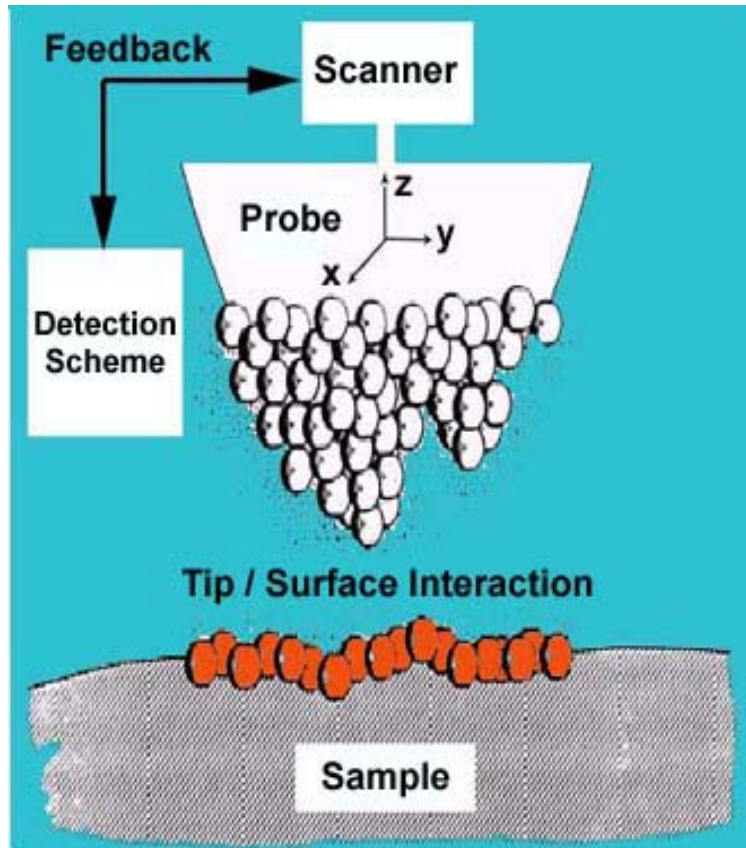


Scanning Probe Microscopy (SPM)



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)

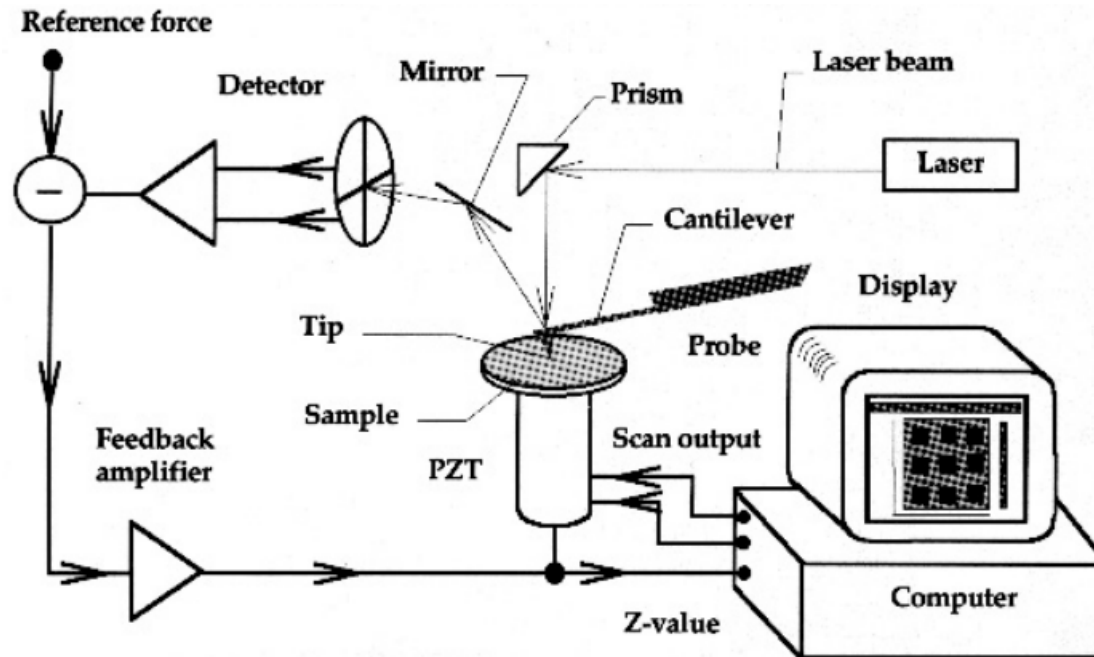
Scanning Capacitance Microscopy (SCM)

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

Force Modulation Microscopy (FMM)

--- P. Maivald et al (1991)

Atomic Force Microscopy (AFM)



$$F = k\Delta z$$

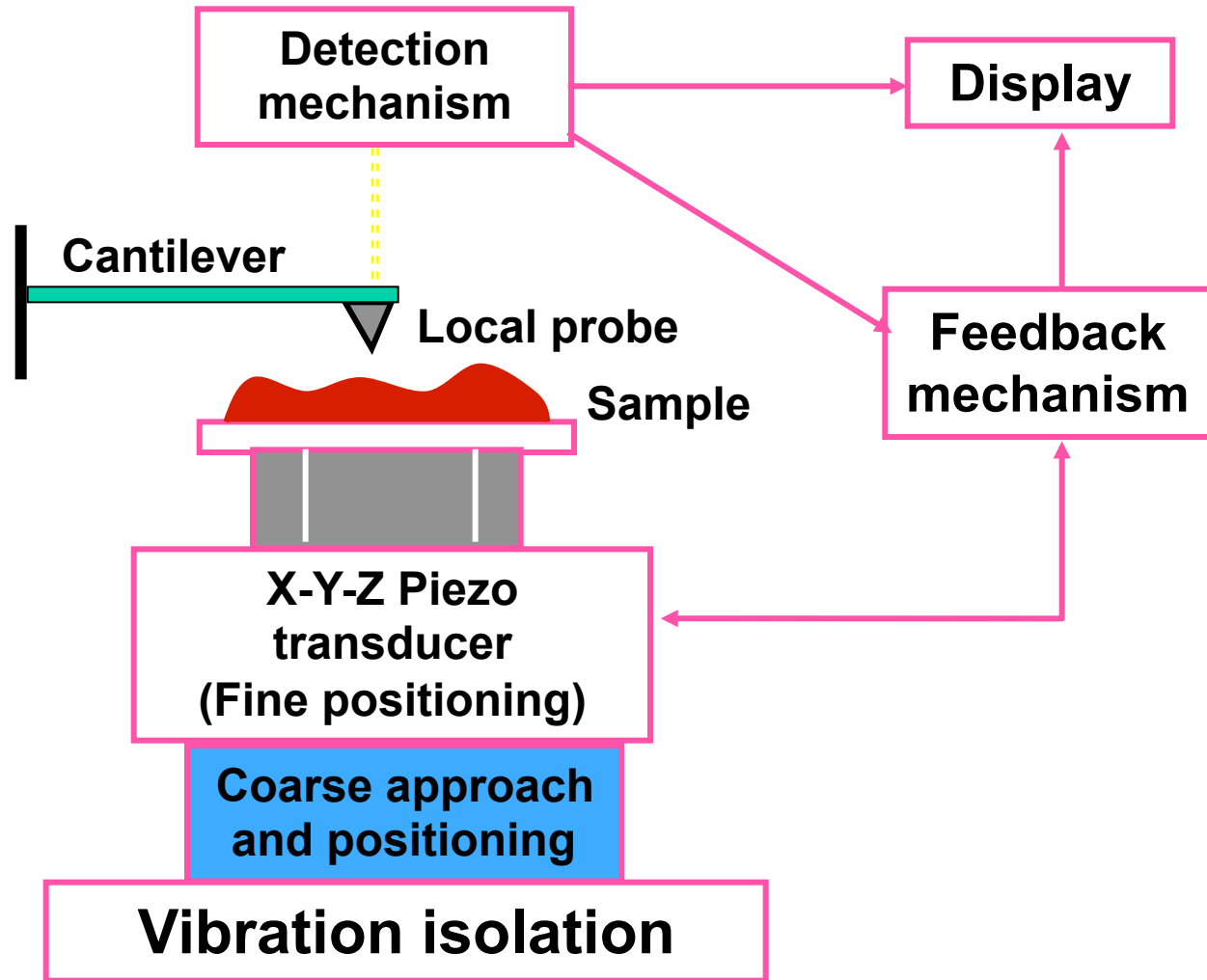
$$F = 10^{-9} - 10^{-6} \text{ N}$$

$$k = 0.1 - 1 \text{ N/m}$$

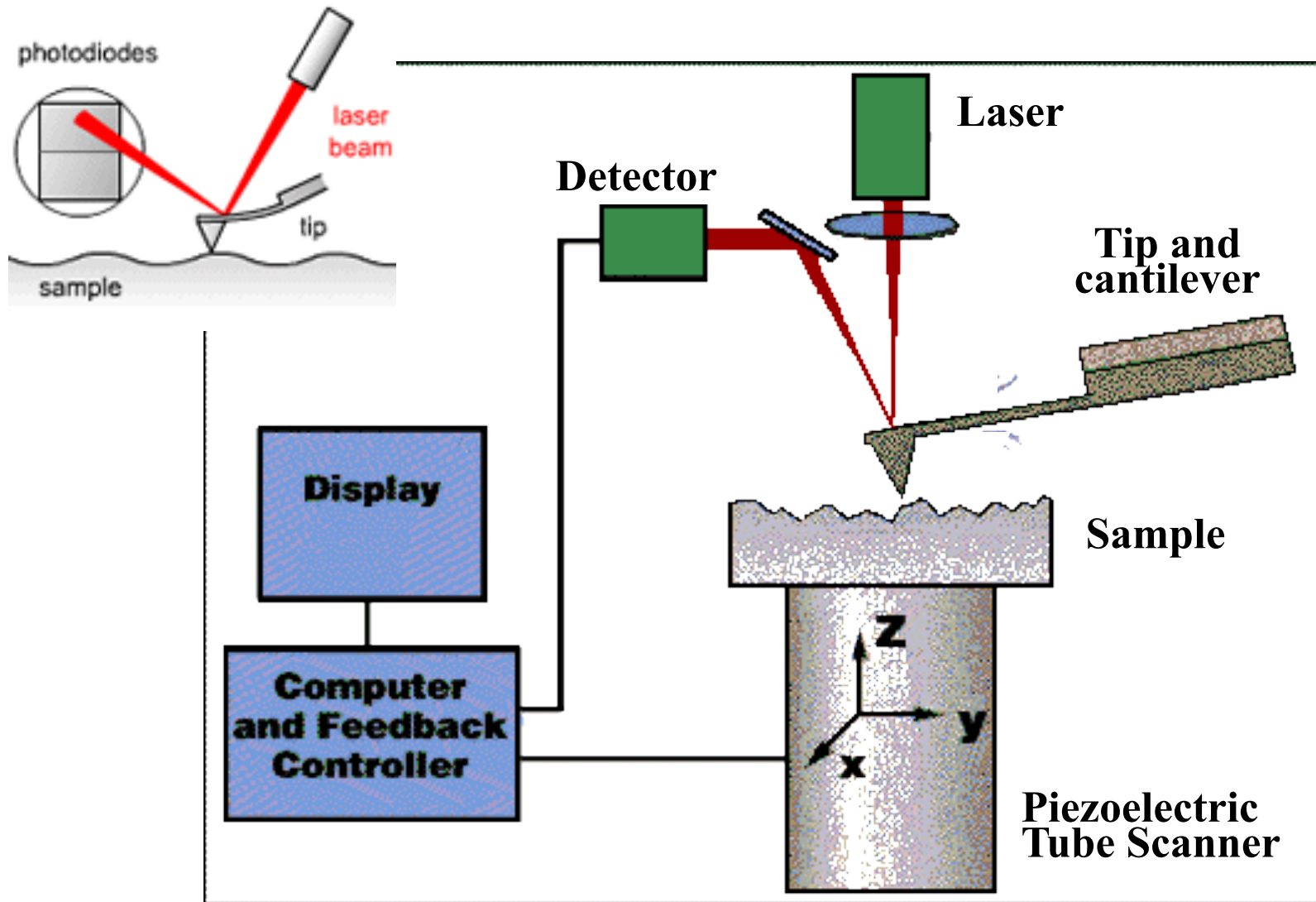
References:

- 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).

Structure of AFM



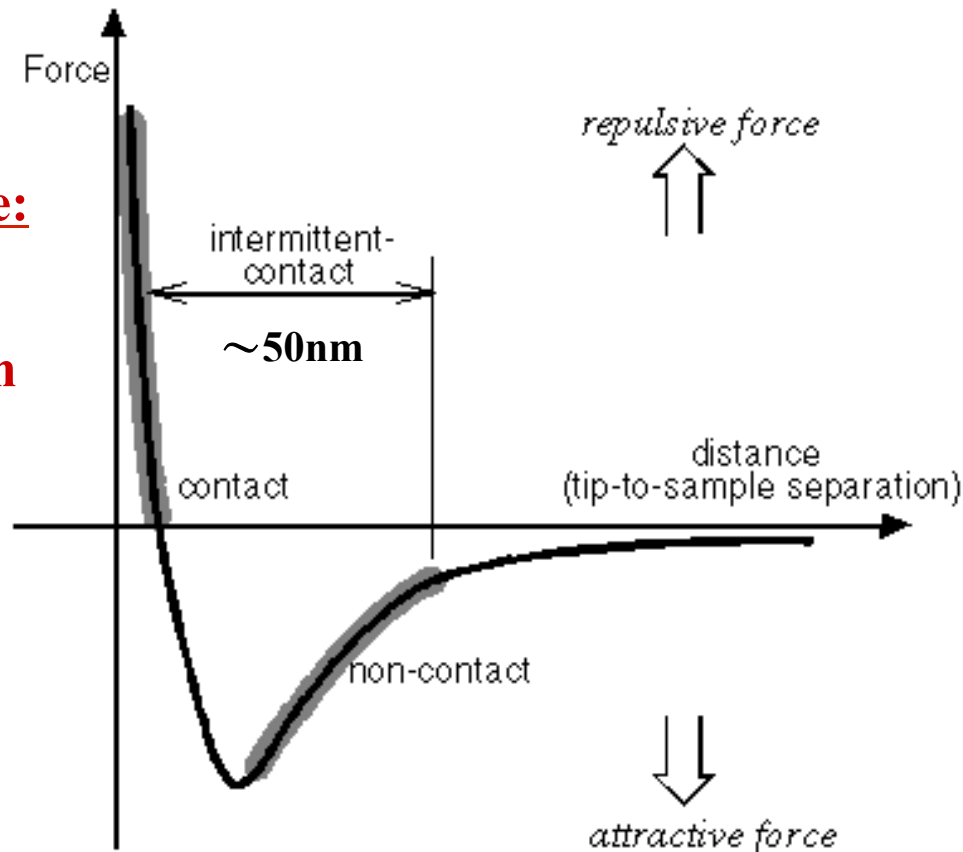
Core components of AFM



Interaction between the probe and sample

Short-range:

- 1) Bonding
- 2) Repulsion

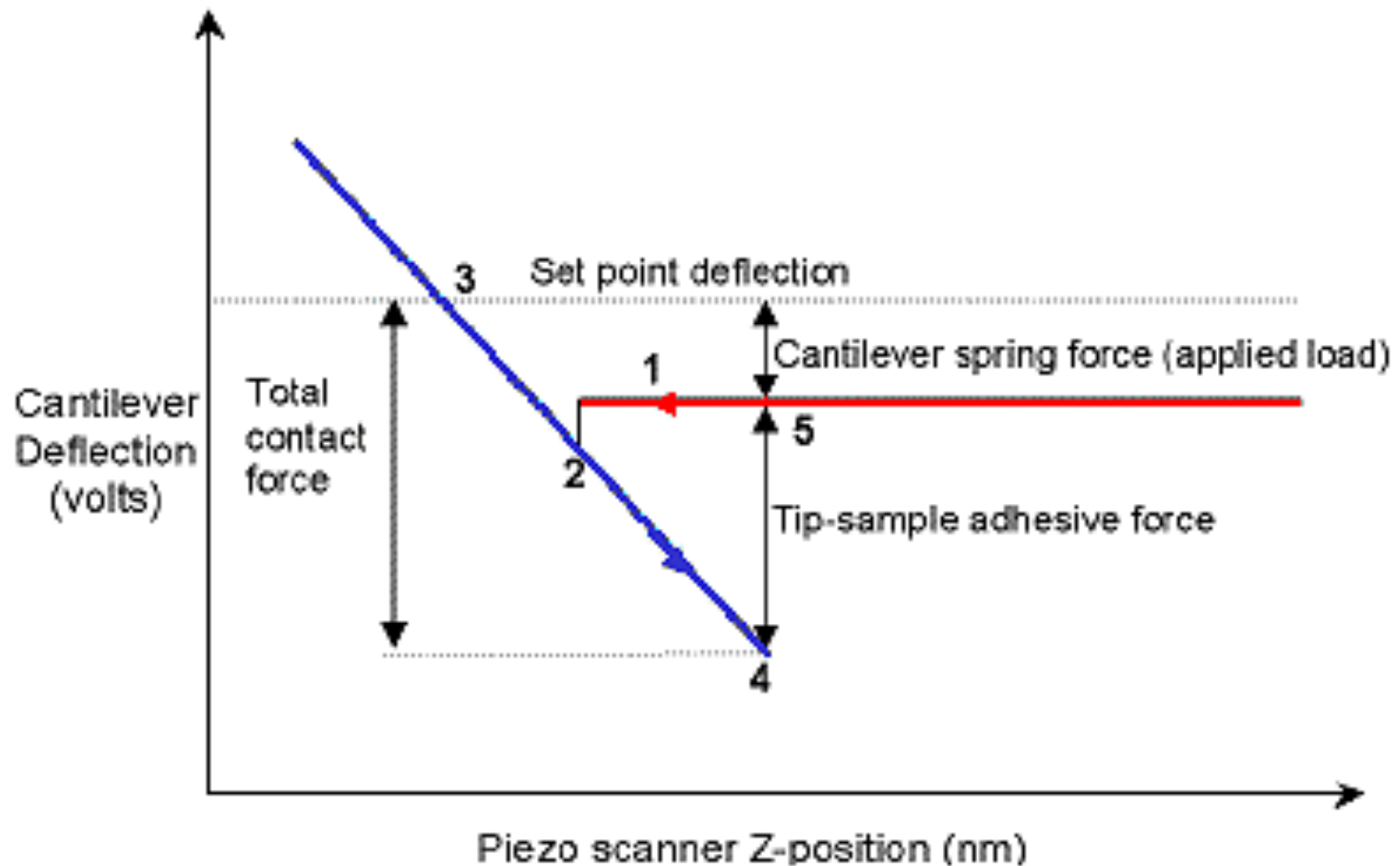


Long-range:

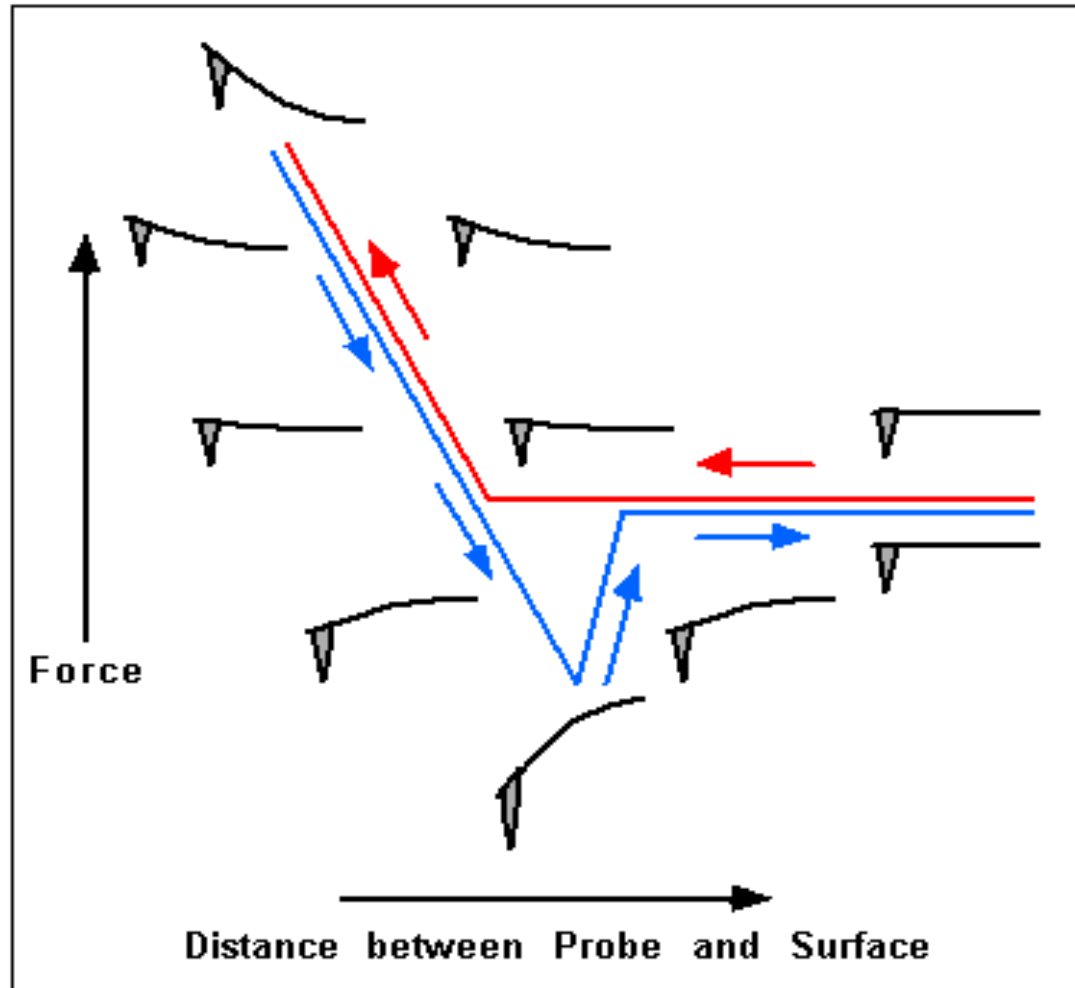
- 1) Van der Waal
- 2) Capillary
- 3) Magnetic
- 4) Electrostatic

