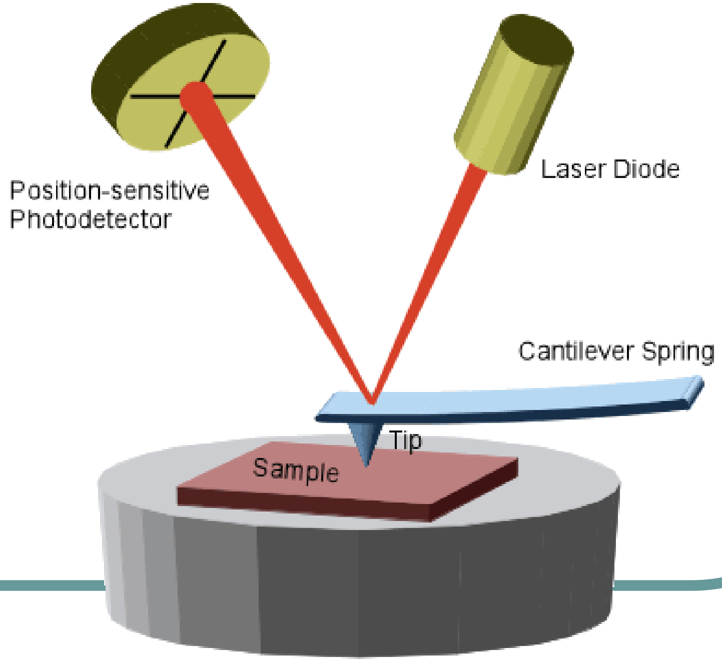
Lecture 10: Basics of Atomic Force Microscope (AFM)

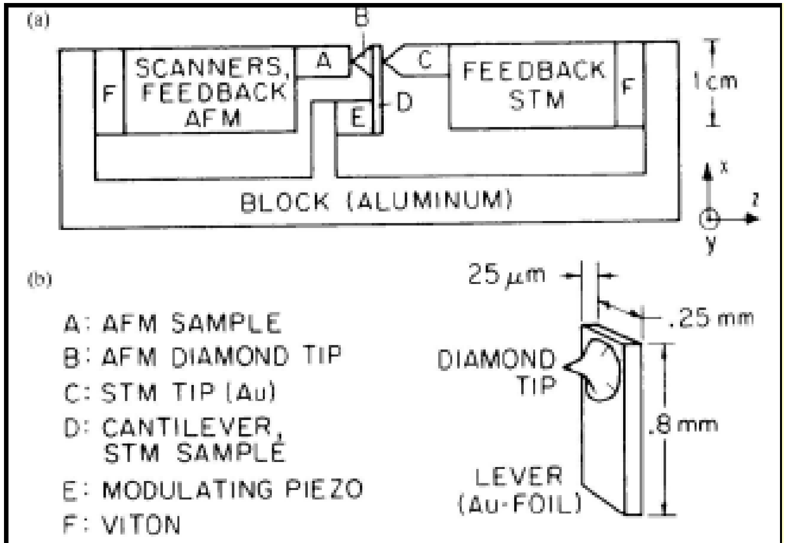
- History and background of AFM;
- Basic component of an AFM;
- Tip-Sample interactions and feedback mechanism;
- Atomic force and different scanning modes;
- AFM tips and resolution.



Brief History of AFM

- Atomic force microscopy (AFM) was developed when people tried to extend STM technique to investigate the electrically non-conductive materials, like proteins.
- In 1986, Binnig and Quate demonstrated for the first time the ideas of AFM, which used an ultra-small probe tip at the end of a cantilever (*Phys. Rev. Letters*, 1986, Vol. 56, p 930).
- In 1987, Wickramsinghe et al. developed an AFM setup with a vibrating cantilever technique (*J. Appl. Phys.* 1987, Vol. 61, p 4723), which used the light-lever mechanism.

The first AFM based on STM sensing



A: AFM SAMPLE
B: AFM DIAMOND TIP
C: STM TIP (Au)
D: CANTILEVER, STM SAMPLE
E: MODULATING PIEZO
F: VITON

Fig. 2.3. Experimental set-up for the first AFM. The cantilever is not to scale in (*a*). Its dimensions are given in (*b*). The STM and AFM piezoelectric drives face each other, sandwiching the diamond tip that is glued to the cantilever (Binnig *et al*., 1986c).

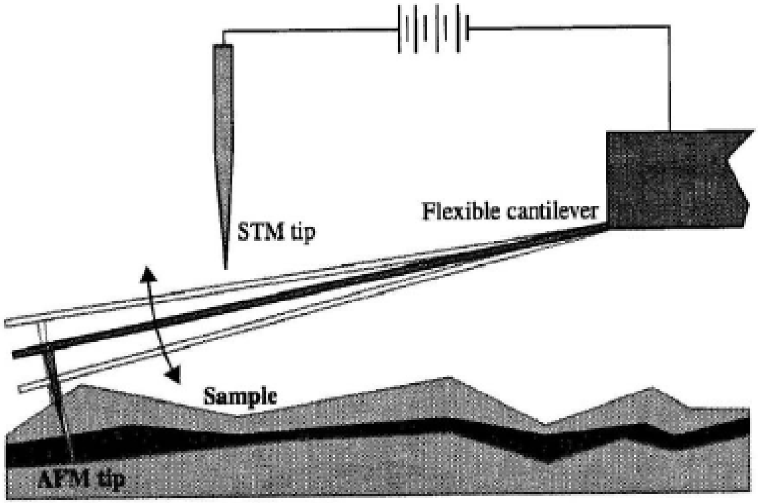


Figure 2.12. Early contact AFM which allowed imaging non-conductive samples. In this scheme, a contact AFM tip was monitored using the STM tip directly above it.

Phys. Rev. Letters, 1986, Vol. 56, p 930

STM based AFM

Cantilever Deflection Measured by Tunneling current.

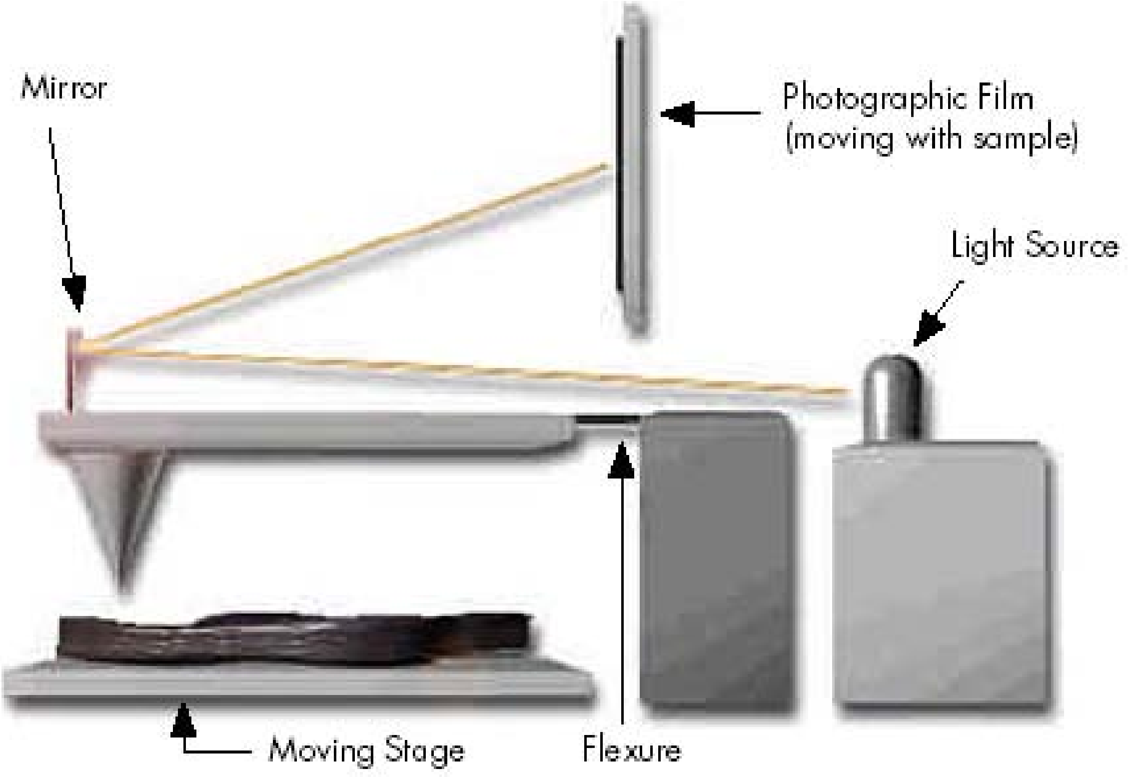
Disadvantages:

- Difficult alignment;
- Sensitivity of ~0.01 Å, but extremely sensitive to surface conditions,
- Thermal drifts, local changes in barrier height affect force measurements

But it opens the idea to develop a wide variety of SPM techniques.

A surface profiler invented in 1929 by Schmalz

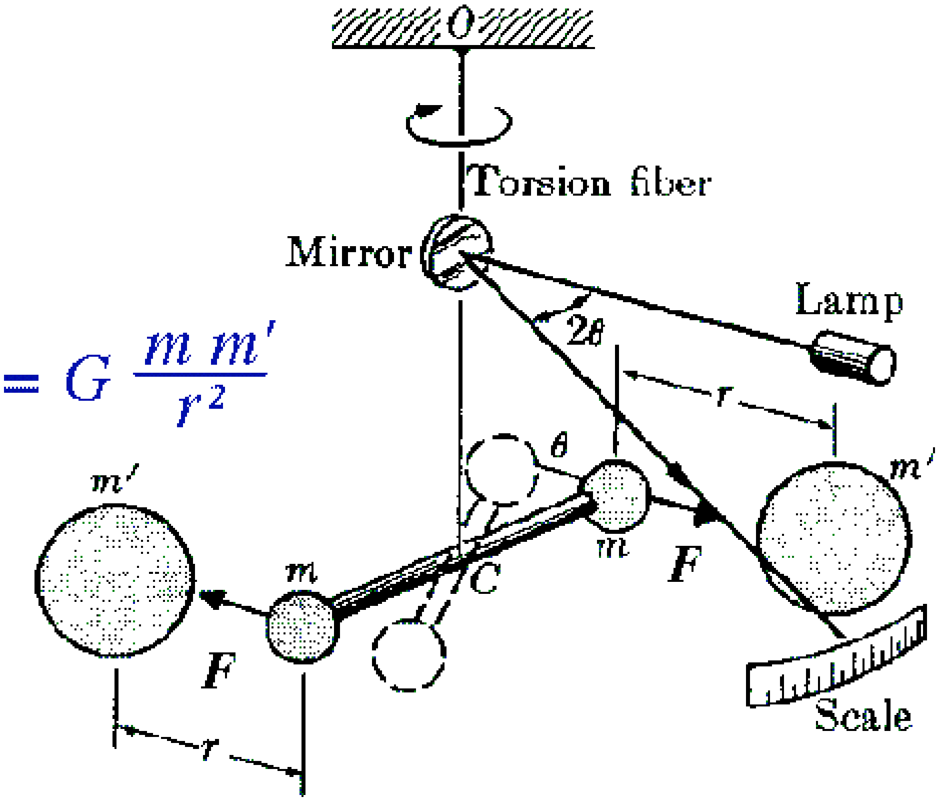
- **Light lever** --- used for the first time, to amplify the distance of movement;
- Magnification: 1000X.



The Cavendish Experiment: another example of light lever for precise spatial measurement

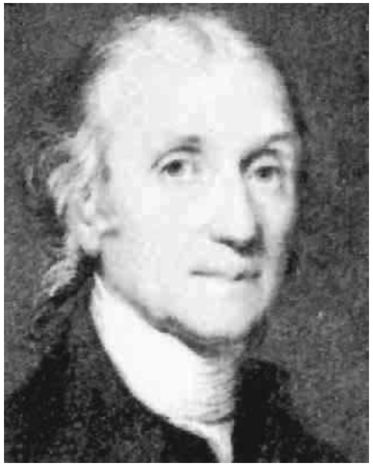
Since 1679, **Sir Isaac Newton** proposed the law of universal gravitation;

In 1798, **Sir Henry Cavendish** determined, for the first time, the constant G.

