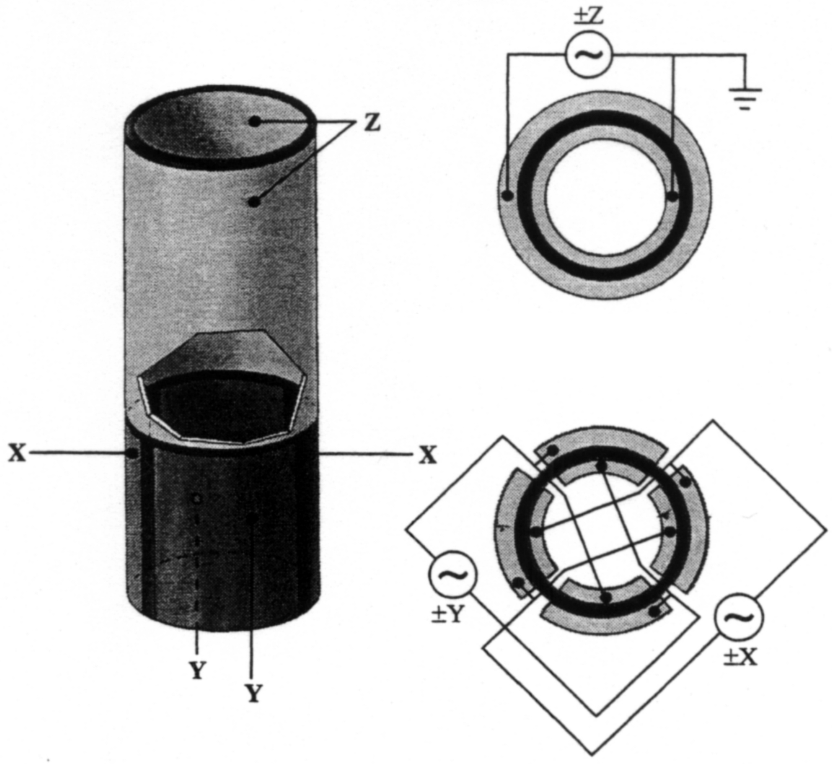


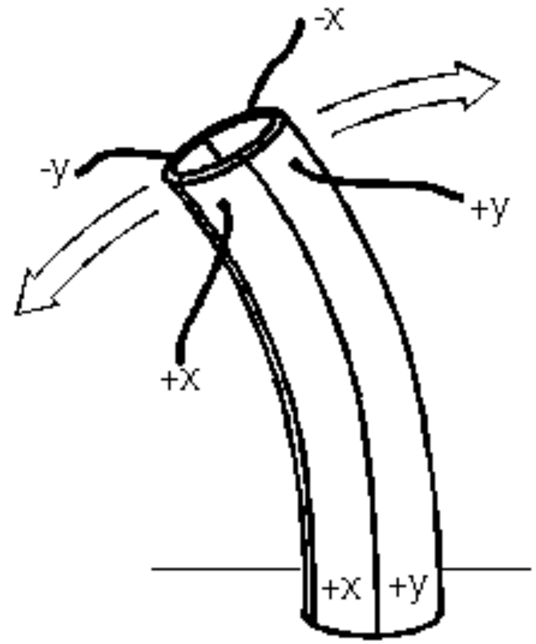




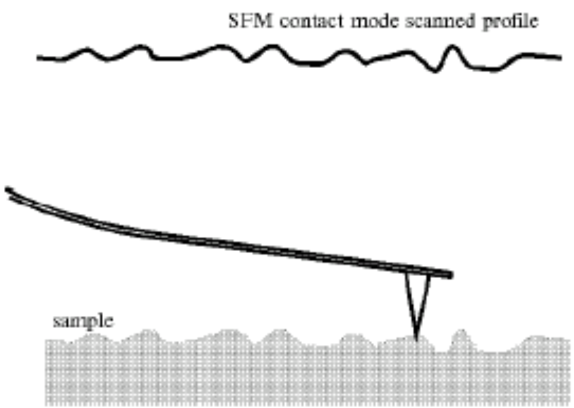
D ~ 10mm   d ~ 1mm   s ~ 0.01mm

Piezo Scanner

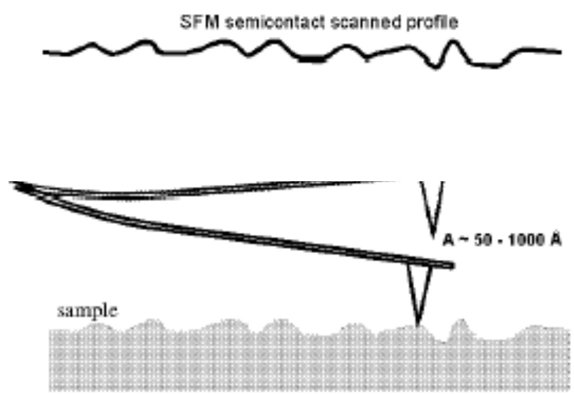




Three scanning modes of AFM

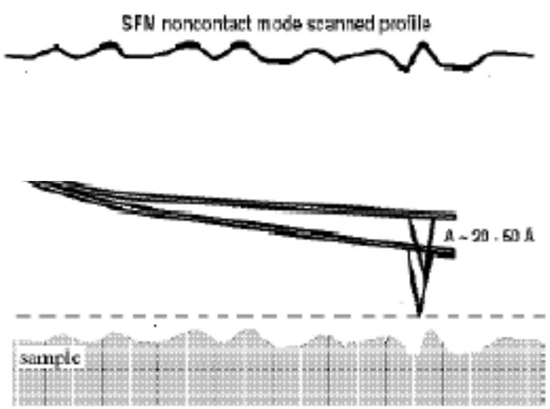


Contact Mode AFM



Semicontact Mode AFM

(Tapping mode)



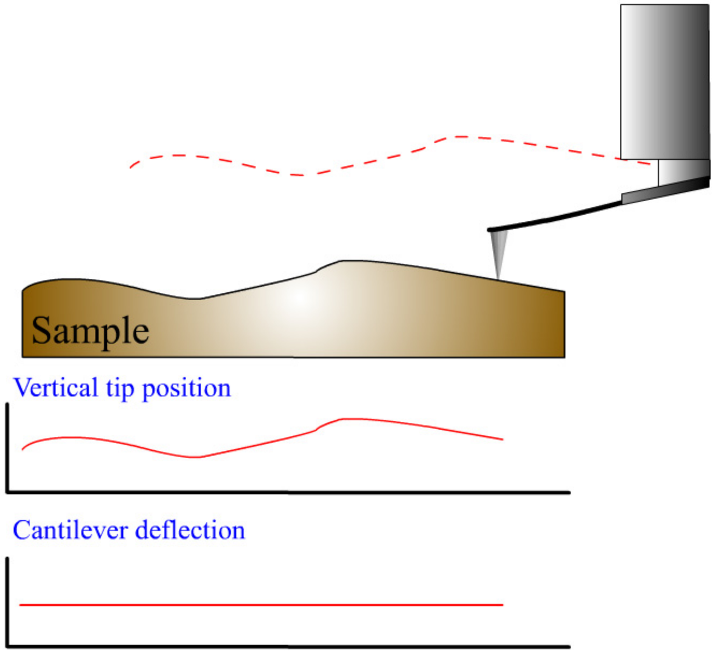
Non-contact Mode AFM

Two imaging methods in contact mode

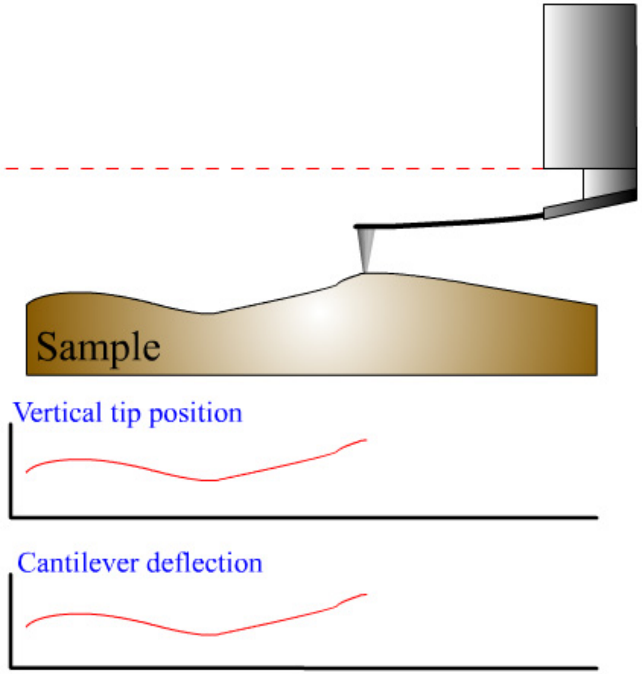
- Constant force method： By using a feedback loop the tip is vertically adjusted in such a way that the force always stays constant. The tip then follows a contour of a constant contact force during scanning. A kind of a topographic image of the surface is generated by recording the vertical position of the tip.

- Constant height method： In this mode the vertical position of the tip is not changed, equivalent to a slow or disabled feedback. The displacement of the tip is measured directly by the laser beam deflection. One of its advantages is that it can be used at high scanning frequencies.

Constant-force scan vs. constant-height scan



Constant-force mode



Constant-height mode

Constant-force scan vs. constant-height scan

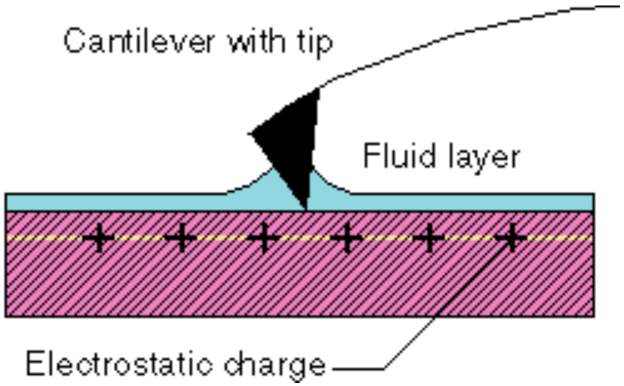
Constant-force

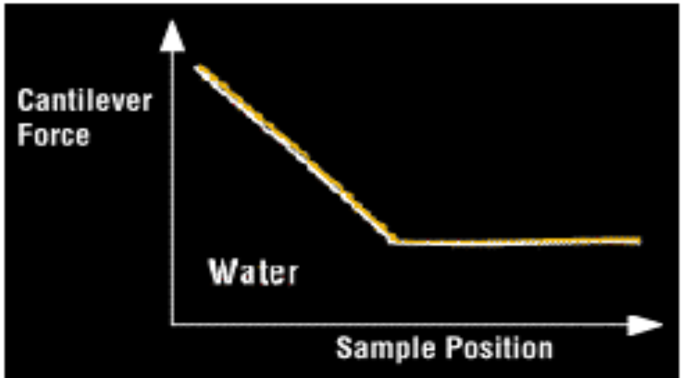
- Advantages:
  - Large vertical range
  - Constant force (can be optimized to the minimum)
- Disadvantages:
  - Requires feedback control
  - Slow response

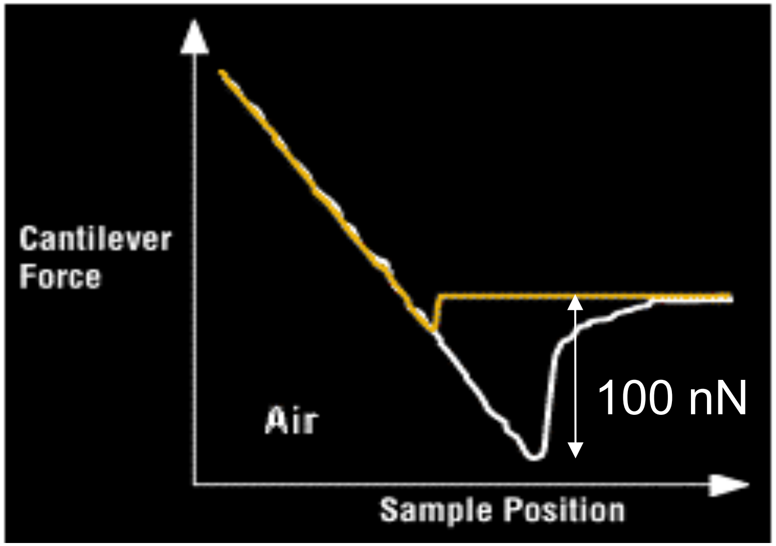
Constant-height

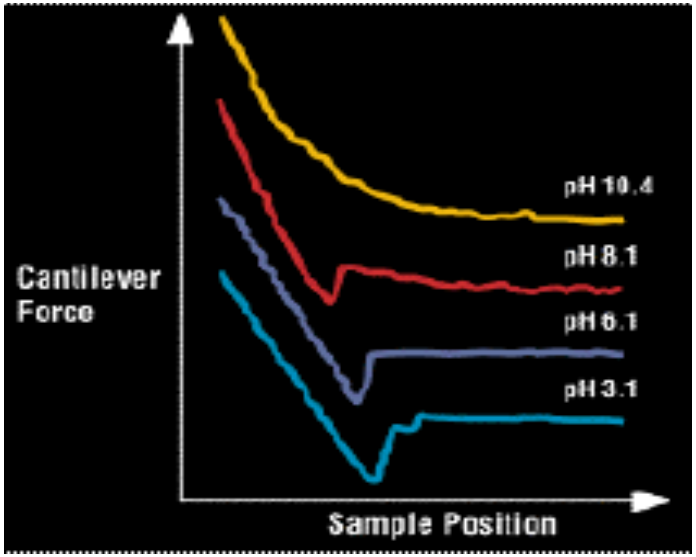
- Advantages:
  - Simple structure (no feedback control)
  - Fast response
- Disadvantages:
  - Limited vertical range (cantilever bending and detector dynamic range)
  - Varied force