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

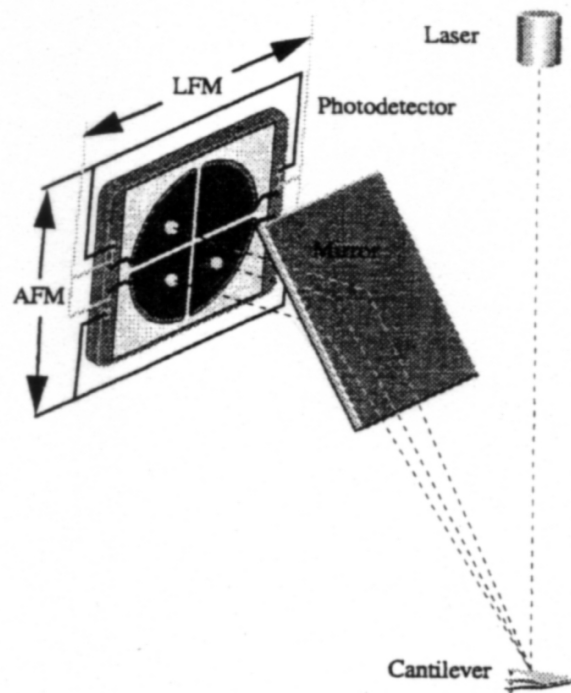
Deflection of Cantilever vs Piezo displacement



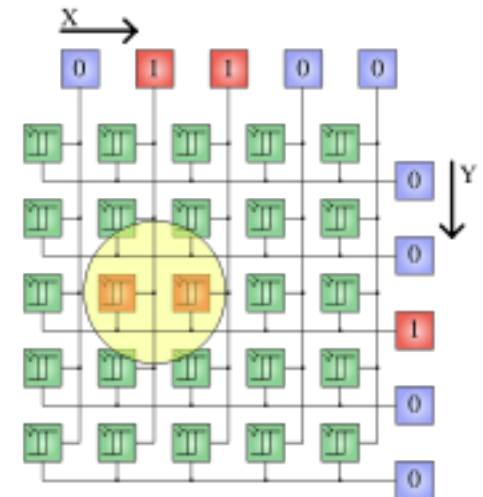
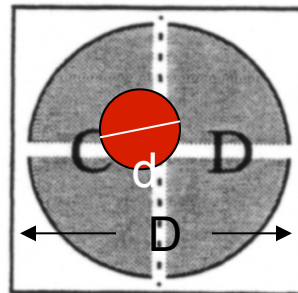
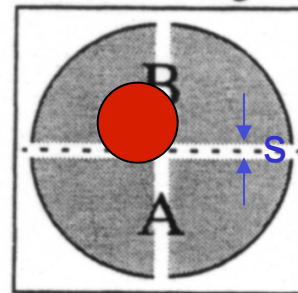
Reaction of the probe to the force



Position-sensitive Photo Diode (PSPD)

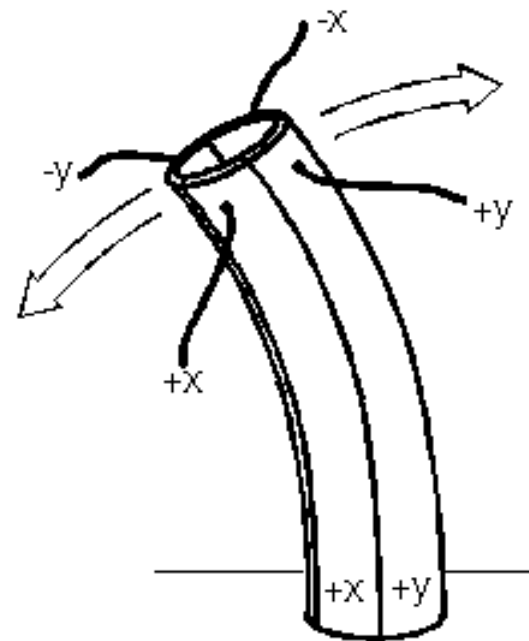
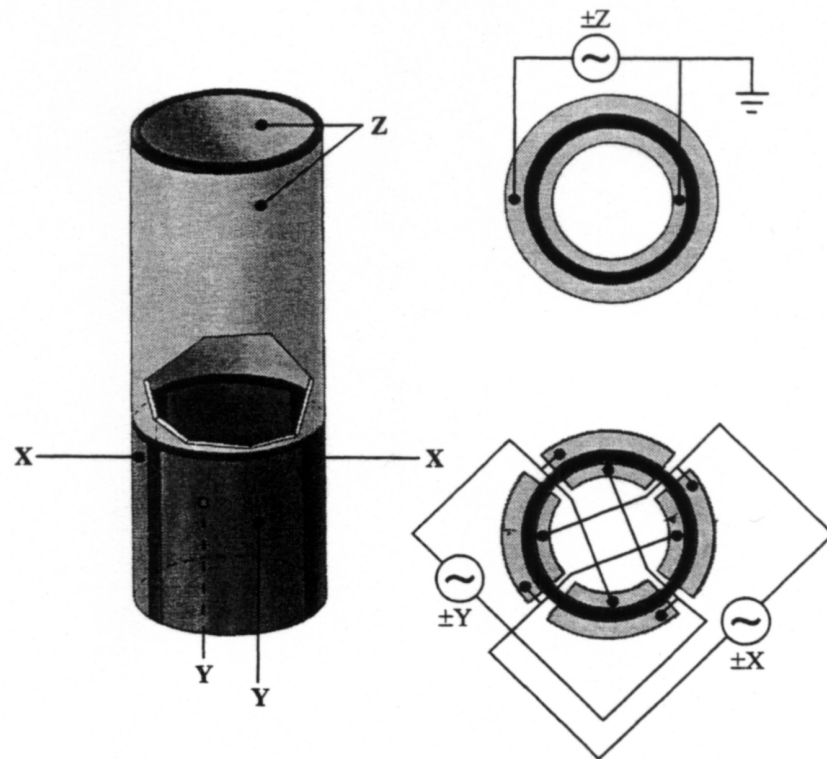


Photodetector segments

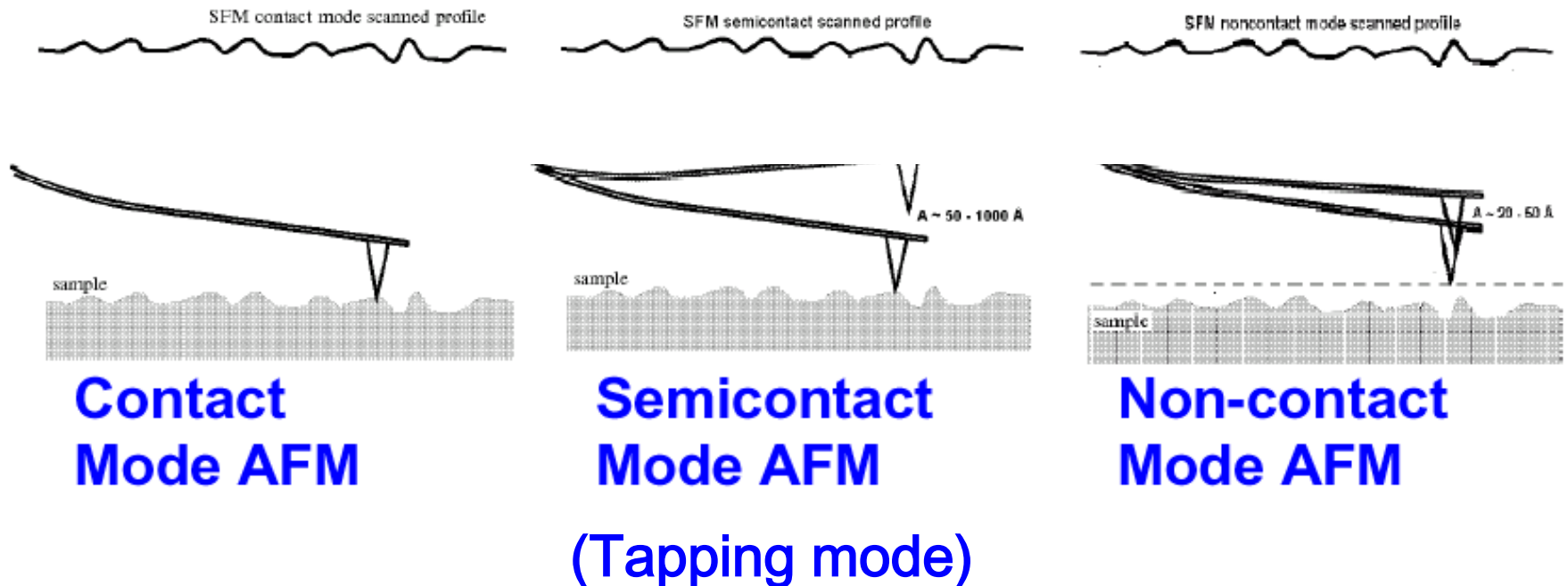


$D \sim 10\text{mm}$ $d \sim 1\text{mm}$ $s \sim 0.01\text{mm}$

Piezo Scanner



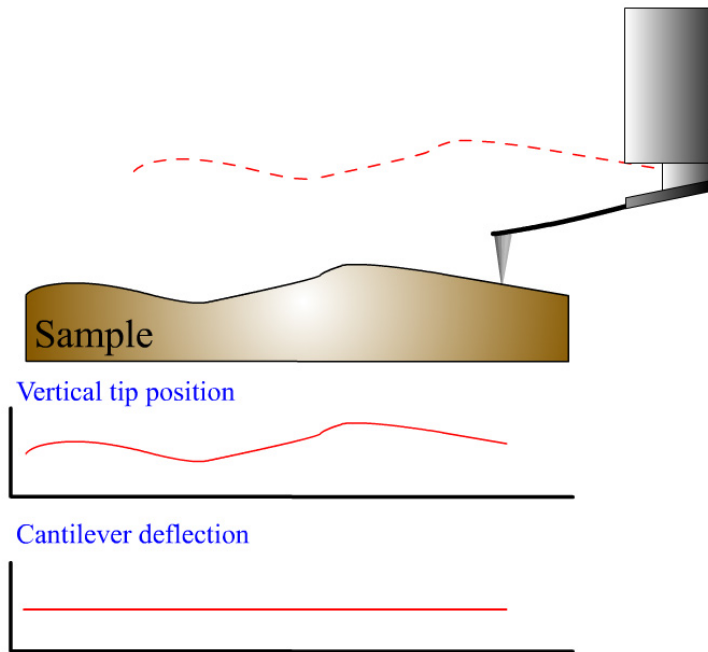
Three scanning modes of AFM



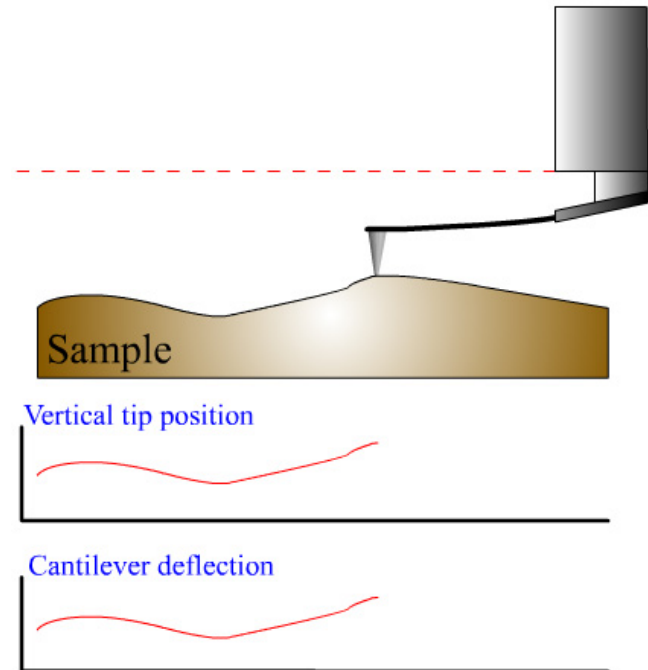
Two imaging methods in contact mode

- 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

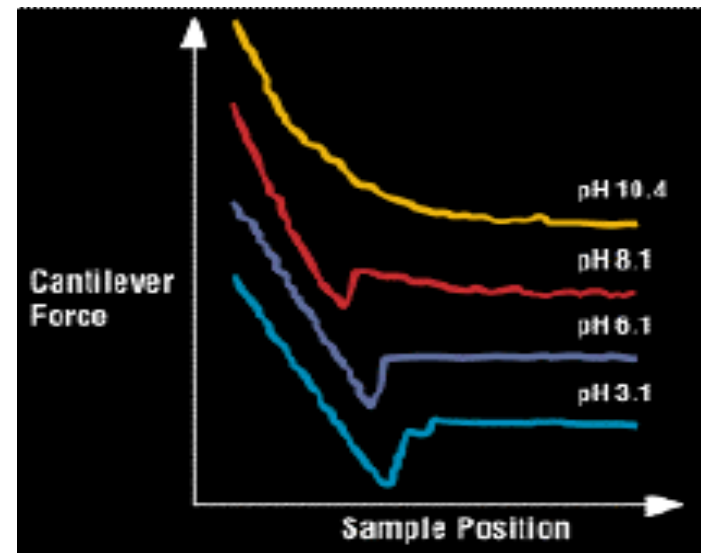
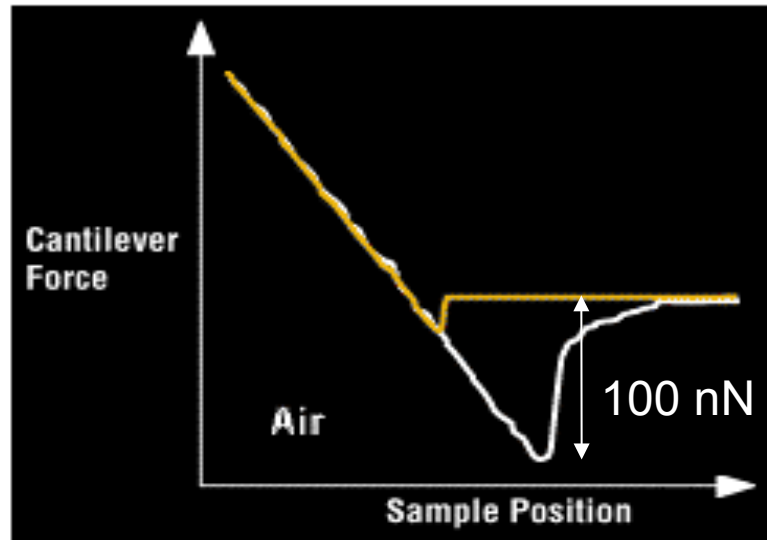
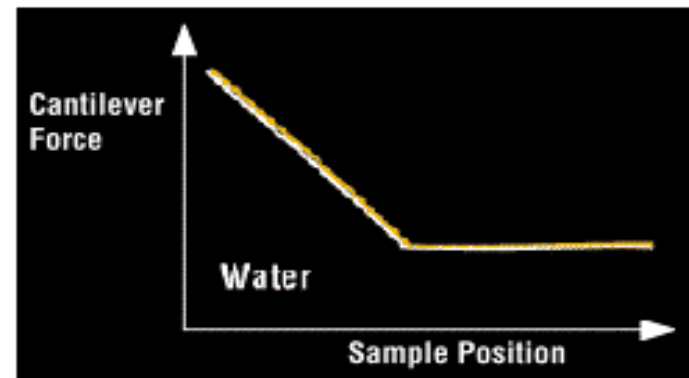
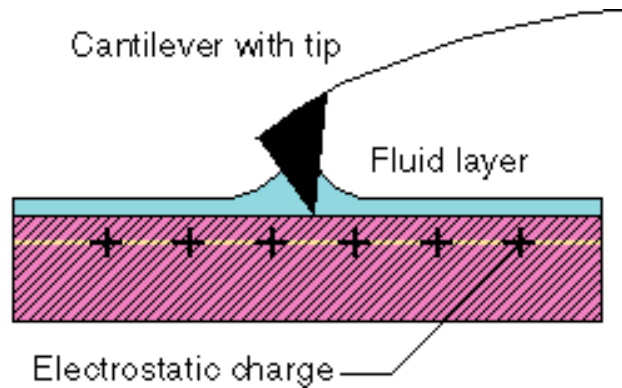
Constant-force

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

Constant-height

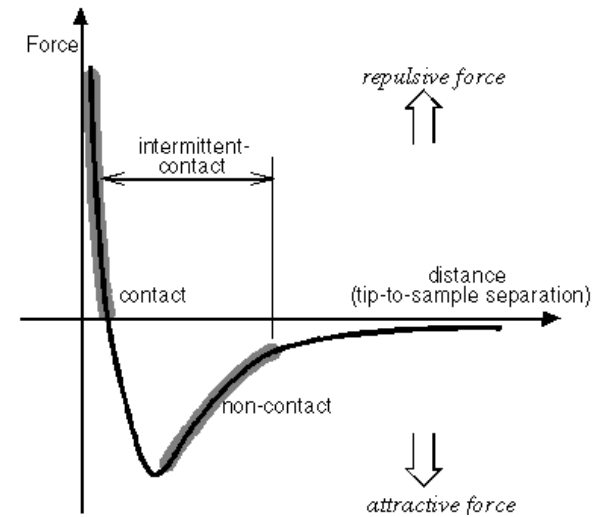
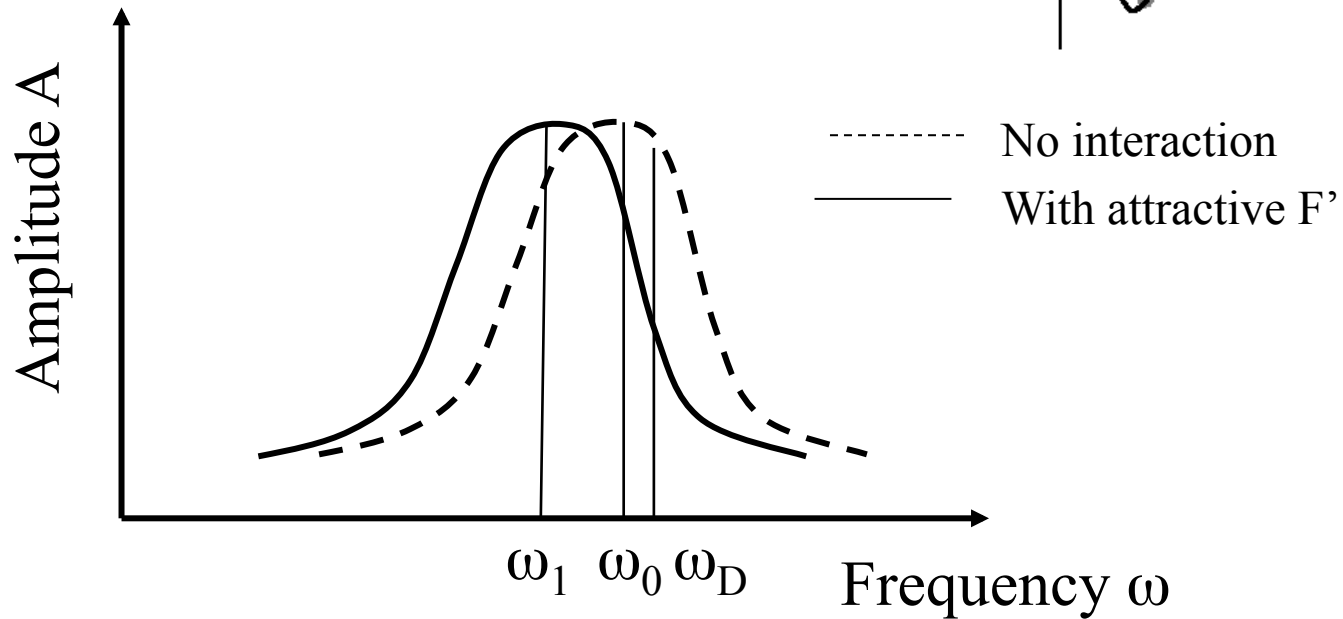
- Advantages:
 - Simple structure (no feedback control)
 - Fast response
- Disadvantages:
 - Limited vertical range (cantilever bending and detector dynamic range)
 - Varied force