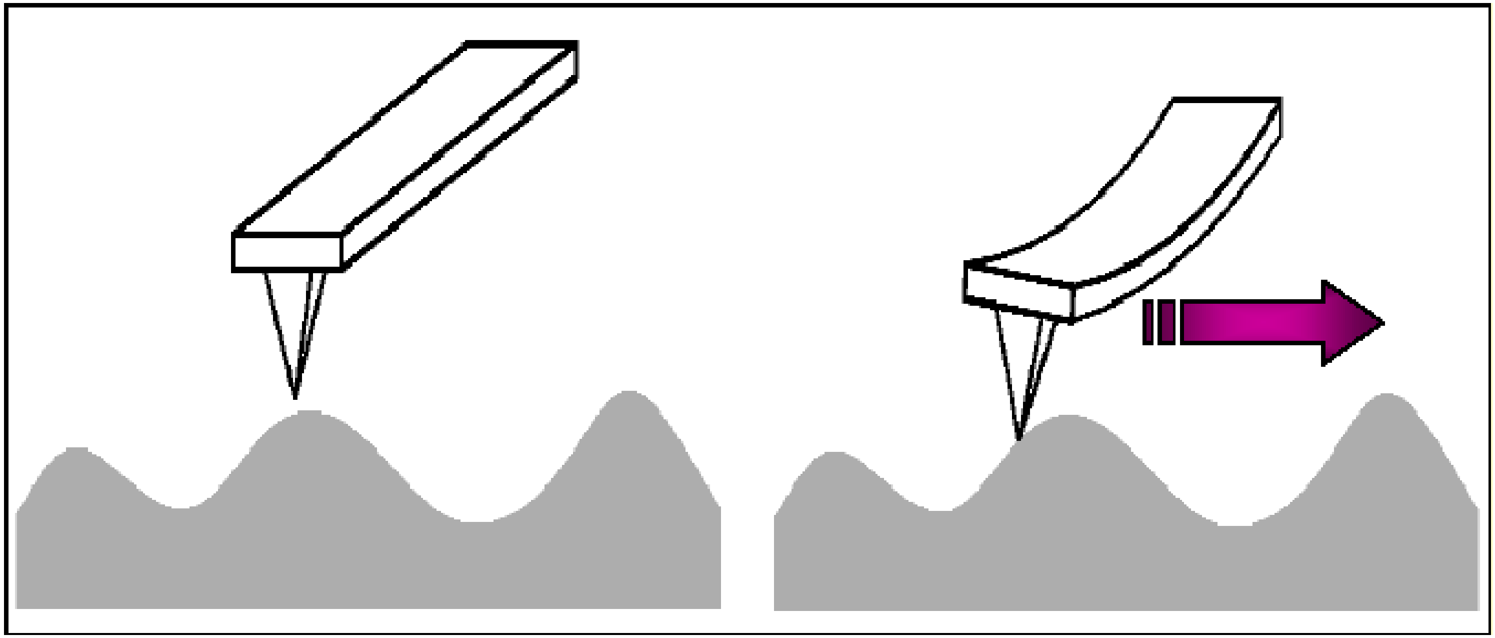


$$F = G\frac{m\,m'}{r^2}$$

$$G = 6.673 \times 10^{-11}\ \frac{\mathrm{Nm}^2}{\mathrm{kg}^2}$$



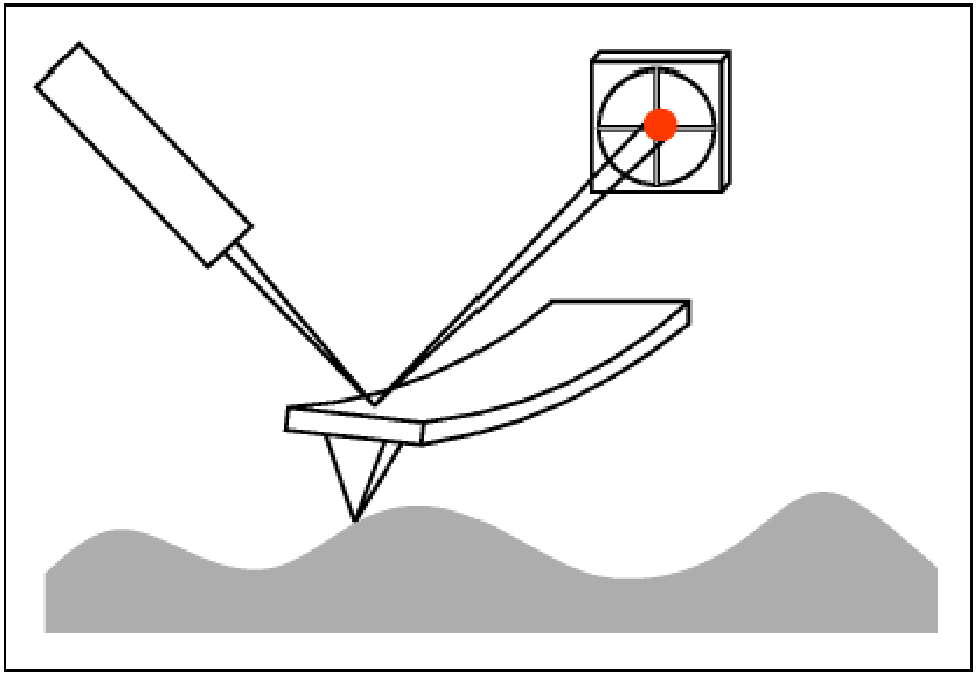
Atomic force causes bending of cantilever



Light-lever detection based on laser and photodiode array

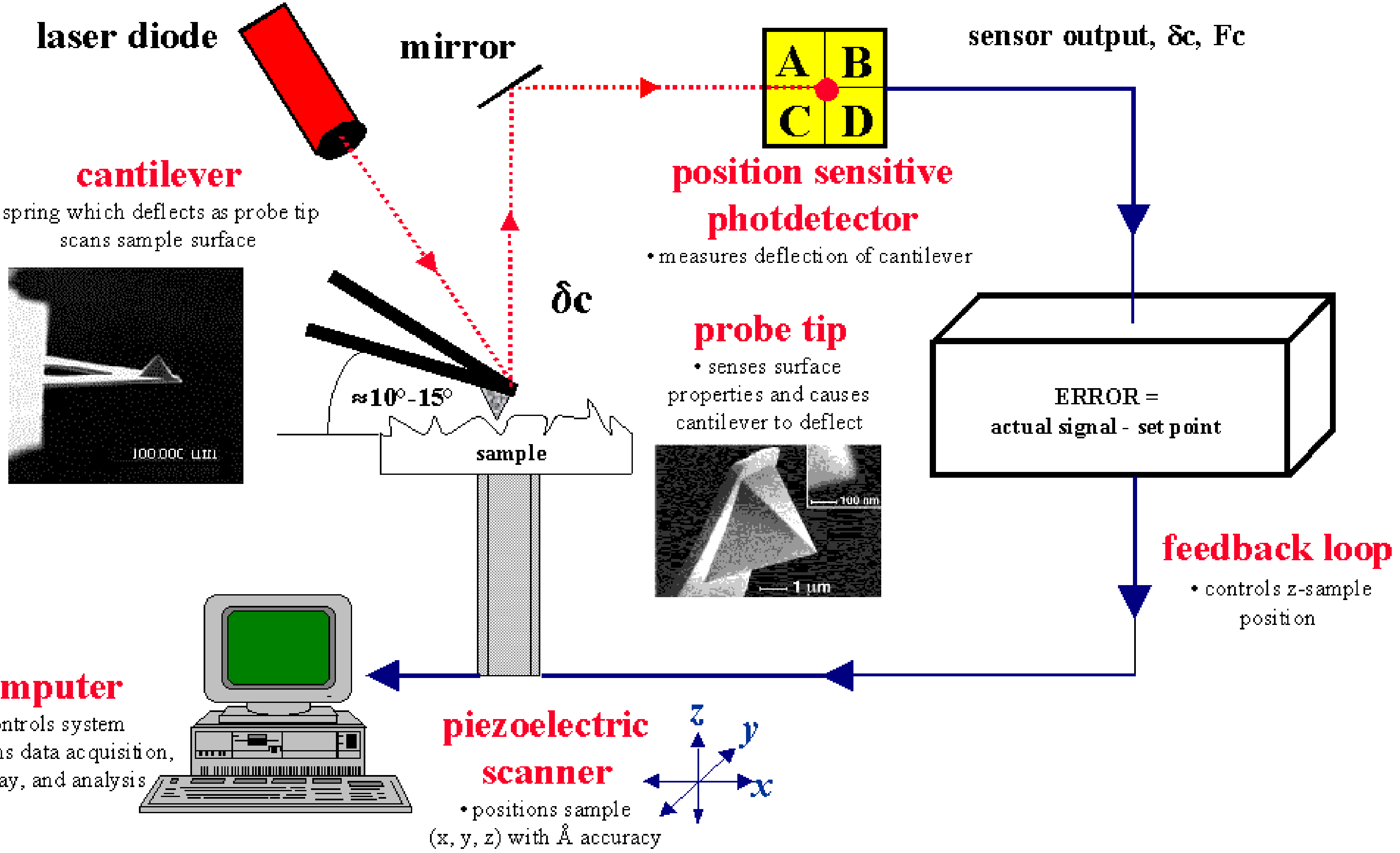
Laser: highly dense and thus excellent spatial resolution, as small as high sensitivity over the photodiode detector;

Photodiode: high sensitivity for detection at 2 dimension.



Atomic Force Microscopy (AFM) :

General Components and Their Functions



cantilever
- spring which deflects as probe tip scans sample surface

position sensitive photdetector
- measures deflection of cantilever

probe tip
- senses surface properties and causes cantilever to deflect

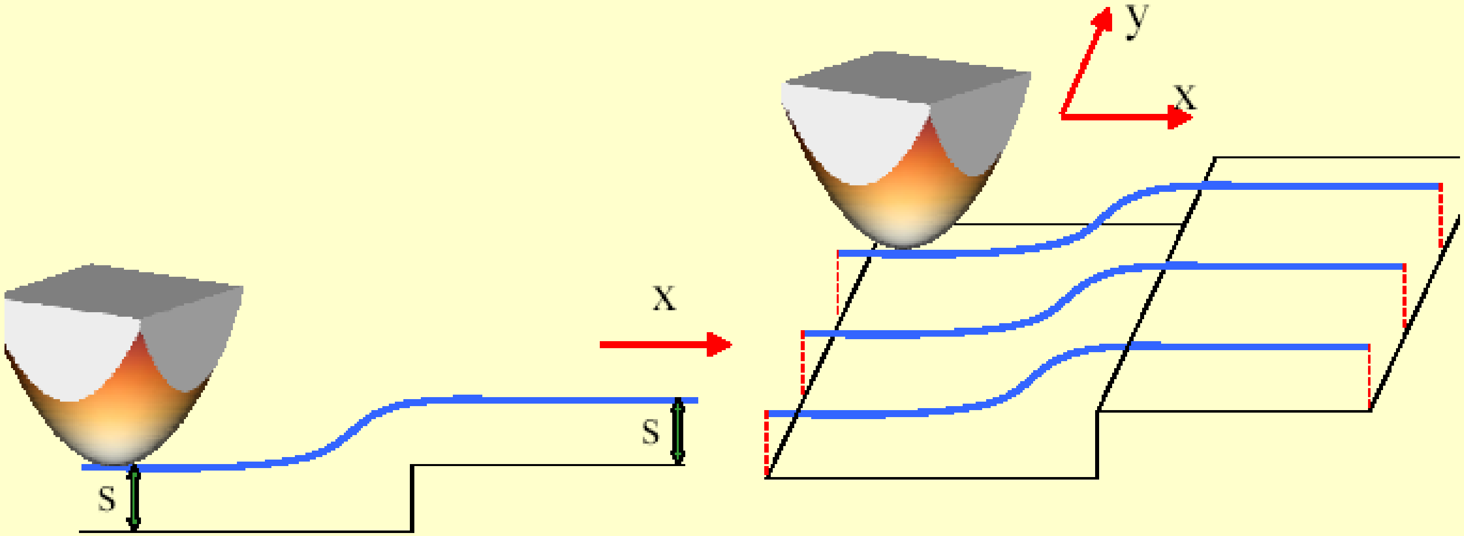
ERROR =
actual signal - set point

feedback loop
- controls z-sample position

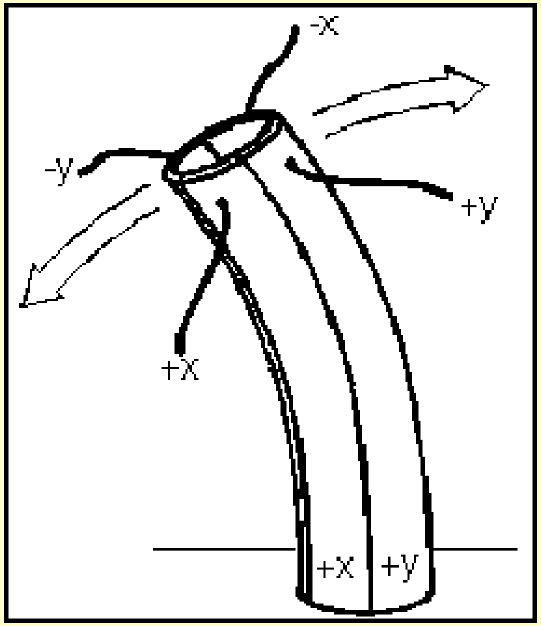
computer
- controls system
- performs data acquisition, display, and analysis

piezoelectric scanner
- positions sample (x, y, z) with Å accuracy

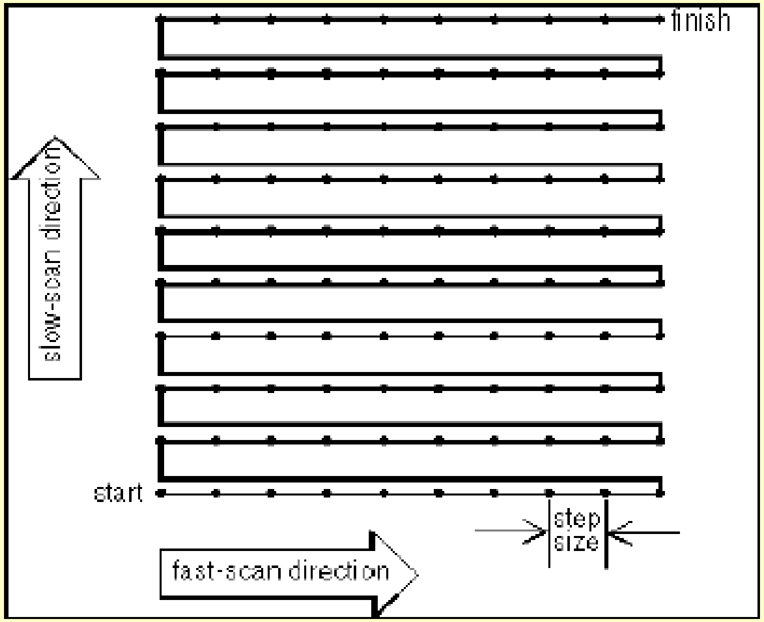
AFM imaging: *Raster Scanning + height profiling*


Obtaining Surface Profiles

AFM raster scanning by piezoelectric tube (the sample stage)



Piezo tube



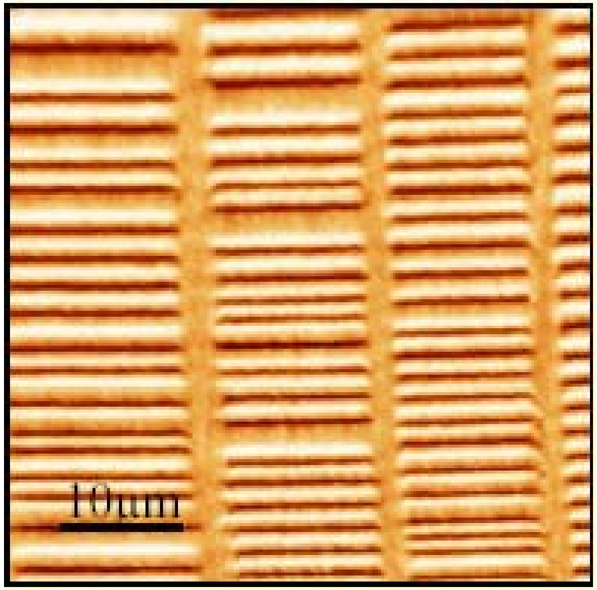
Raster scan

The resolution of the 2D scanning depends on the step size and the number of scanning lines, as well as the tip sharpness as to be discussed below.