Problems with the contact mode

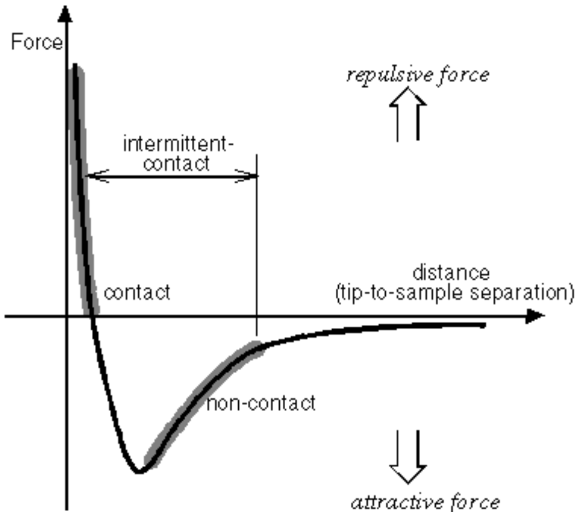




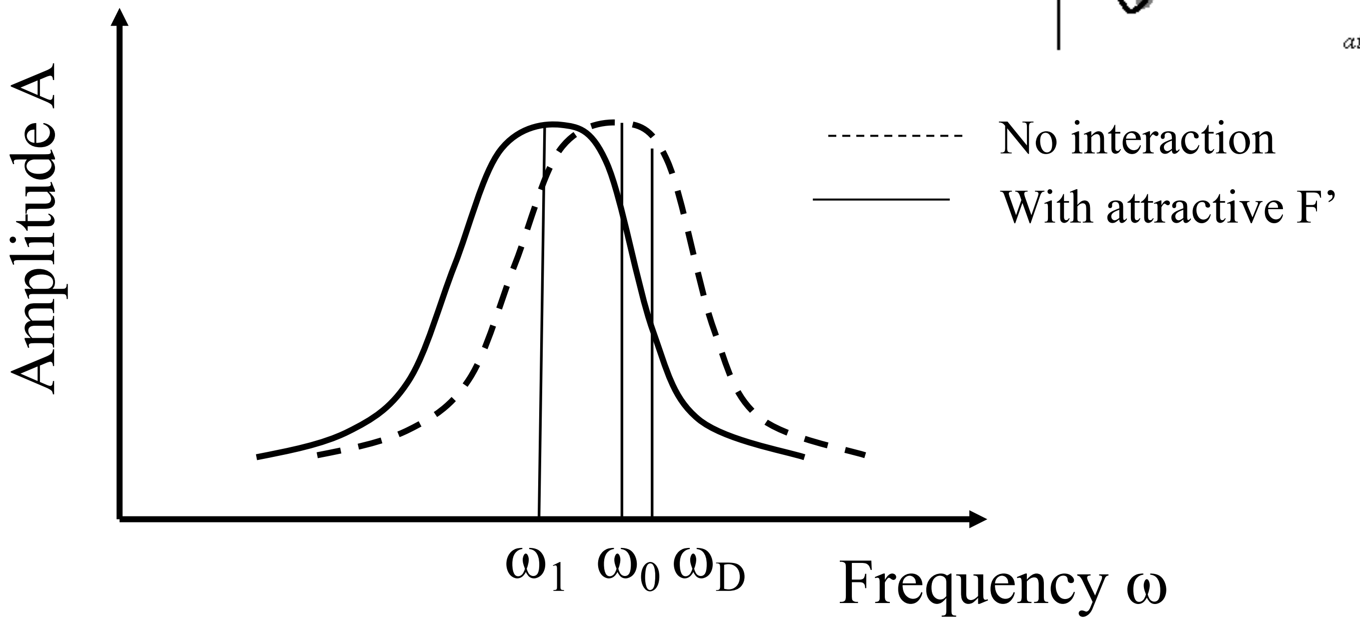




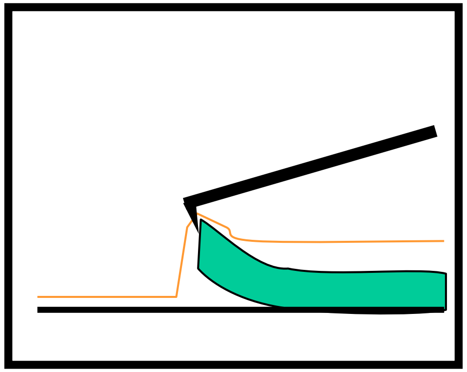
AC imaging mode



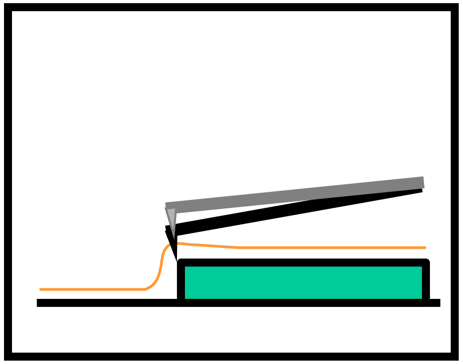
$$\omega_1 = \omega_0\,(1 + F'/2c)$$



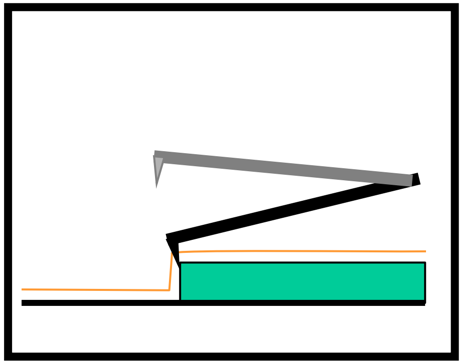
Comparison of three scanning modes



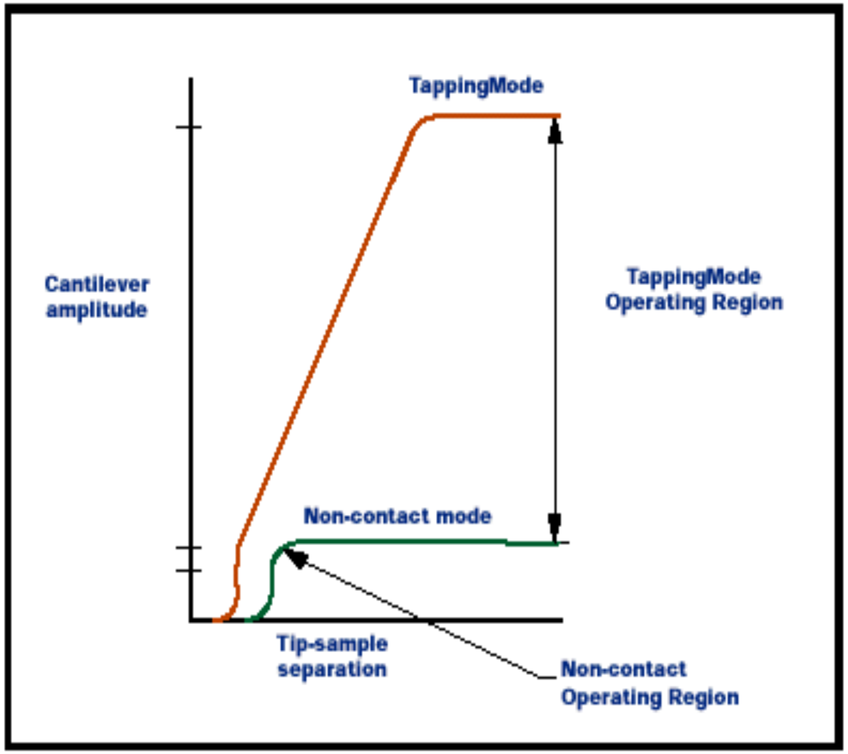
Contact



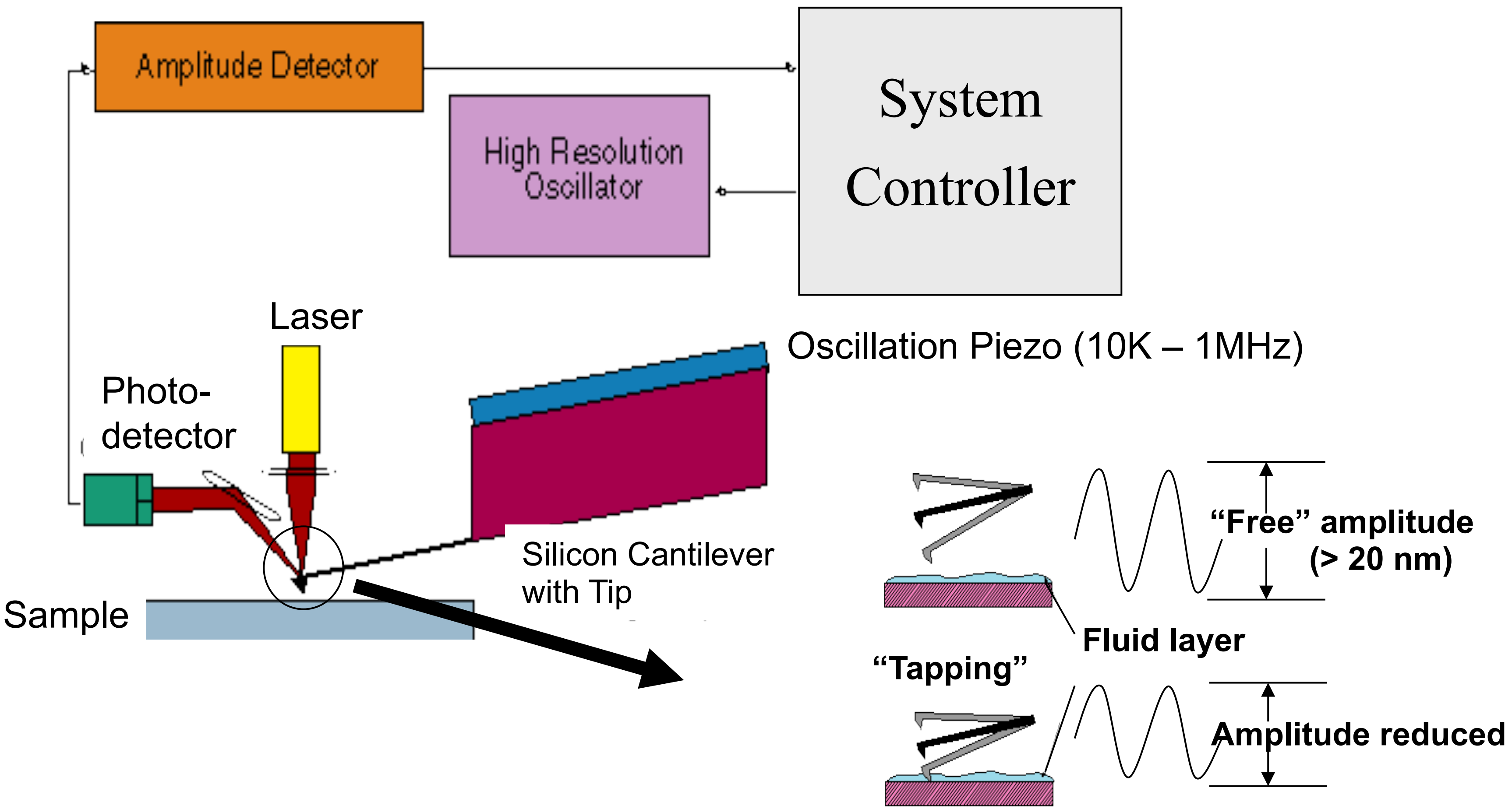
Non-contact



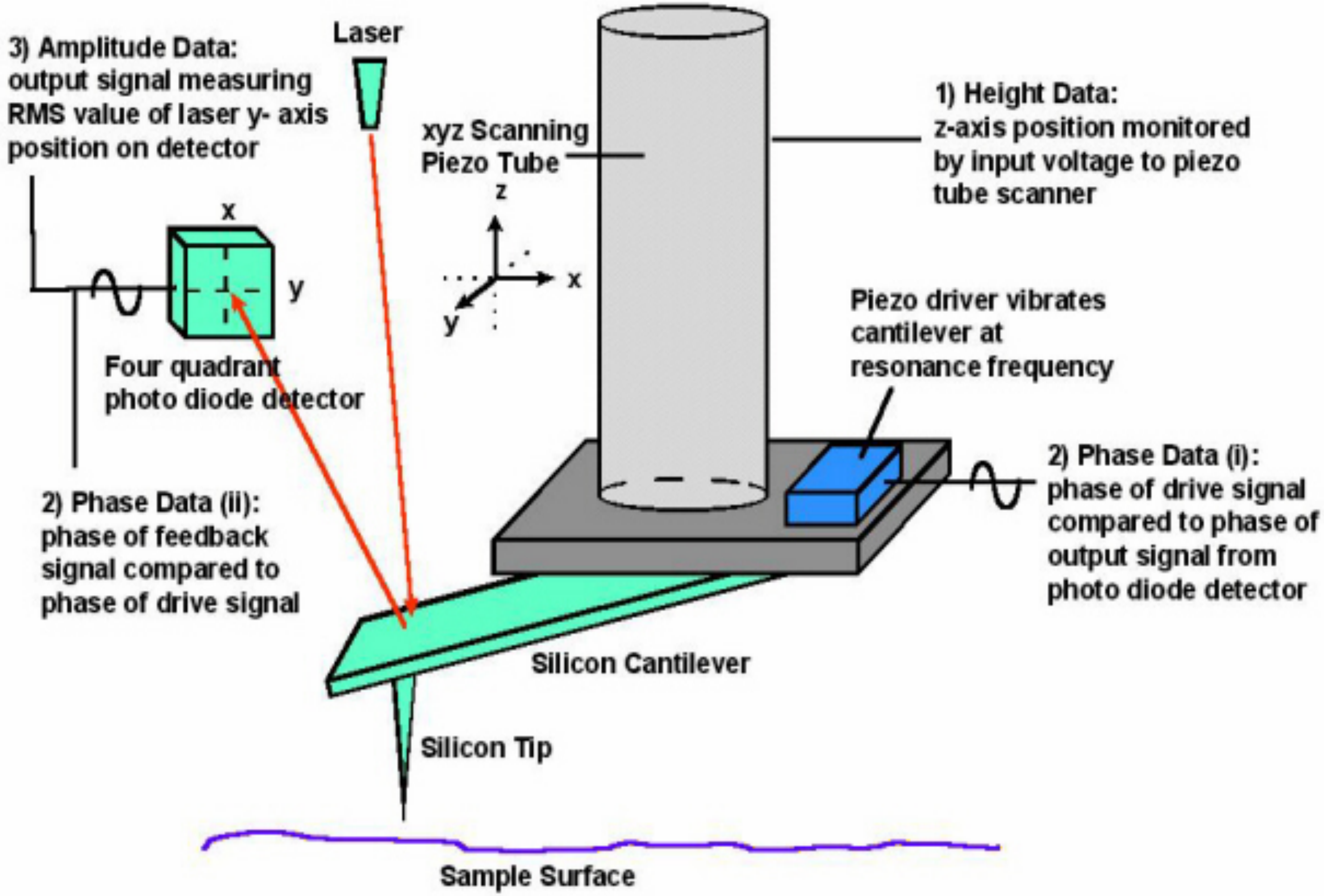
Intermittent-contact



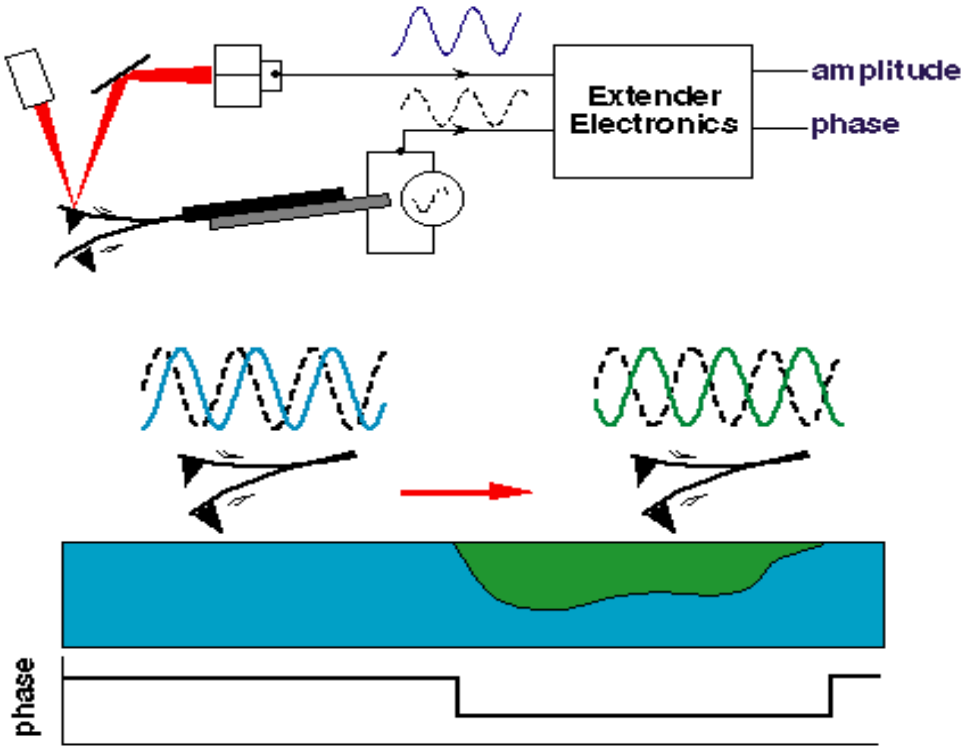
Tapping mode

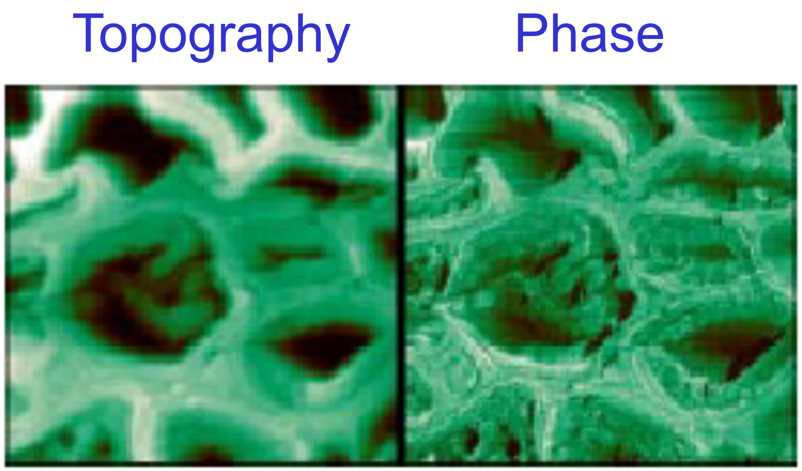


Three Types of Data Collected in Tapping Mode



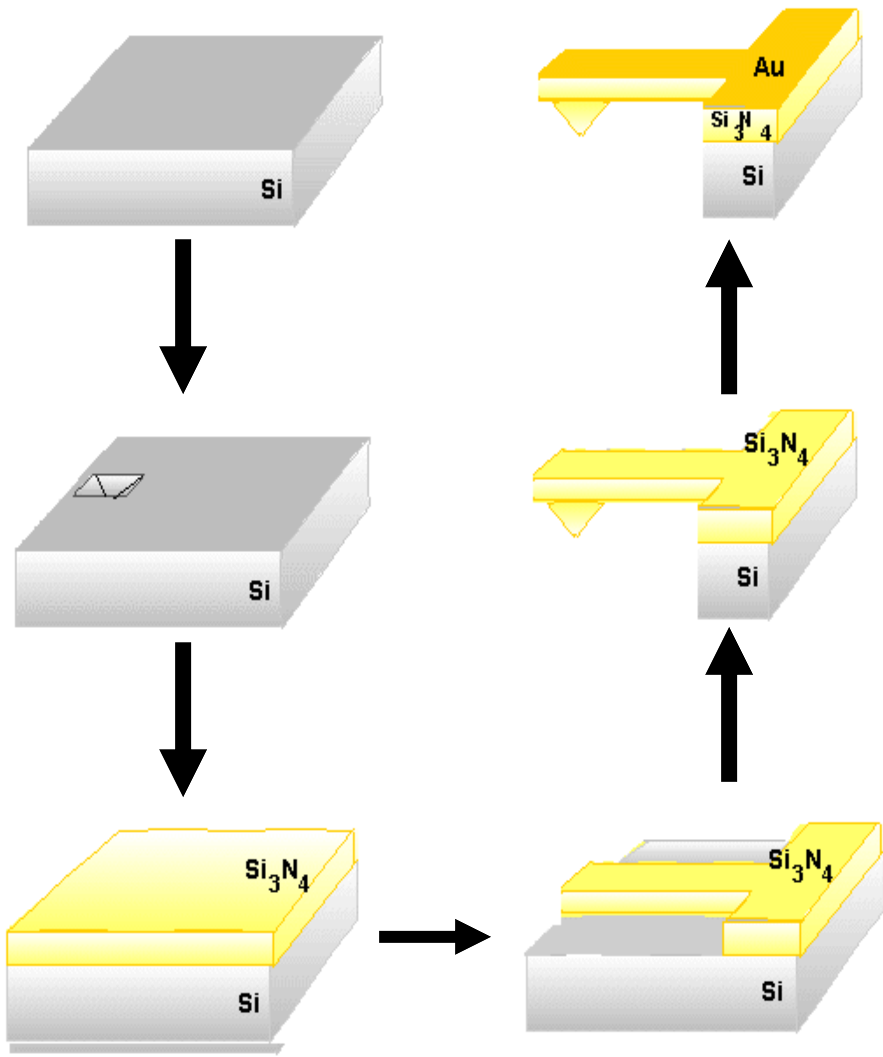
Images by tapping mode

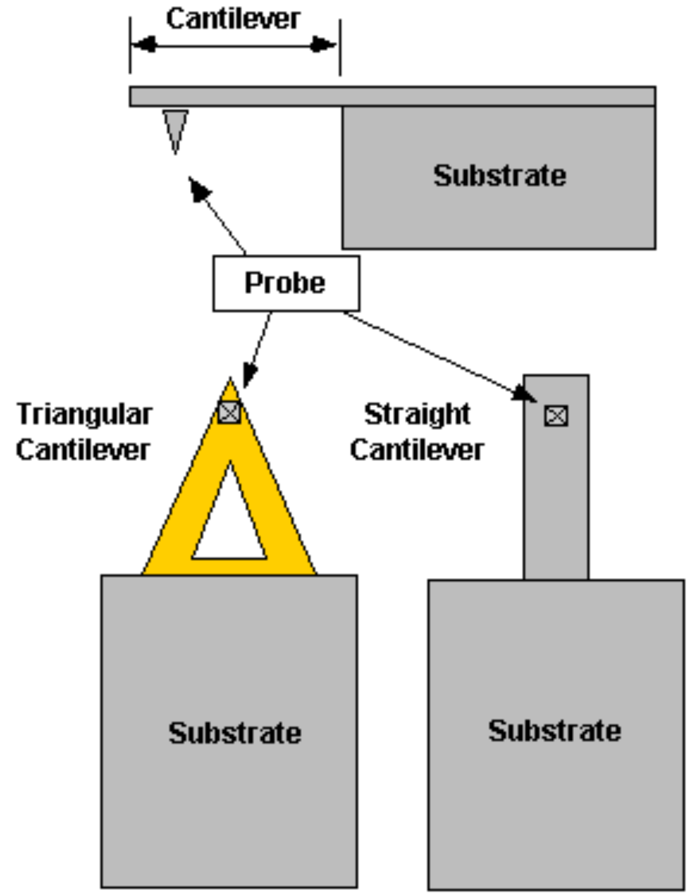




AFM image of a fresh Alfalfa root section

Fabrication of AFM probes

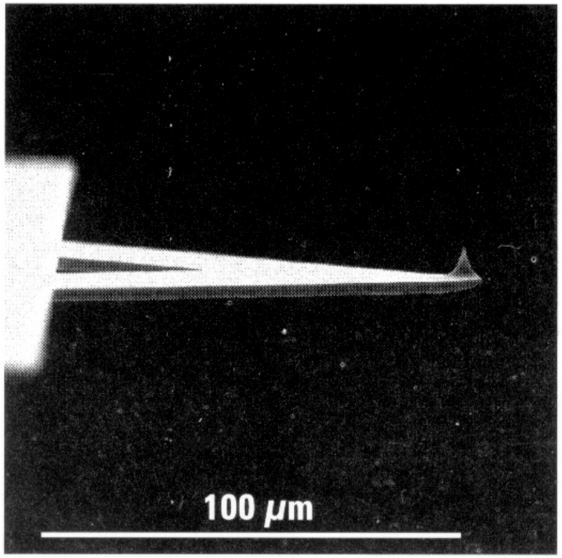




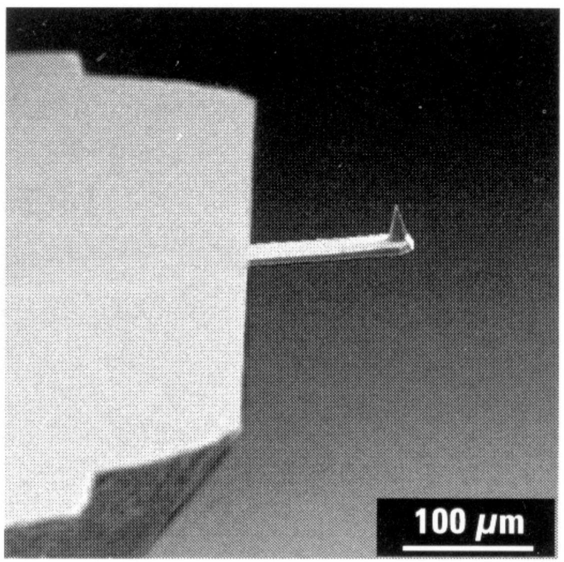
Typical Tip Dimension:
150µm x 30µm x 0.5µm
k ~ 0.1 N/m
Materials: Si_3N_4

Typical Tip Dimension:
150µm x 30µm x 3µm
f_r ~ 100 kHz
Materials: Si

V-shaped



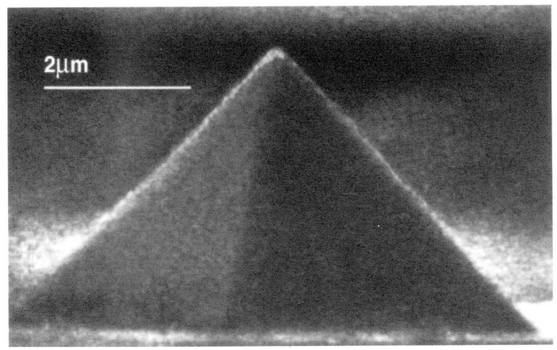
Rectangular-shaped



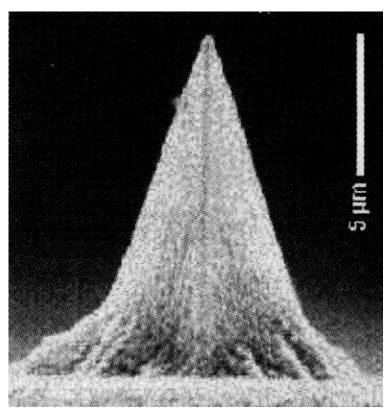
Materials: Si, SiO_2, Si_3N_4

Ideal Tips: hard, small radius of curvature, high aspect ratio

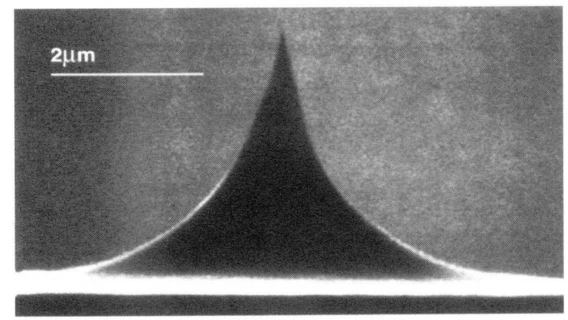
Pyramid Tip

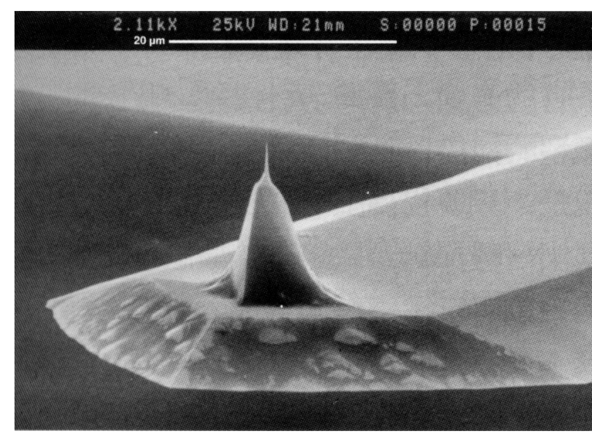


Diamond-coated Tip



Ultrasharp Tip





Criteria for AFM probe

1) Small spring constant (k) $F = k\,\Delta z$

To detect force of ~ nN

2) High resonant frequency (f_r) $f_r \propto (k/m)^{1/2}$