Problems with the contact mode

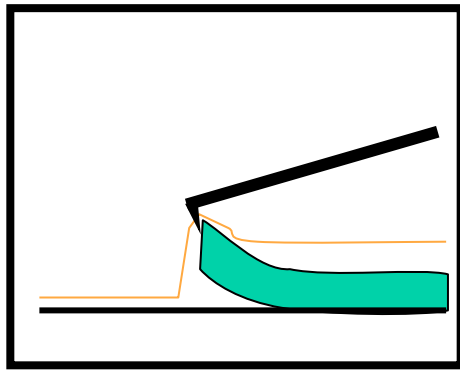


AC imaging mode

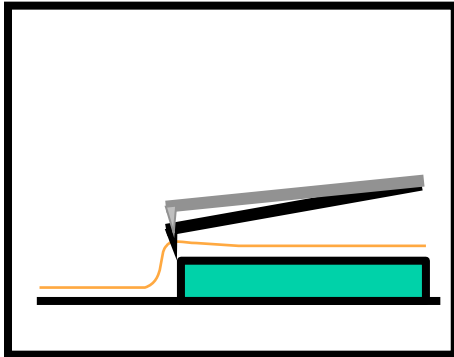
$$\omega_1 = \omega_0 (1 - F'/2c)$$



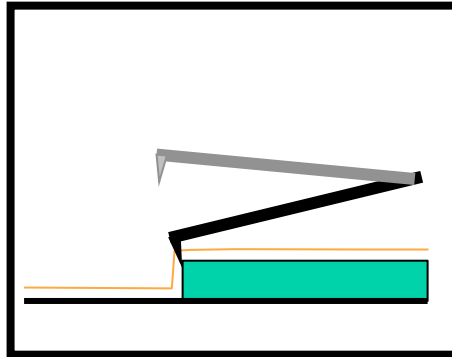
Comparison of three scanning modes



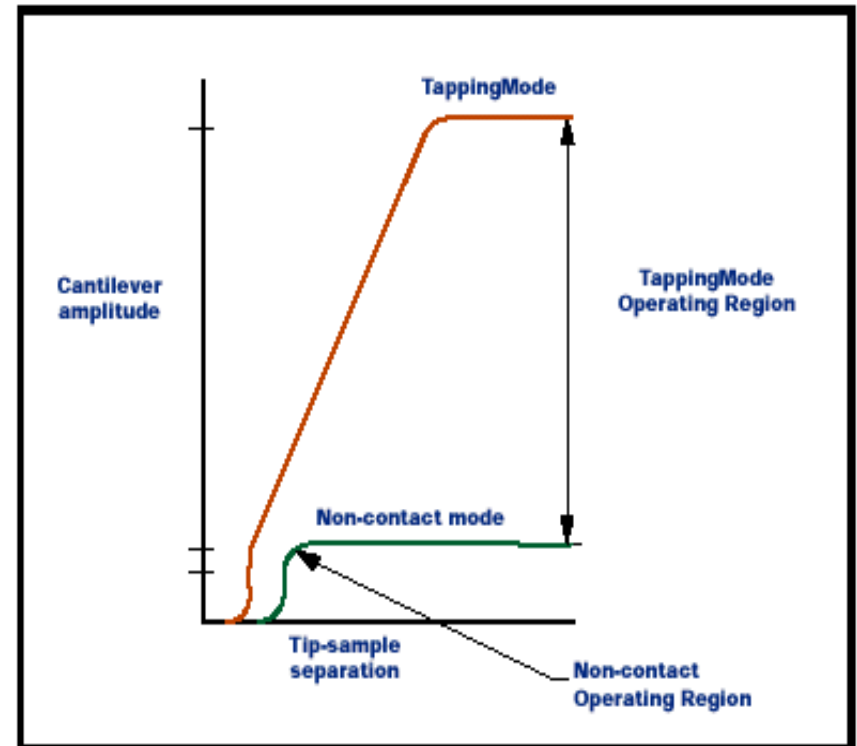
Contact



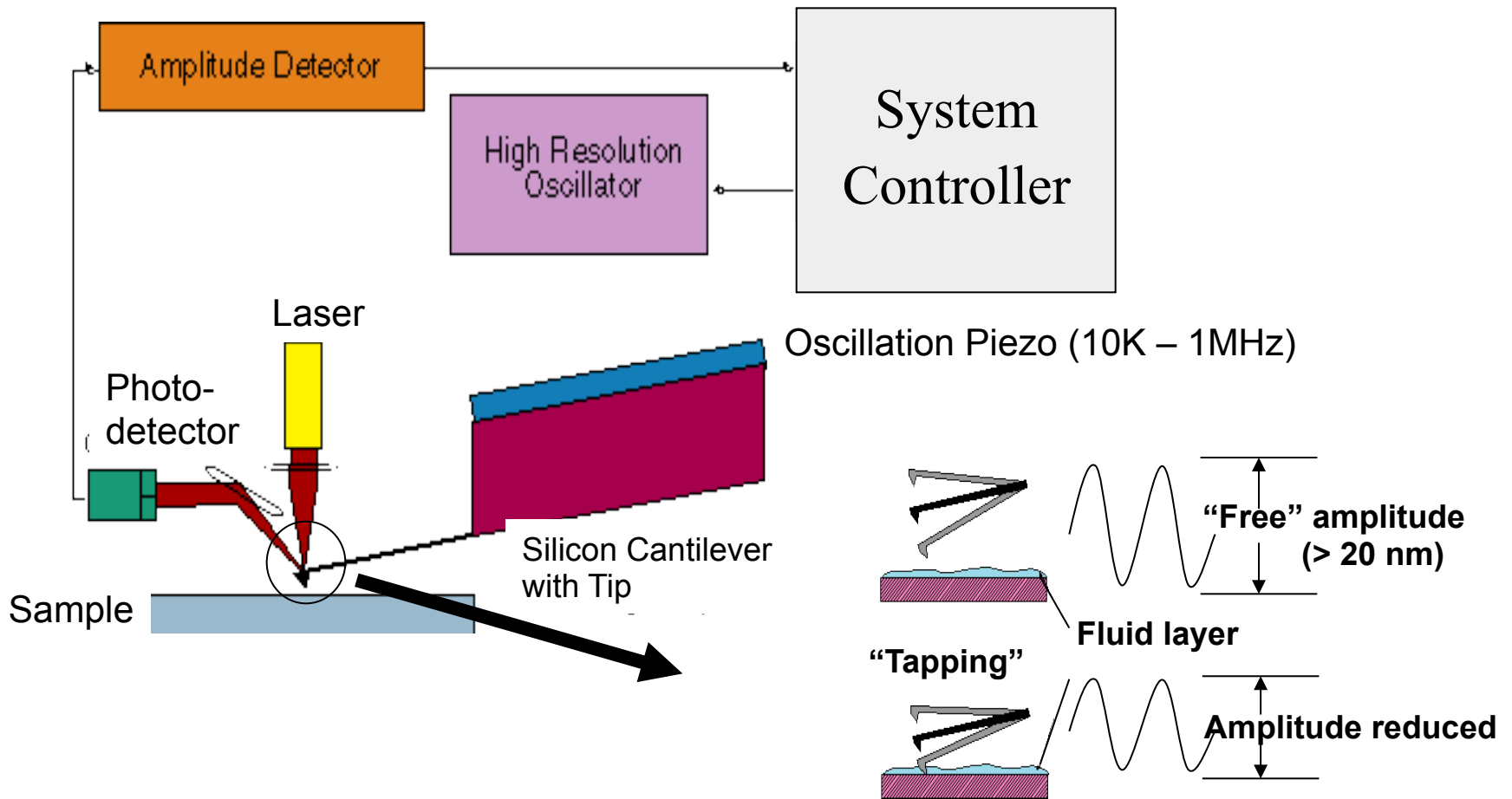
Non-contact



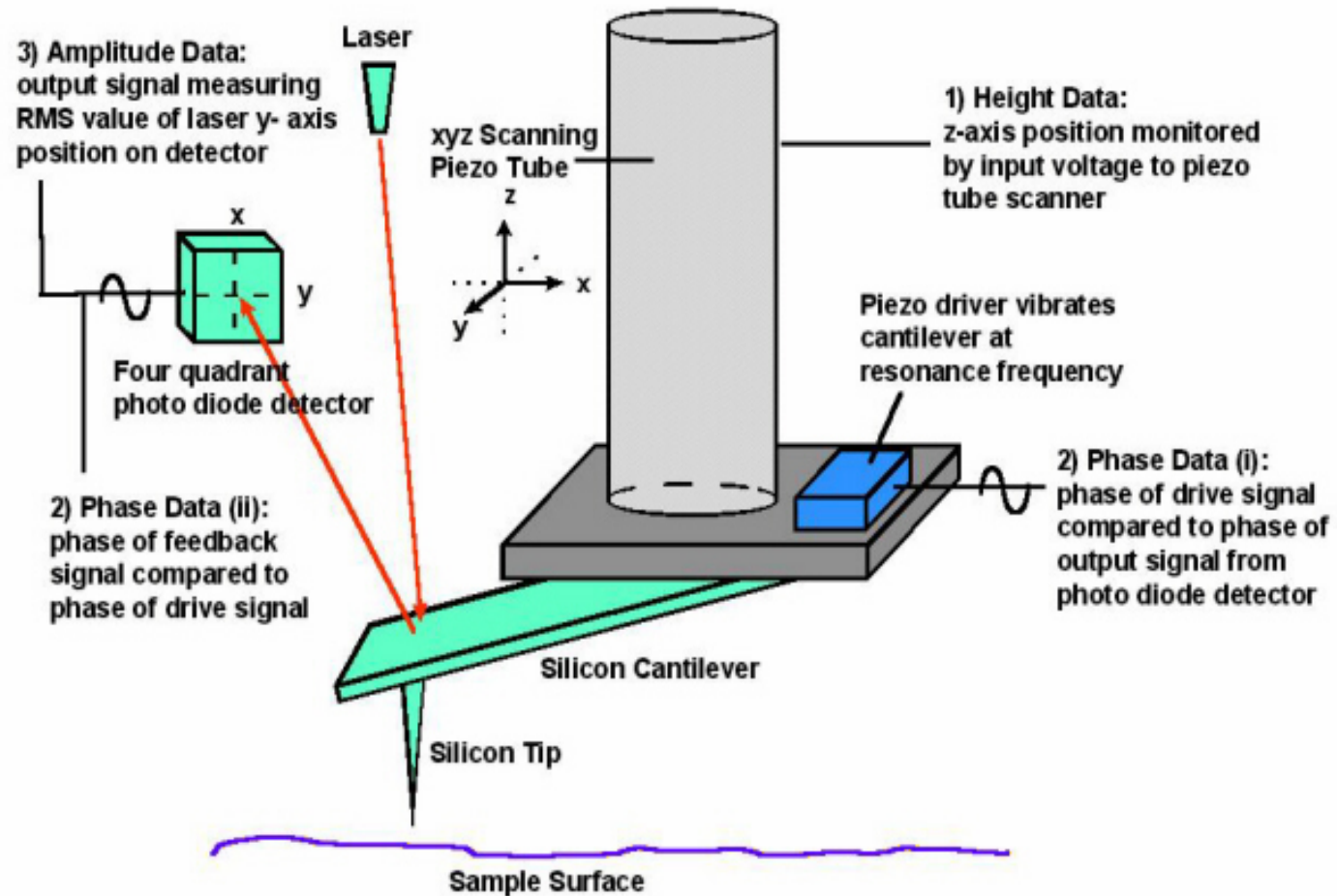
Intermittent-contact



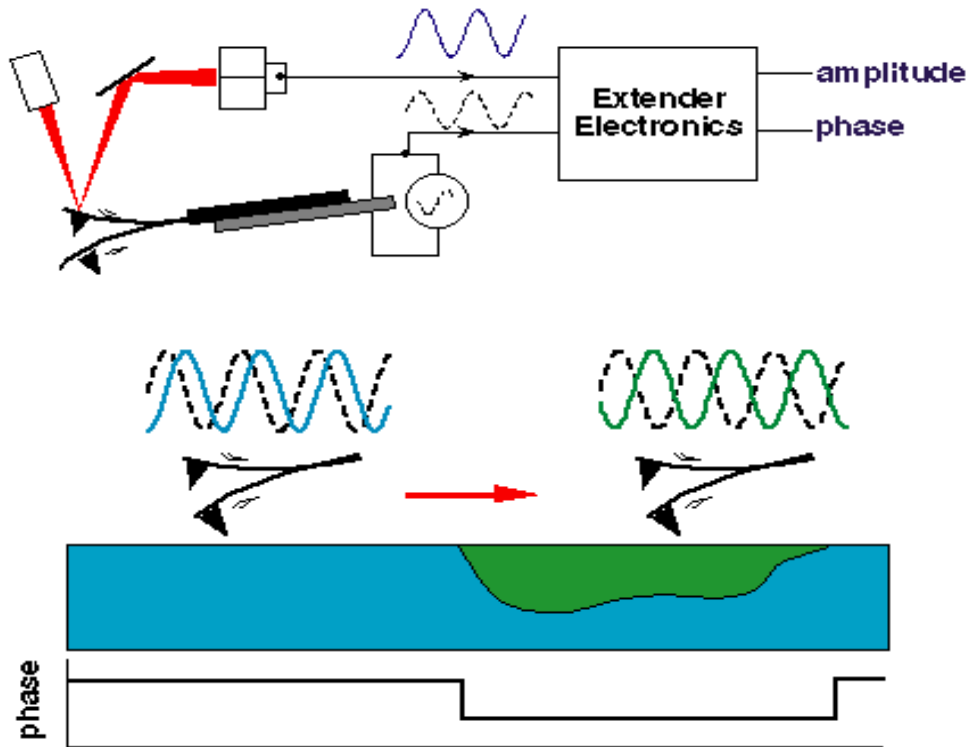
Tapping mode



Three Types of Data Collected in Tapping Mode

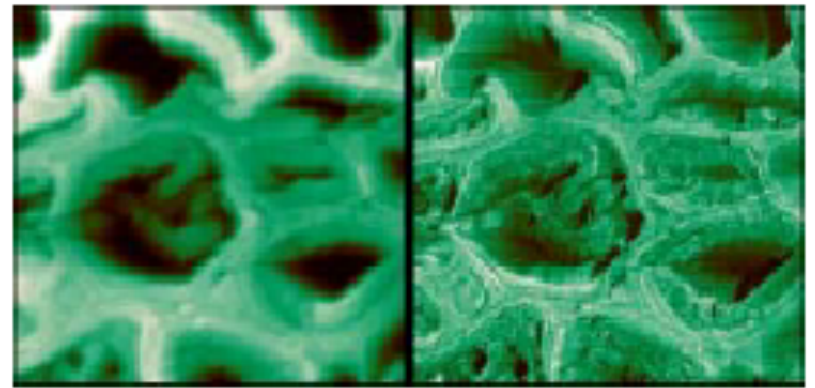


Images by tapping mode



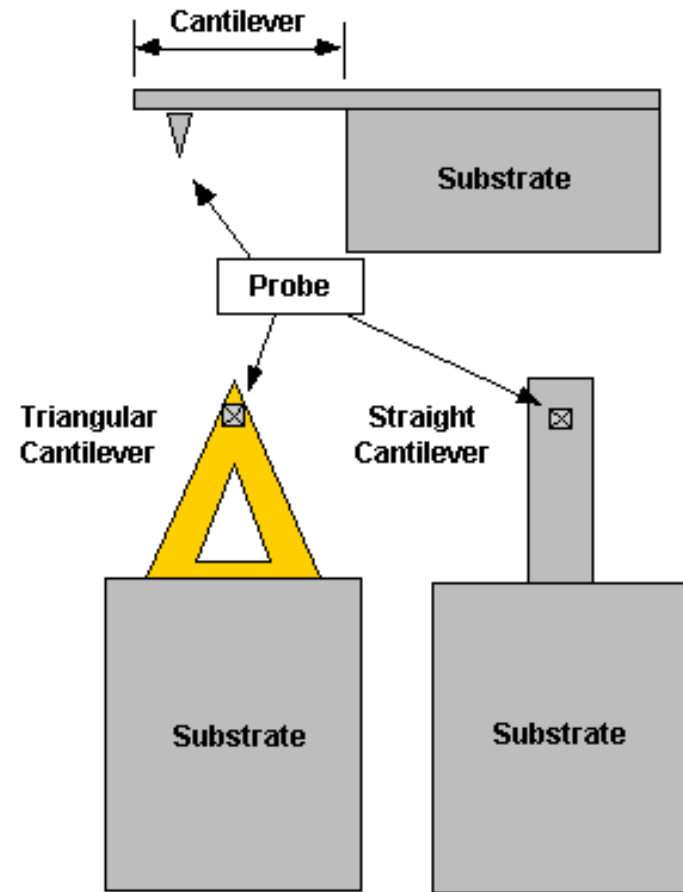
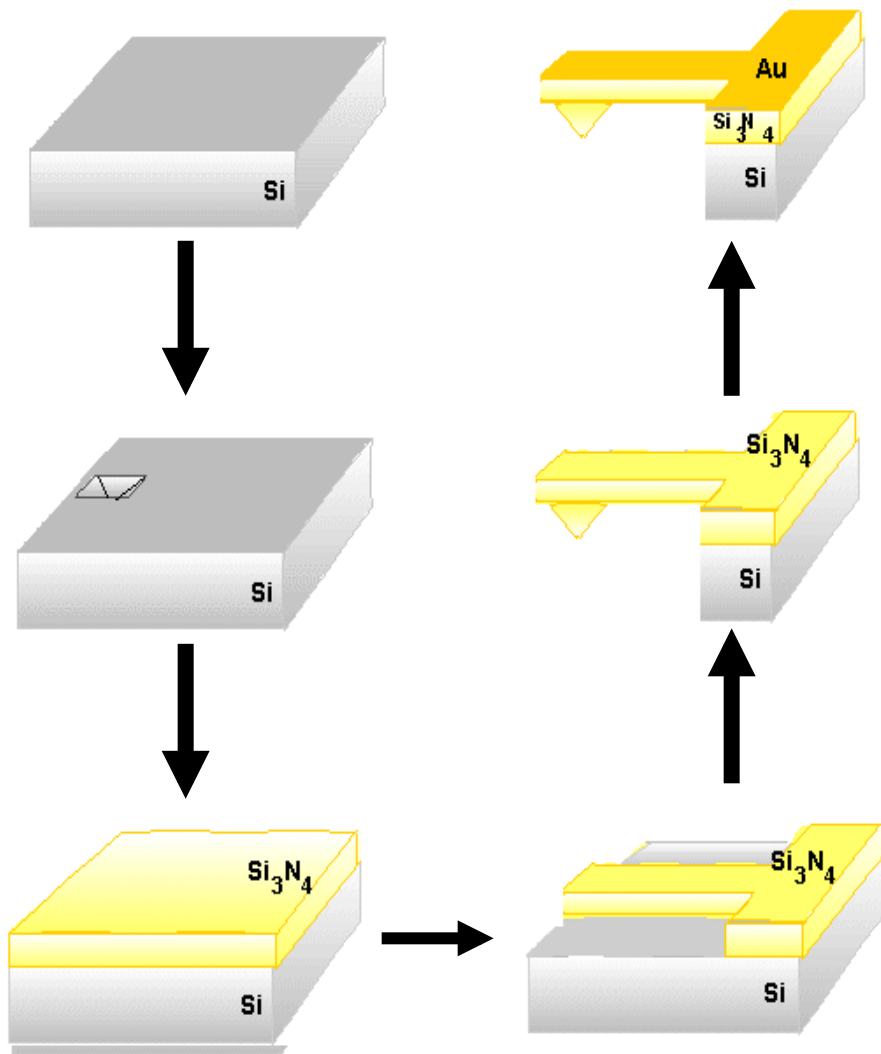
Topography

Phase



AFM image of a fresh
Alfalfa root section

Fabrication of AFM probes



Typical Tip Dimension:
 $150\mu\text{m} \times 30\mu\text{m} \times 0.5\mu\text{m}$

$k \sim 0.1 \text{ N/m}$

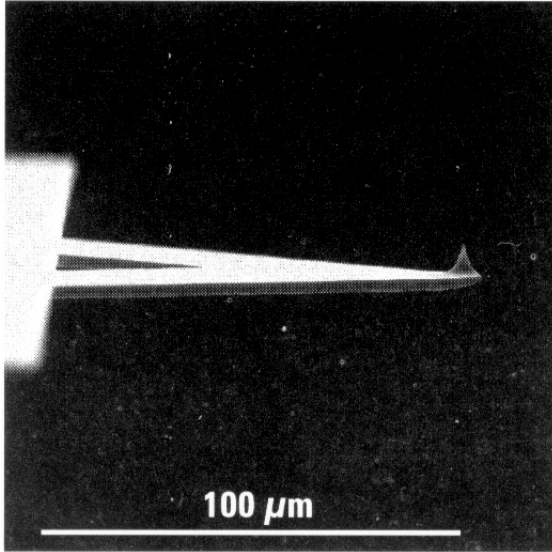
Materials: Si_3N_4

Typical Tip Dimension:
 $150\mu\text{m} \times 30\mu\text{m} \times 3\mu\text{m}$

$f_r \sim 100 \text{ kHz}$

Materials: Si

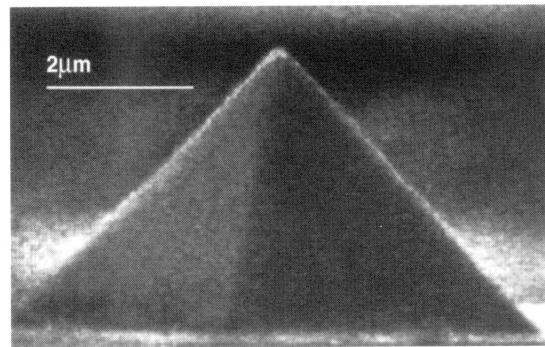
V-shaped



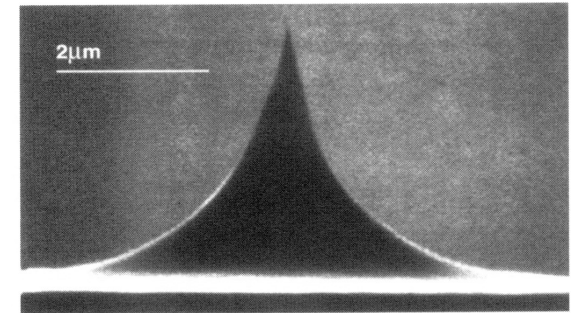
Materials: Si, SiO₂, Si₃N₄

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

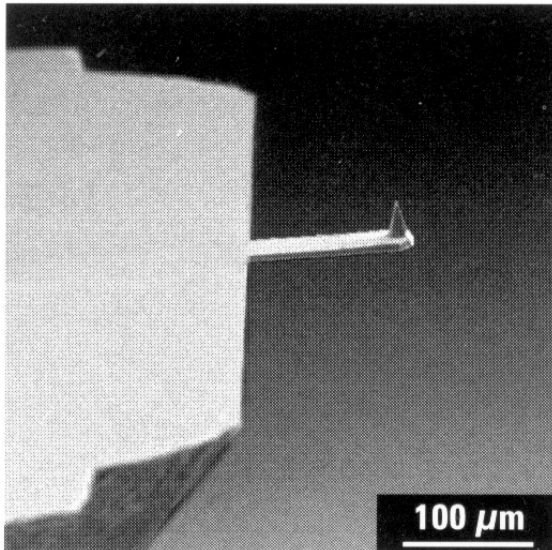
Pyramid Tip



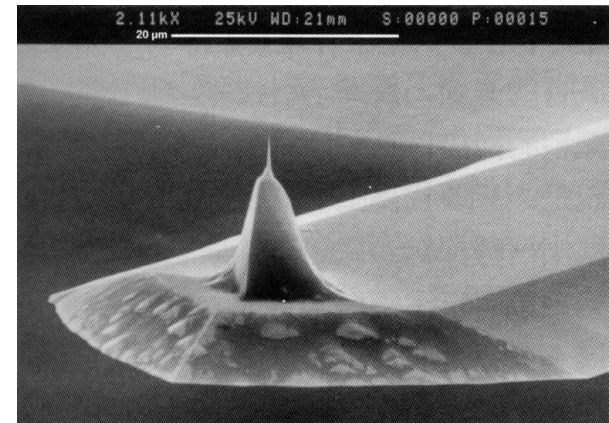
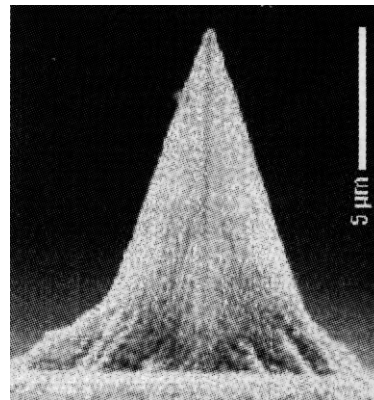
Ultrasharp Tip



Rectangular-shaped



Diamond-coated Tip



Criteria for AFM probe

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

To detect force of $\sim$ nN

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

To enable scanning and other operations

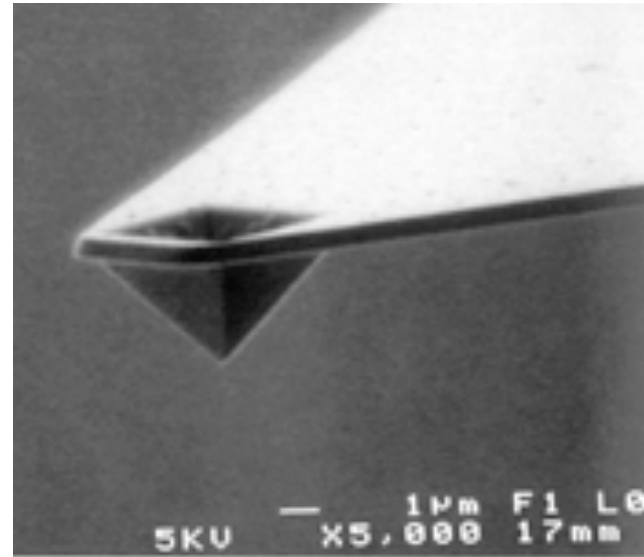
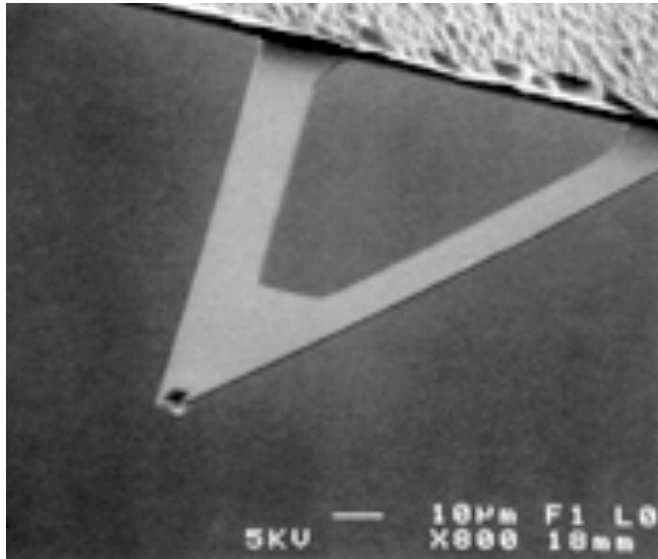
- 3) Highly anisotropic stiffness