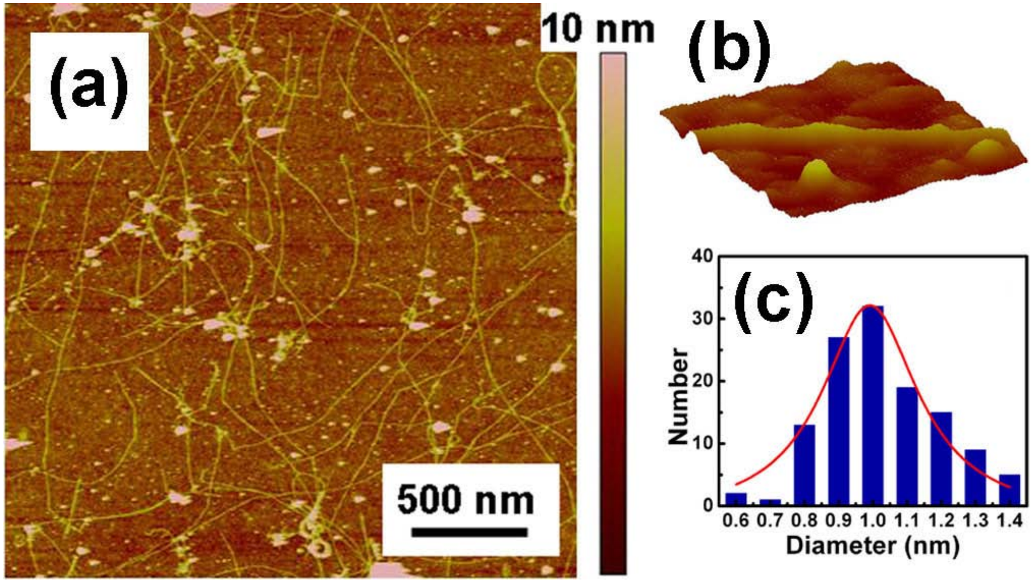
Comparison between AFM and Electronic Microscopes

- Optical and electron microscopes can easily generate two dimensional images of a sample surface, with a magnification as large as 1000X for an optical microscope, and a few hundreds thousands ~100,000X for an electron microscope.
- However, these microscopes cannot measure the vertical dimension (z-direction) of the sample, the height (e.g. particles) or depth (e.g. holes, pits) of the surface features.
- AFM, which uses a sharp tip to probe the surface features by raster scanning, can image the surface topography with extremely high magnifications, up to 1,000,000X, comparable or even better than electronic microscopes.
- The measurement of an AFM is made in three dimensions, the horizontal X-Y plane and the vertical Z dimension. Resolution (magnification) at Z-direction is normally higher than X-Y.

AFM imaging: *from mm to nm, to Å*

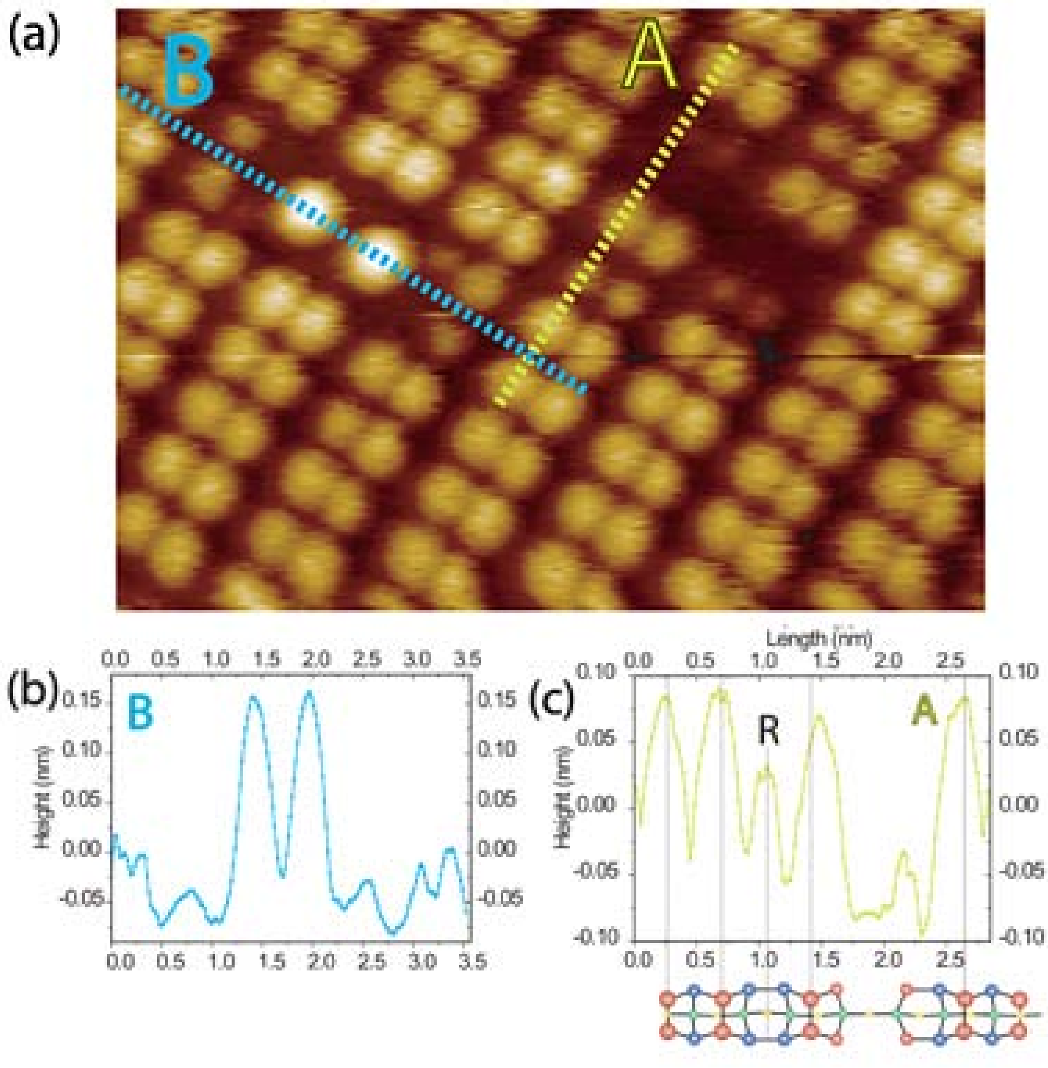


Magnetic bits of a zip disk



Carbon Nanotubes

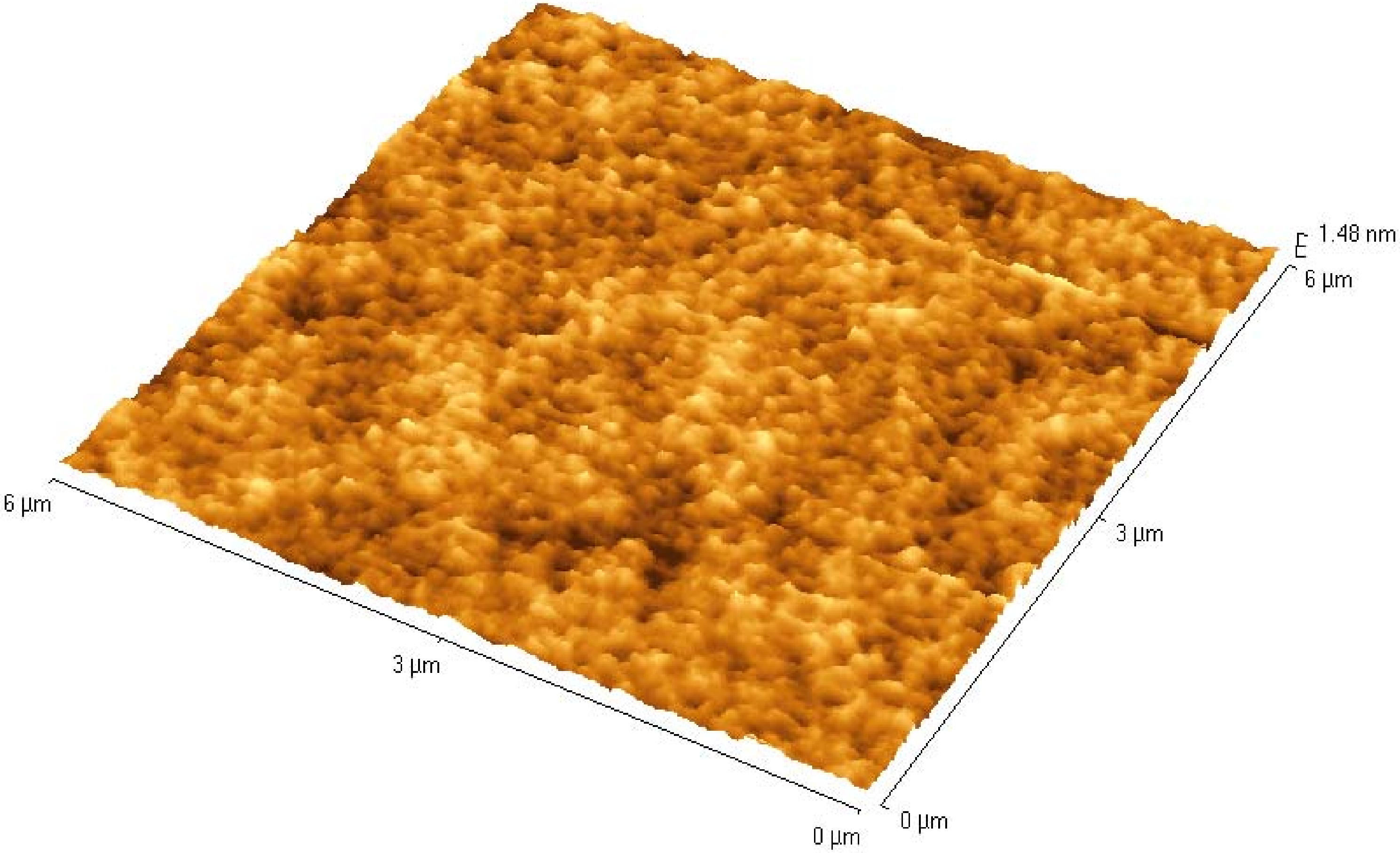
AFM imaging: *from mm to nm, to Å*



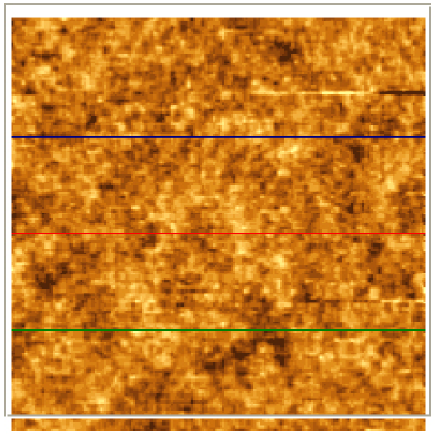
Si(100) surface taken at 77 K.

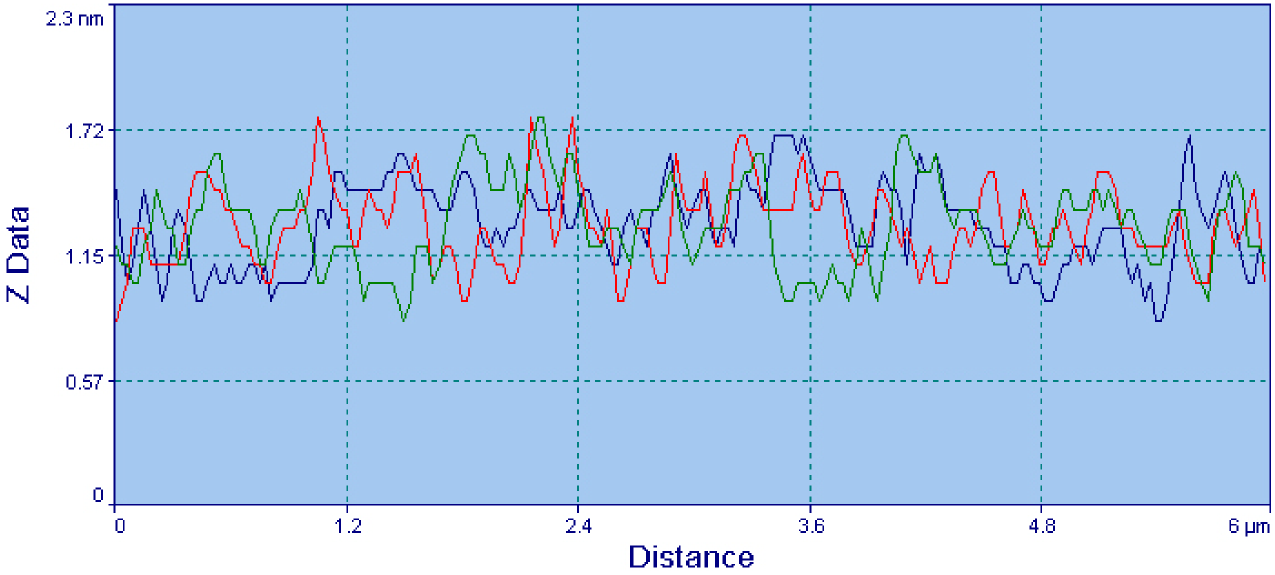
Scanned with non-contact mode.