To enable scanning and other operations

3) Highly anisotropic stiffness

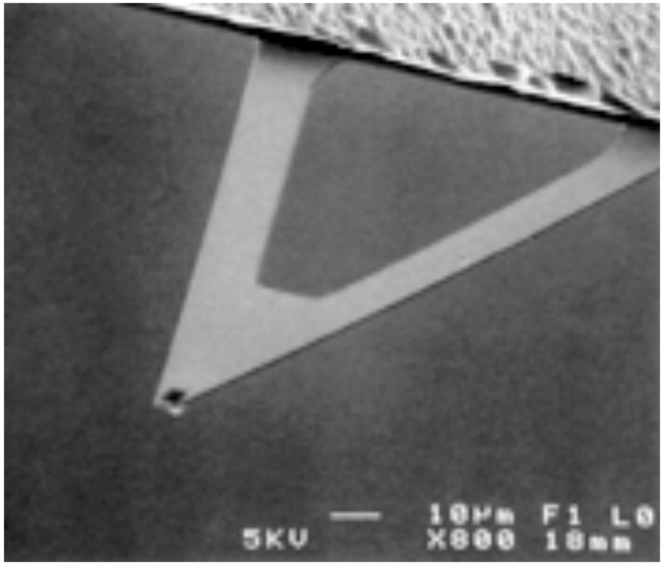
Easy to bent and difficult to twist

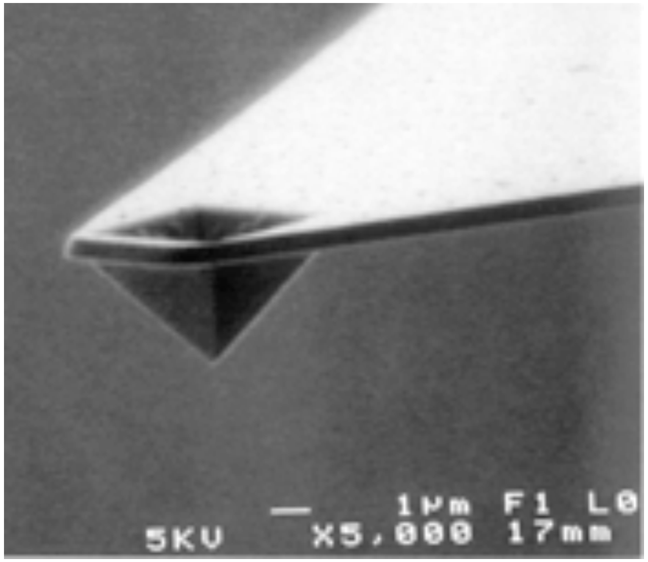
4) Sharp protrusion at the apex

To better define the tip-sample interaction

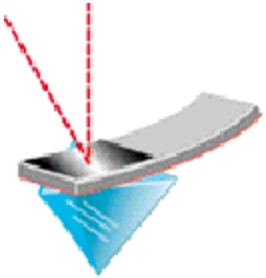
Tip of small shear force

(for Contact mode)



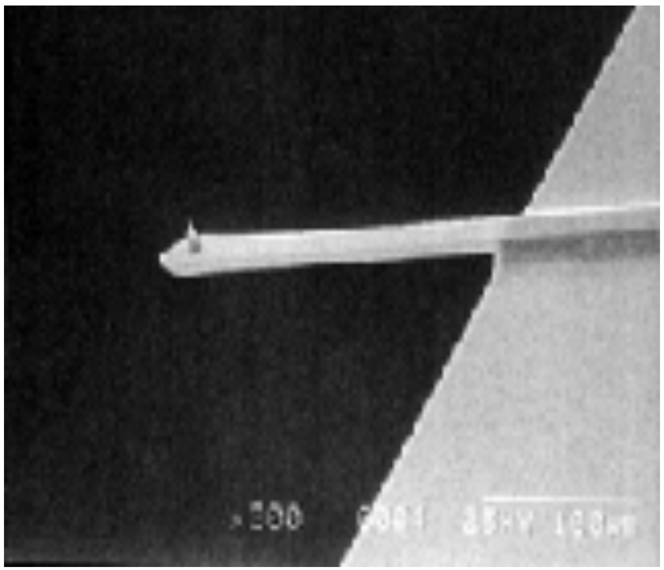


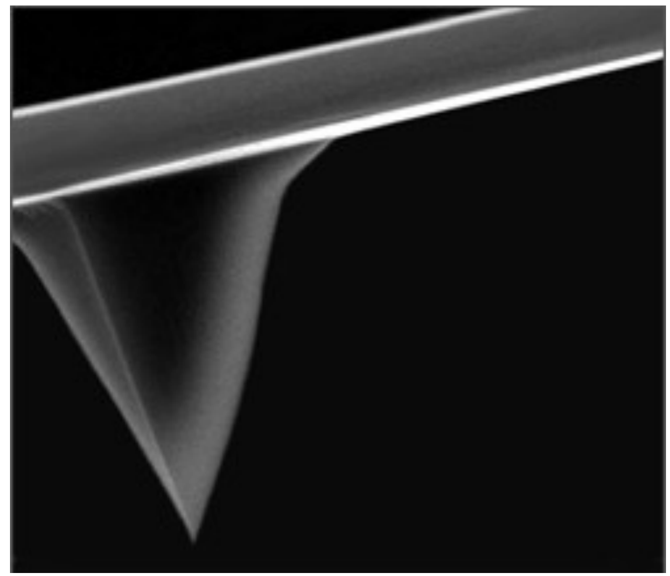
Typical Tip Dimension:
150µm x 30µm x 0.5µm
k ~ 0.1 N/m
Materials: Si_3N_4



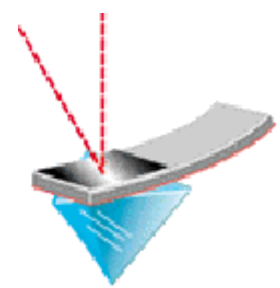
Tip of high resonant frequency

(for Tapping mode)





Typical Tip Dimension:
150µm x 30µm x 3µm
f_r ~ 100 kHz
Materials: Si



AFM versus STM

1. Generally, STM has “better” resolution than AFM.
2. The force-distance dependence in AFM is much more complex when characteristics such as tip shape and contact force are considered.
3. STM is generally applicable only to conducting samples while AFM is applied to both conductors and insulators.
4. AFM offers the advantage that the writing voltage and tip-to-substrate spacing can be controlled independently, whereas with STM the two parameters are integrally linked.

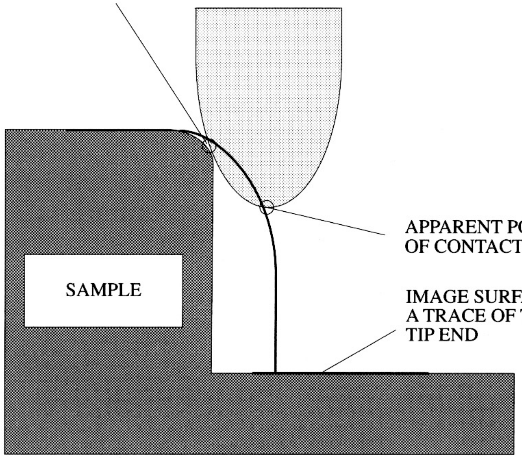
AFM versus EM

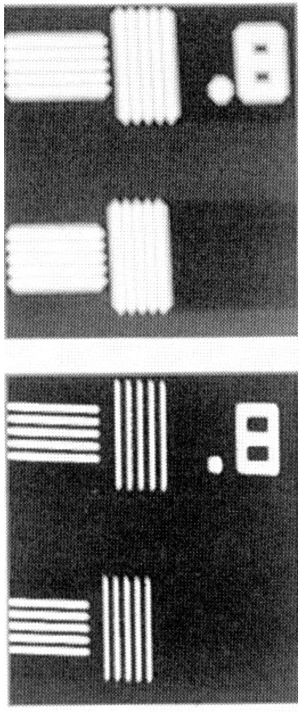
1. AFM only reveal the surface and EM can probe the interior structure of the sample with higher resolution.
2. AFM provides direct topographic measurements and EM provides only 2D projection of the sample structure.
3. No charging effect occurs in AFM. So, for insulating samples, no metallic coating is necessary.