Easy to bent and difficult to twist

- 4) Sharp protrusion at the apex

To better define the tip-sample interaction

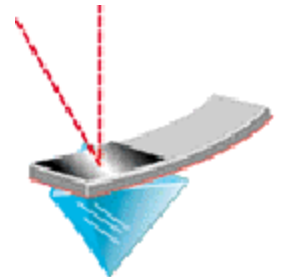
Tip of small shear force (for Contact mode)



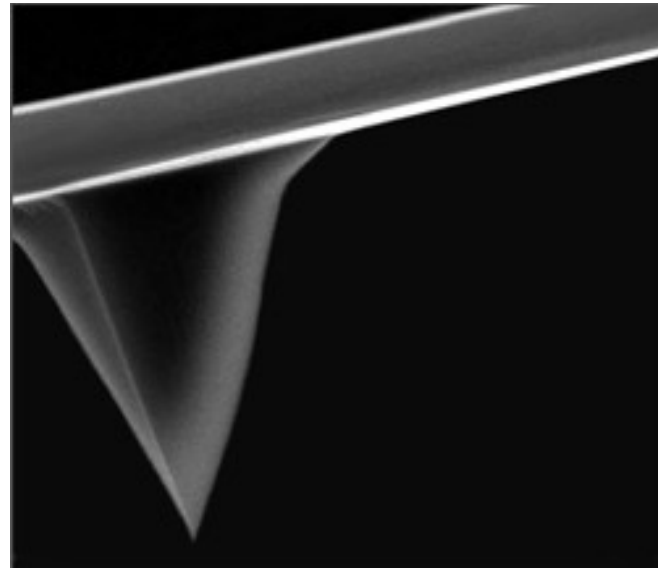
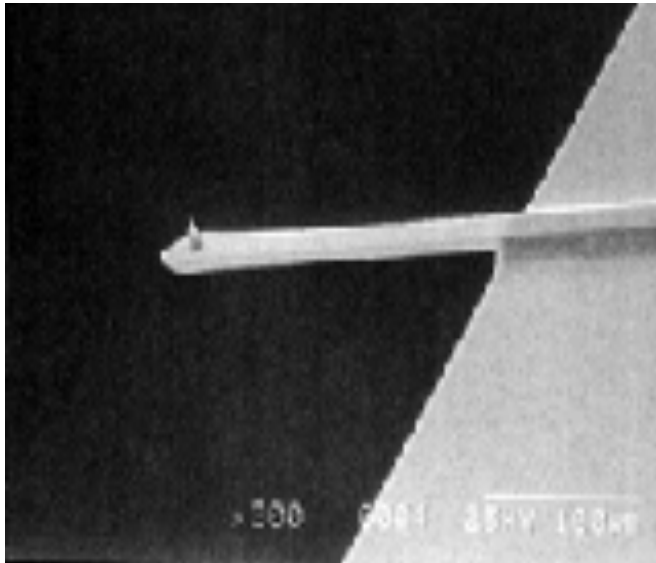
Typical Tip Dimension:
 $150\mu\text{m} \times 30\mu\text{m} \times 0.5\mu\text{m}$

$k \sim 0.1 \text{ N/m}$

Materials: Si_3N_4



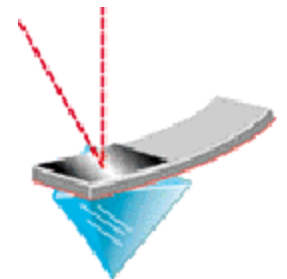
Tip of high resonant frequency (for Tapping mode)



Typical Tip Dimension:
 $150\mu\text{m} \times 30\mu\text{m} \times 3\mu\text{m}$

$f_r \sim 100 \text{ kHz}$

Materials: Si



AFM versus STM

1. 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.

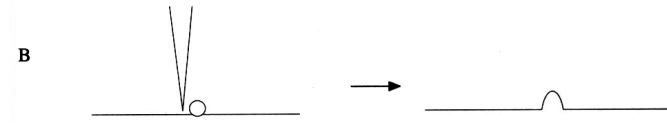
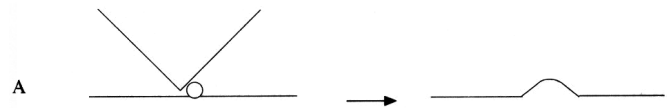
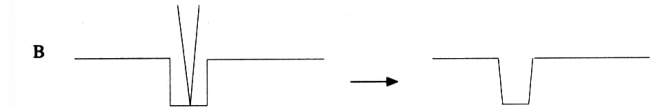
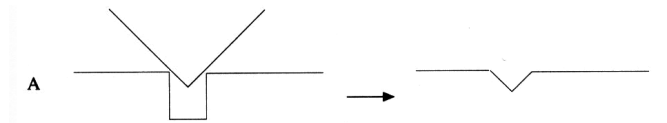
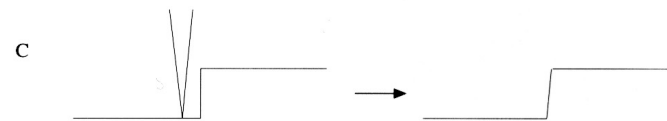
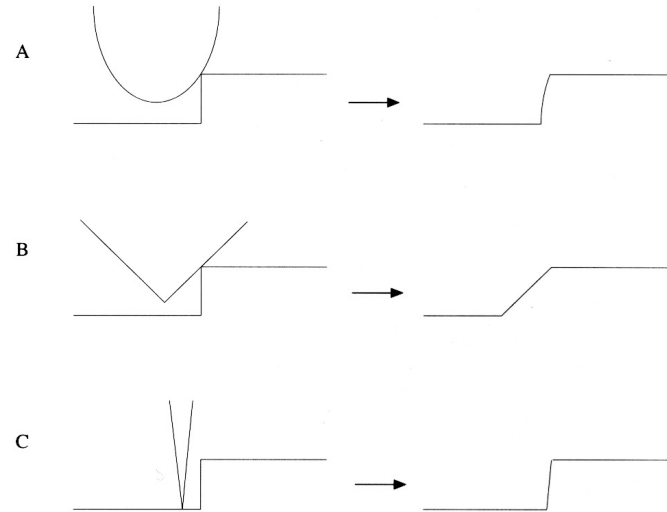
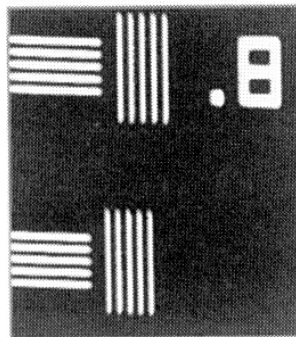
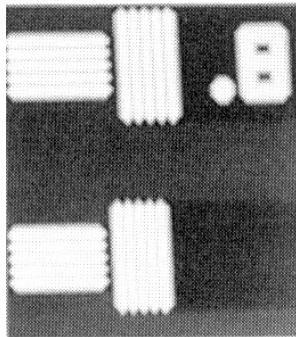
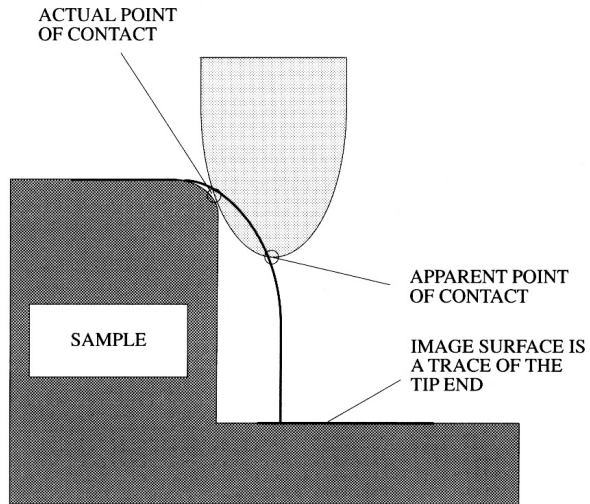
AFM versus EM

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.

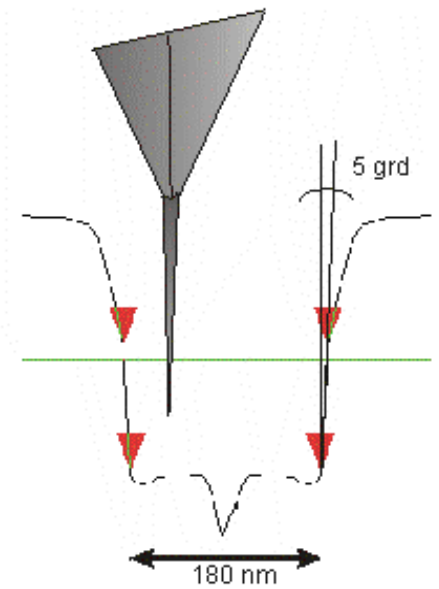
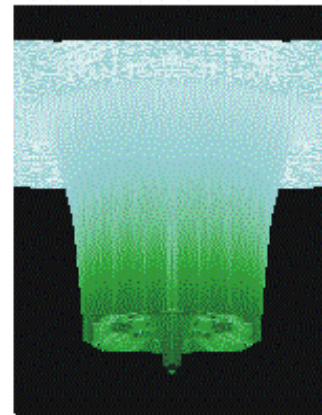
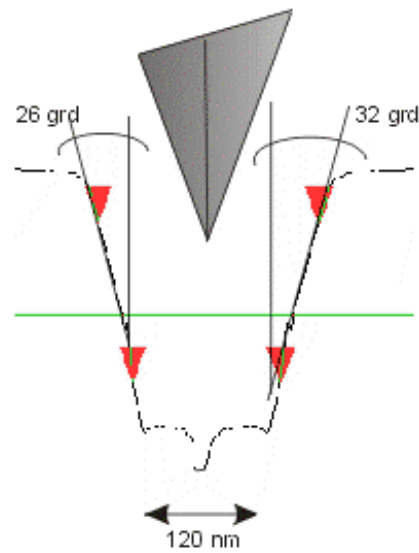
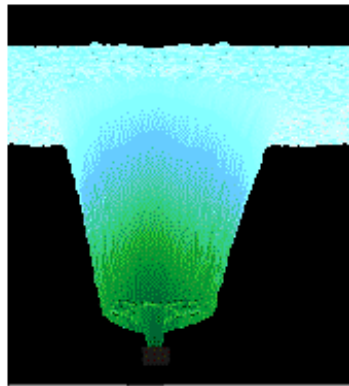
AFM versus Optical Microscope

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

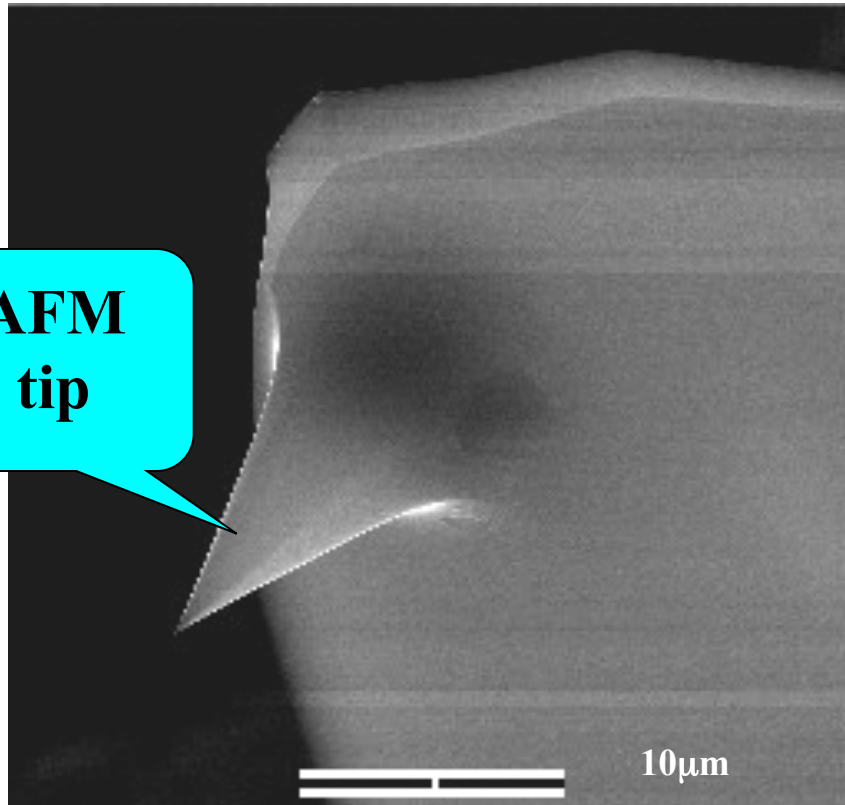


Ultra-sharp tip



AFM Tip + Carbon Nanotube

**AFM
tip**



**Carbon
Nanotube**
 $\phi \approx 20\text{nm}$
 $L \approx 80\text{nm}$

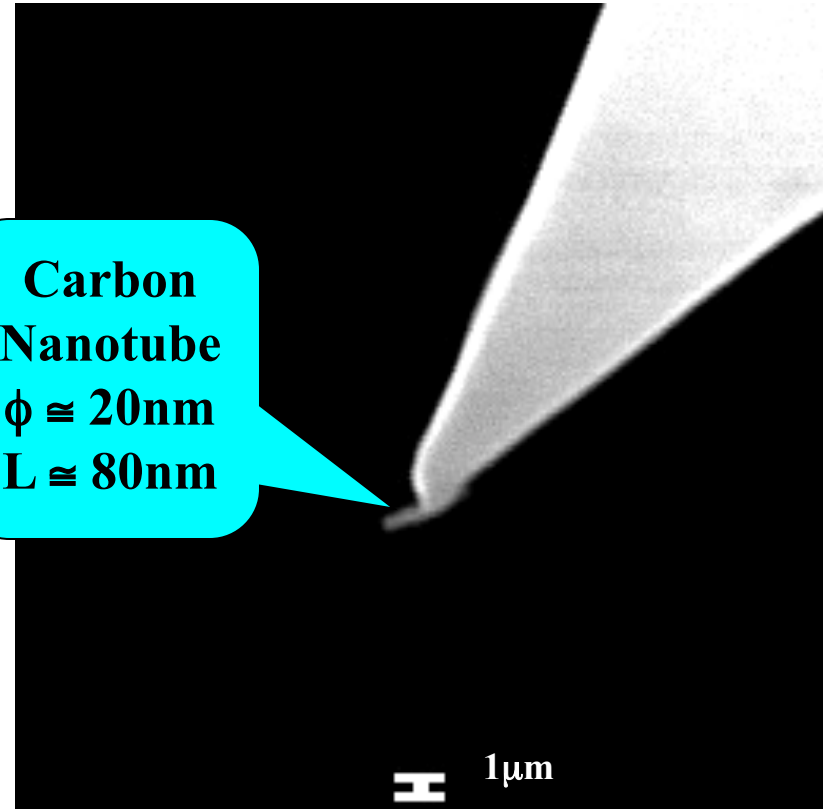
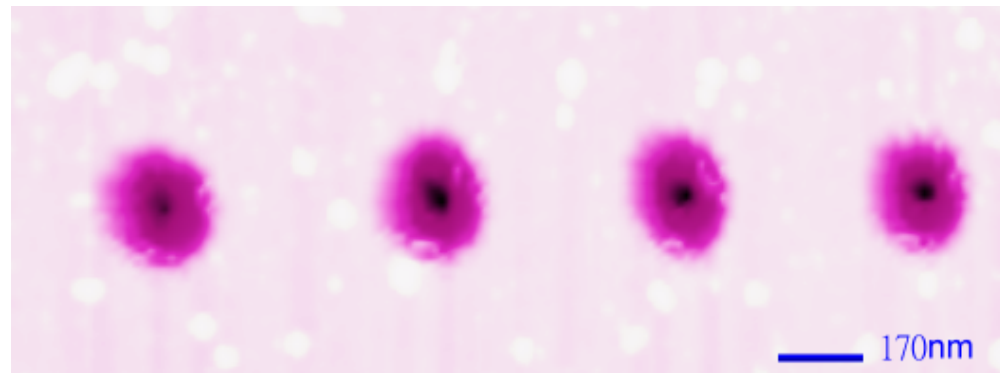
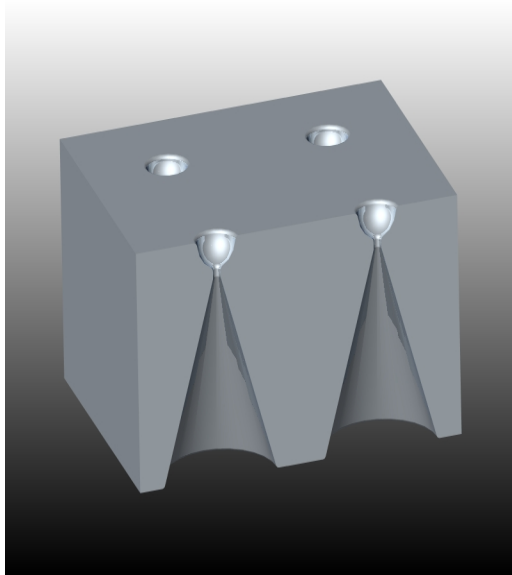
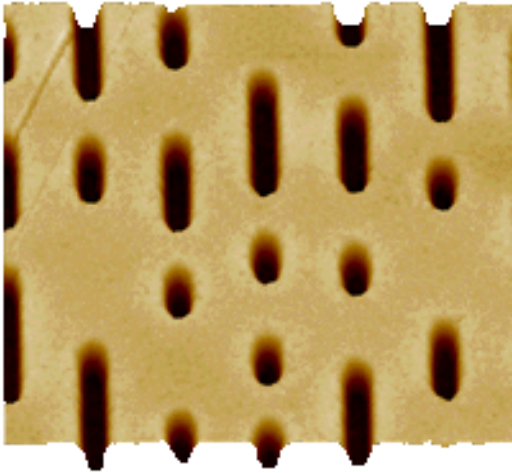


Image of high aspect ratio

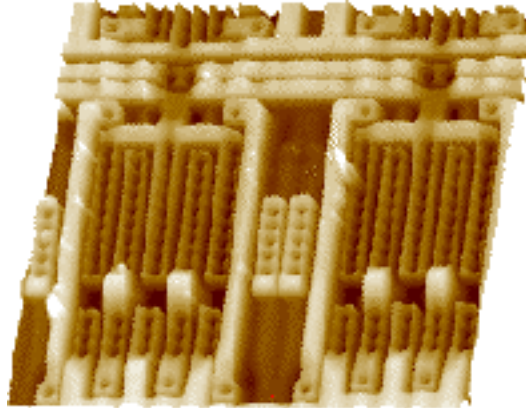


AFM images

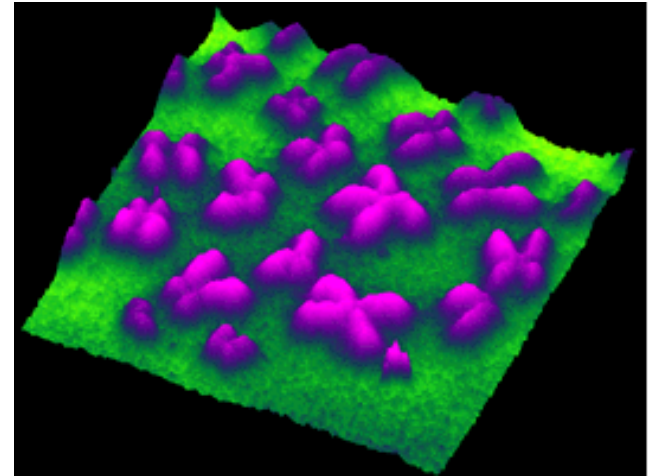
CD pits



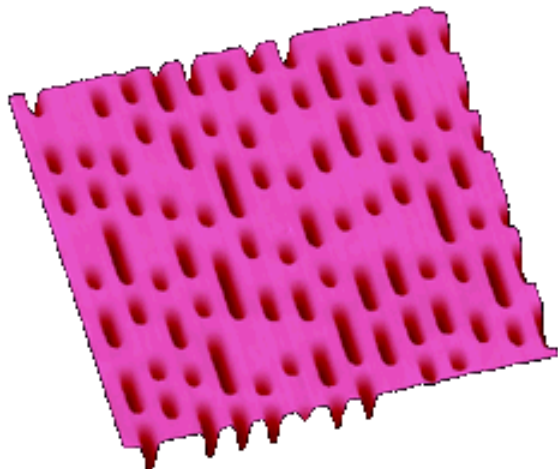
Integrated circuit



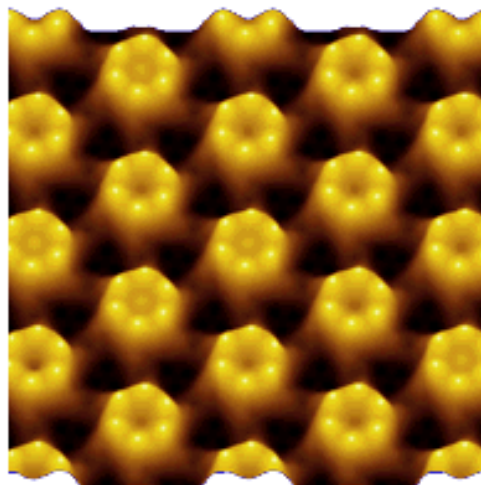
Chromosomes



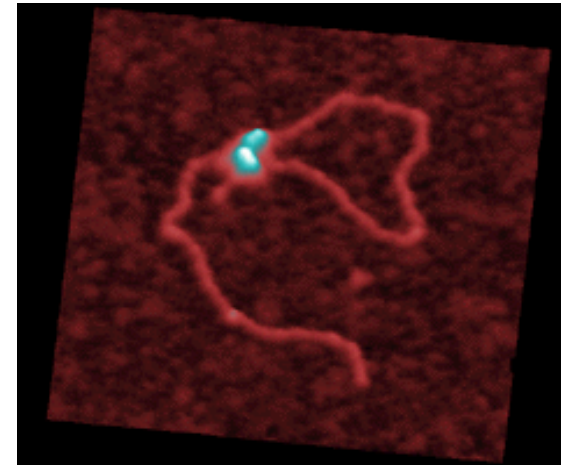
DVD pits



Bacteria



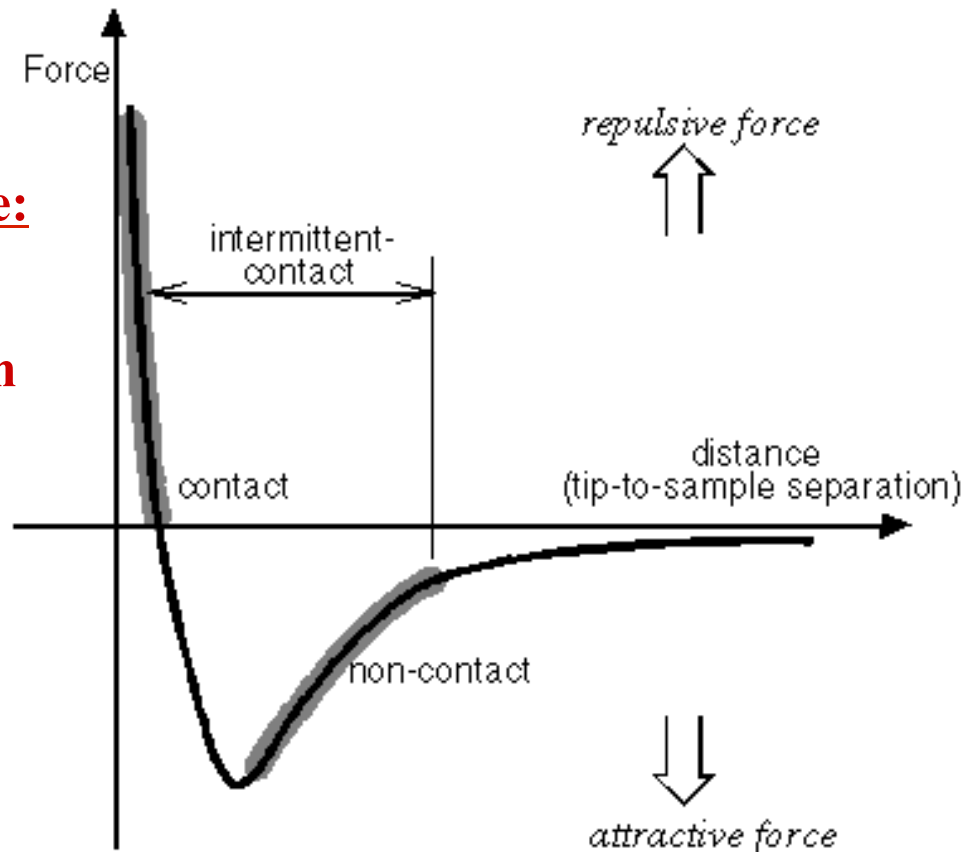
DNA



Interaction between the probe and sample

Short-range:

- 1) Bonding
- 2) Repulsion

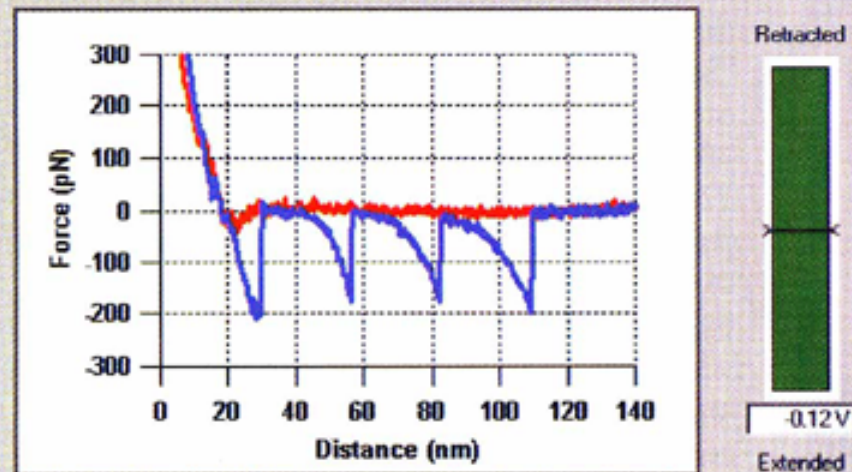
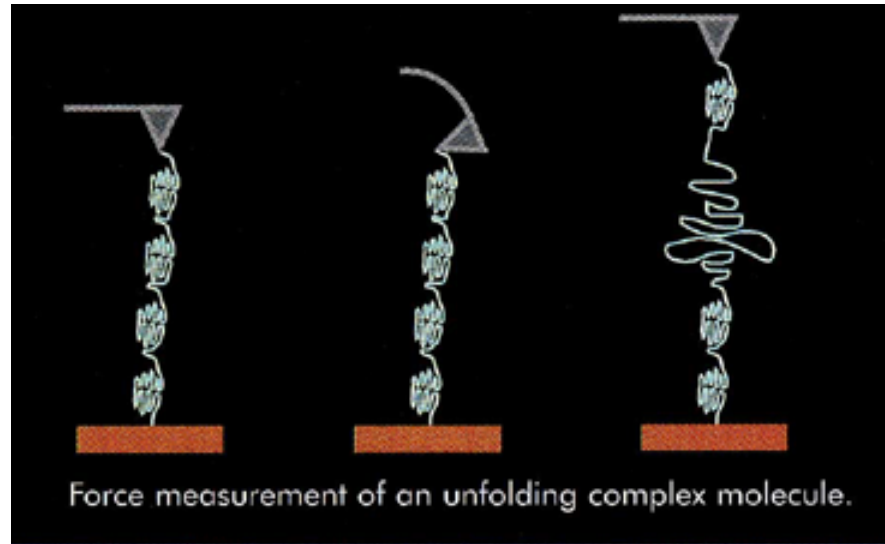


Long-range:

- 1) Van der Waal
- 2) Capillary
- 3) Magnetic
- 4) Electrostatic

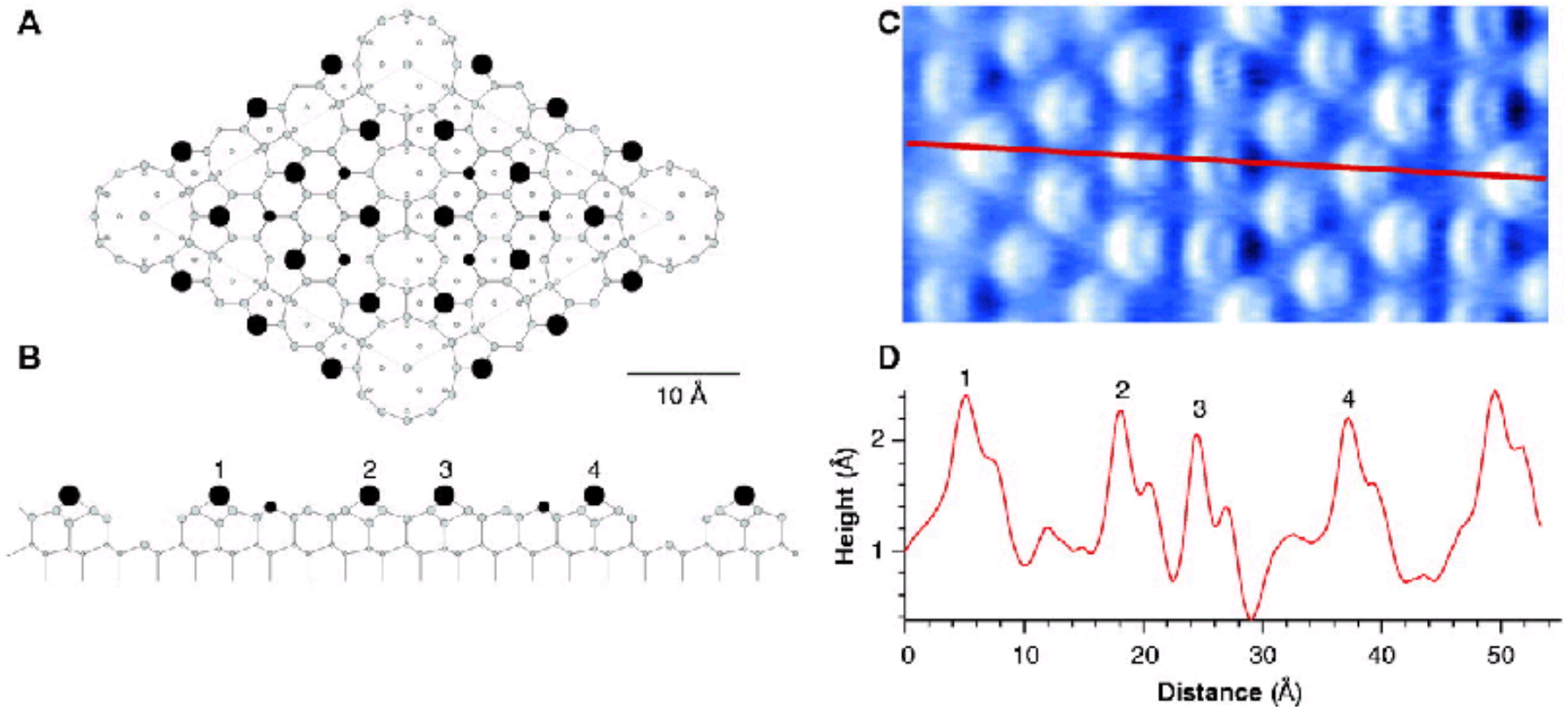
Lennard-Jones potential $\phi(r) = -A/r^6 + B/r^{12}$

Force spectroscopy by AFM



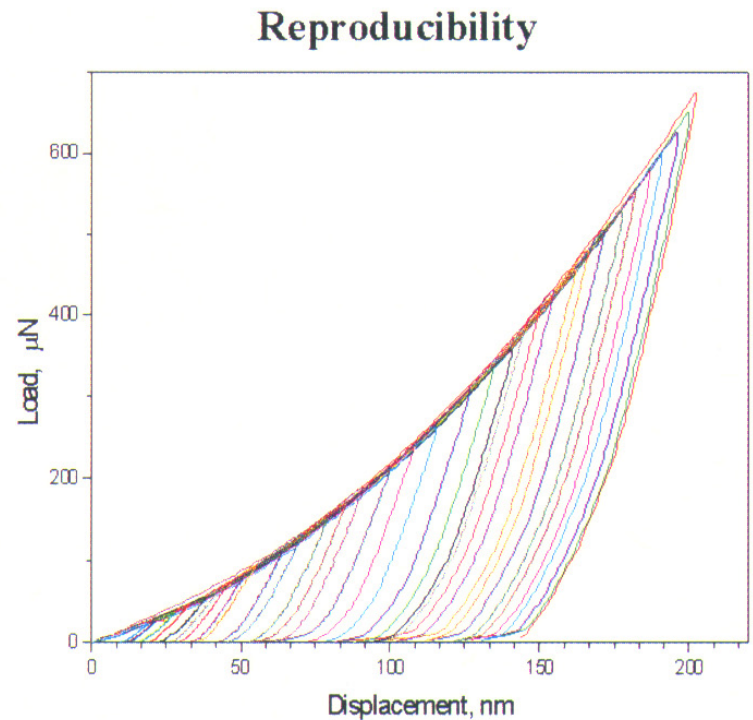
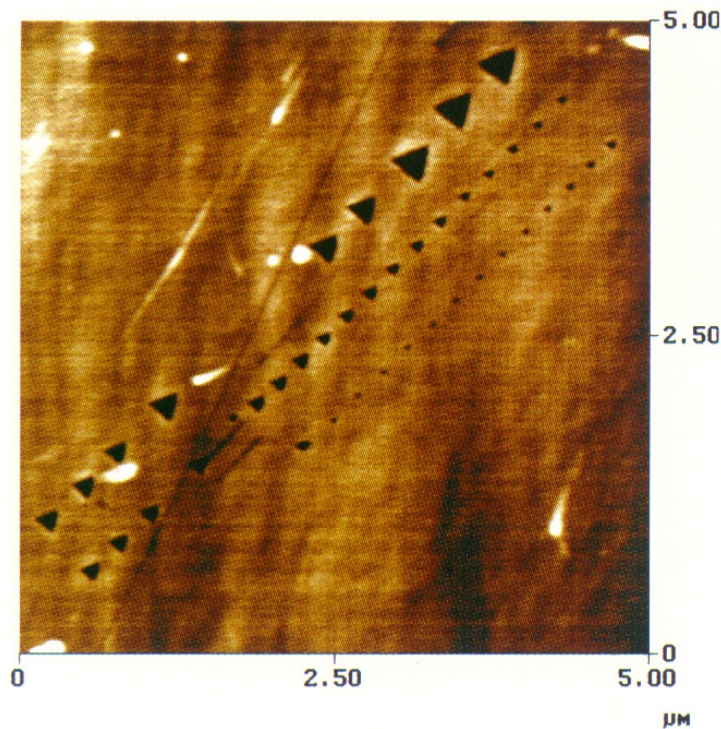
Advanced graphical user interface shows titin muscle molecule force curve.

Atomic Image of Si(111)-(7×7) Taken with AFM



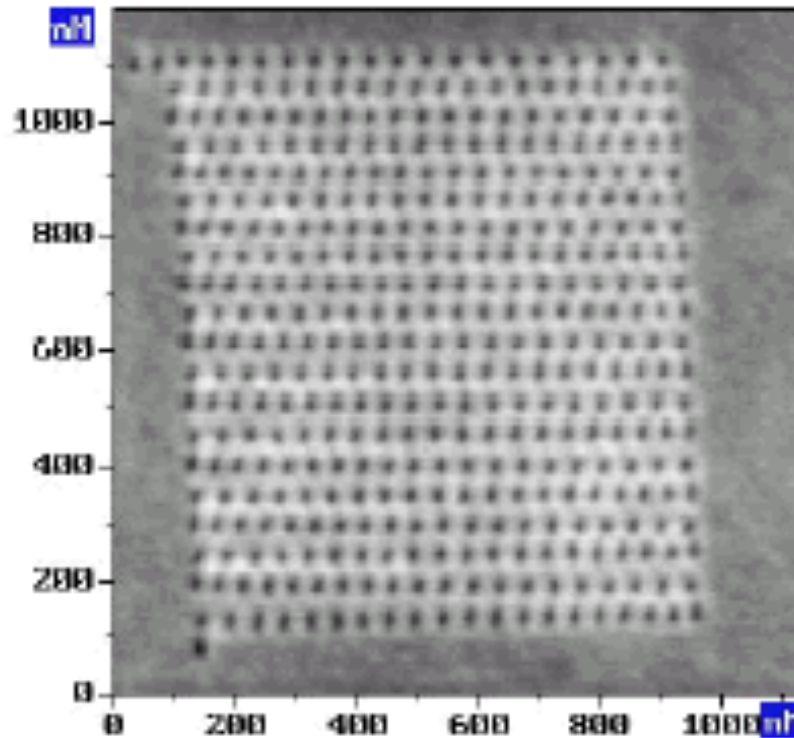
F.J. Giessibl *et al.*, Science 289, 422 (2000)

Measurement of Mechanical Properties

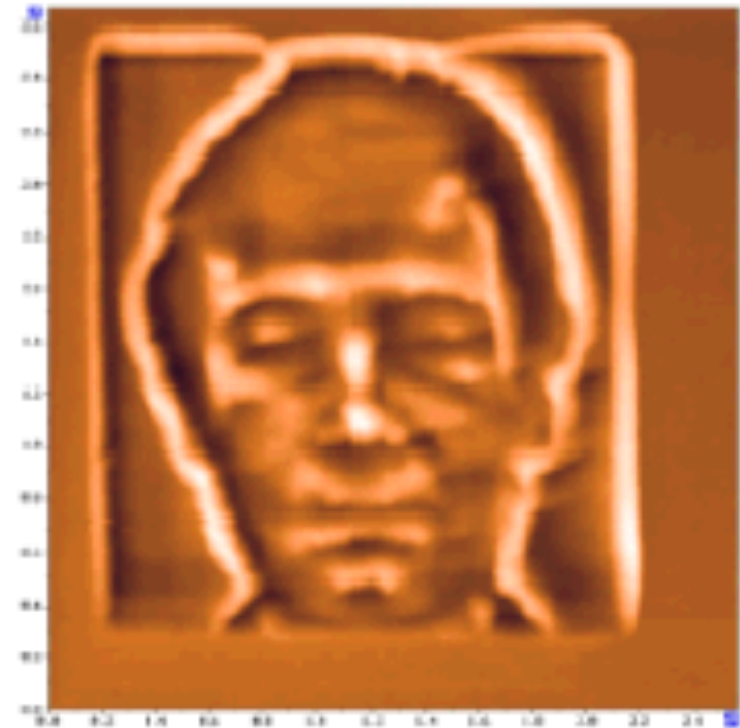


1. The load-displacement curves provide a “mechanical fingerprint” of material’s response to deformation, from which parameters such as hardness and young’s modulus of elasticity can be determined.
2. In measuring the mechanical properties of thin coated system, the size of contact impression should be kept small relative to the film thickness.

Nanolithography of Tapping-Mode AFM



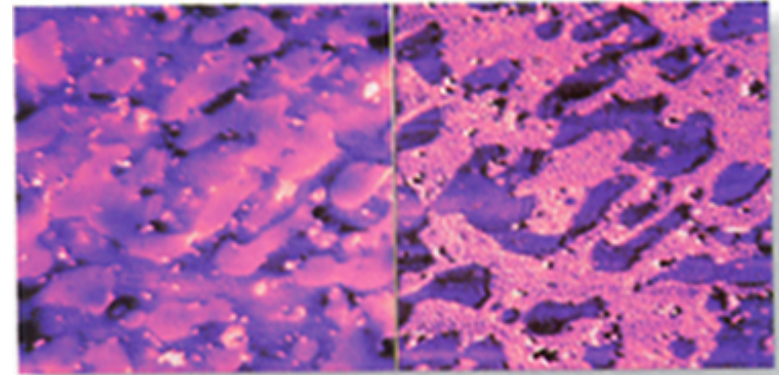
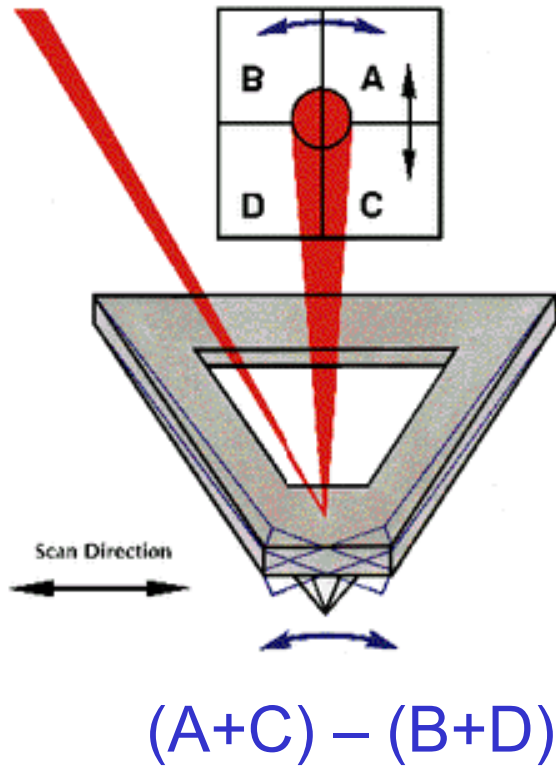
$(1.2 \mu\text{m} \times 1.2 \mu\text{m})$



$(2.5 \mu\text{m} \times 2.5 \mu\text{m})$

Image of polycarbonated film on silicon surface

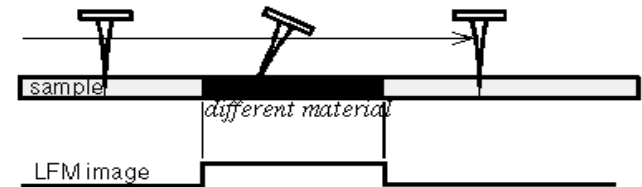
Lateral Force Microscopy



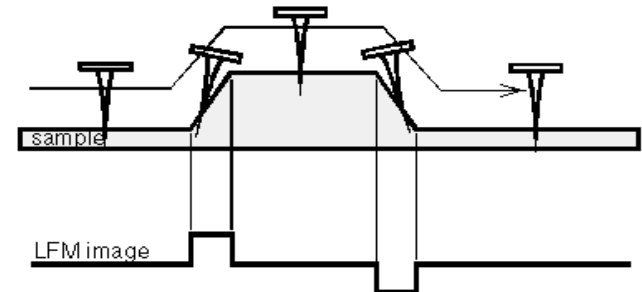
Topography

LFM

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.