Clean glass surface: roughness ~ 0.8 nm

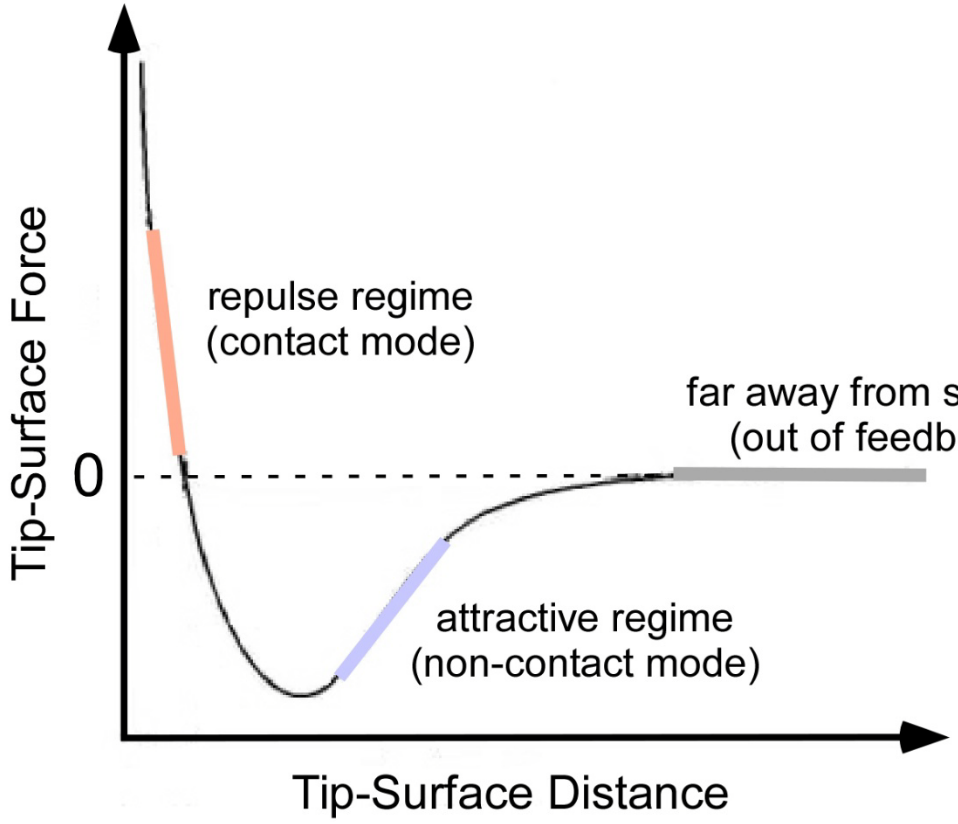


Clean glass surface: roughness ~ 0.8 nm





Atomic interaction



Atomic interaction at different tip-sample distances

Repulsion:

At very small tip-sample distances (a few angstroms) a very strong repulsive force appears between the tip and sample atoms. Its origin is the so-called exchange interactions due to the overlap of the electronic orbitals at atomic distances. When this repulsive force is predominant, the tip and sample are considered to be in "**contact**".

Attraction (Van der Waals):

A polarization interaction between atoms: An instantaneous polarization of an atom induces a polarization in nearby atoms – and therefore an attractive interaction.

Different modes of tip-sample interaction when in contact

Friction:

The cantilever bends laterally due to a friction force between the tip and the sample surfaces --- lateral force microscope (LFM).

Adhesion:

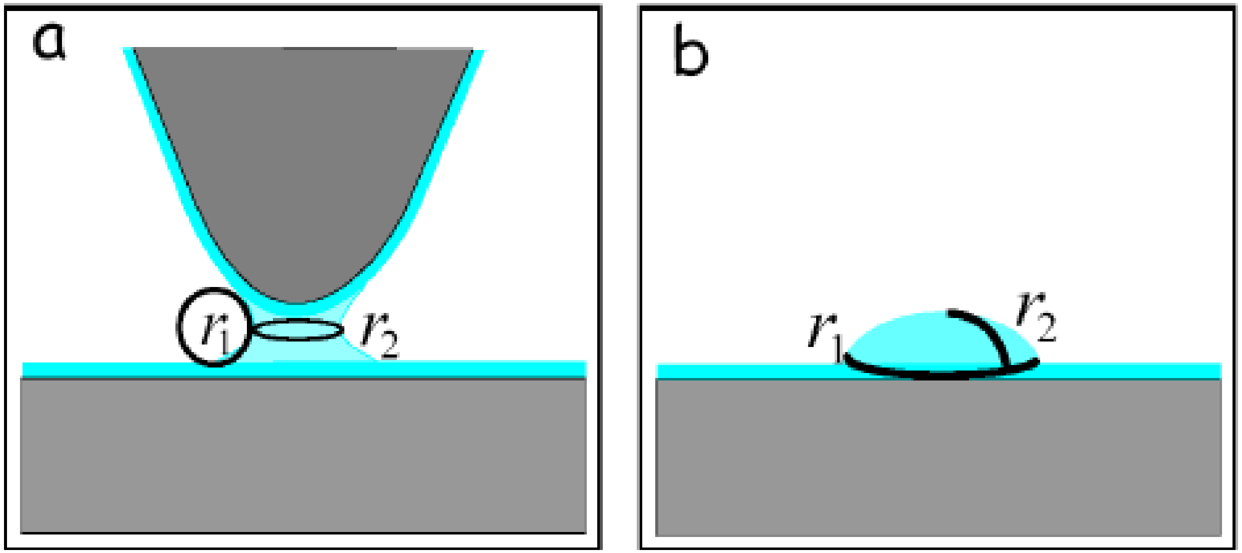
- Adhesion can be defined as “the free energy change to separate unit areas of two media from contact to infinity in vacuum or in a third medium”.

- In general, care has to be taken with the term adhesion, since it is also used to define a force - the adhesion force, as for example in force modulation microscope (FMM).

- In FMM at ambient conditions, in addition to the intrinsic adhesion between tip and sample, there is another one from the capillary neck condensing between the tip and water meniscus --- interference from the huminity.

- The pull-off force is considered as the adhesion force, which is in the range of a few nanonewton to tens of nanonewton.

Adhesion (due to water meniscus)



The snap-in distance increases with the relative humidity, up to 10-15 nm.

Electromagnetic interactions between tip and sample

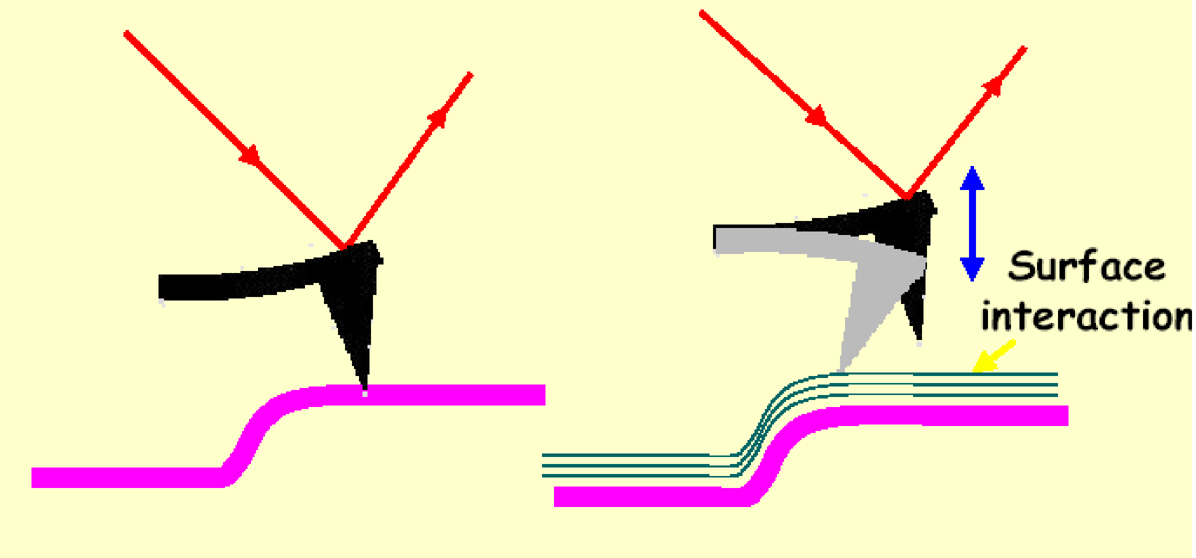
Electrostatic interaction:
Caused by both the localized charges and the polarization of the substrate due to the potential difference between the tip and the sample. It has been used to study the electrostatic properties of samples such as microelectronic structures, charges on insulator surfaces, or ferroelectric domains.

EFM --- to be discussed in Lecture #15

Magnetic interaction:
Caused by magnetic dipoles both on the tip and the sample. This interaction is used for Magnetic Force Microscopy to study magnetic domains on the sample surface.

MFM --- to be discussed in Lecture #15

AFM imaging modes: Contact *vs.* Non-contact



Contact mode (left): the deflection of cantilever is kept constant.

Non-contact mode (right): the tip is oscillated at the resonance frequency and the amplitude of the oscillation is kept constant.

Tapping mode: somewhere between the contact and non-contact mode.

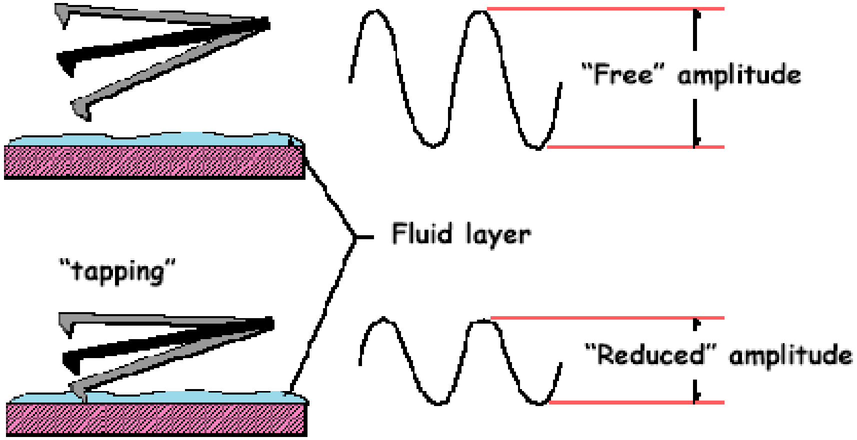
Non-contact vs. tapping mode

- Both are based on a Feedback Mechanism of constant oscillation amplitude.

- Non-contact mode: amplitude set as ~ 100% of “Free” amplitude;
- Tapping mode: amplitude set as ~ 50 -60% of “Free” amplitude.

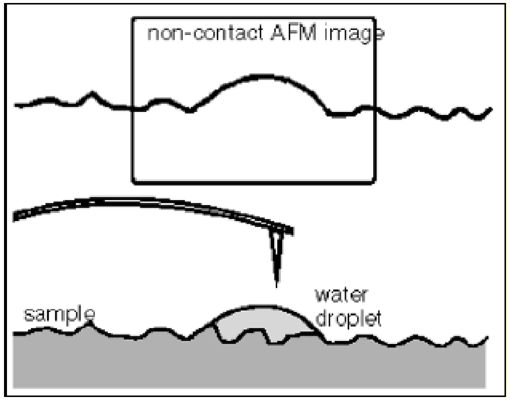
- Tapping mode provides higher resolution with minimum sample damage.

- **Most of times, non-contact mode is operated as tapping mode.**

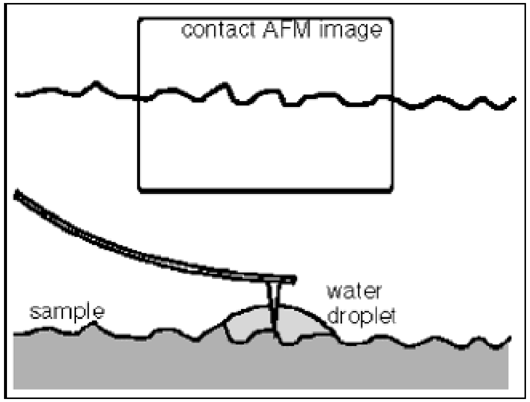


Advantages of contact mode scanning

Non-Contact vs. Contact Through Water



Non-Contact



Contact

Two contact scanning modes: *Constant Height and Constant Force*

Constant Height (of Scanner):

- In this mode, the spatial variation of the cantilever deflection is used directly to generate the topographic data set because the height of the scanner is fixed as it scans.
- Constant-height mode is often used for taking atomic-scale images of atomically flat surfaces, where the cantilever deflections and thus variations in applied force are **small**.
- Constant-height mode is also essential for recording real-time images of changing surfaces, where high scan speed is essential.

Constant Force:

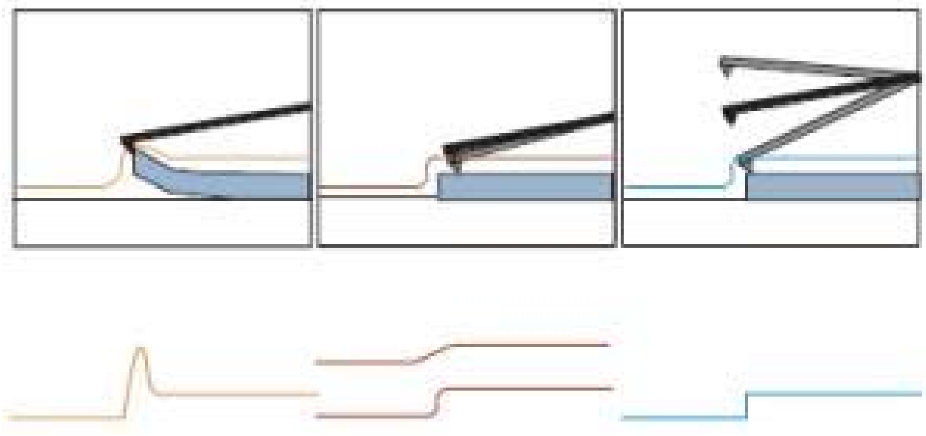
- In this mode, the deflection of the cantilever can be used as input to a feedback circuit that moves the scanner up and down in ***z***, responding to the topography by keeping the cantilever deflection constant.
- With the cantilever deflection held constant, the total force applied to the sample is constant.
- In this mode, the image is generated from the scanner's *z*-motion. The scanning speed is thus limited by the response time of the feedback circuit.
- **Constant-force mode is generally preferred for most applications.**

Comparison between the three scanning modes: *damage to the sample*

- **Contact mode imaging** (left) is heavily influenced by frictional and adhesive forces, and can damage samples and distort image data.

- **Non-contact imaging** (center) generally provides low resolution and can also be hampered by the contaminant (e.g., water) layer which can interfere with oscillation.

- **Tapping Mode imaging** (right) takes advantages of the two above. It eliminates frictional forces by intermittently contacting the surface and oscillating with sufficient amplitude to prevent the tip from being trapped by adhesive meniscus forces from the contaminant layer.



Contact, Non-Contact, Tapping

Imaging by contact and non-contact (tapping) mode

Contact vs. Tapping – Si (100)

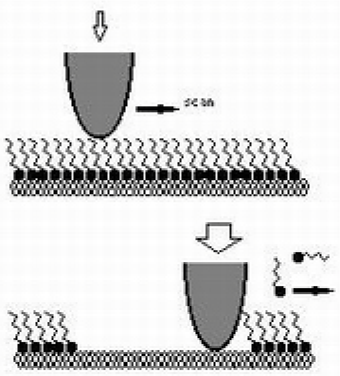
- Tapping Mode images show no surface alteration and better resolution.
- Contact imaging shows clear surface damage. Material has been removed by the scanning tip, while in other cases, additional oxide growth or more subtle changes may occur.
- This type of surface alteration often goes undetected since not all researchers check for damage by rescanning the affected area at lower magnification.