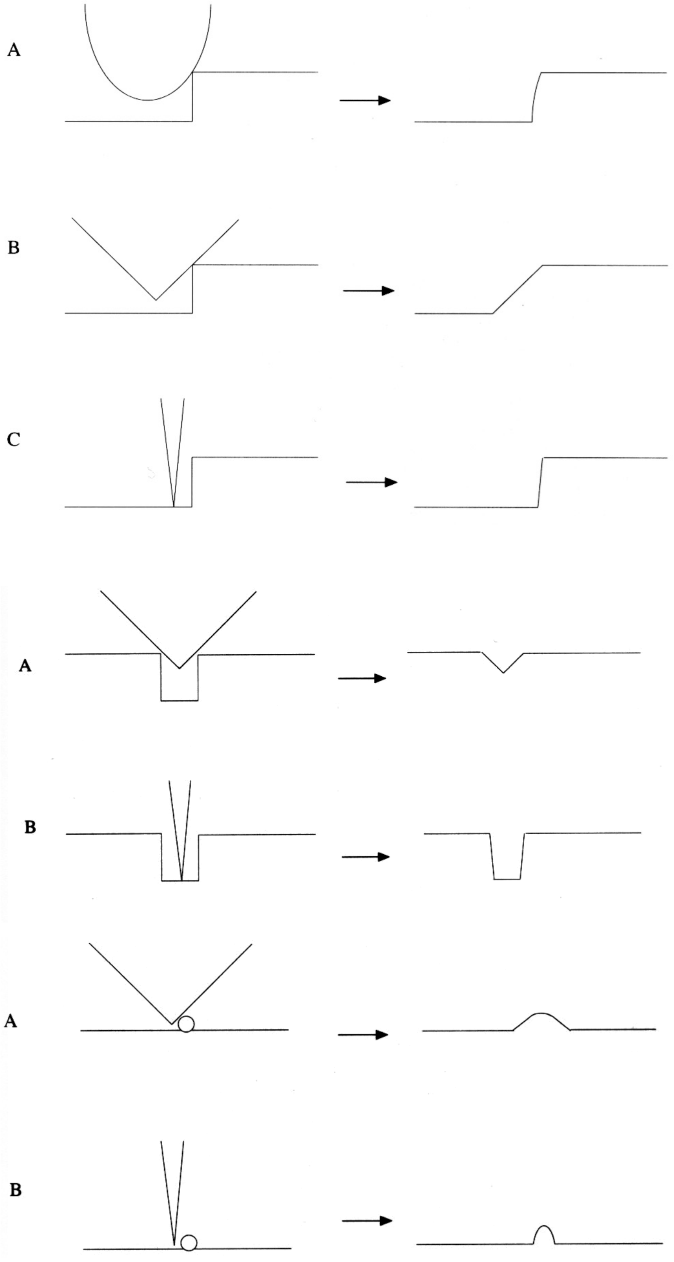
AFM versus Optical Microscope

1. AFM has much better resolution than Optical Microscope (OM).
2. AFM provides unambiguous measurement of step heights, independent of reflectivity differences between materials.
3. OM can be applied to much faster dynamic studies with the pump-probe method.

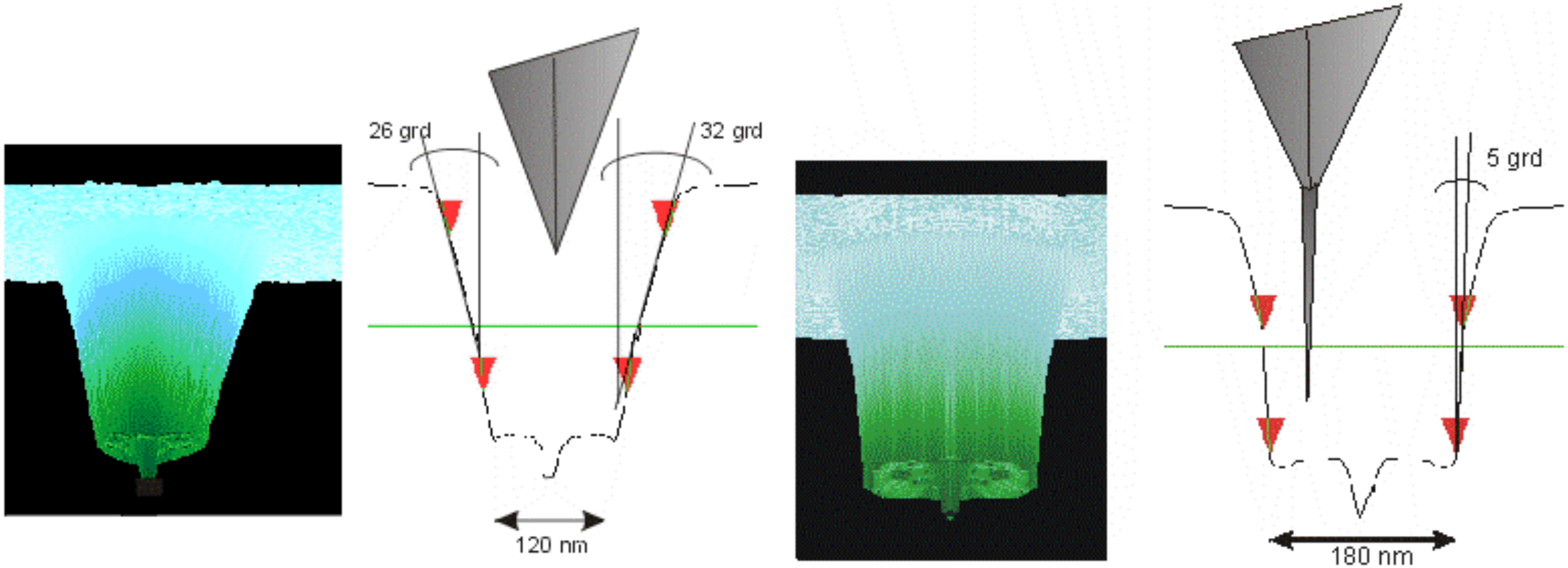
Effects of the Tip Shape



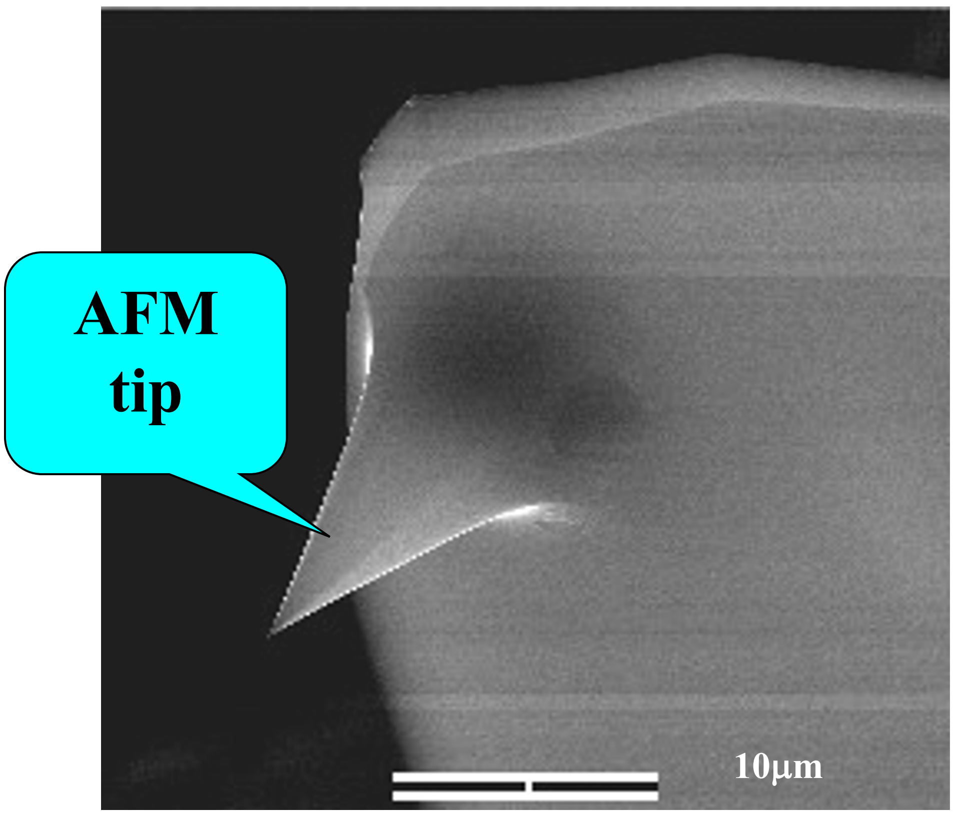




Ultra-sharp tip



AFM Tip + Carbon Nanotube



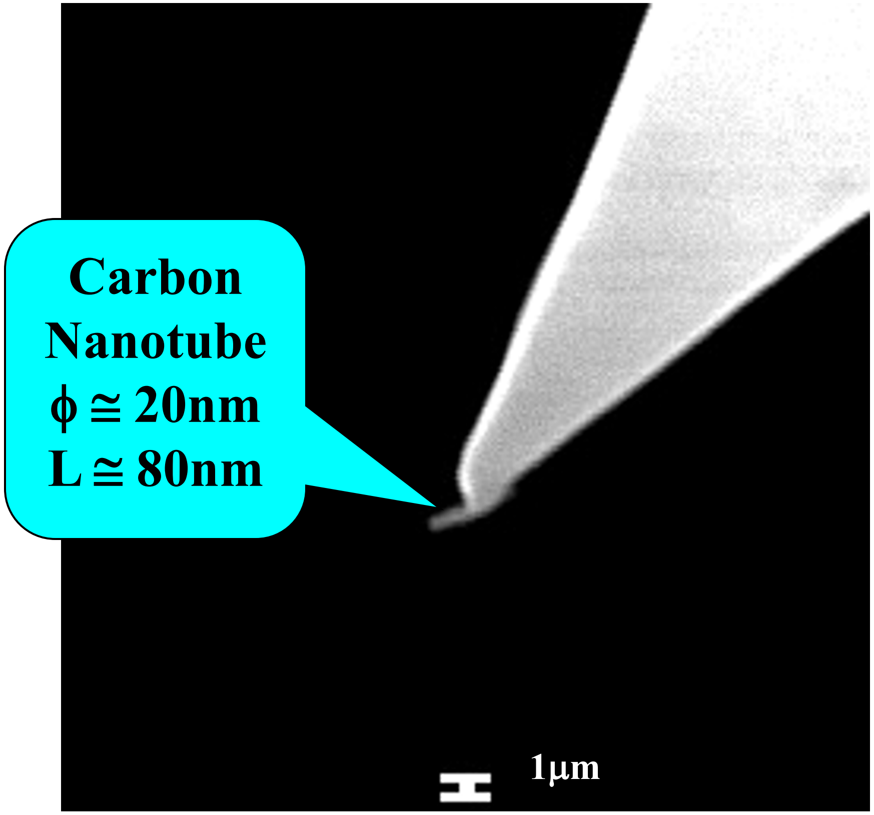
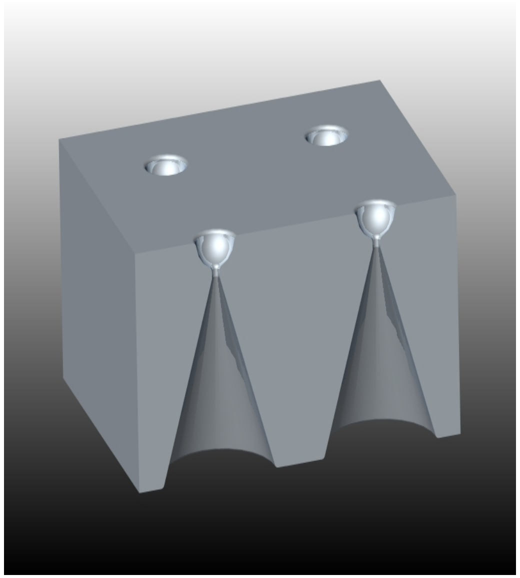
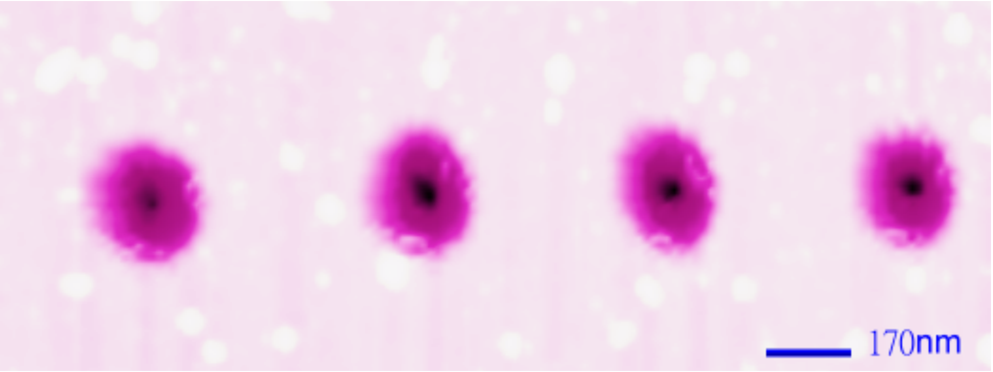