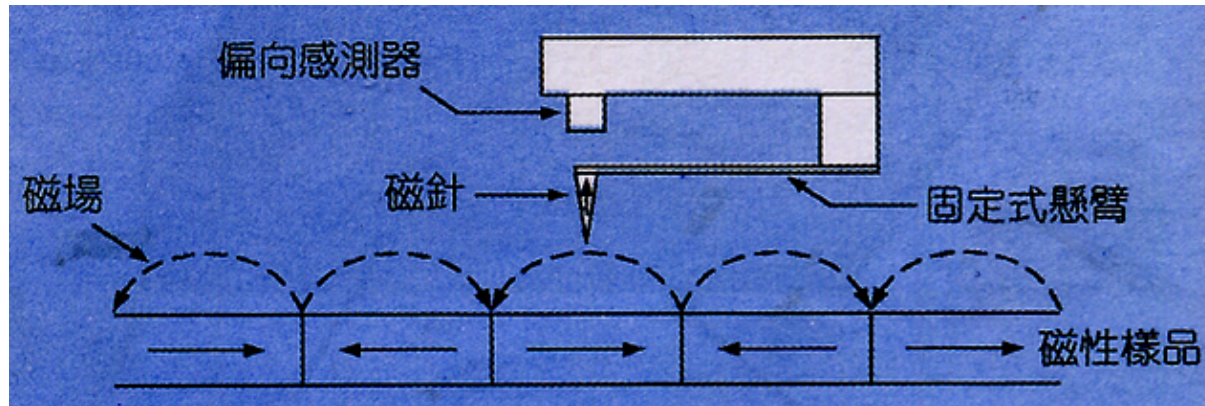


Chemical Force Microscopy



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 acid-terminated regions exhibit greater frictional force (lighter color) than the methyl-terminated regions. When a methyl-terminated 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)



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

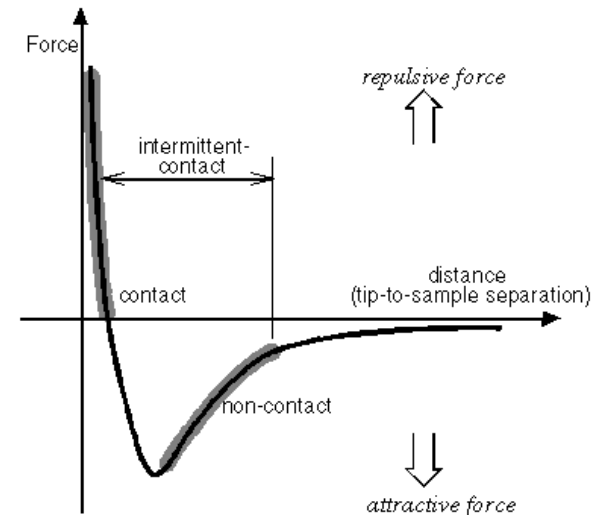
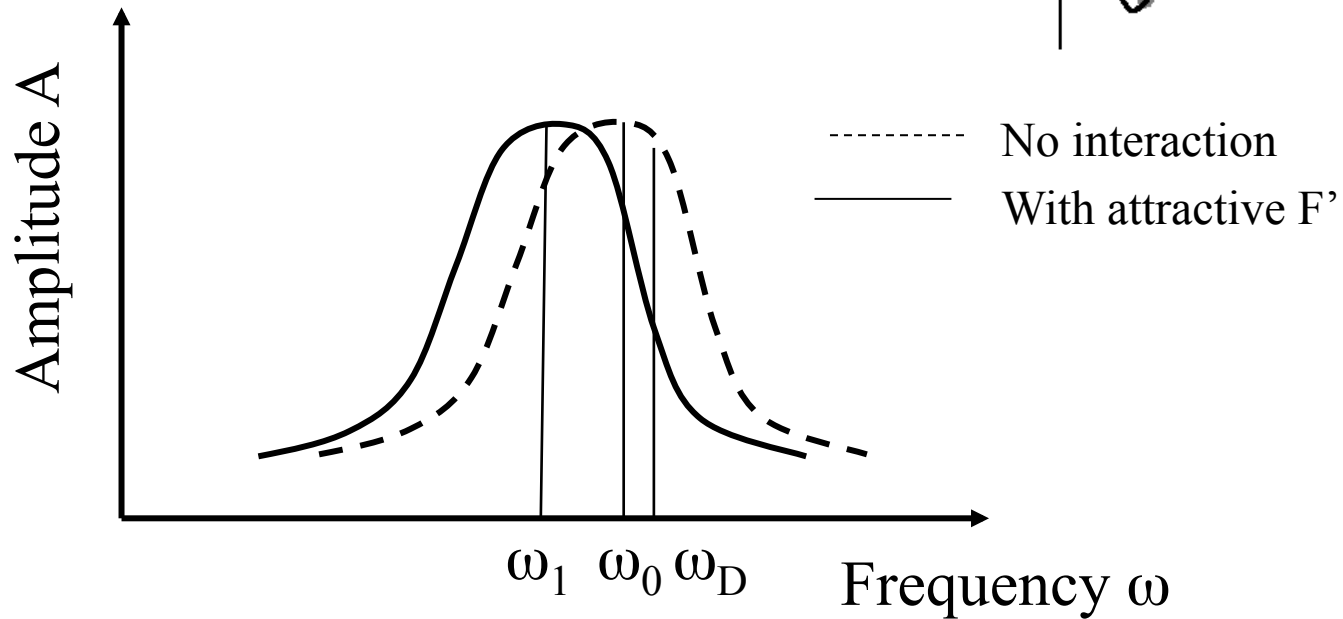
Tips: silicon probes are magnetically sensitized by sputter coating with a ferromagnetic material.

Resolution: 10 ~ 25 nm.

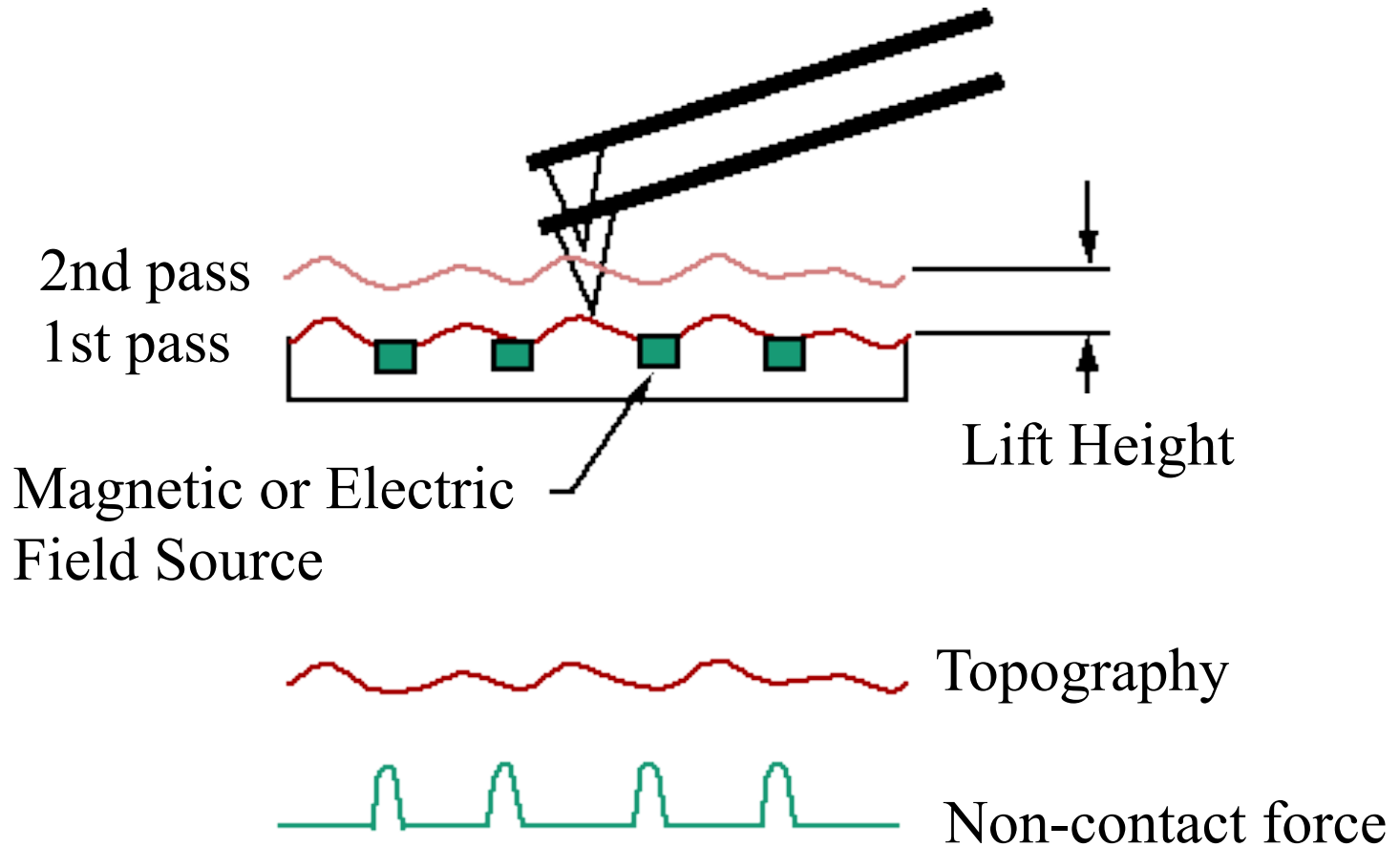
Applications: hard disks, magnetic thin film materials, micromagnetism.

AC imaging mode

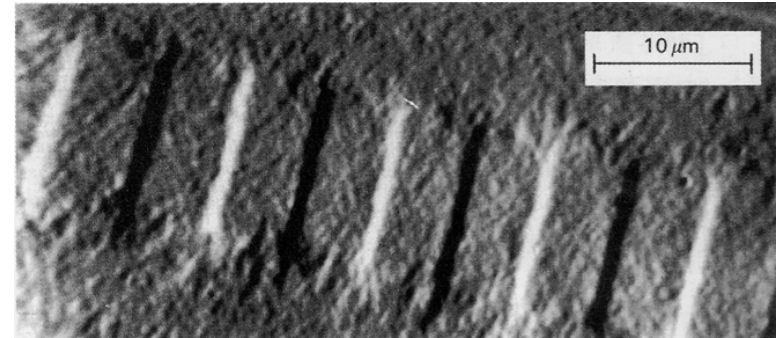
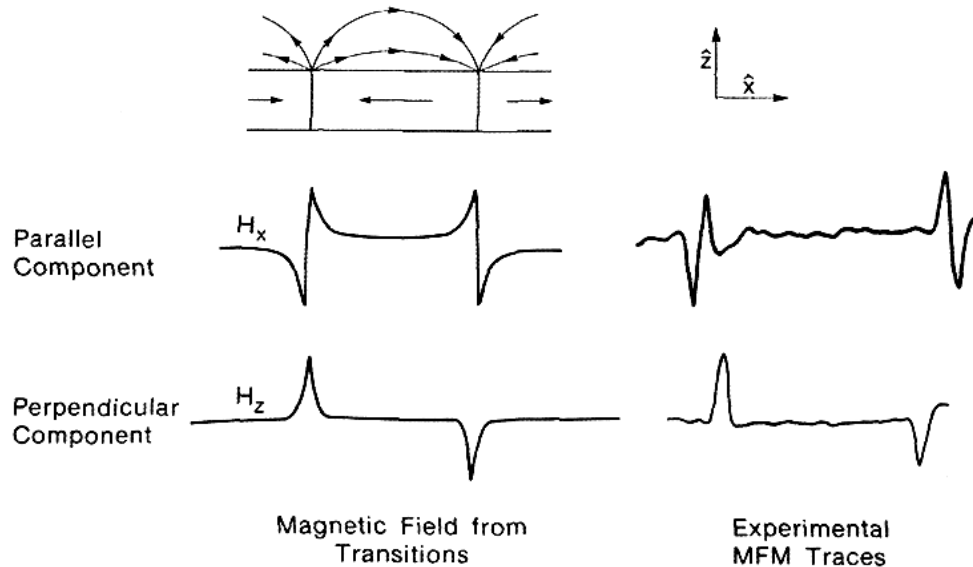
$$\omega_1 = \omega_0 (1 - F'/2k)$$



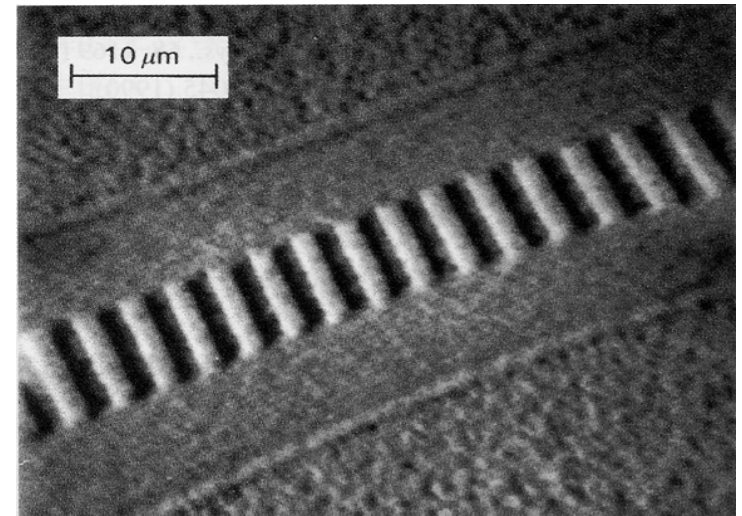
Lift mode of AFM



MFM Images



$$m^T = m_z$$



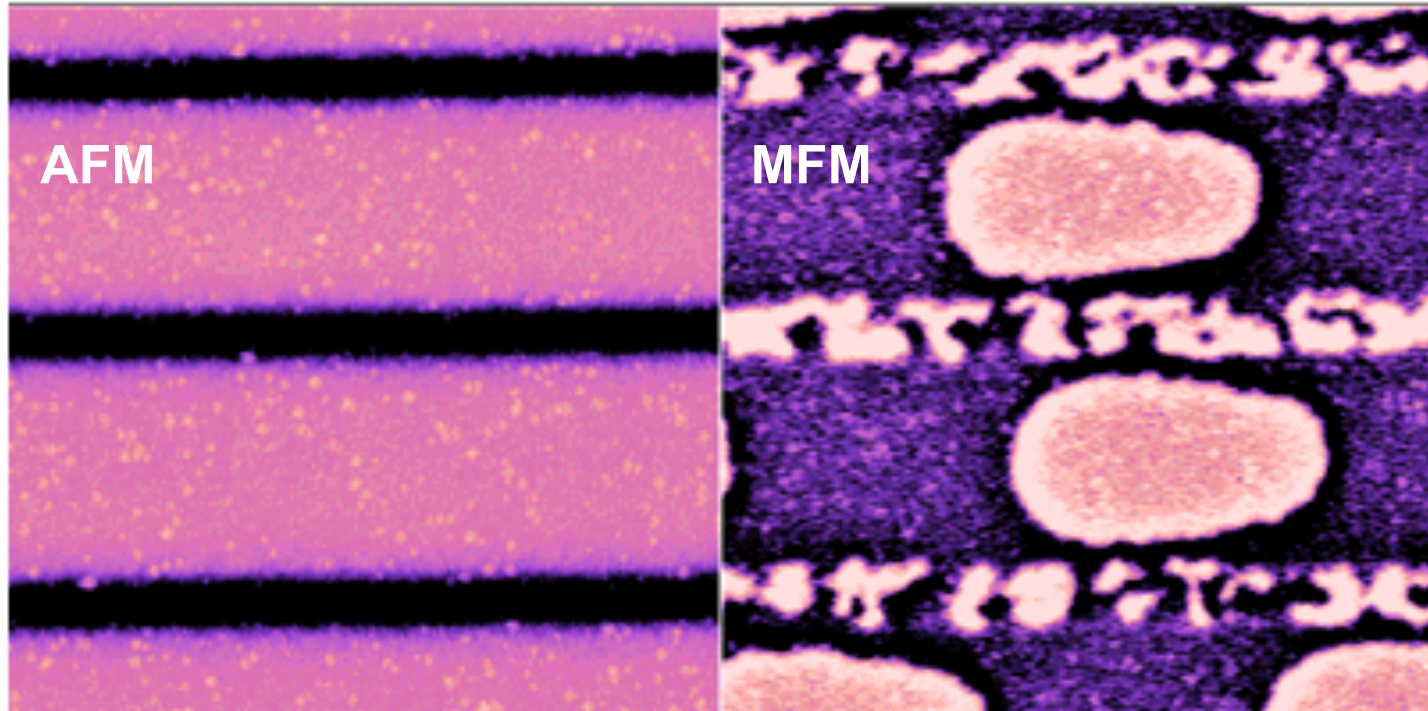
$$m^T = m_x + m_z$$

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

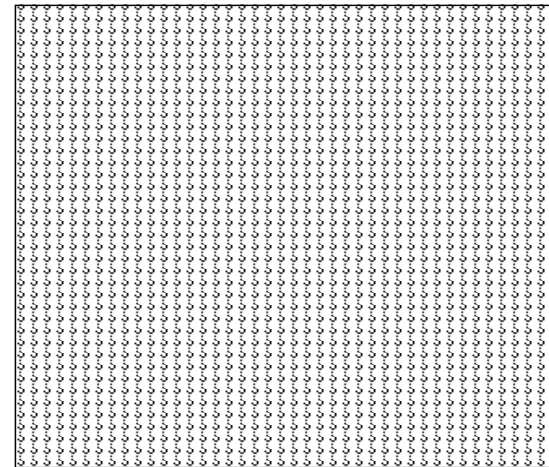
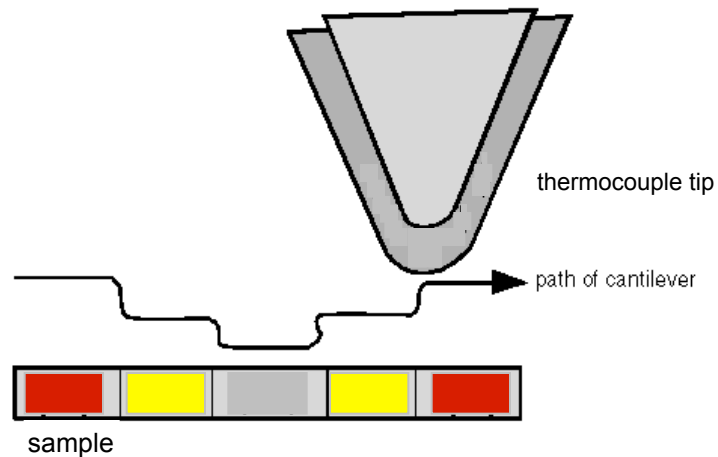
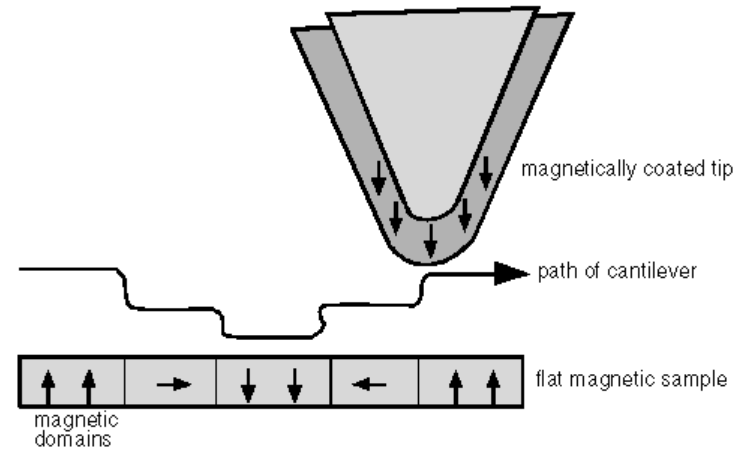
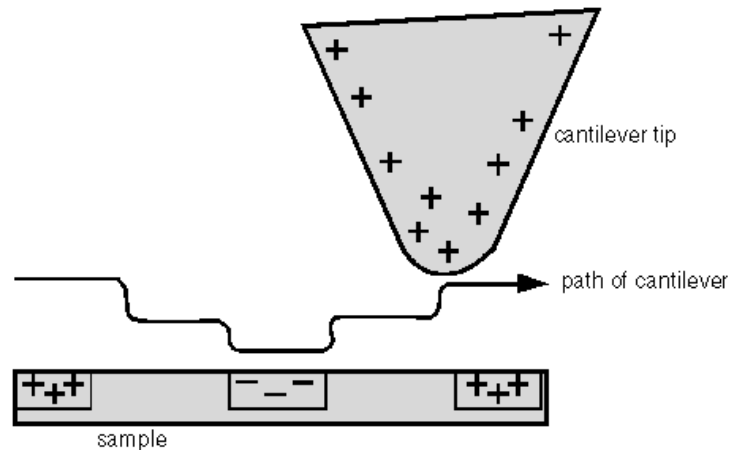
$$F_z = m_z H_z$$



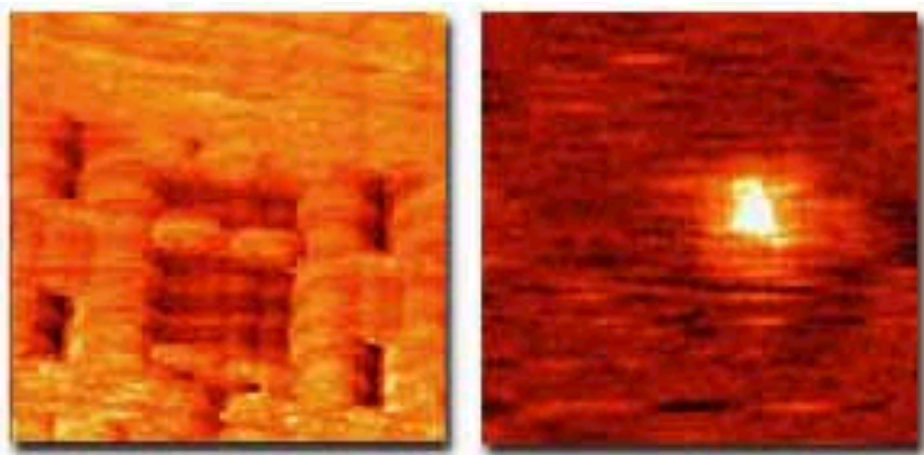
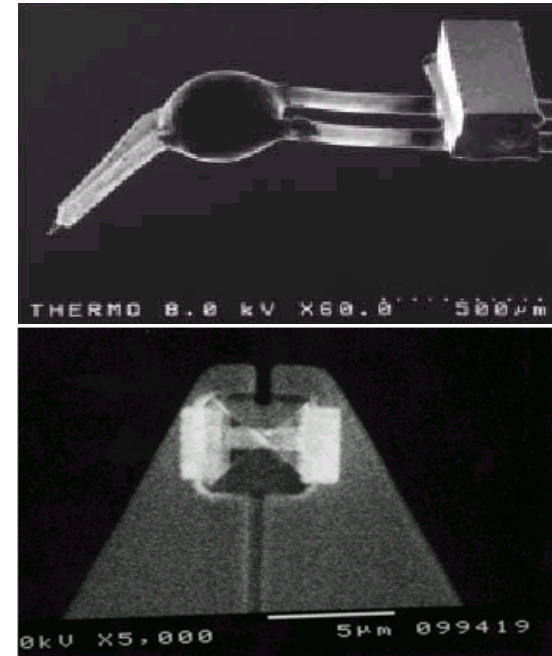
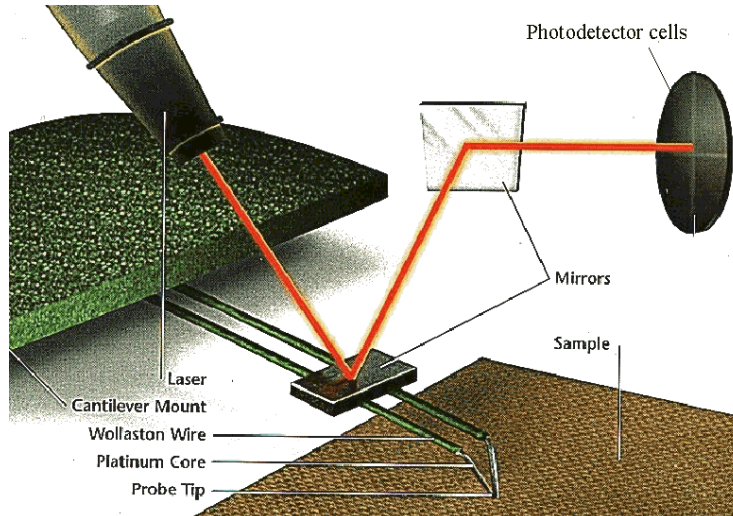
Bits (50 nm) on a magneto-optical disk

Scan area (5 μ m $\times$ 5 μ m)