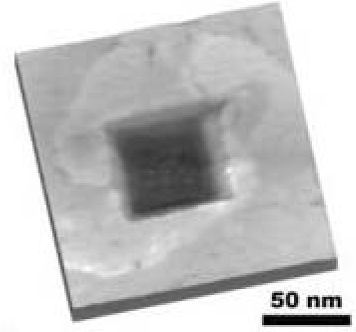
Intended Damage

==>   Lithography

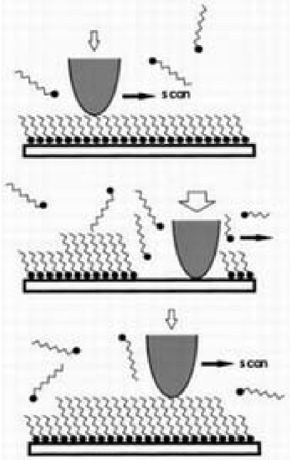
AFM nanolithography based on contact mode

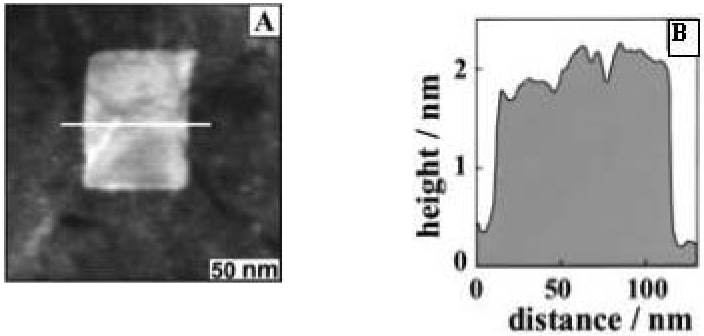
Nanoshaving





Nanografting





Prof. Gang-Yu Liu group @ UC Davis

Advanced imaging techniques of AFM

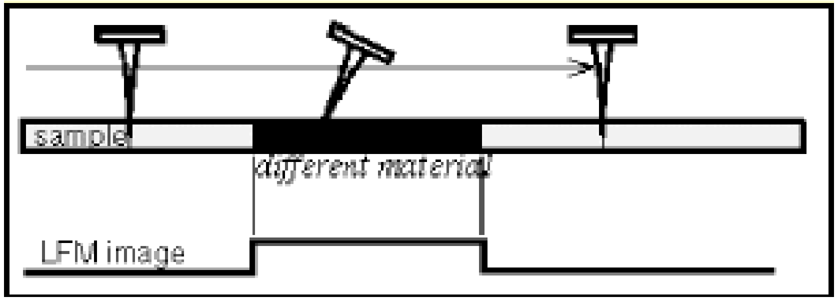
<u>Contact-Mode scanning</u>

- Lateral force microscope (LFM) --- surface friction.
- Force modulation microscope (FMM) --- detecting surface stiffness or elasticity;

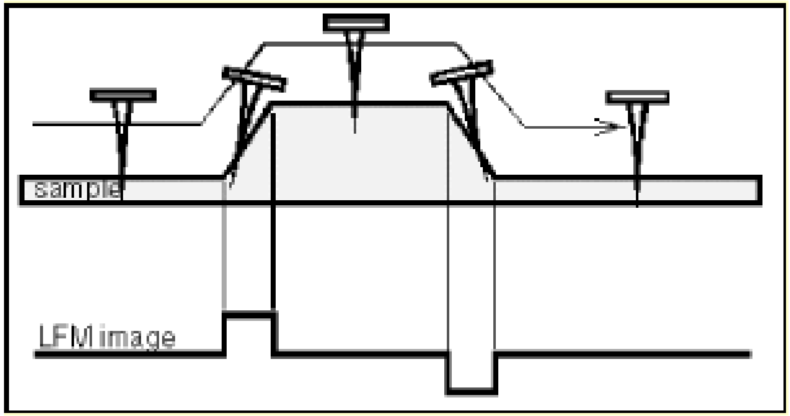
<u>Tapping-Mode scanning</u>

- Phase mode imaging --- detecting surface structure or elasticity property.

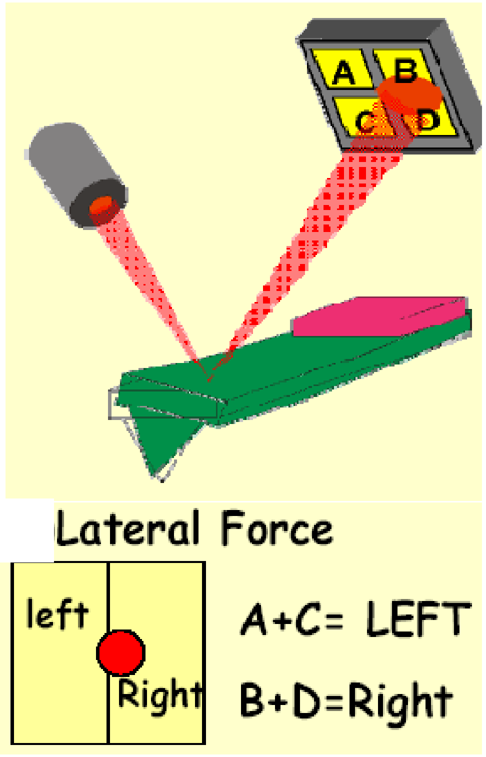
Lateral Force Microscopy (LFM)



Lateral deflection by friction variations



Lateral deflection by slope variations



Friction: lateral force microscope (LFM)

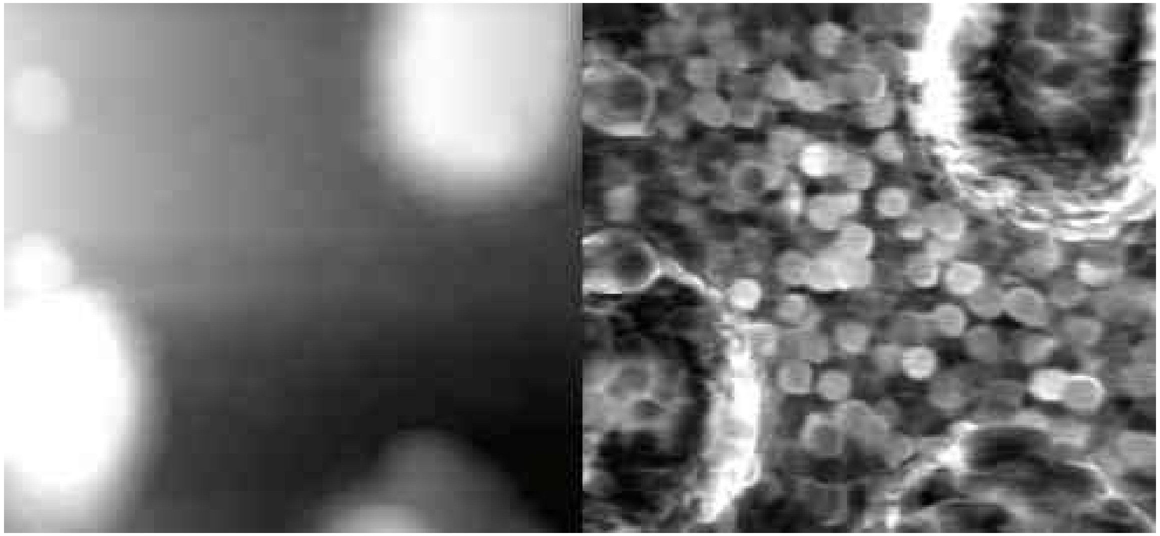
Lateral Force Microscopy (LFM)

- LFM measures lateral deflections (twisting) of the cantilever that arise from forces on the cantilever parallel to the plane of the sample surface.

- LFM images variations in surface friction, arising from inhomogeneity in surface material.

- LFM imaging is also enhanced by edge deflection (slope variations) of surface feature. This differentiates from the imaging of different materials by two sharp changes (up/down) at both sides.

Lateral Force Microscopy (LFM)



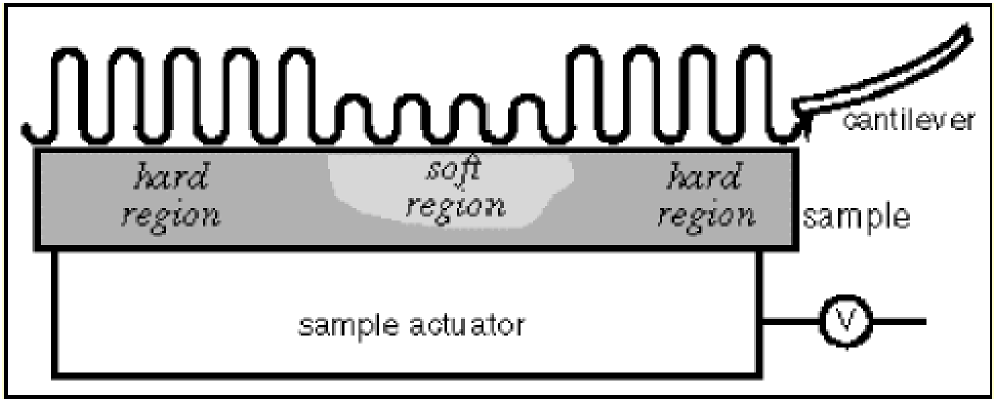
Topography *Friction image*

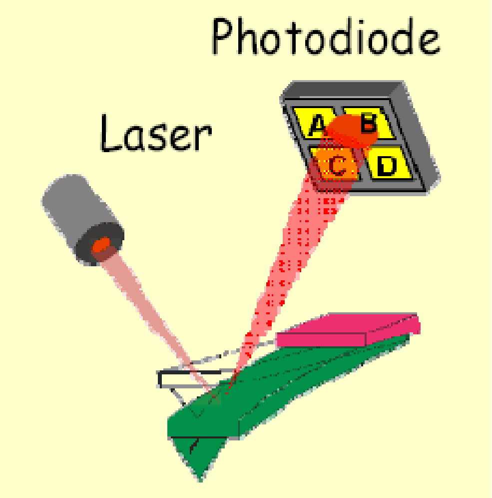
2µm x 2µm section on a Ni CD-stamper matrix.

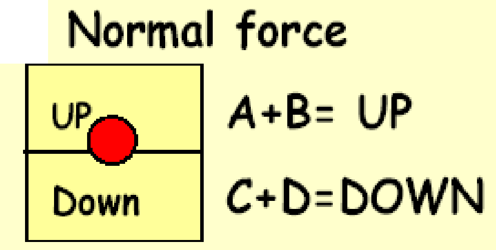
The friction image presents a more detailed surface structure than the topography image. Bright areas correspond to higher friction.

T.Göddenhenrich, S.Müller and C.Heiden, ***Rev. Sci. Instrum***. 65, (1994) 2870

Force Modulation Microscopy (FMM): *a secondary imaging technique*





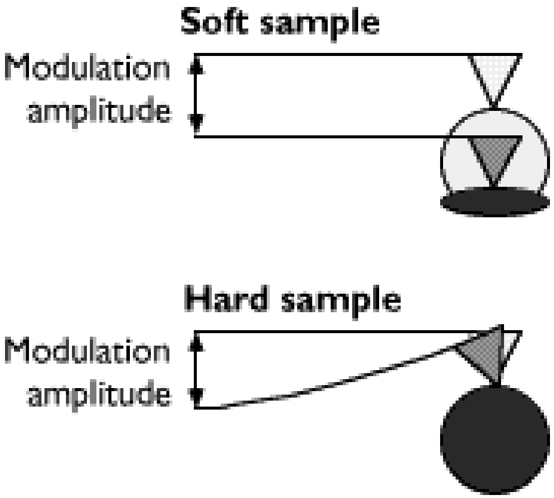


Force Modulation Microscopy (FMM): *a secondary imaging technique*

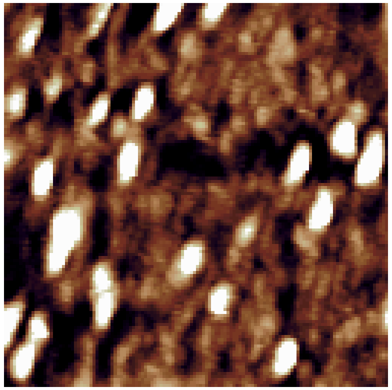
- In FMM mode, the tip is scanned in contact with the sample, and the **z** feedback loop maintains a constant cantilever deflection (as for constant-force mode AFM).
- A periodic vertical oscillation signal is applied to either the tip or the sample. The amplitude of cantilever modulation that results from this applied signal varies according to the elastic properties of the sample.
- From the changes in the amplitude of cantilever modulation, the system generates a force modulation image --- a map of the sample's elastic properties.
- The frequency of the applied signal is on the order of hundreds of kHz, which is

  faster than the rater scan rate (i.e. the z-feedback loop set up to track the scanning).
- Under the same force, a stiff area on the sample deforms less than a soft area; i.e., stiffer areas put up greater resistance to the cantilever's vertical oscillation, and, consequently cause greater bending of the cantilever. The variation in cantilever deflection amplitude at the frequency of modulation is a measure of the relative stiffness of the surface.
- Topographic information can be separated from local variations in the sample's elastic properties, and the two types of images can be collected simultaneously --- *direct correlation between topographic structure and elastic properties*.