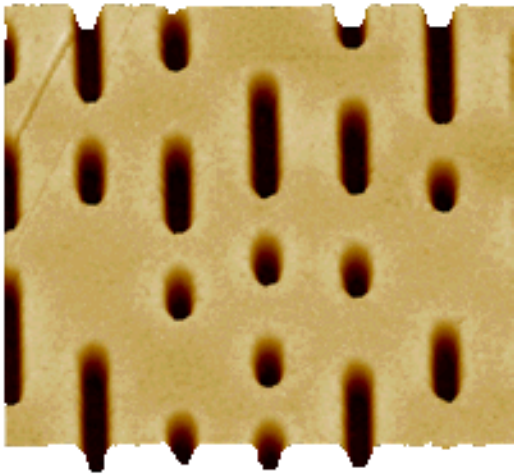
Image of high aspect ratio



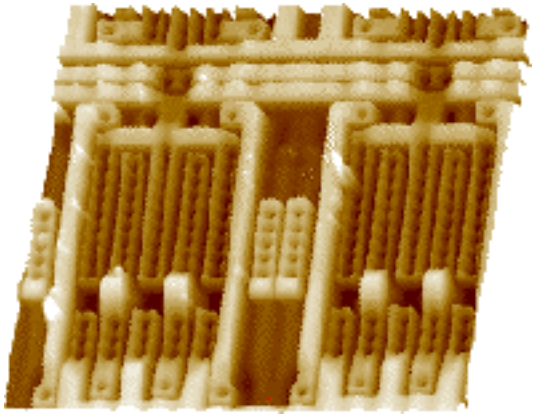


AFM images

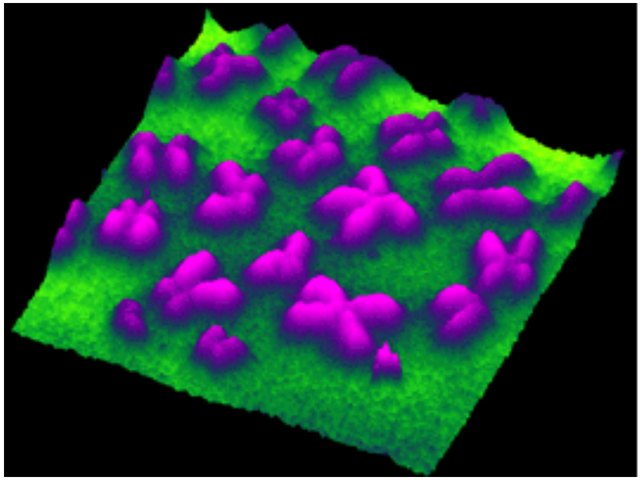
CD pits



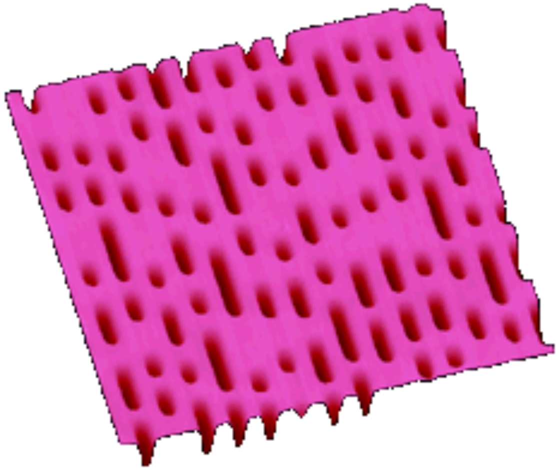
Integrated circuit



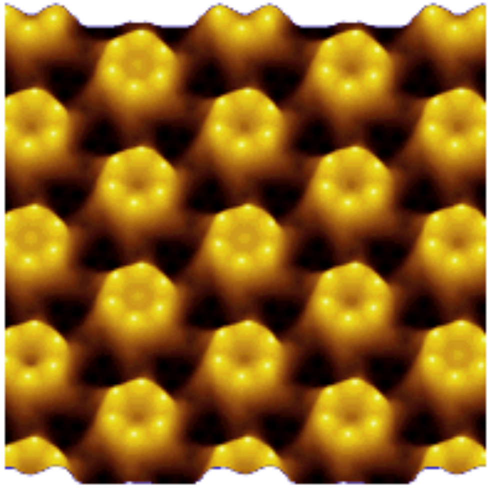
Chromosomes



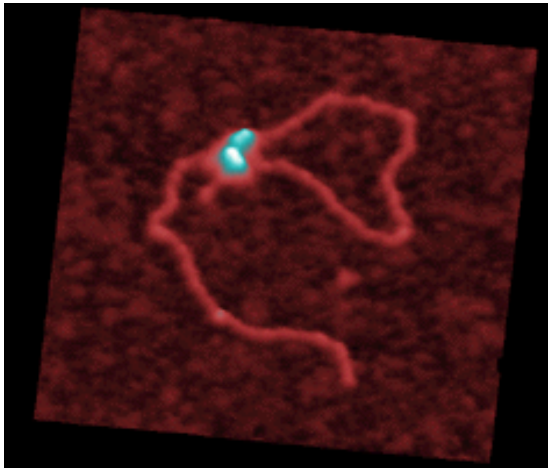
DVD pits



Bacteria



DNA

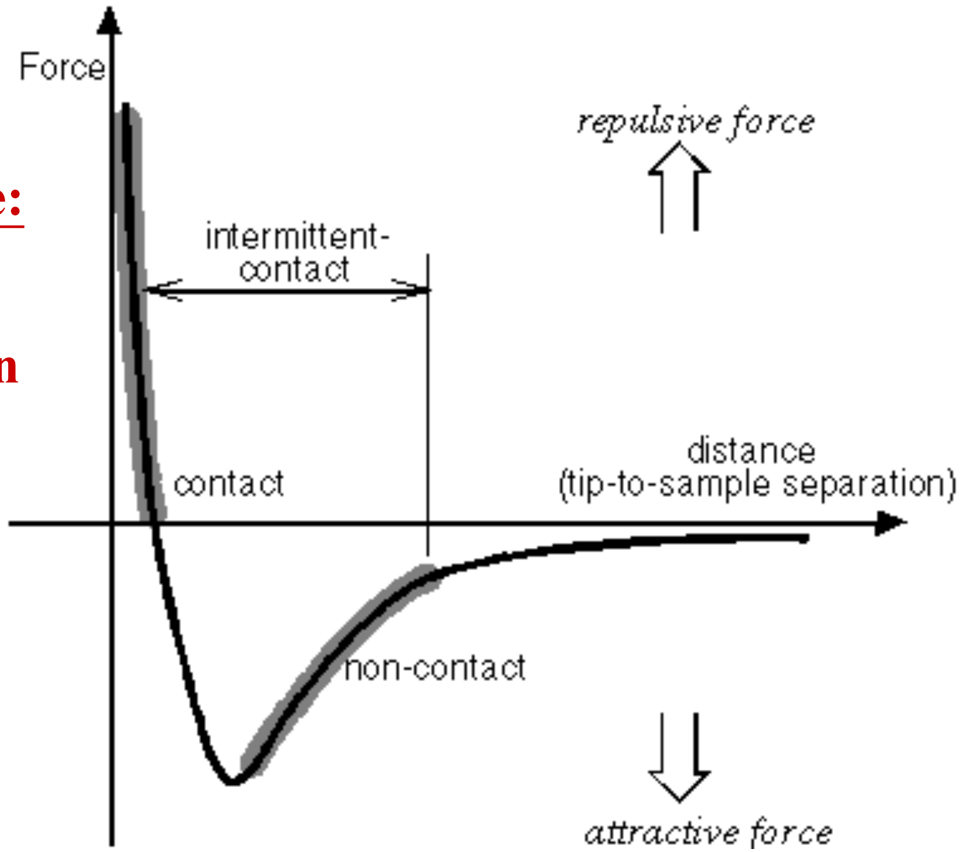


Interaction between the probe and sample

Short-range:

1) Bonding

2) Repulsion



Long-range:

1) Van der Waal

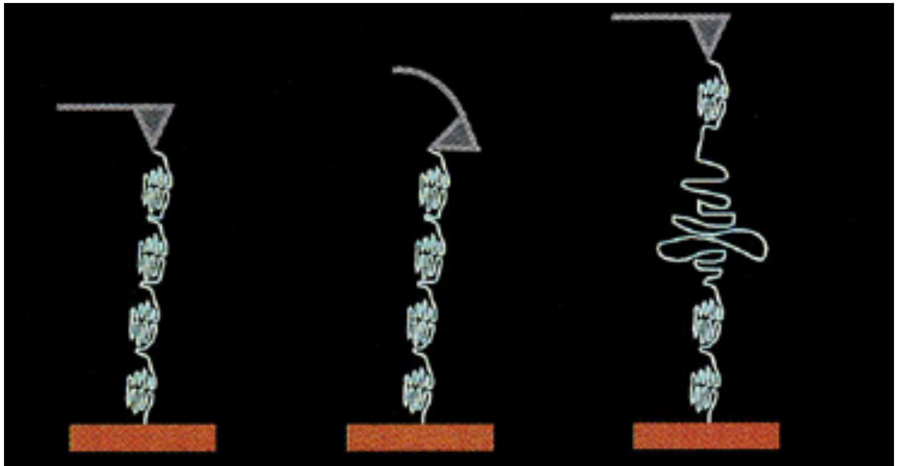
2) Capillary

3) Magnetic

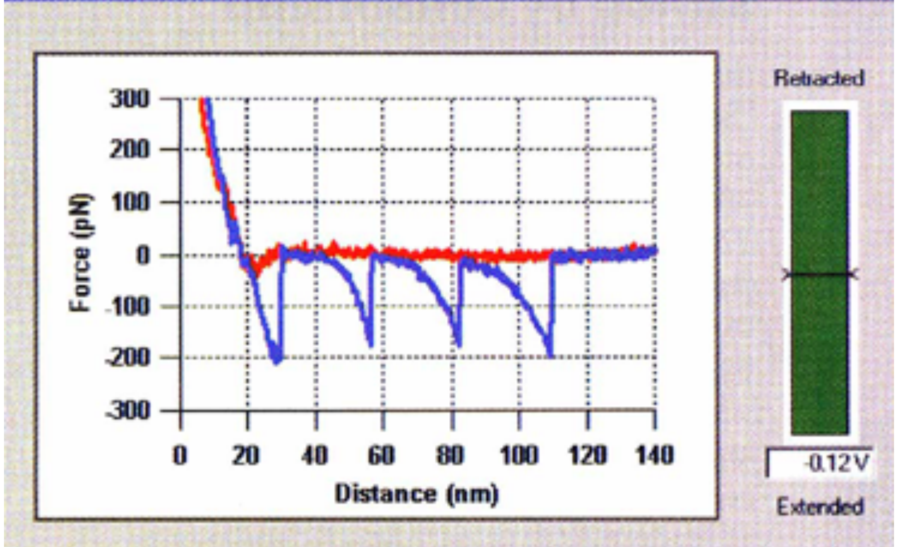
4) Electrostatic

Lennard-Jones potential $\phi(r) = - A/r^6 + B/r^{12}$

Force spectroscopy by AFM

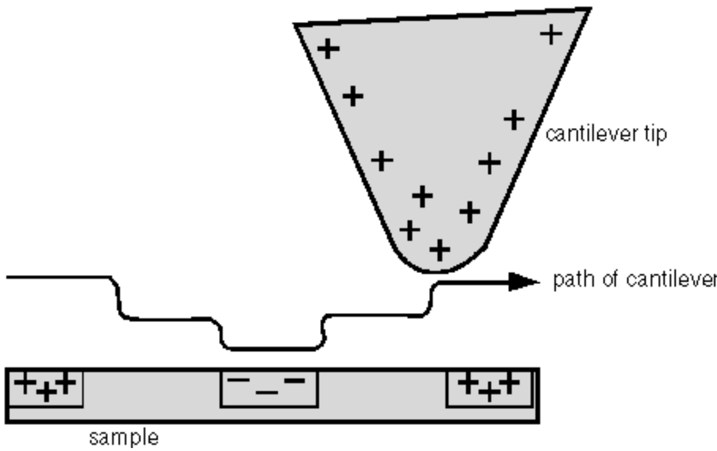


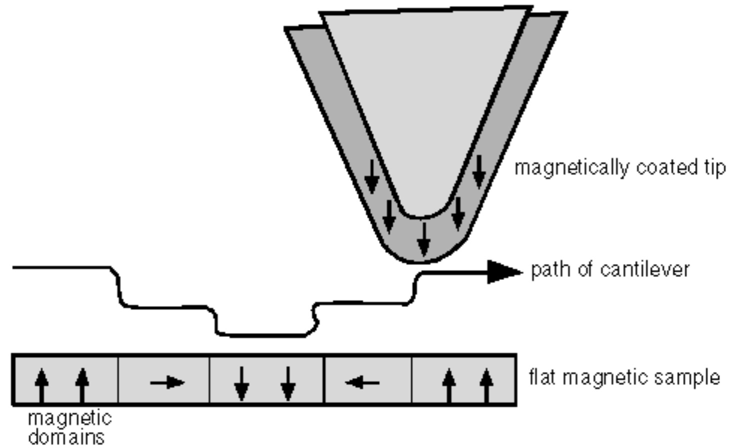
Force measurement of an unfolding complex molecule.

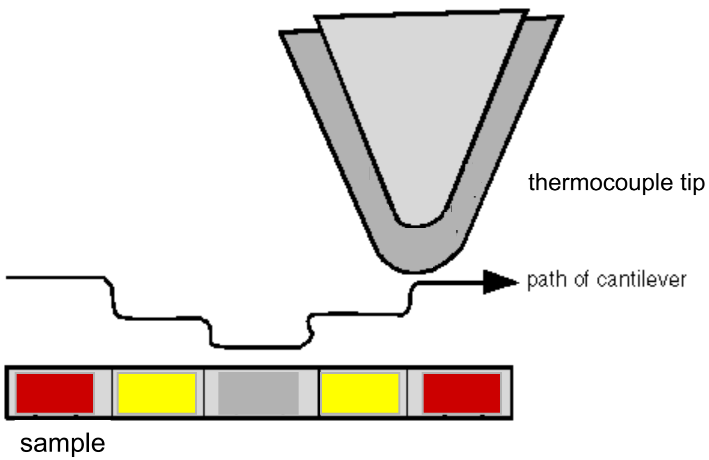


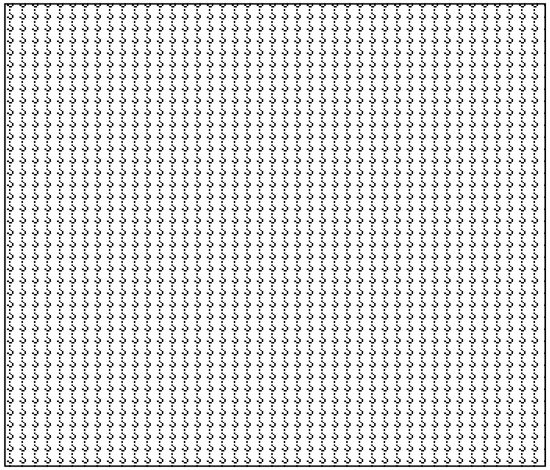
Advanced graphical user interface shows titin muscle molecule force curve.

Probes of various functions

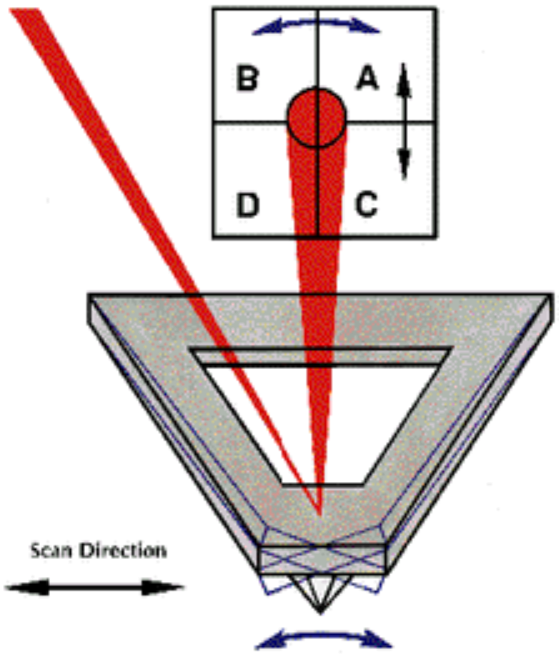




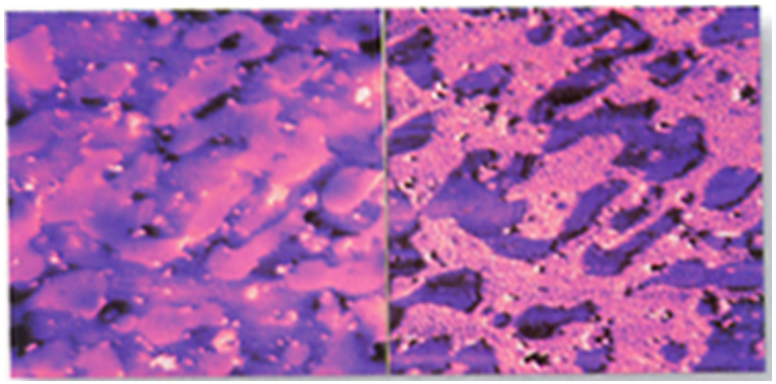




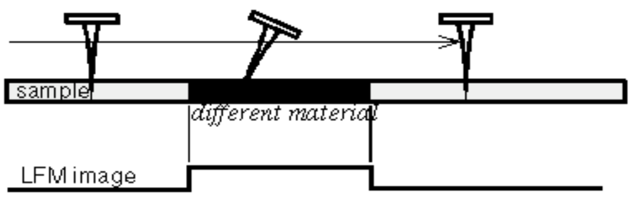
Lateral Force Microscopy



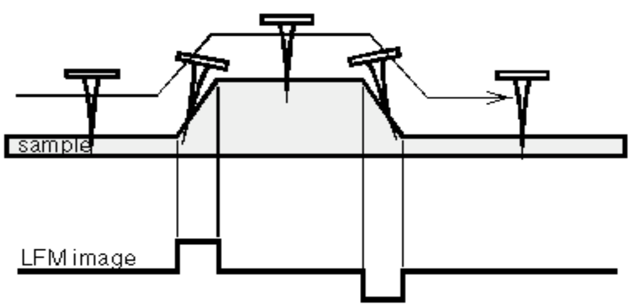
(A+C) – (B+D)



Nature rubber/EDPM blend



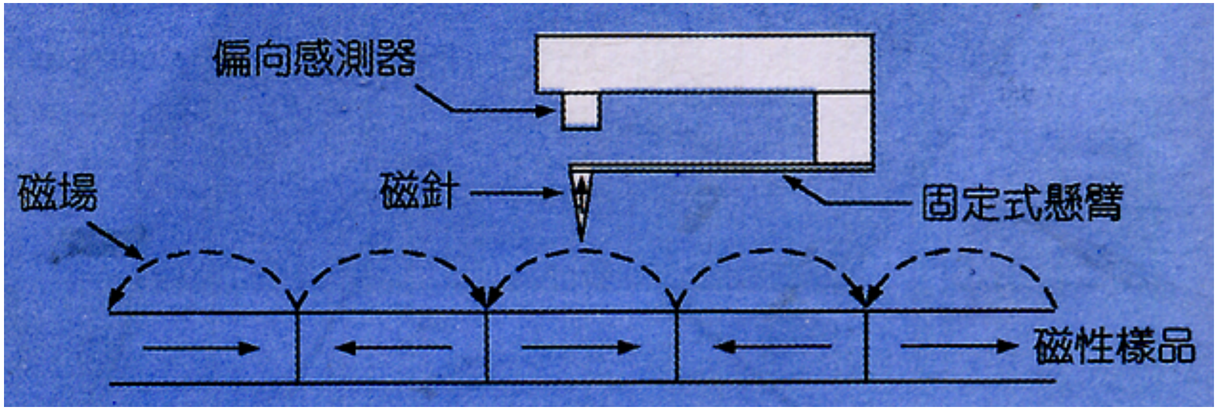
- LFM is sensitive to friction and chemical forces.
- Image contrast depends on the scanning direction.
- Surface roughness will contribute to the contrast.