Probes of various functions

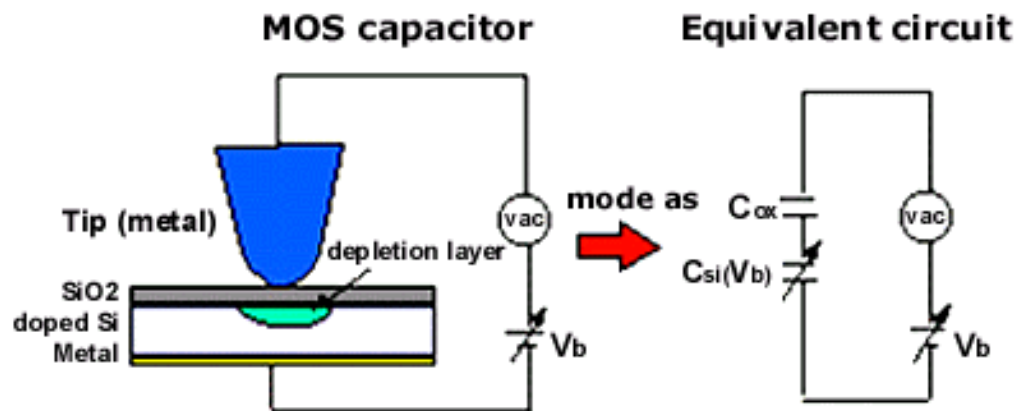
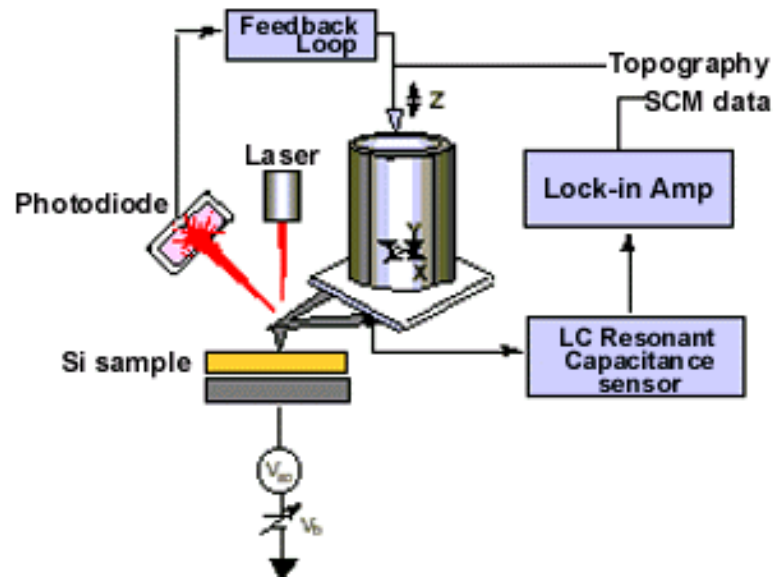


Scanning Thermal Microscopy (SThM)

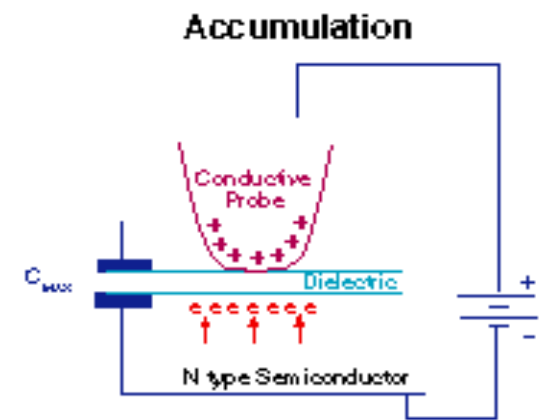
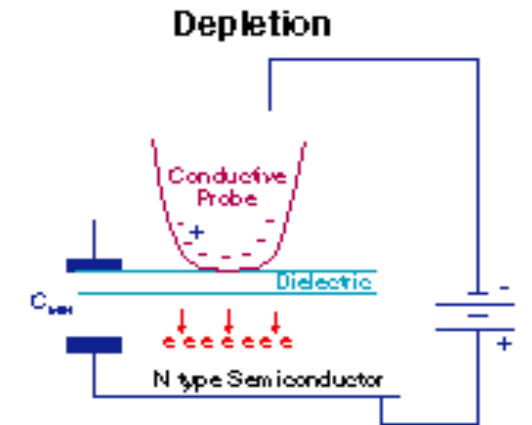
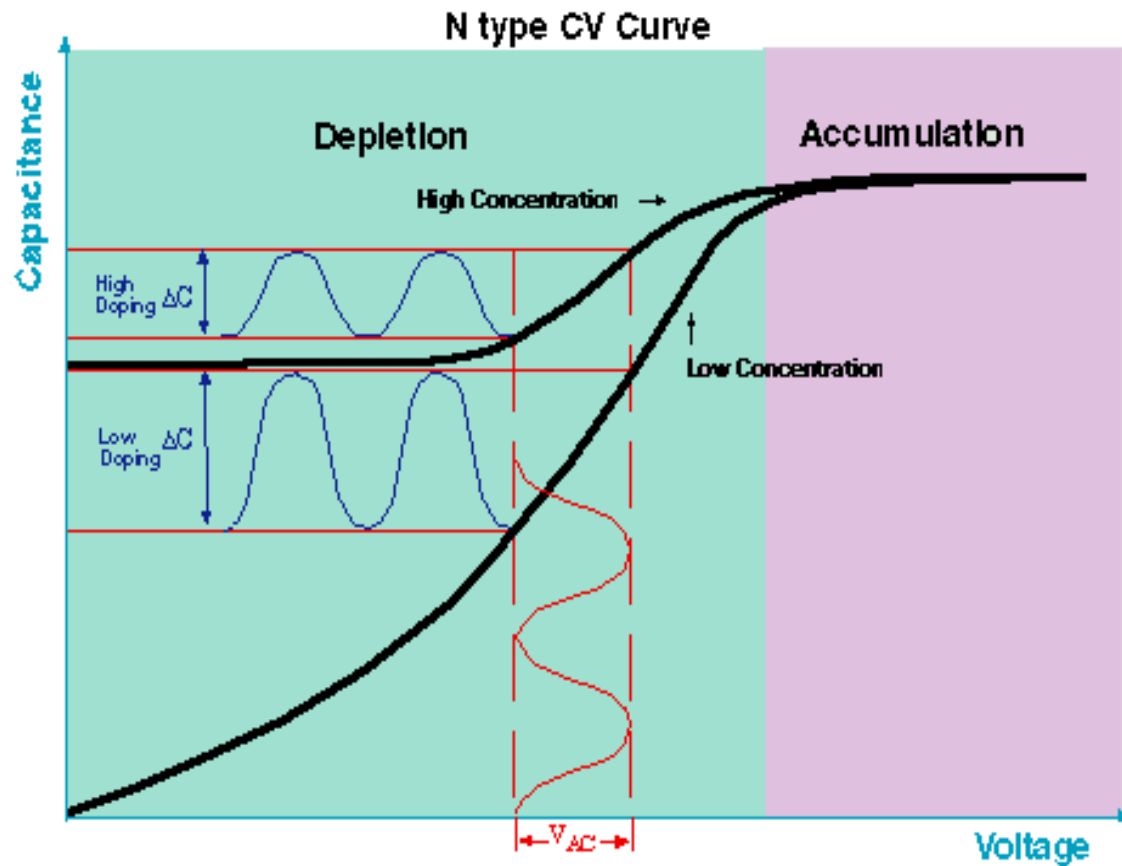


Scanning Capacitance Microscopy (SCM)

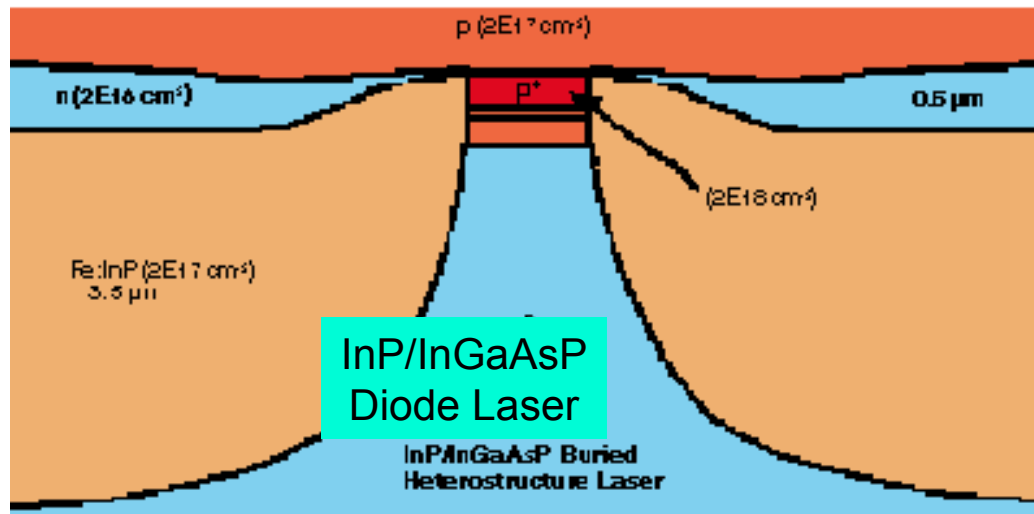
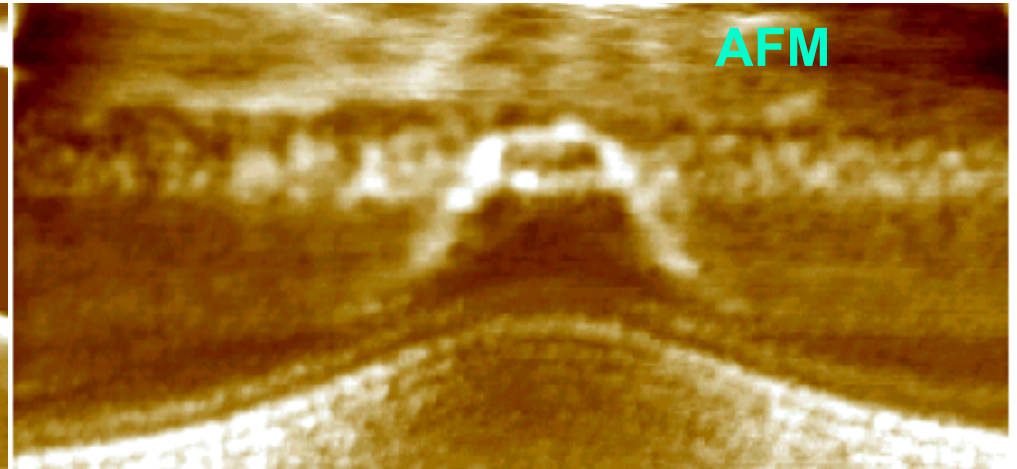
Operational principle of the SCM



SCM CV Curve

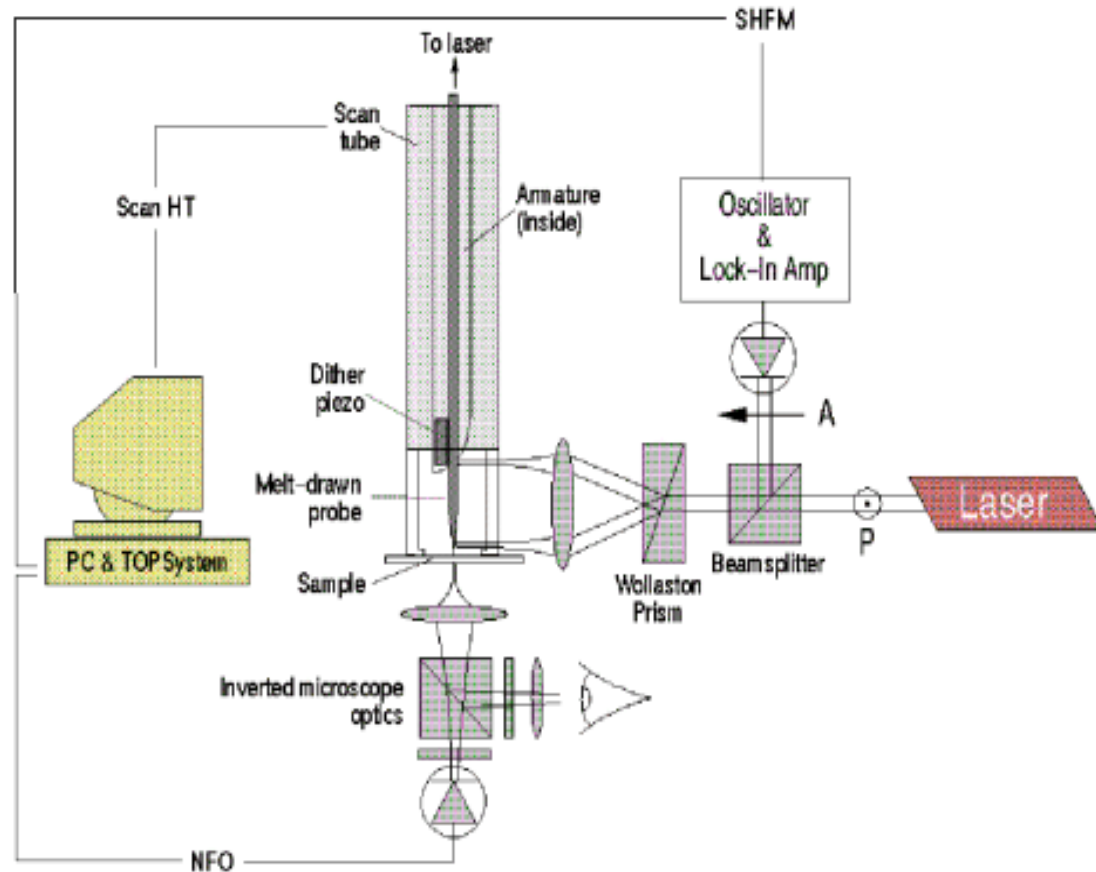


Scanning Capacitance Microscopy



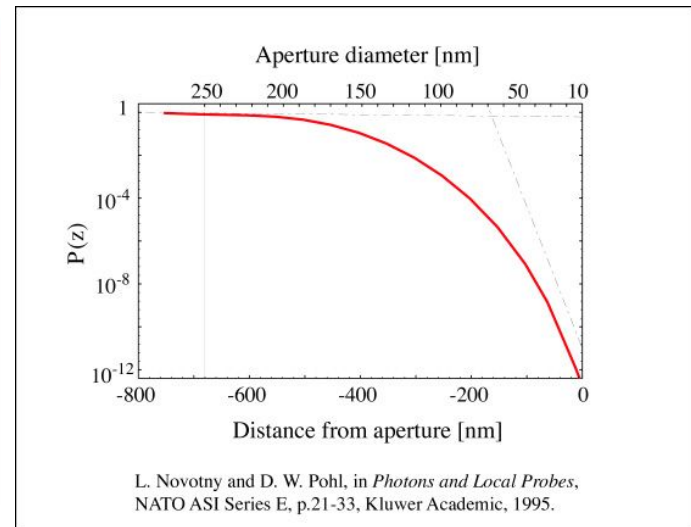
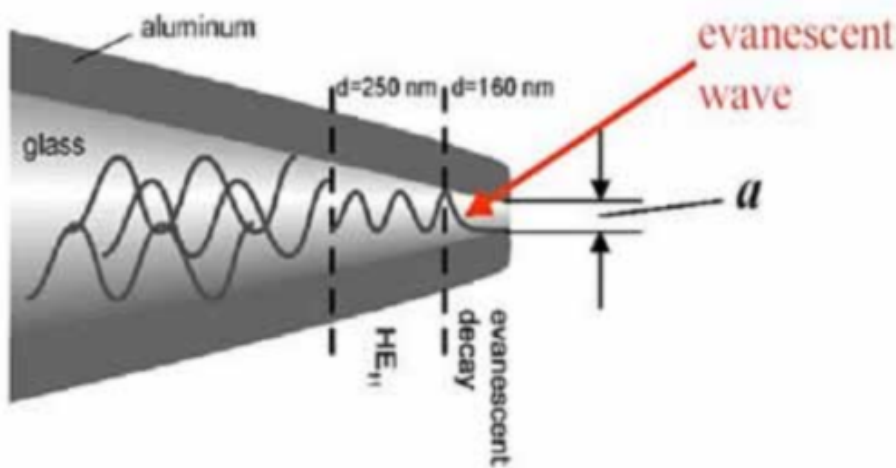
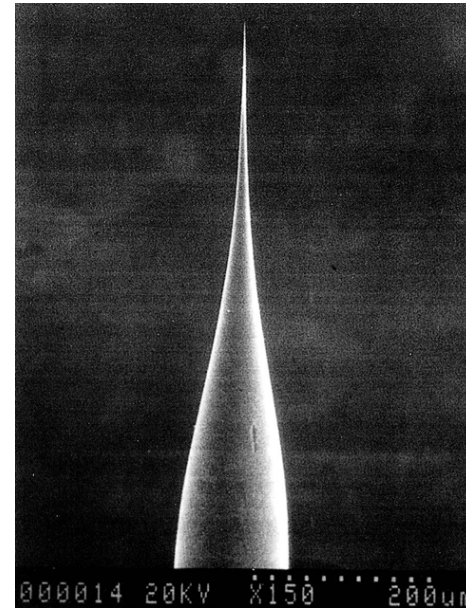
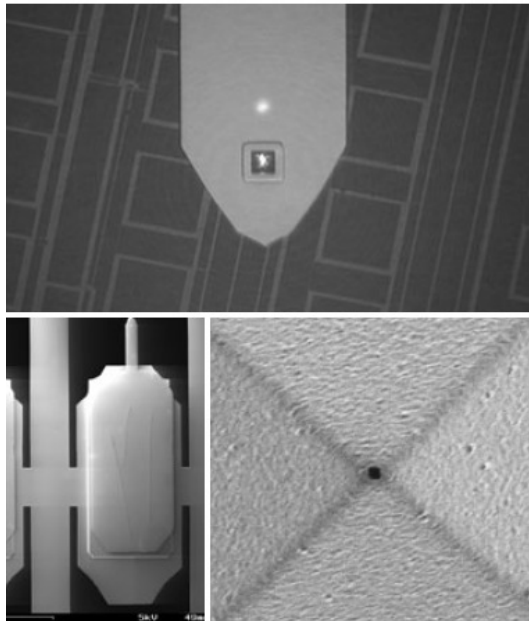
- 1. All SPMs are based on the ability to position various types of probes in very close proximity with extremely high precision to the sample under investigation.**
- 2. These probes can detect electrical current, atomic and molecular forces, electrostatic forces, or other types of interactions with 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.**

Near-field Scanning Optical Microscopy (NSOM)