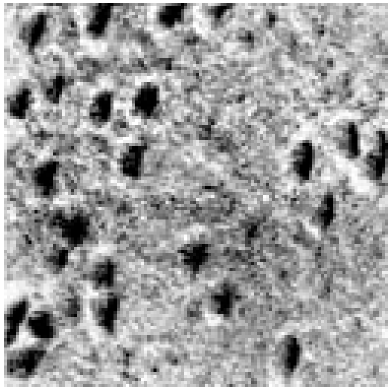
Measurement of Elasticity by FMM



bovine serum albumen on silicon



topography

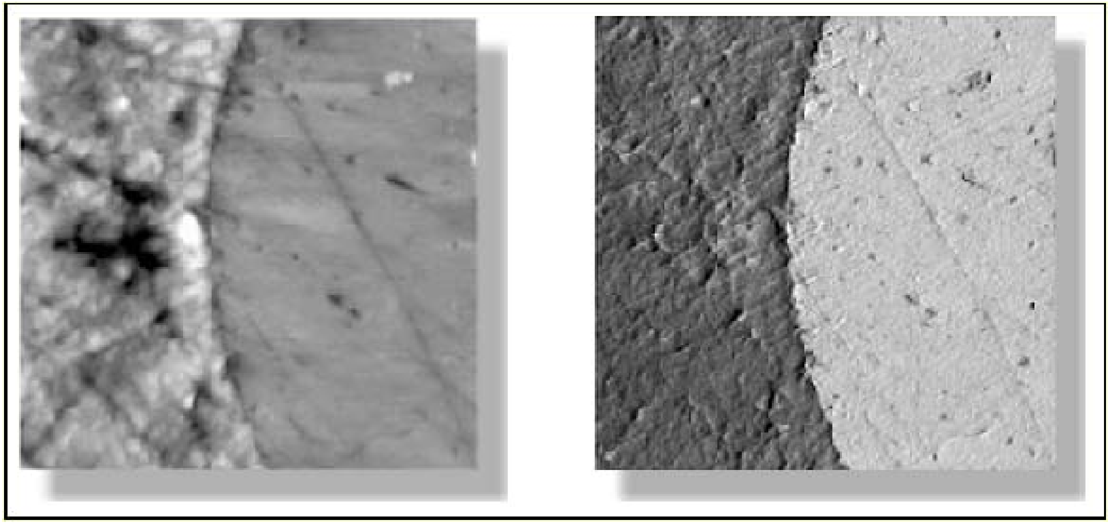


Elasticity by FMM

The elasticity image reveals that each of the bumps is soft relative to the silicon substrate, a reasonable result for protein molecules.

Measurement of Elasticity by FMM

Carbon fiber/polymer Composite Collected Simultaneously (5μm)

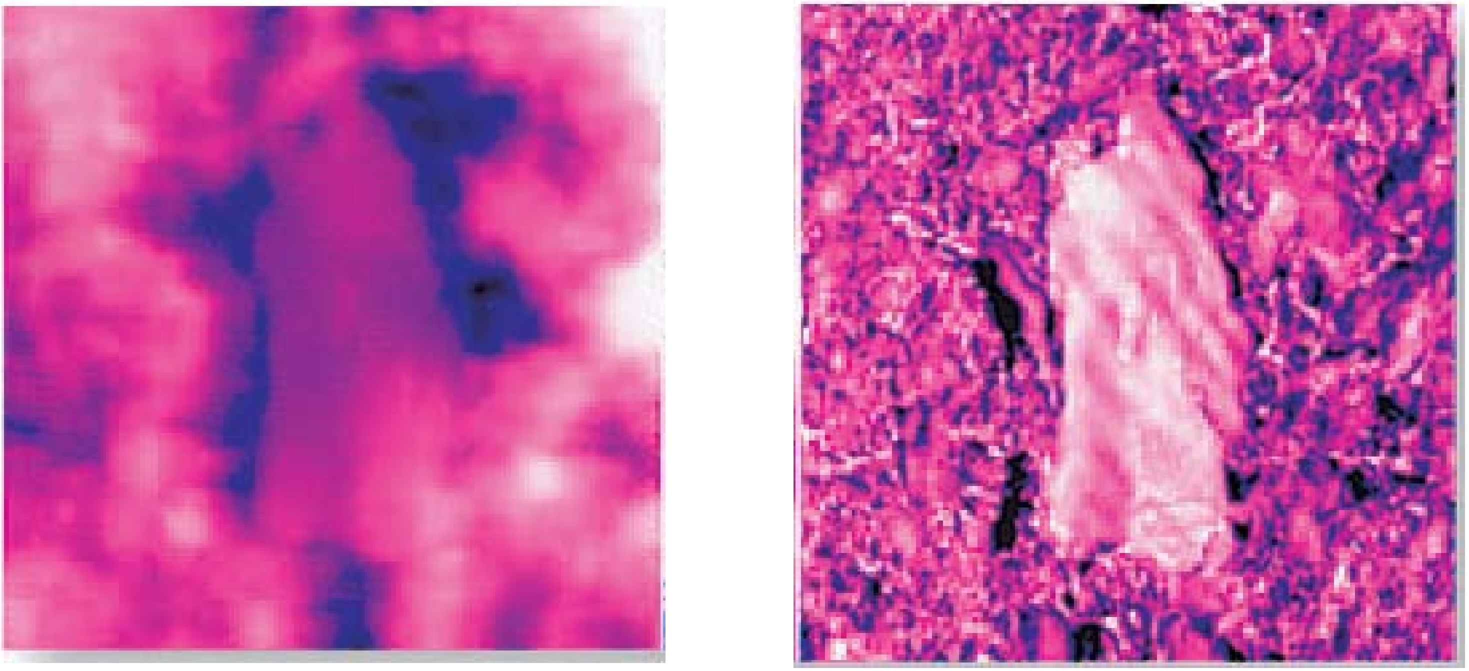


FMM

Topography by contact AFM

FMM gives more detailed information about the composition and distribution of the two components --- soft polymer (dark area) and hard carbon fiber.

Carbon in tire rubber by FMM

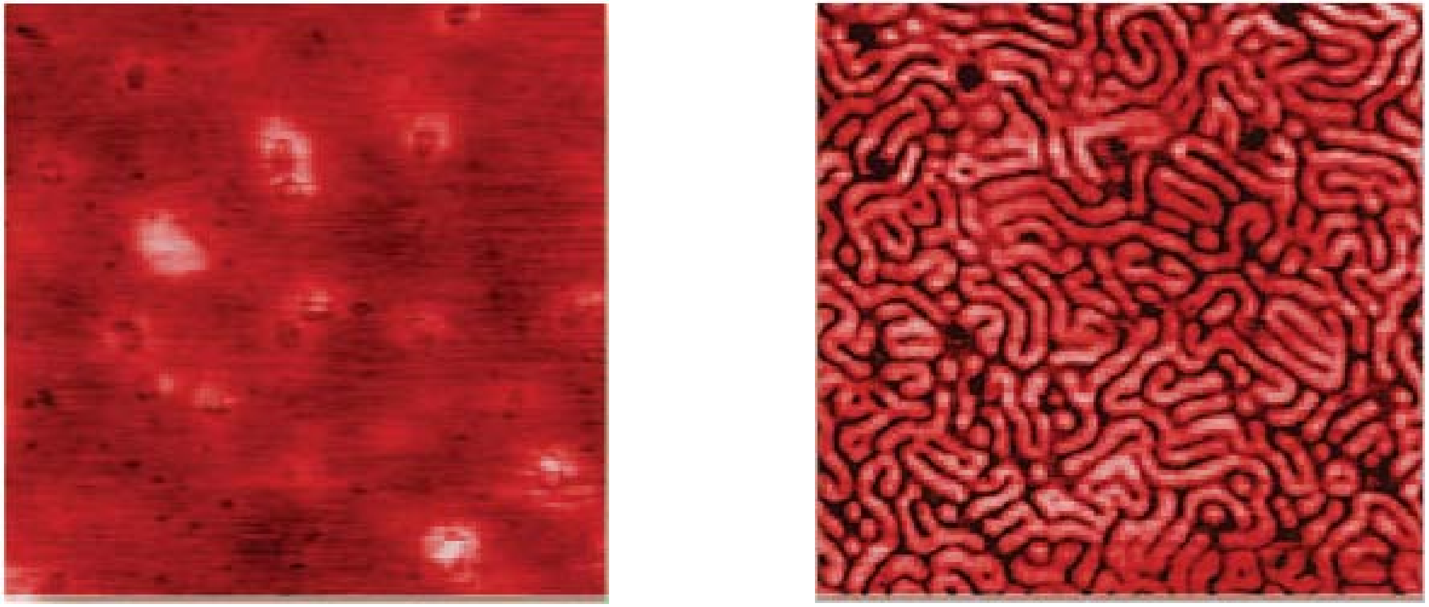


topography

FMM

Contact mode topography (left) and force modulation image (right) of carbon black deposit in automobile tire rubber. 15µm scan. Veeco.

Two-phase block polymer by FMM

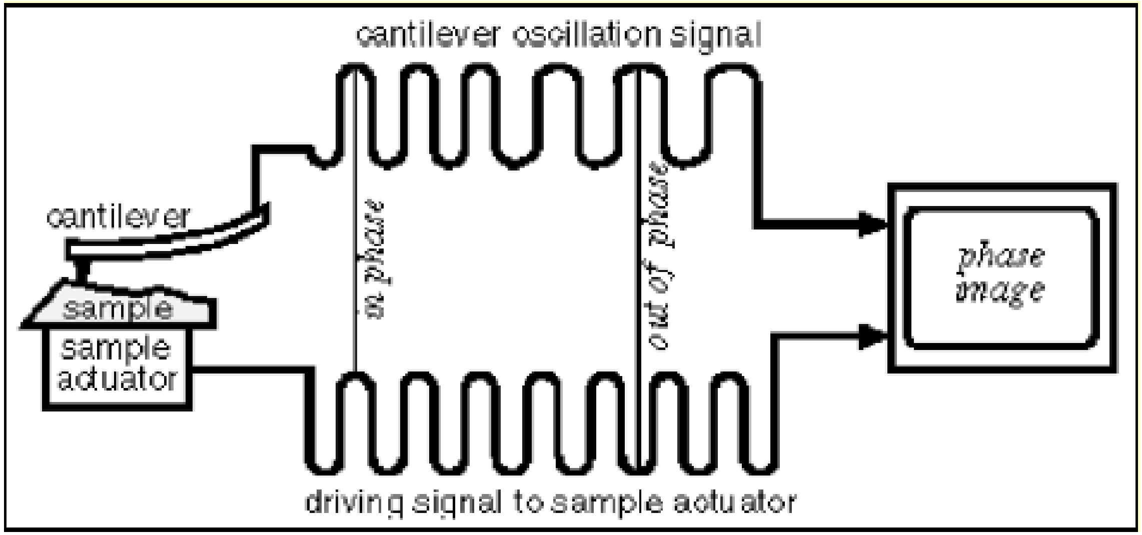


topography

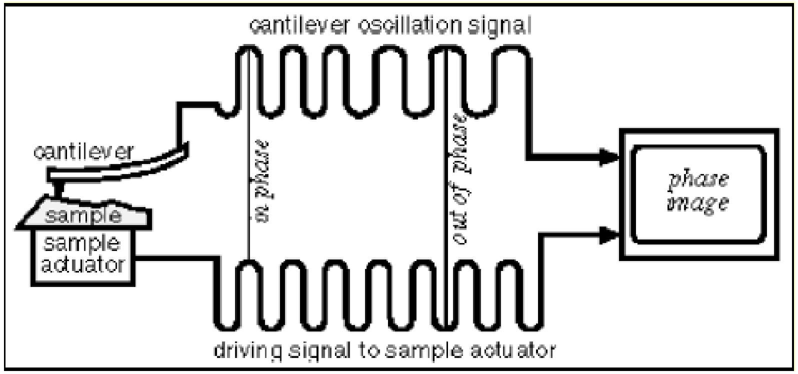
FMM

Contact mode topography (left) and force modulation image (right) of a two-phase block copolymer. The softer, more compliant component of the polymer maps in black. 900nm scans. Veeco.

Phase Imaging: *a secondary imaging technique for tapping mode*



Phase Imaging: *a secondary imaging technique for tapping mode*



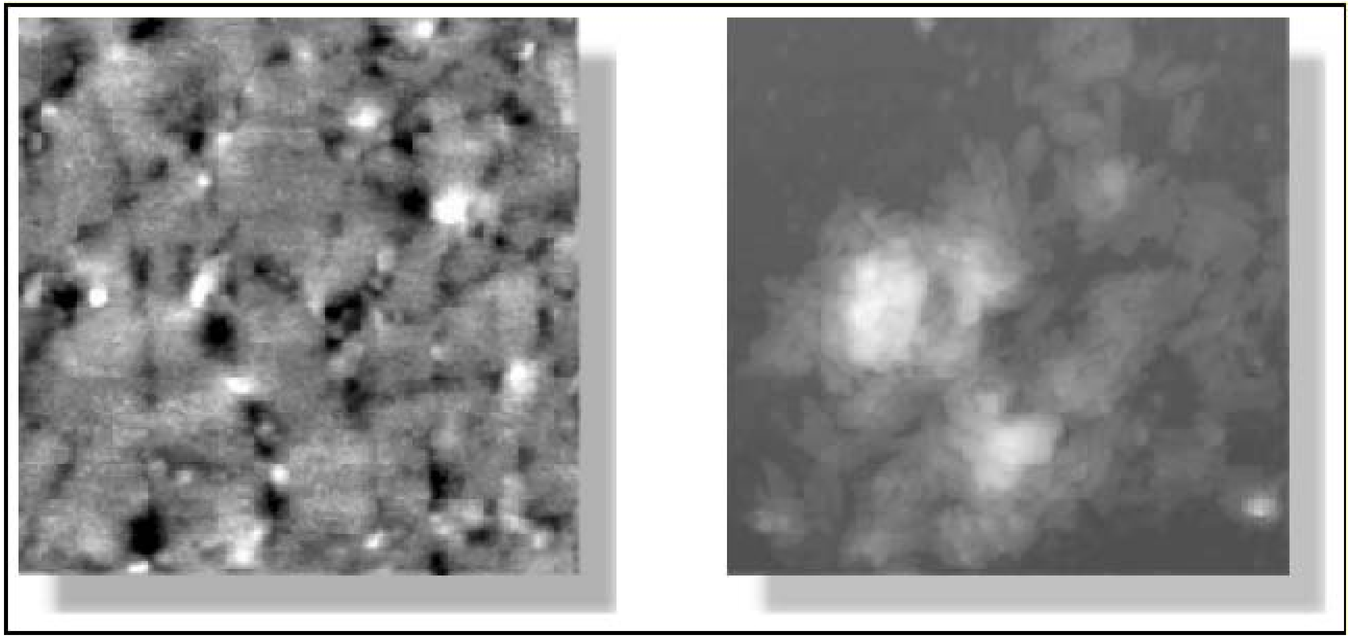
- Phase imaging monitors the phase lag between the signal that drives the cantilever to oscillate and the cantilever oscillation output signal. In Tapping-Mode AFM, the cantilever is excited into resonance oscillation with a piezoelectric driver.
- Phase imaging is used to map variations in surface properties such as elasticity, adhesion and friction, which all may cause the phase lag.
- Phase detection images can be produced while an instrument is operating in any vibrating cantilever mode, such as tapping mode AFM, MFM, EFM.
- The phase lag is monitored while the topographic image is being taken so that images of topography and material properties can be collected simultaneously ---- direct correlation between surface properties and topographies.