Chemical Force Microscopy

CFM scan of well-defined regions that terminate in either methyl or carboxylic acid groups. When a carboxylic acid-terminated tip is used for imaging (left), the carboxylic acidterminated regions exhibit greater frictional force (lighter color) than the methyl-terminated regions. When a methylterminated tip is used (right), the friction contrast is reversed. No differences are revealed by the topographic AFM scan (not shown) since the functional groups are structurally quite similar. Image courtesy of Dr. C. Lieber, Harvard University.

Magnetic Force Microscopy (MFM)



$$\mathbf{F} = \nabla(\mathbf{m} \bullet \mathbf{H})$$

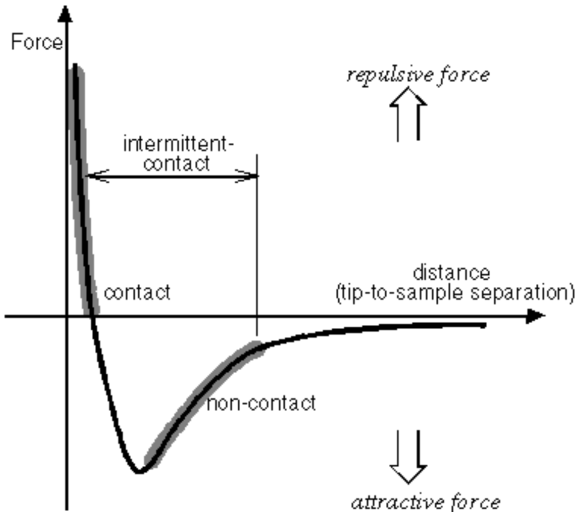
Tips: silicon probes are magnetically sensitized by sputter coating with a ferromagnetic material.

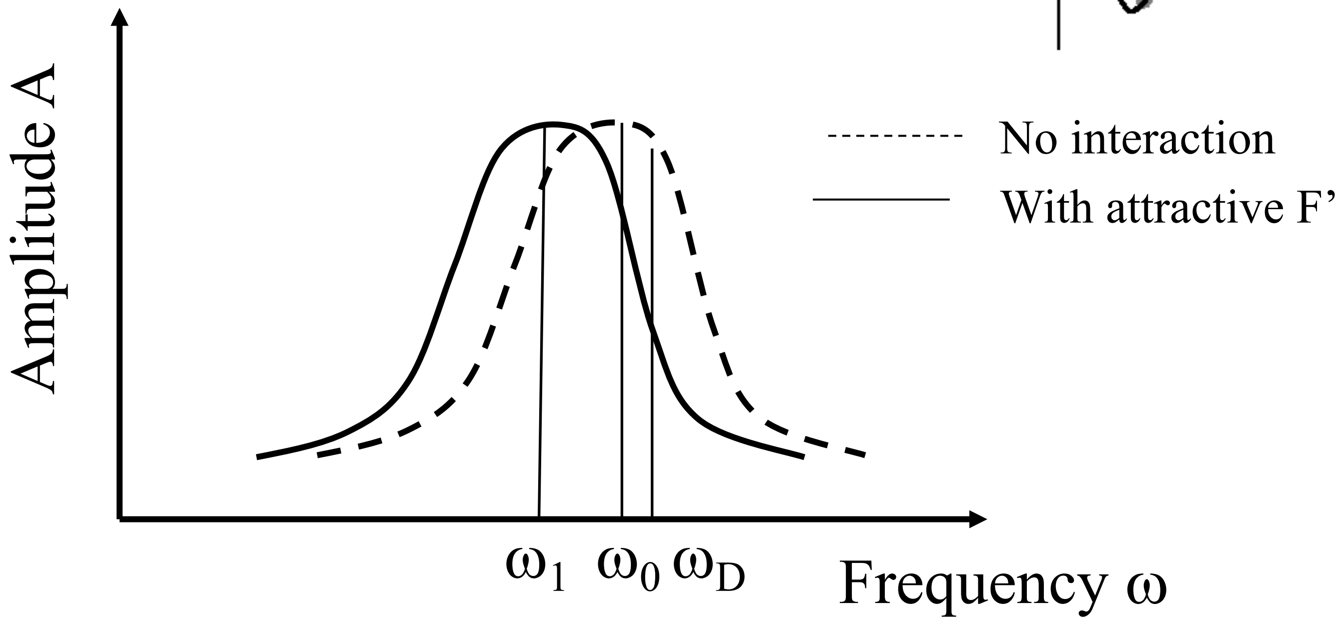
Resolution: 10 ~ 25 nm.

Applications: hard disks, magnetic thin film materials, micromagnetism.

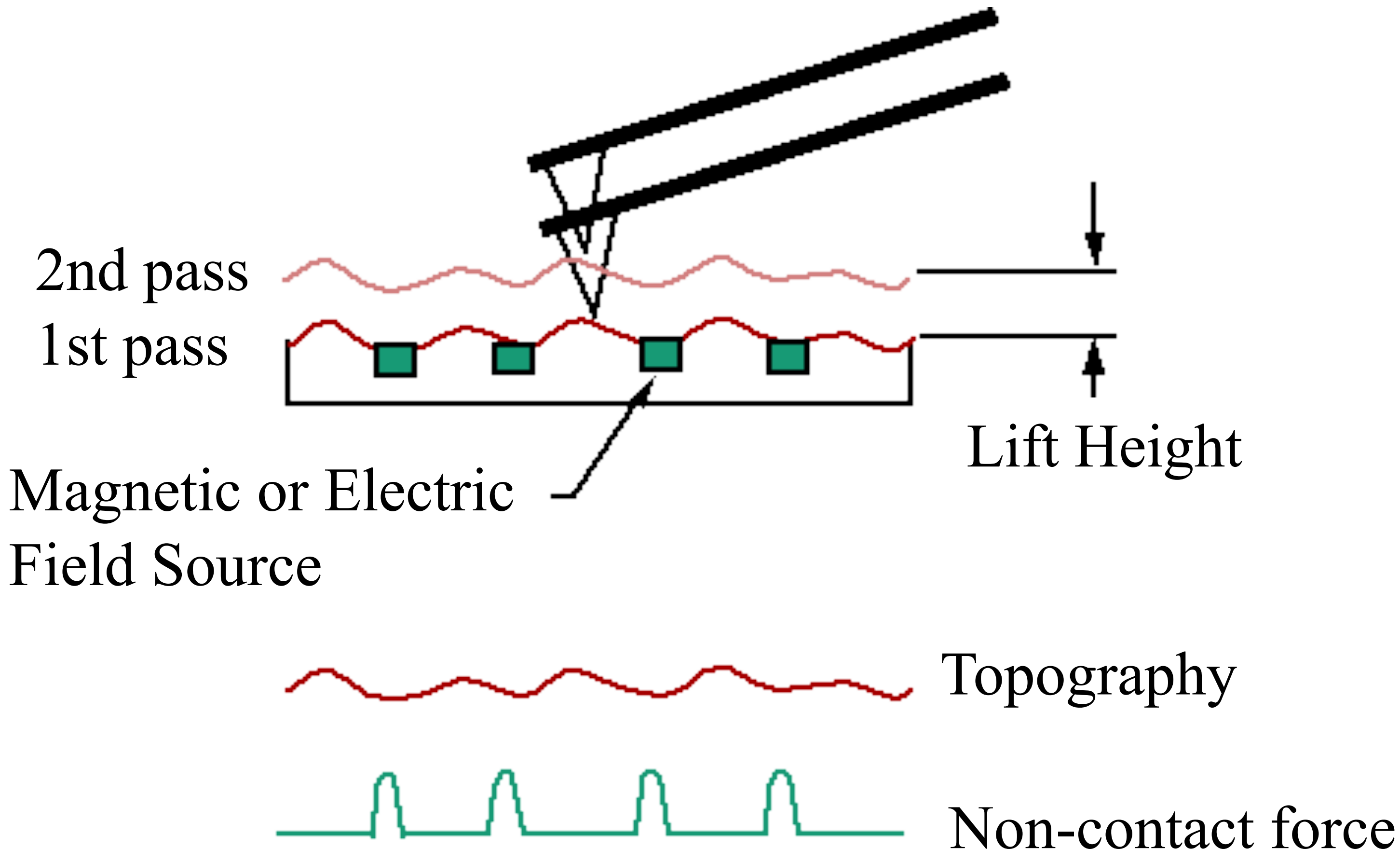
AC imaging mode

$$\omega_1 = \omega_0 \, (1 + F'/2k)$$

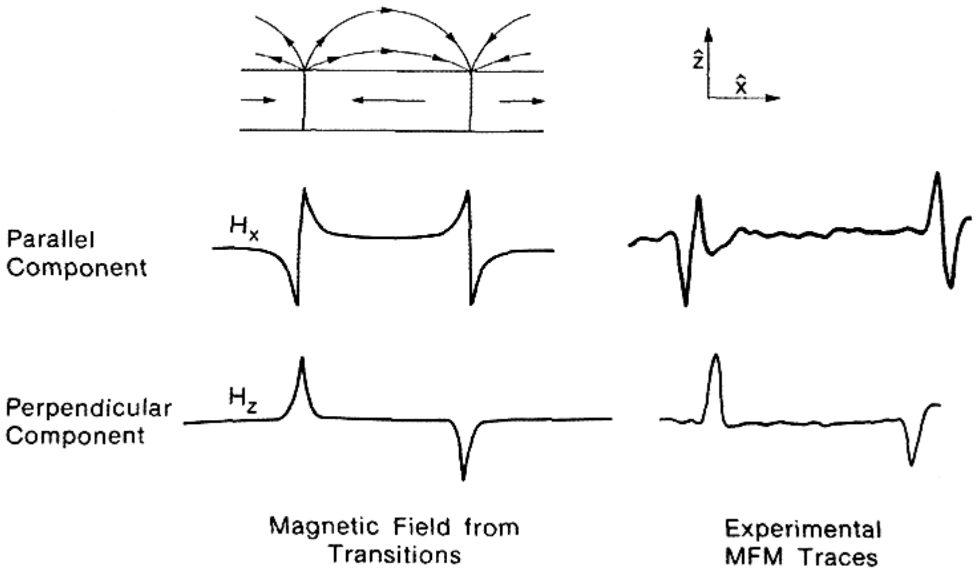


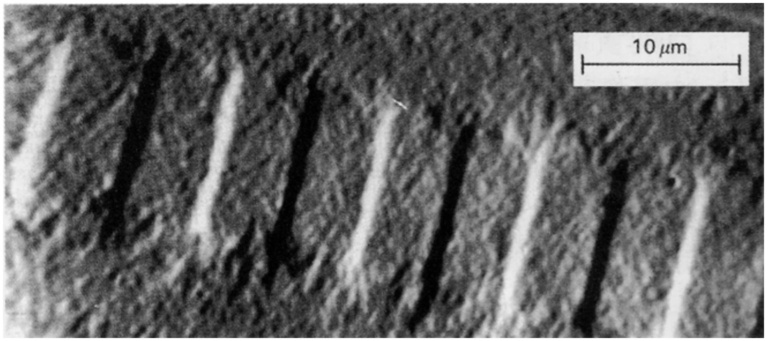


Lift mode of AFM

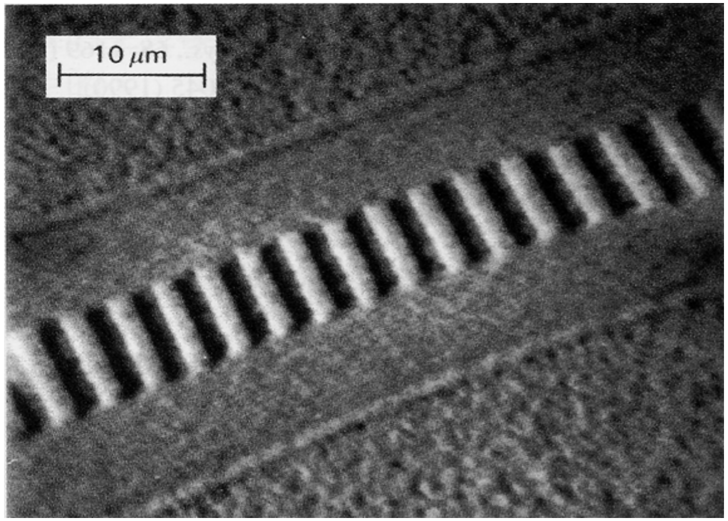


MFM Images





$m^T = m_Z$



$m^T = m_x + m_z$

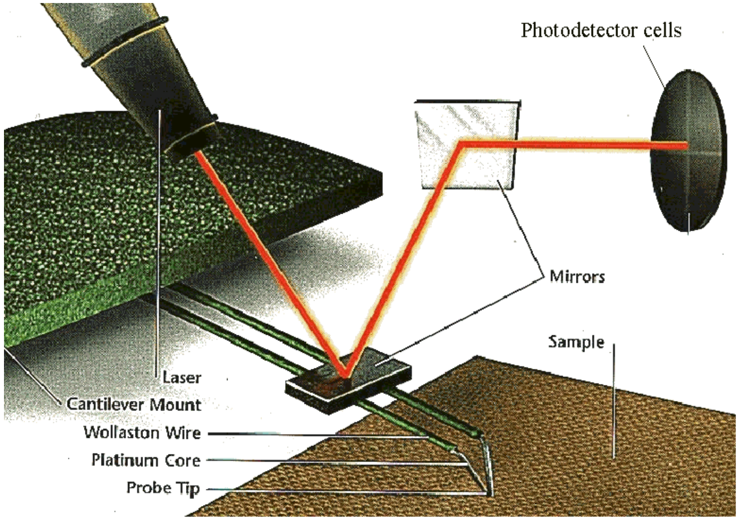
Tip as a point dipole

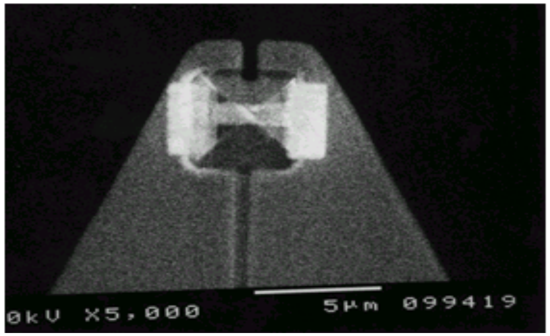
$$F_z = \partial(m_x H_x + m_y H_y + m_z H_z)/\partial z$$

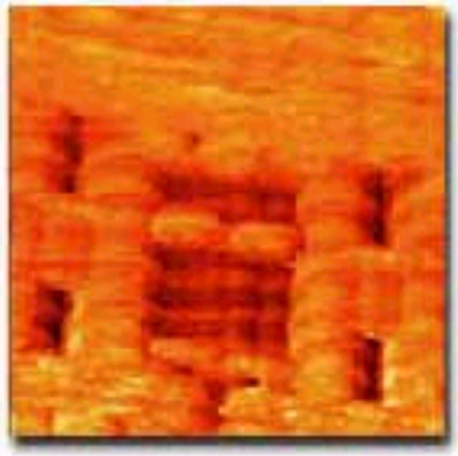
Tip as a long rod

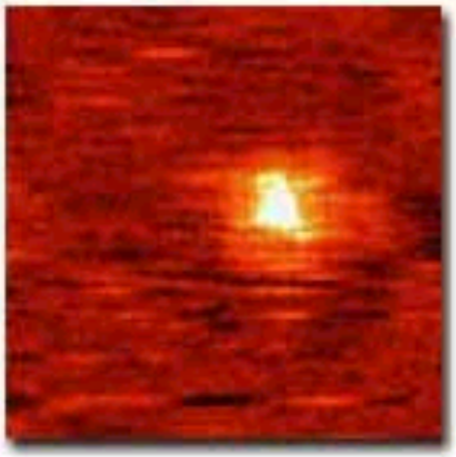
$$F_z = m_z H_z$$

Scanning Thermal Microscopy (SThM)