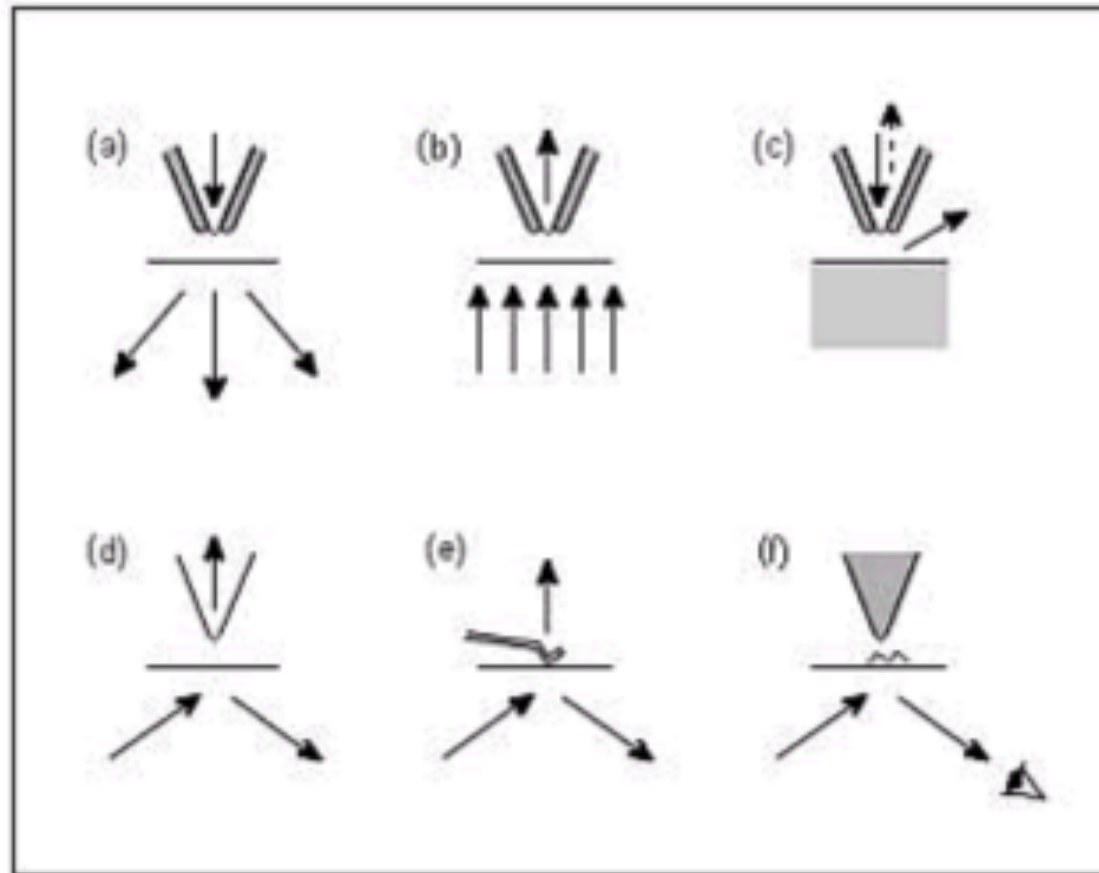


Shear force detection is used to regulate the tip/sample separation

Probes for NSOM

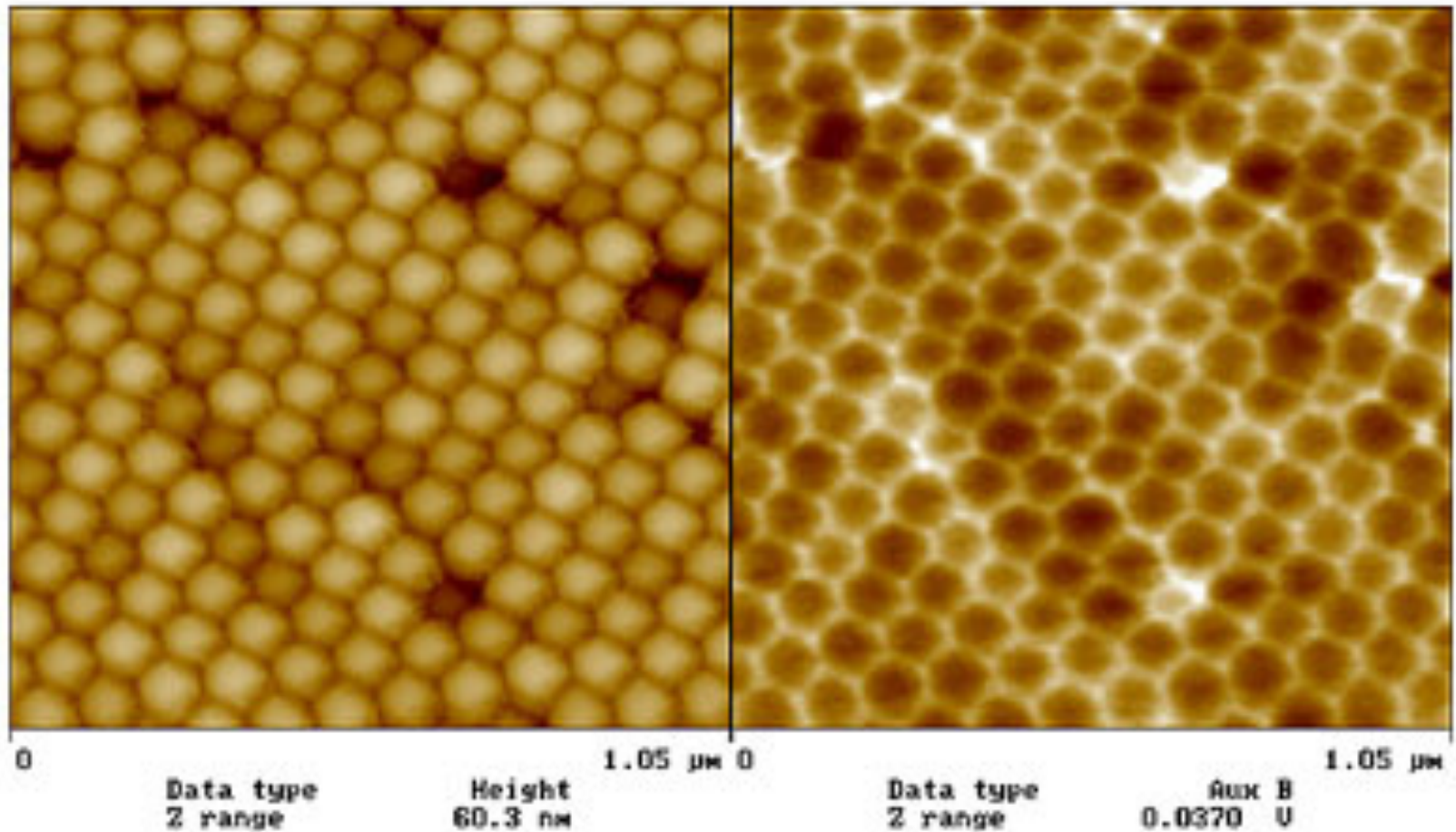


Imaging modes for NSOM



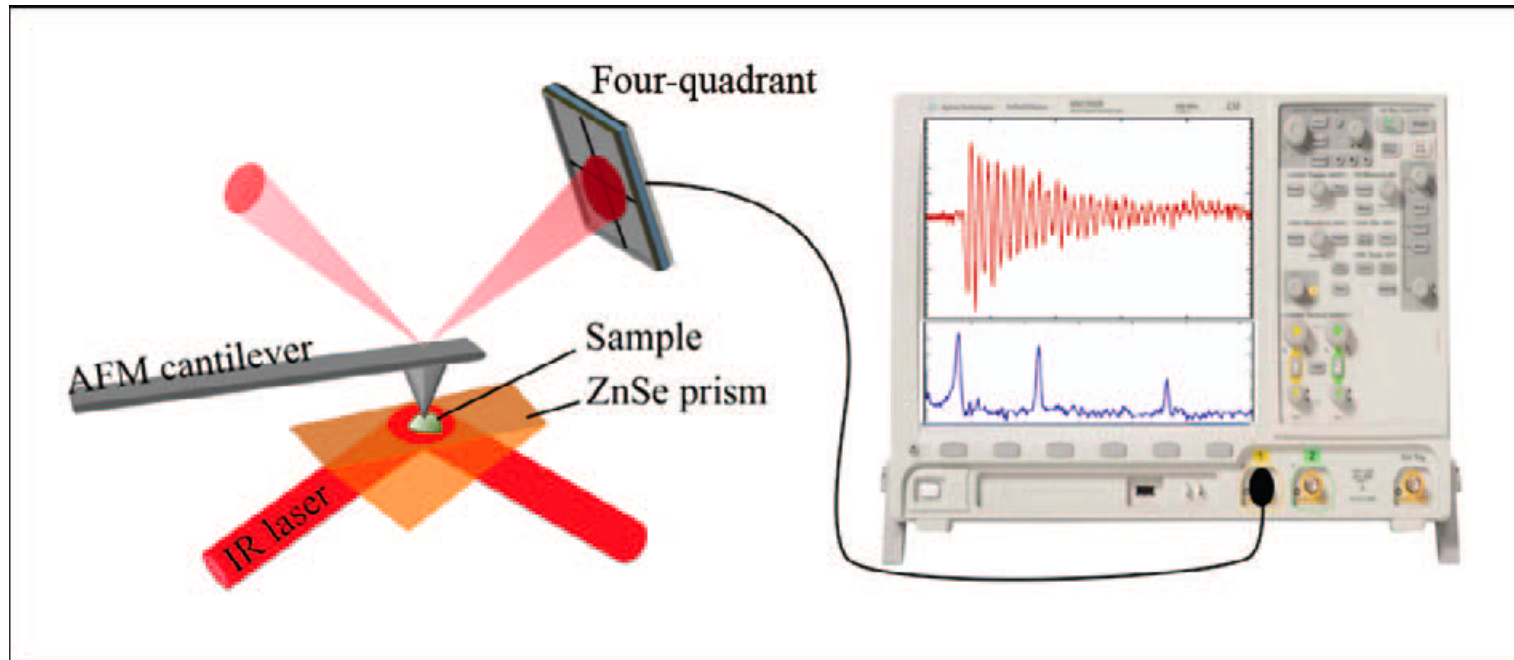
Topography

NSOM Image



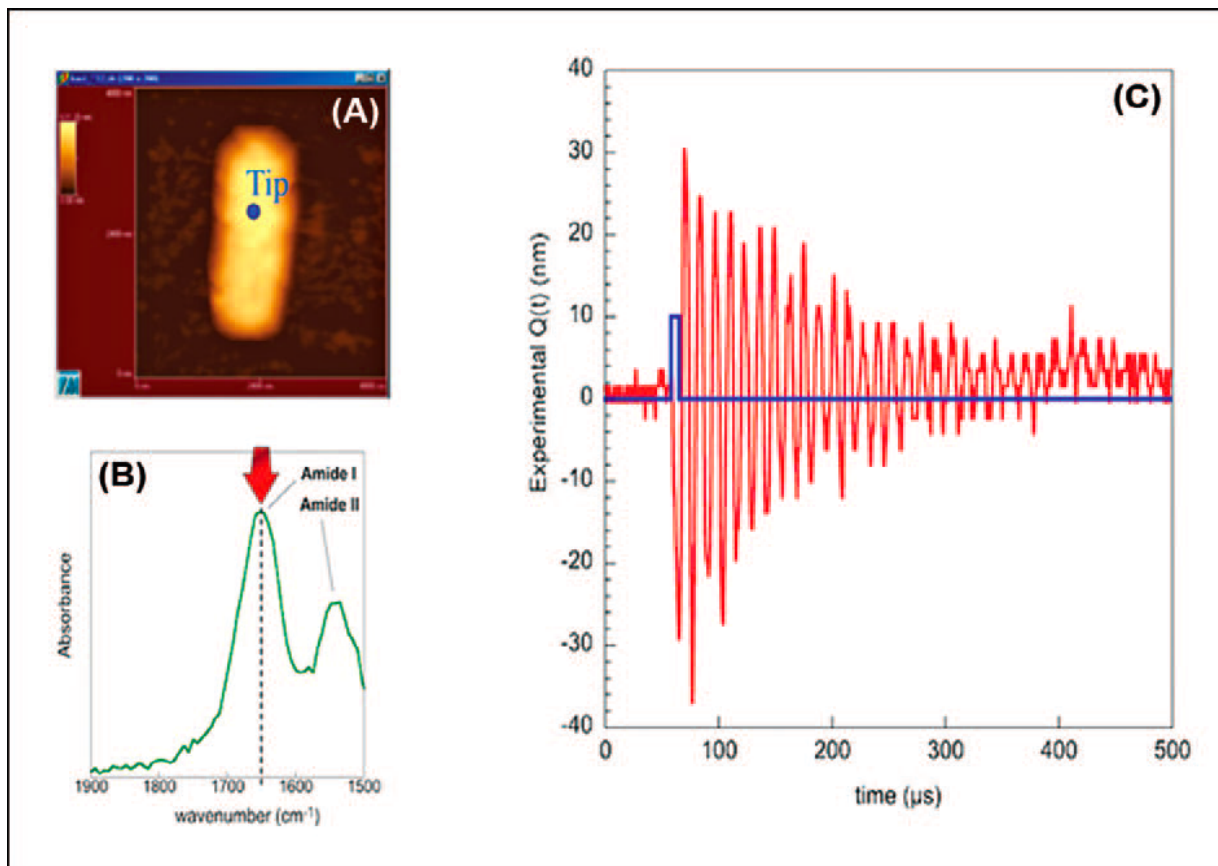
Polystyrenes of 100 nm on glass

Recent development: AFM-IR system



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

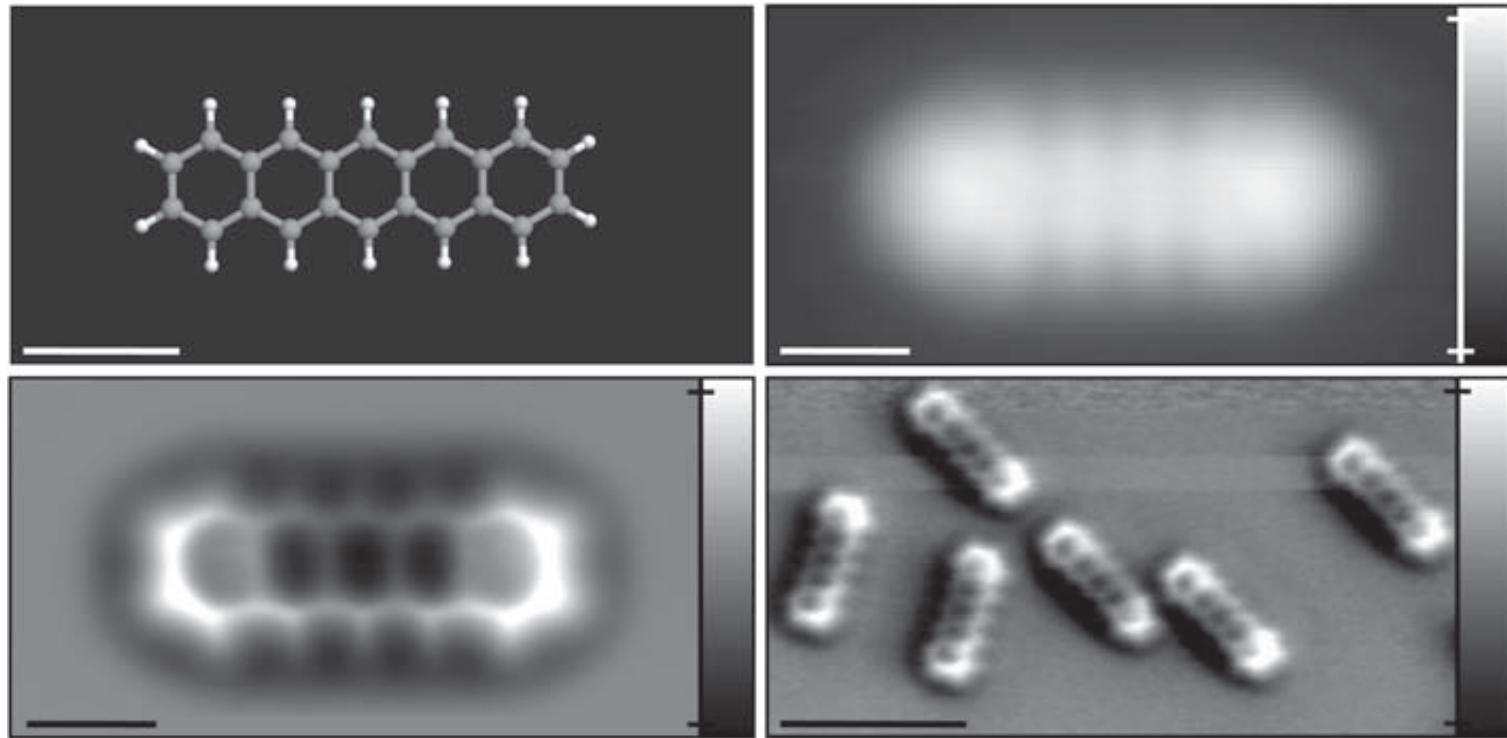
Alexandre Dazzi et al., APPLIED SPECTROSCOPY OA 66, 1365 (2012)



(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).

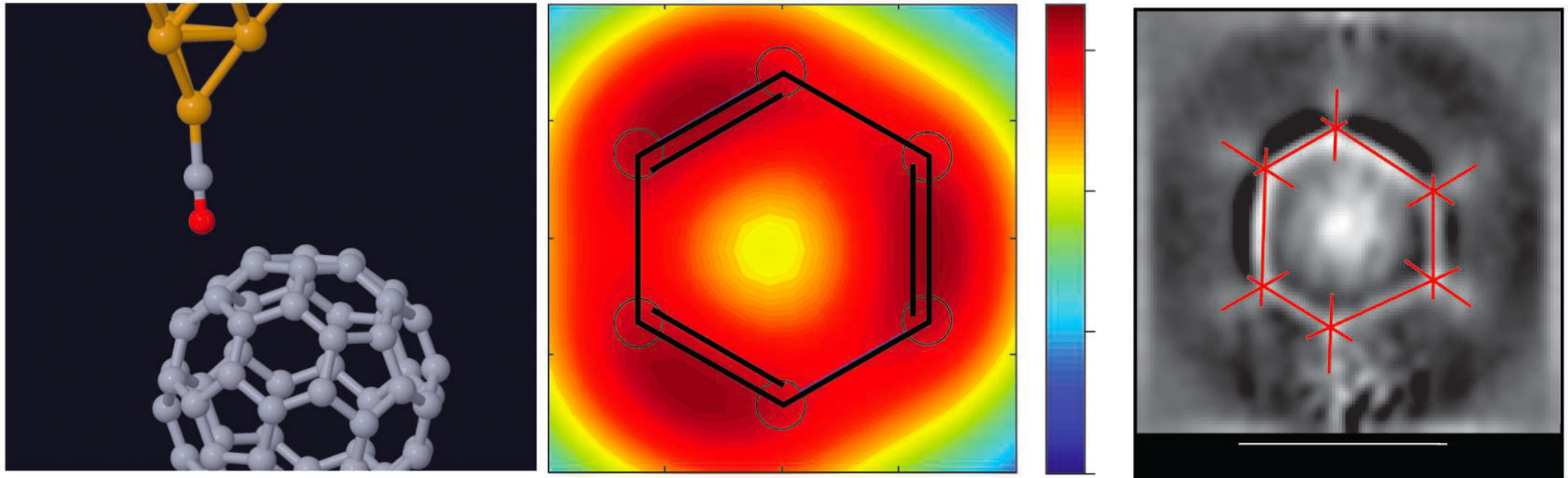
Alexandre Dazzi et al., APPLIED SPECTROSCOPY OA 66, 1365 (2012)

Recent development: High resolution AFM



STM and AFM imaging of pentacene on Cu(111). (A) Ball-and-stick model of the pentacene molecule. (B) Constant-current STM and (C and D) constant-height AFM images of pentacene acquired with a CO-modified tip

Discriminating Chemical Bonds



Forceful discrimination. Gross et al. used AFM with a CO-functionalized tip (A) to map the subtle differences in charge density (B) and bond length (C) associated with nonequivalent C-C bonds in a fullerene (C60) molecule and to correlate them with their bond order.

L. Gross et al., Science 337, 1326 (2012)

Recent development: High speed AFM

Why High Speed AFM?

- Most dynamic biological processes occur within milliseconds time scale or less.

Example:

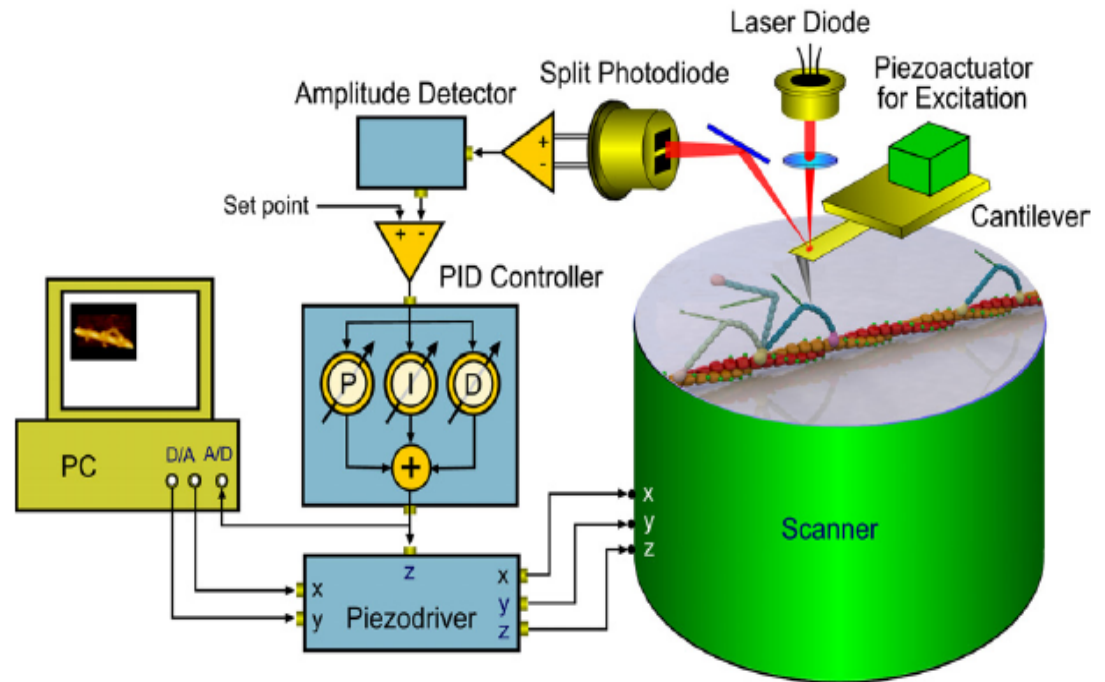
1. Photocycle of Bacteriorhodopsin (*Nature Nanotechnology* **5**, 208-212 (2010))

WT: ~10 ms D96N bR mutant: ~10 s

2. F_1 -ATPase's rotary rate: 0.5 r.p.s (*Nature* 386, 299-302(1997))

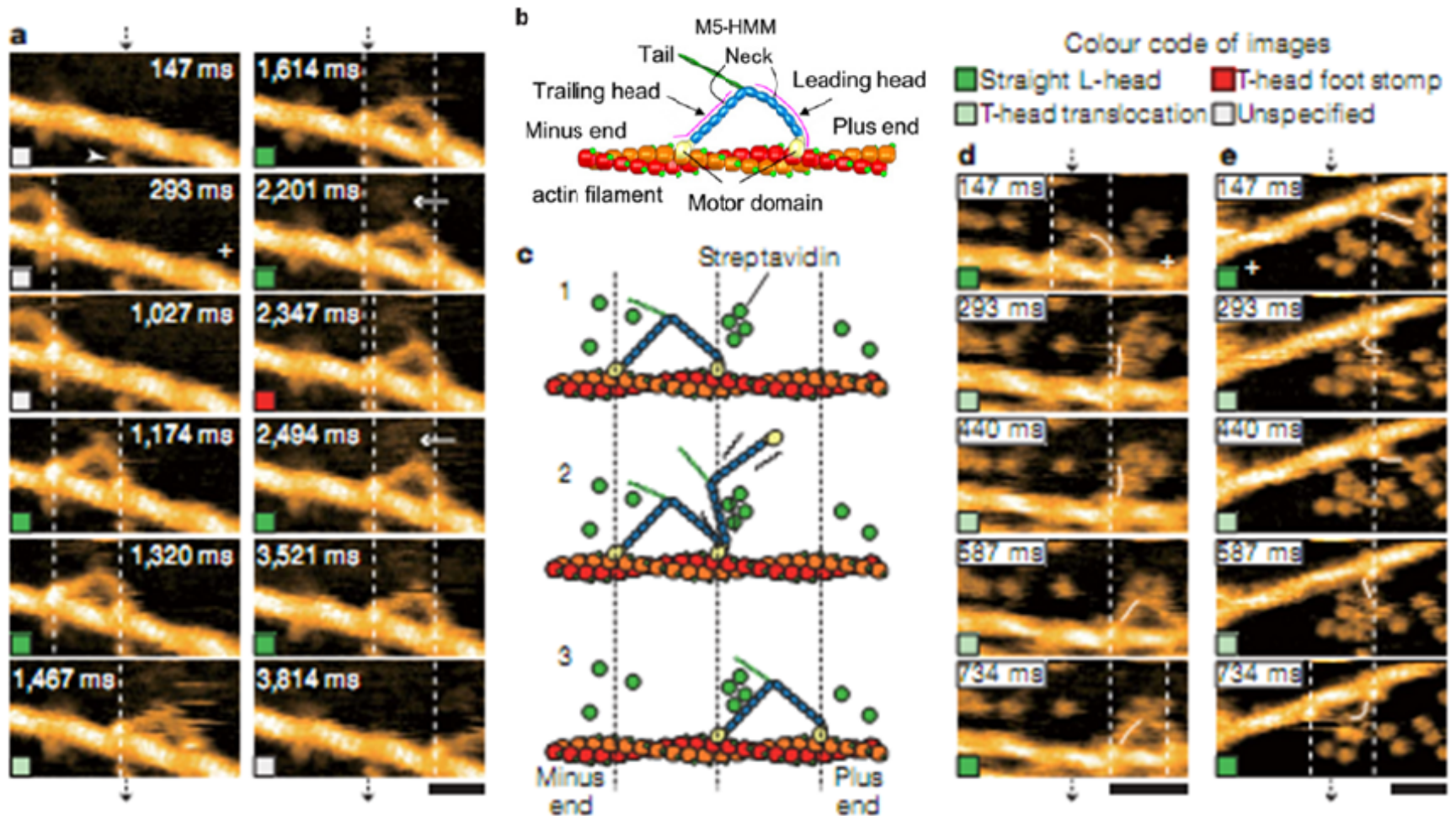
Ando's High-Speed AFM

- Small cantilever of small spring constant (150-280 pN/nm) to reduce tip forces
- Small cantilever of high resonance frequency (450-650 kHz) to decrease response time
- Effective dynamic PID control
- Low-noise sensor amplifier

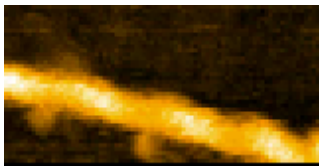


Obtain consecutive images of a 240 x 240 nm² area in as little as 80 ms

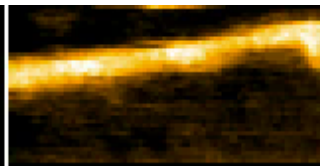
Hand-Over-Hand Movement



Noriyuki Kodera, Daisuke Yamamoto, Ryoki Ishikawa & Toshio Ando, Nature 468, 72 (2010)



a



b

c



d



myosin V motor
Erin M. Craig and Heiner Linke.
Mechanochemical model for myosin V. PNAS 2009
Basic model; no fine-tuning of parameters

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adamw@animetix.com