Applications of Phase Imaging

- Identification of contaminants;
- Mapping of different components in composite materials;
- Differentiating regions of high and low surface adhesion or hardness;
- Mapping of electrical and magnetic properties with wide-ranging implications in data storage and semiconductor industries.

- In many cases, phase imaging complements lateral force microscopy (LFM), and force modulation microscopy (FMM), often providing additional information more <u>rapidly</u> and with <u>higher resolution</u>.
- Phase imaging is as fast and easy to use as Tapping-Mode AFM — with all its benefits for imaging soft, adhesive, easily damaged or loosely bound samples.

Phase Imaging of an Adhesive Label (3 μm)

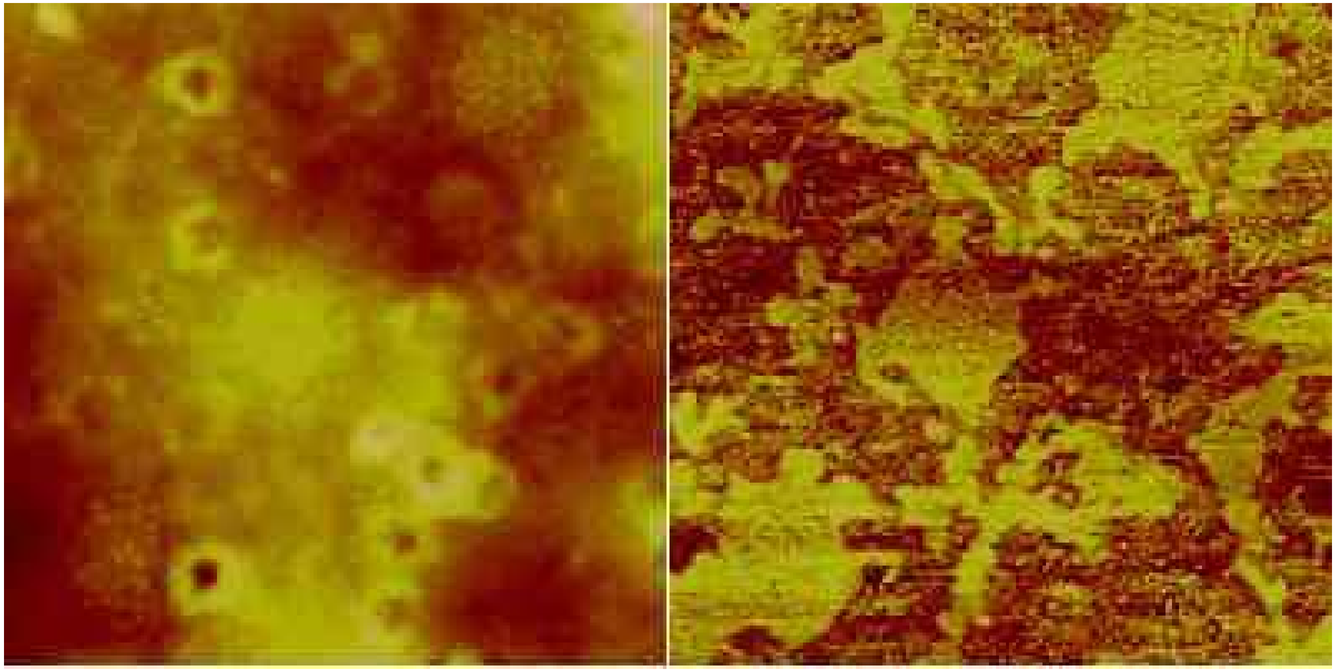


Non-contact AFM

Phase image

Phase imaging indicates the surface distribution of different materials.

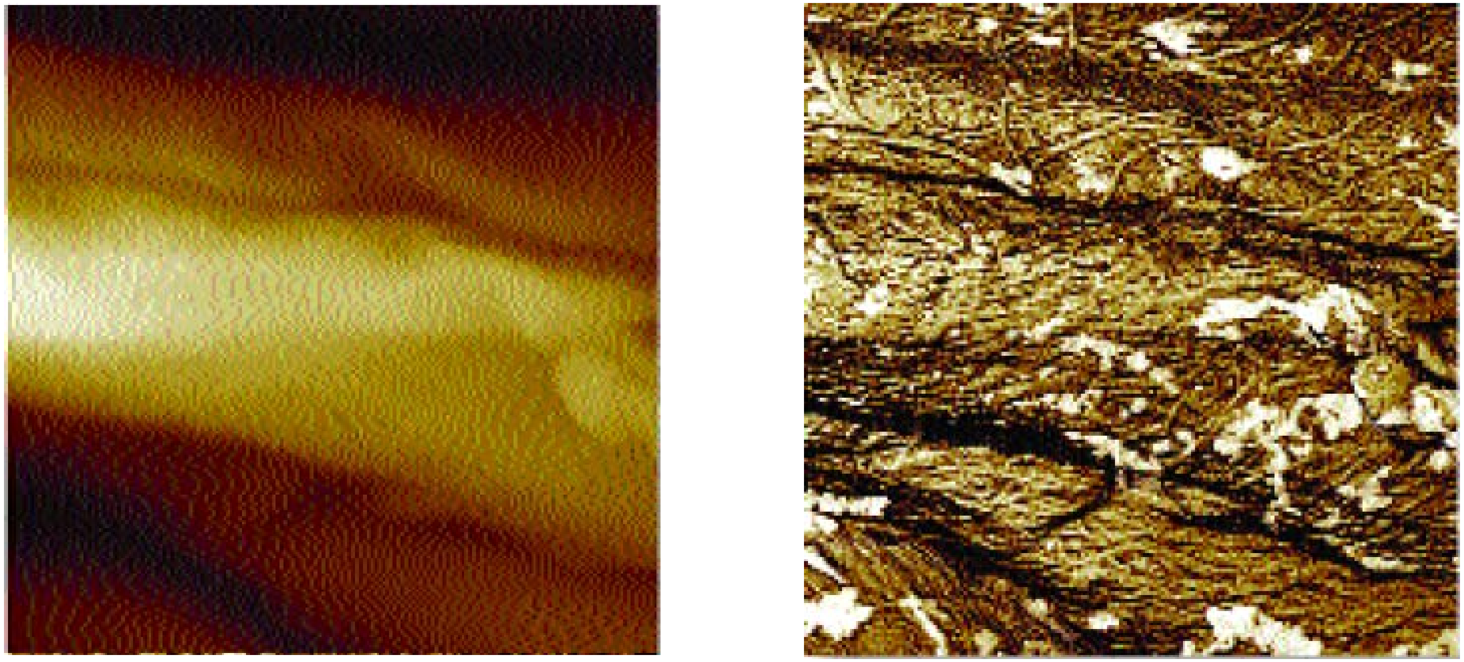
Phase Imaging of polymer coating on inside of aluminum beverage can



Non-contact AFM **Phase image**

Phase imaging tells the homogeneity of the coating regardless the roughness of the original alumina surface. This cannot be seen from the height imaging (left).

wood pulp fiber

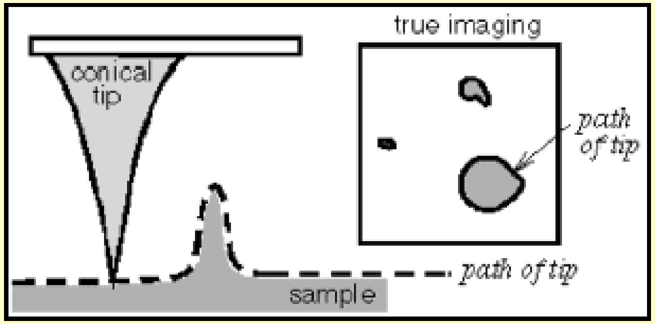


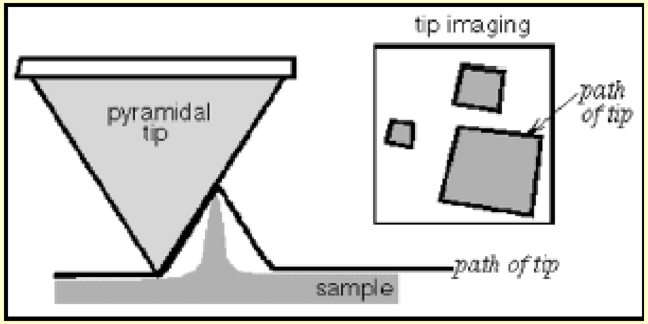
Non-contact AFM

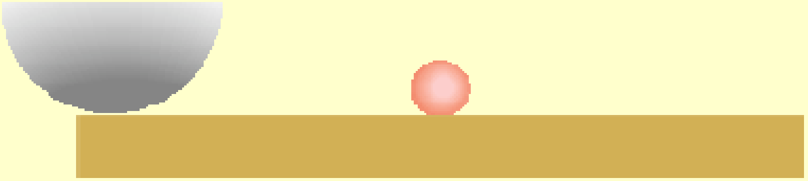
Phase image

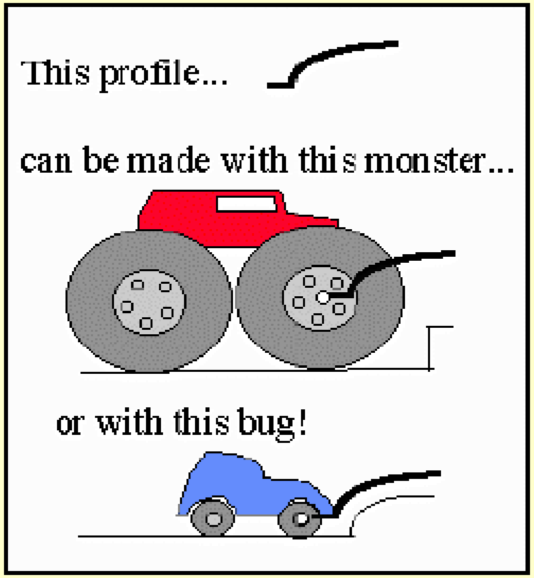
Phase (top) and Tapping-Mode (bottom) images of wood pulp fiber. The phase image highlights cellulose microfibrils. In addition, a lignin component appears as light areas in the phase image, but is not apparent in the topography image. 3µm scan. Veeco.

Lateral resolution depends on tip sharpness







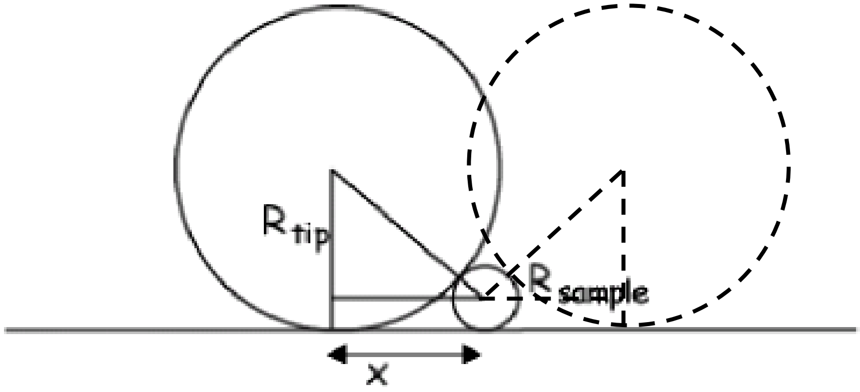


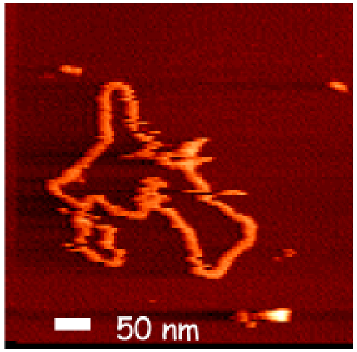
This profile...

can be made with this monster...

or with this bug!

Fat-tip Effect: *apparent width measured by large tip*





$$x^2 = (R_{tip} + R_{sample})^2 - (R_{tip} - R_{sample})^2$$

$$x^2 = \cancel{R_{tip}^2} + 2R_{tip}R_{sample} + \cancel{R_{sample}^2} - \cancel{R_{tip}^2} + 2R_{tip}R_{sample} - \cancel{R_{sample}^2}$$

$$x = 2\sqrt{R_{tip}R_{sample}}$$

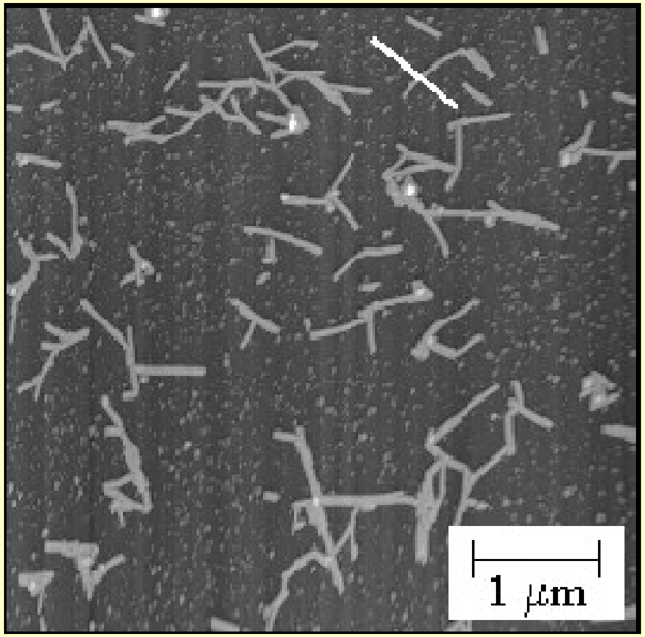
$$w = 2x = 4\sqrt{R_{tip}R_{sample}}$$

DNA: 2 nm.

tip ~ 20 nm => w = 25 nm
tip ~ 10 nm => w = 18 nm

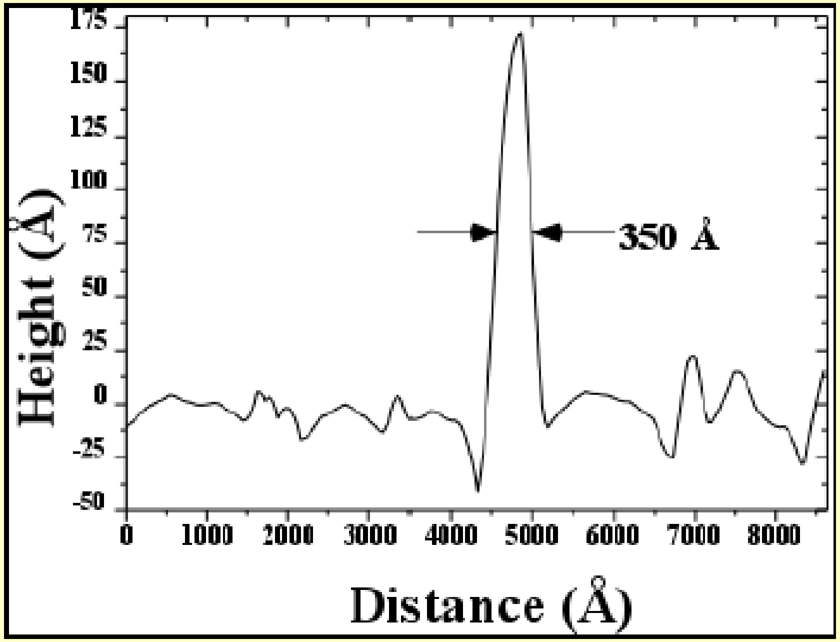
- Measured width: distance between the 1st and last tip/sample contact;
- The smaller the tip (R_{tip}), the smaller the measured width;
- When $R_{tip} \sim \frac{1}{4} R_{sample}$, measured width = $2R_{sample}$;
- For a 5 nm feature (say a particle), the tip apex size must be ~ 1 nm to get a reliable lateral measurement --- quite challenging!
- Normal tip size, ~ 20 nm or larger.
- Another challenge for lateral imaging: to differentiate two adjacent features.