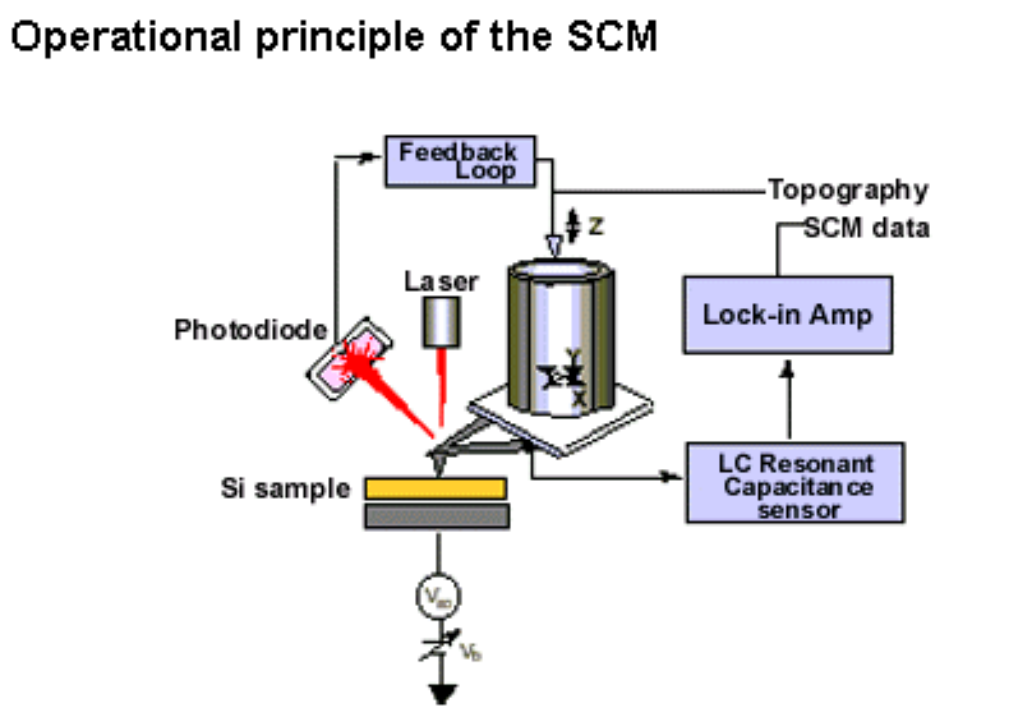


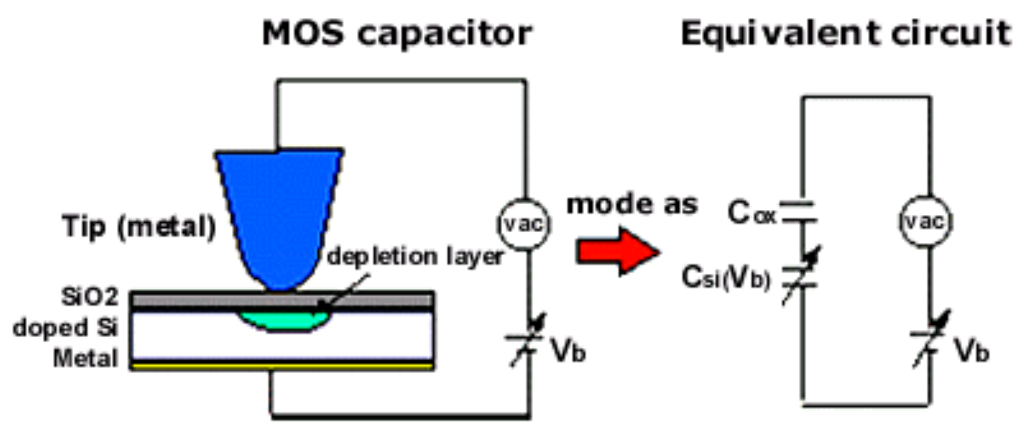




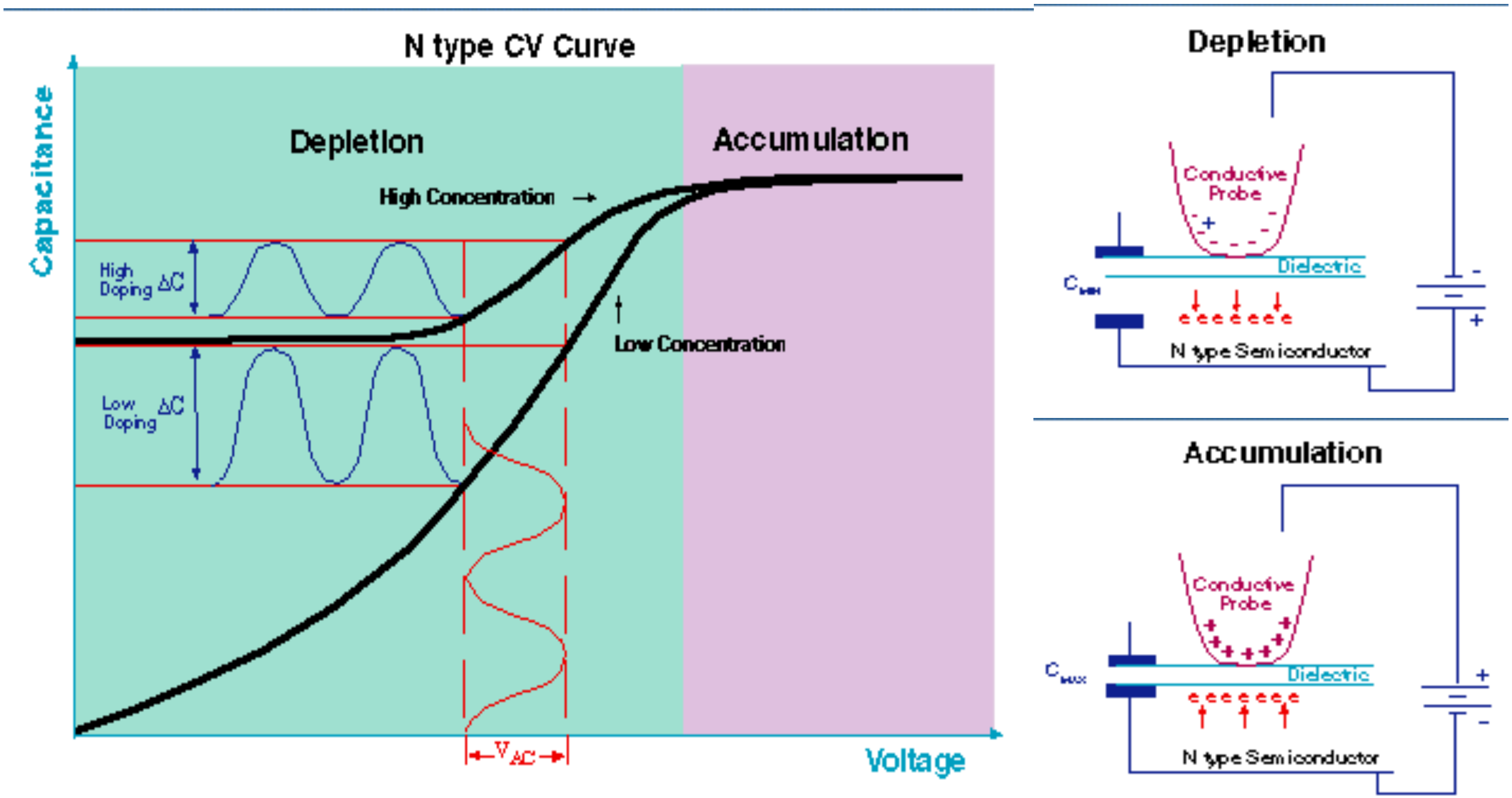


Scanning Capacitance Microscopy (SCM)



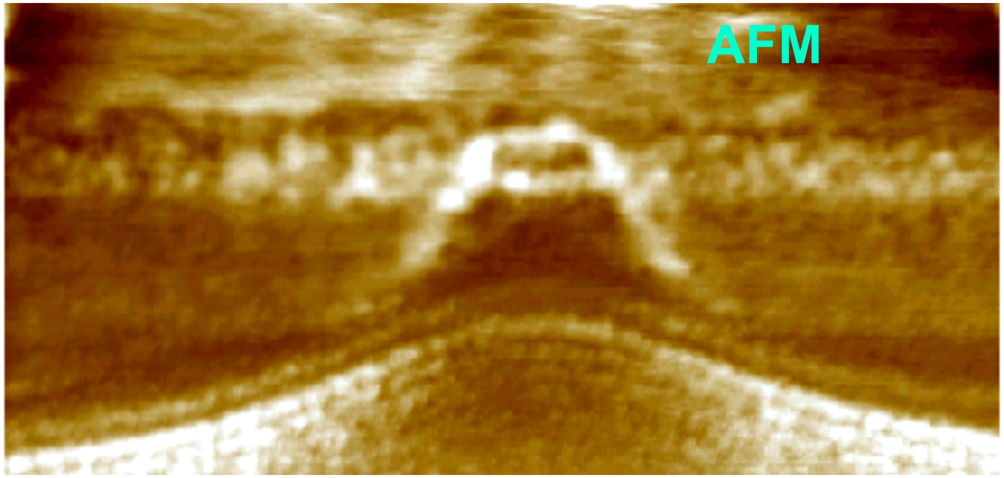


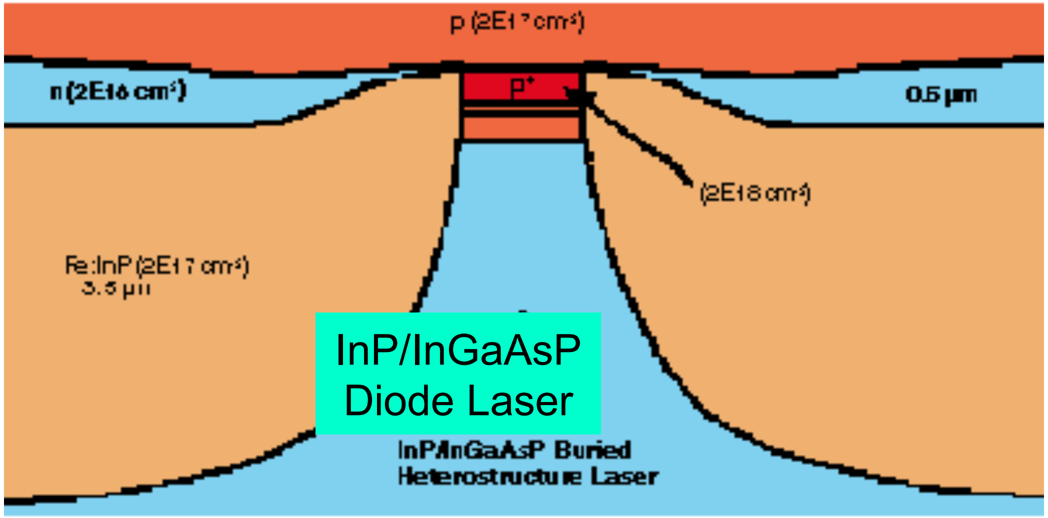
SCM CV Curve



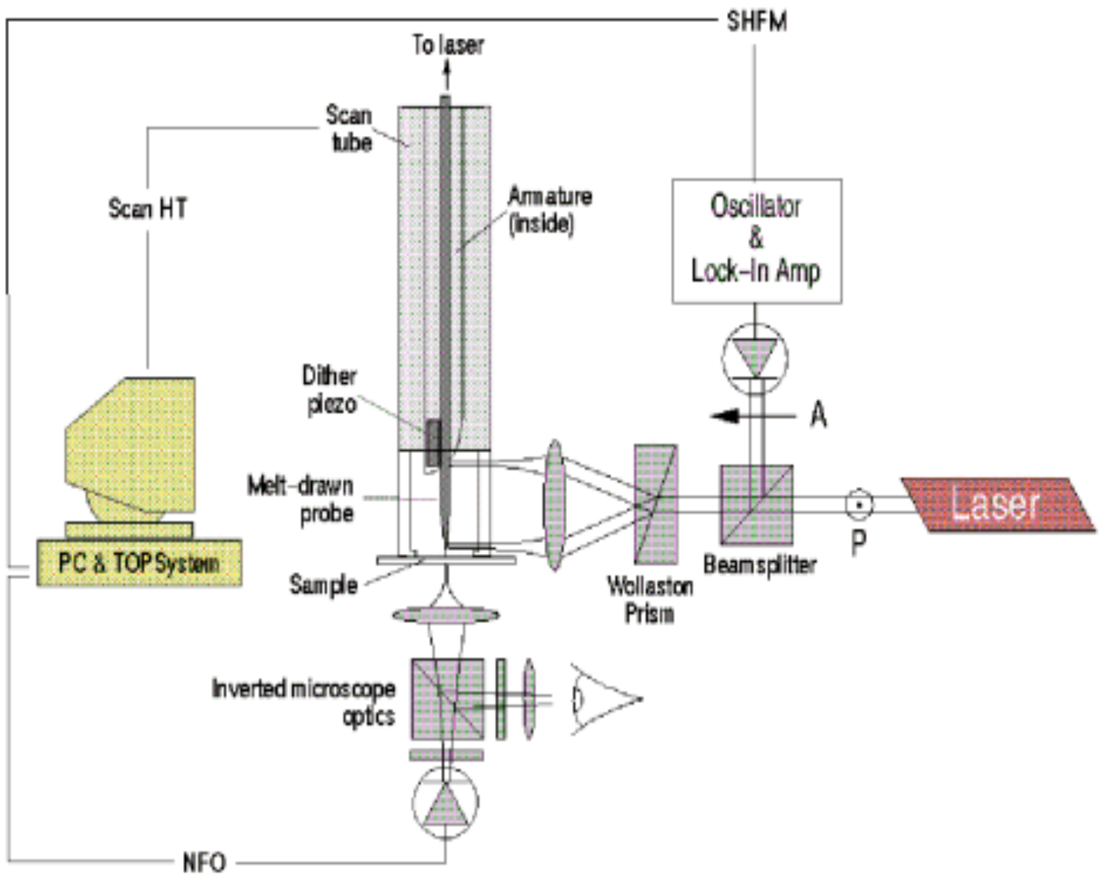
N type CV Curve
Capacitance
Depletion
Accumulation
High Concentration
High Doping ΔC
Low Doping ΔC
Low Concentration
V_AC
Voltage
Depletion
Conductive Probe
Dielectric
N type Semiconductor
Accumulation
Conductive Probe
Dielectric
N type Semiconductor

Scanning Capacitance Microscopy



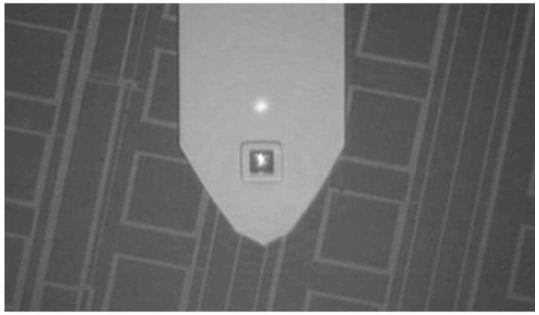


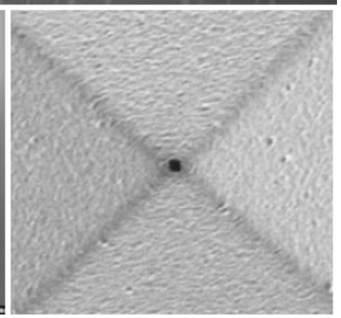
Near-field Scanning Optical Microscopy (NSOM)

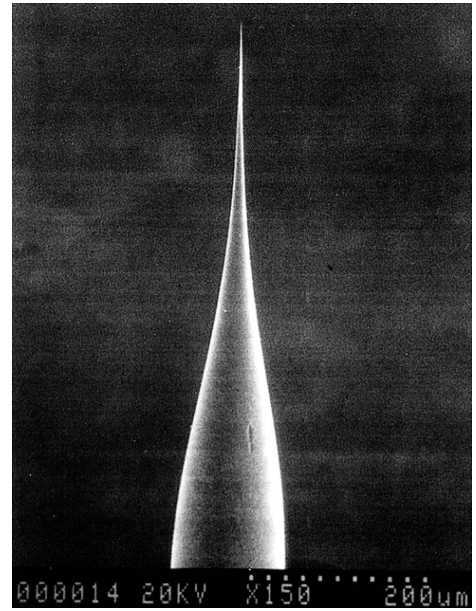


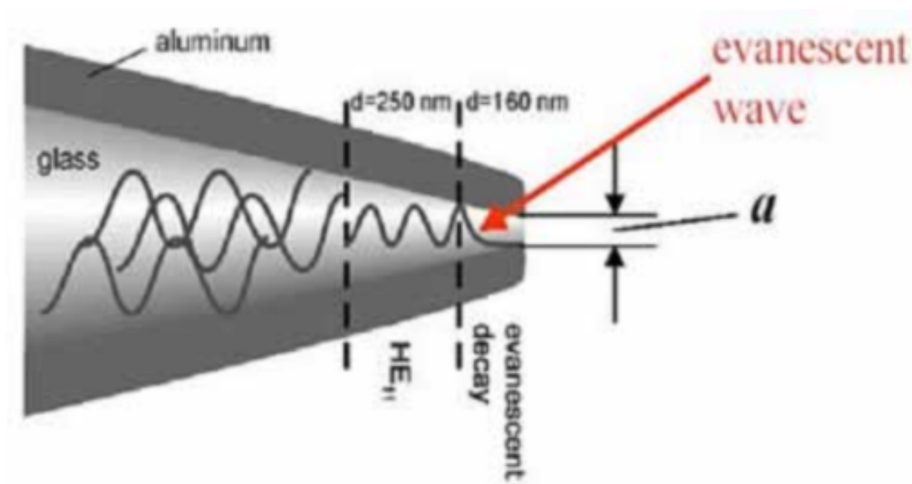
Shear force detection is used to regulate the tip/sample separation

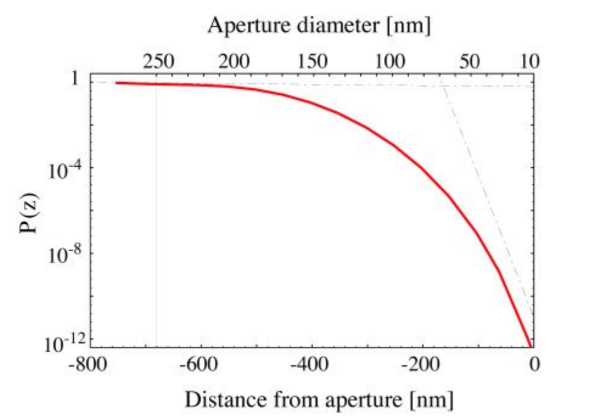
Probes for NSOM





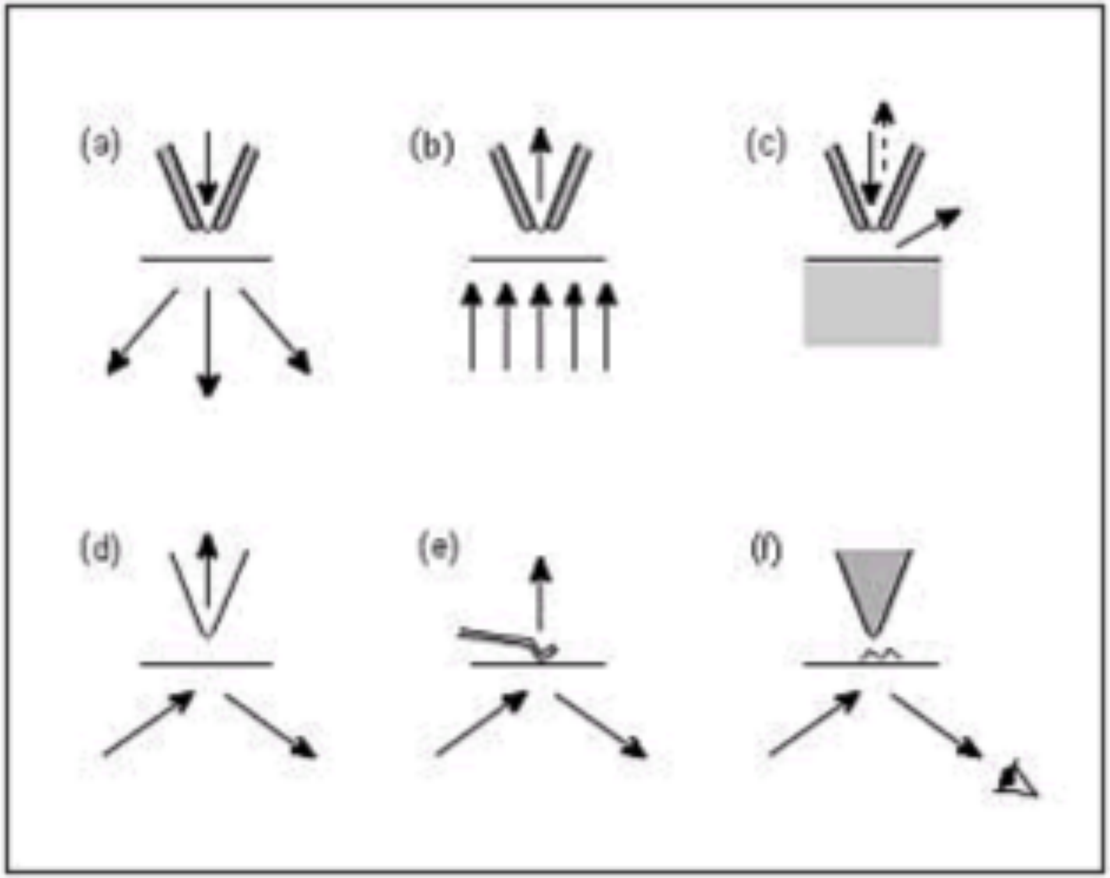




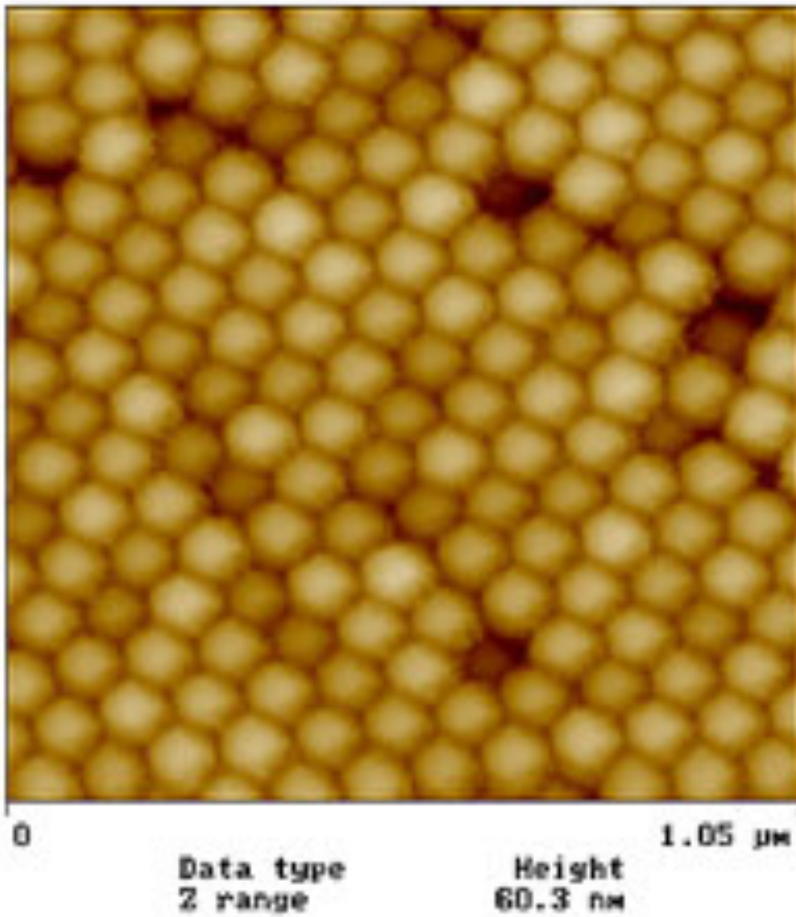


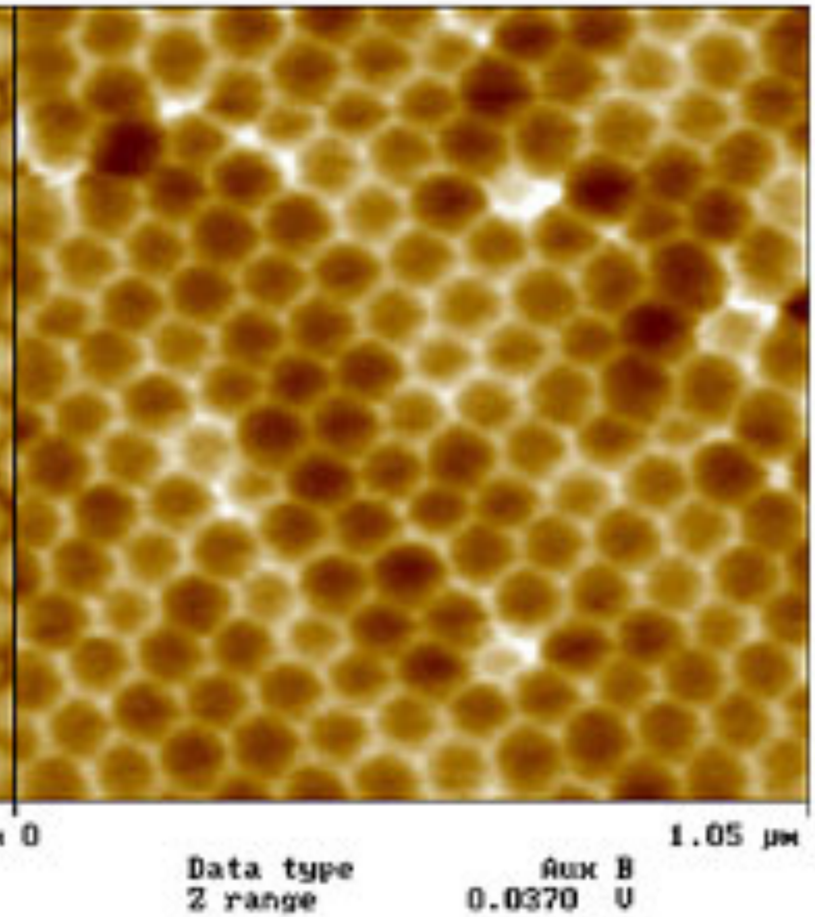
L. Novotny and D. W. Pohl, in *Photons and Local Probes*, NATO ASI Series E, p.21-33, Kluwer Academic, 1995.

Imaging modes for NSOM



Topography **NSOM Image**

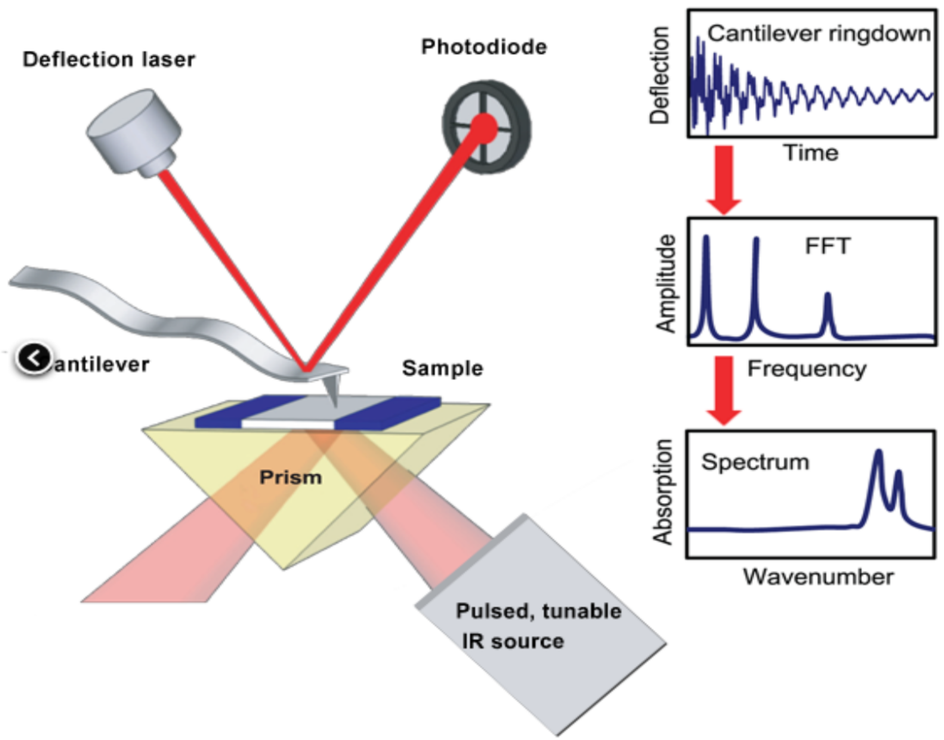




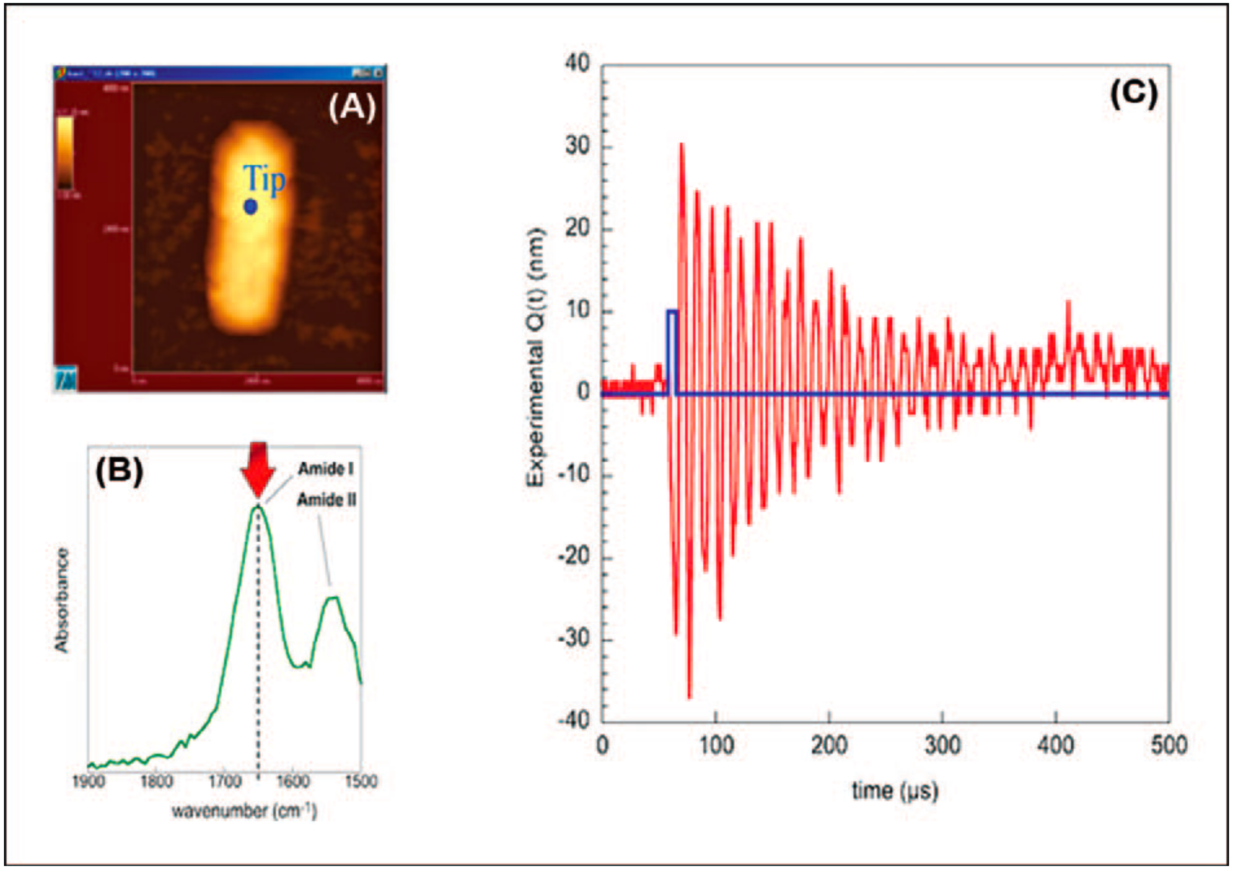
Polystyrenes of 100 nm on glass

Recent development: AFM-IR system

AFM-IR can perform IR spectroscopic chemical identification with sub-100 nm spatial resolution



Scheme of the AFM–IR setup. The AFM cantilever ring-down amplitude plotted as a function of laser excitation wavelength produces the IR spectrum.



(a) AFM topography picture of the bacterium; the position of the tip is indicated in blue. (b) FT–IR spectrum; the bacterium absorption spectrum is drawn in green, and the wavenumber of the CLIO laser is indicated by the red arrow. (c) Oscillations recorded by the four-quadrant detector (in red) as function of time superposed on the CLIO pulse laser (blue).

Alexandre Dazzi et al., APPLIED SPECTROSCOPY OA 66, 1365 (2012)

1. **All SPMs are based on the ability to position various types of probes in very close proximity with extremely high precision to the sample under investigation.**
2. **These probes can detect electrical current, atomic and molecular forces, electrostatic forces, or other types of interactions with the sample.**
3. **By scanning the probe laterally over the sample surface and performing measurements at different locations, detailed maps of surface topography, electronic properties, magnetic or electrostatic forces, optical characteristics, thermal properties, or other properties can be obtained.**
4. **The spatial resolution is limited by the sharpness of the probe tip, the accuracy with which the probe can be positioned, the condition of the surface under study, and the nature of the force being detected.**