A typical AFM image showing fat-tip effect



AFM image

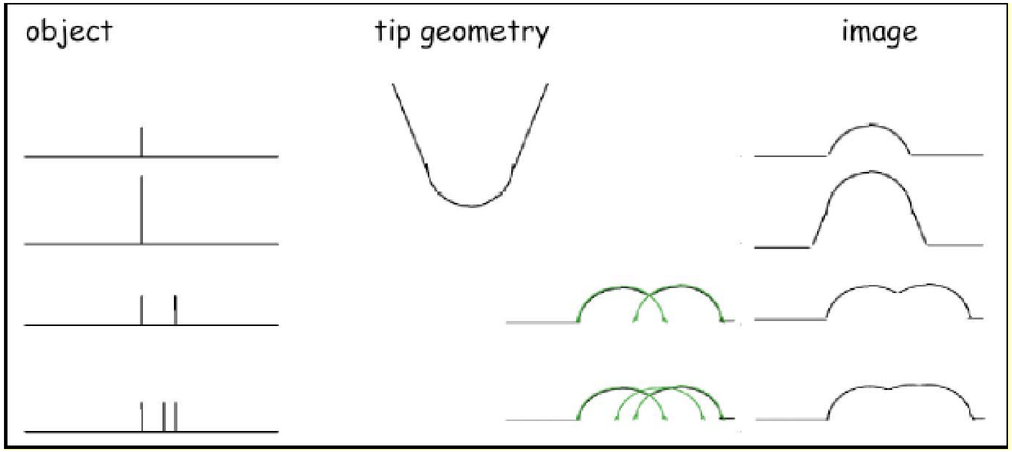
Nominal width 180 Å

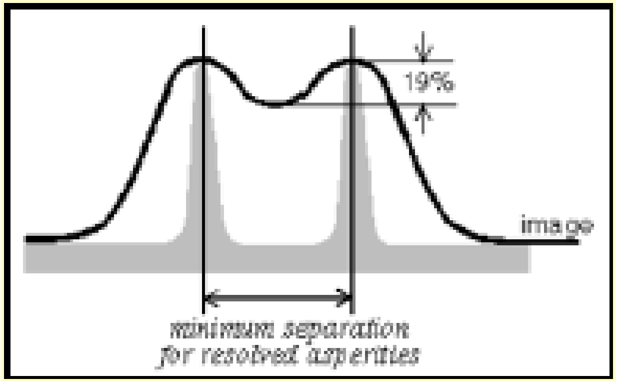


Cross section

Measured width 350 Å

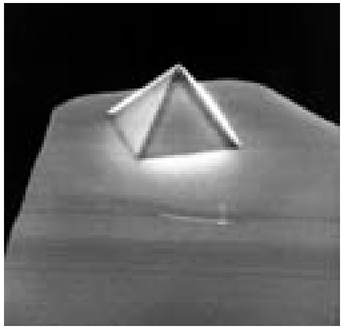
Effect of tip size on imaging resolution



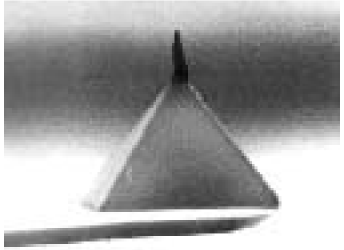


- Large tip measures a feature apparently larger than the real size;
- Larger tip cannot discern two or more adjacent features.
- Higher resolution demands sharper tips.

AFM Tips



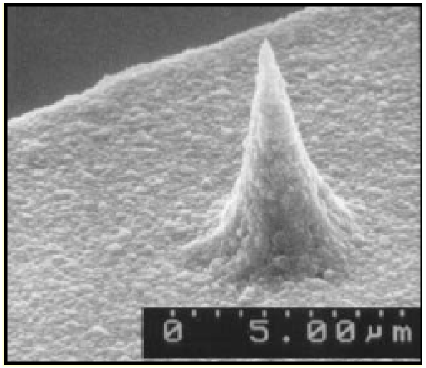
Normal Tip



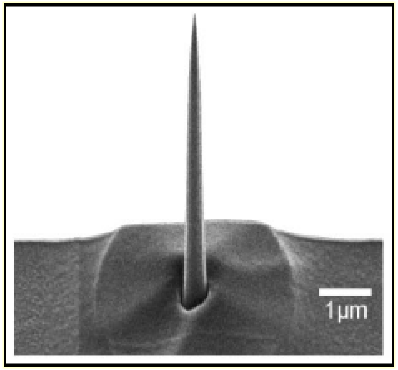
Supertip

Ultralever

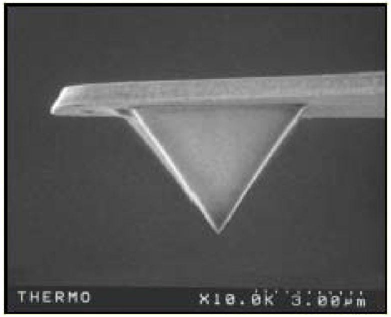
AFM Tips



Diamond-coated tip

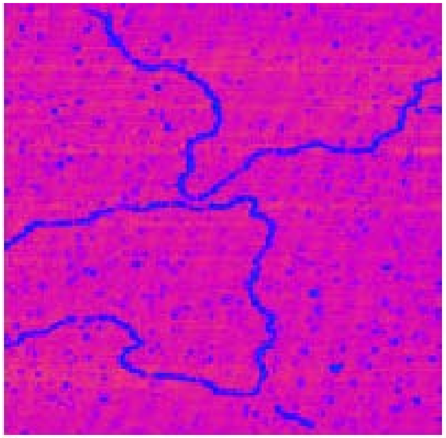


FIB-sharpened tip



Gold-coated Si_3N_4 tip

AFM measurement using ultra-sharp tips



DNA image in fluid at 37°C.
800nm scan size.

Obtained by,
EnviroScope AFM, Veeco.

Imaging in fluid requires
long tips

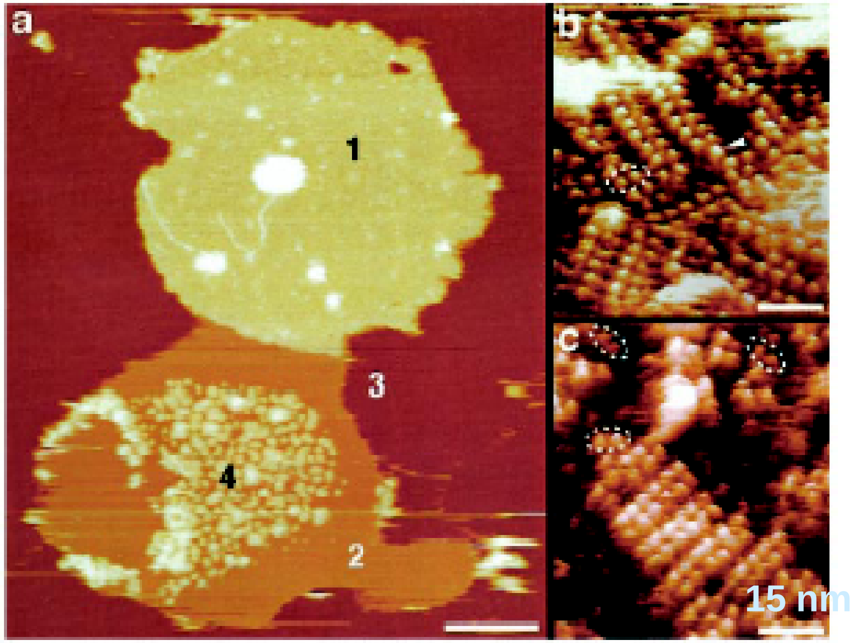


FIG. 3. **Topography of an open, spread-flattened disk adsorbed to mica and imaged in buffer solution.** *a*, height image of the open, spread-flattened disk. Four different surface types are evident: the cytoplasmic surface of the disk (types *1* and *4*), lipid (type *2*), and mica (type *3*). The topographies of regions 1 (*b*) and 4 (*c*) at higher magnification reveal densely packed rows of rhodopsin dimers. Besides paracrystals, single rhodopsin dimers (*broken ellipses*) and occasional rhodopsin monomers (*arrowhead*) are discerned floating in the lipid bilayer. *Scale bars*: 250 nm (*a*) and 15 nm (*b* and *c*). Vertical brightness ranges: 22 nm (*a*) and 2.0 nm (*b* and *c*).

Imaging small features and scanning small area at high resolution require ultra-sharp tips.

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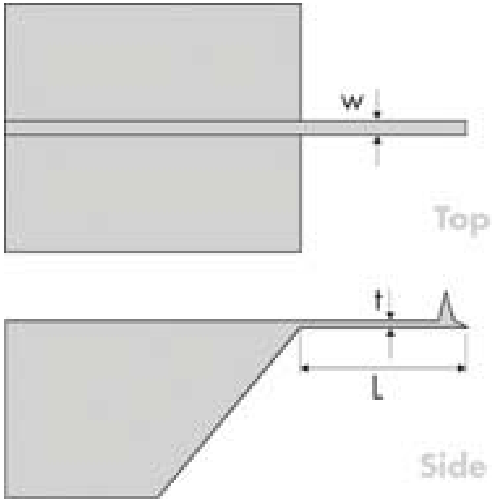
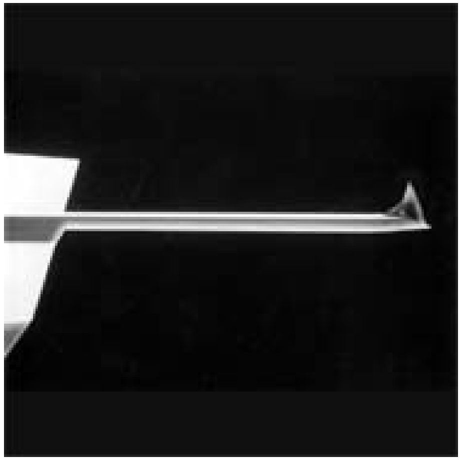
Straight shape cantilever





Common commercial cantilever: Si_3N_4 and SiO_2

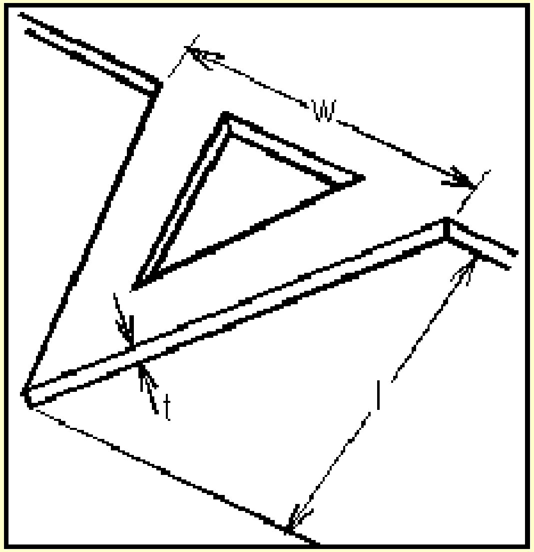
Resonance frequency of the cantilever,

$$f_0 = \frac{1}{2\pi}\left(\frac{k}{m_0}\right)^{0.5} \qquad k = \frac{Ewt^3}{4l^3}$$

k: the spring constant, *E: Young module; t: thickness; l: length;*
w: width, m_0 the effective mass of the lever.

The softer the lever (smaller *k*), the better for sensing the deflection, but requires smaller mass to keep the high frequency. Why high f needed?

V-shape cantilever



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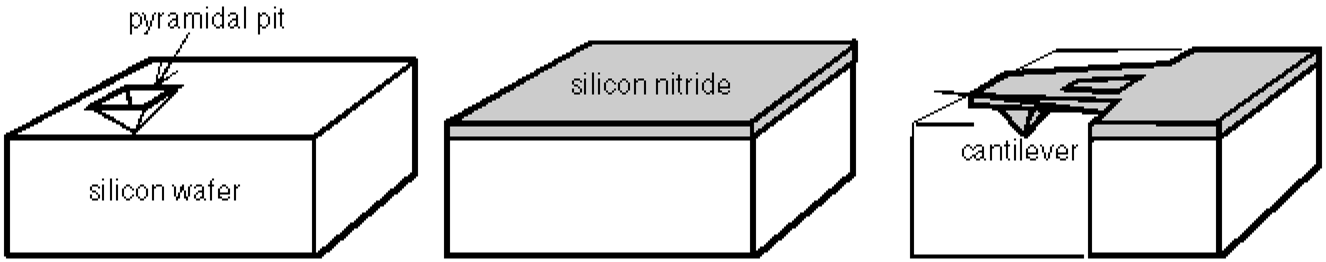
Fabrication of AFM Tip



Pit etching in Si

Si_3N_4 coating

Si underetching