CH62052

Atomic Force Microscope (AFM)

#AFM_by_RM

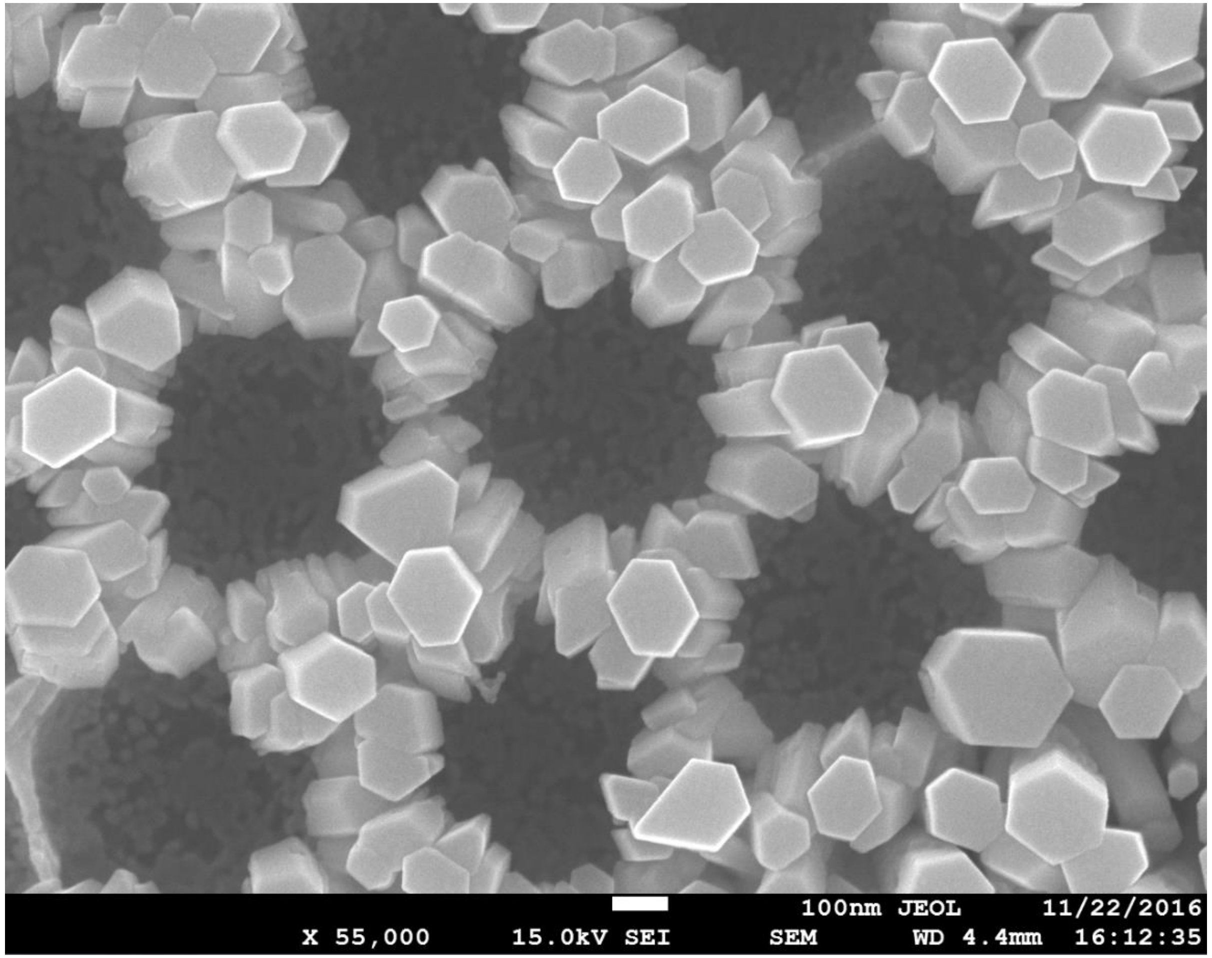
RABIBRATA MUKHERJEE

Why AFM? We all use a microscope to visualize small things! Here is an FESEM image.

Optical Microscope

Scanning Electron Microscope (SEM)

Transmission Electron Microscope (TEM)

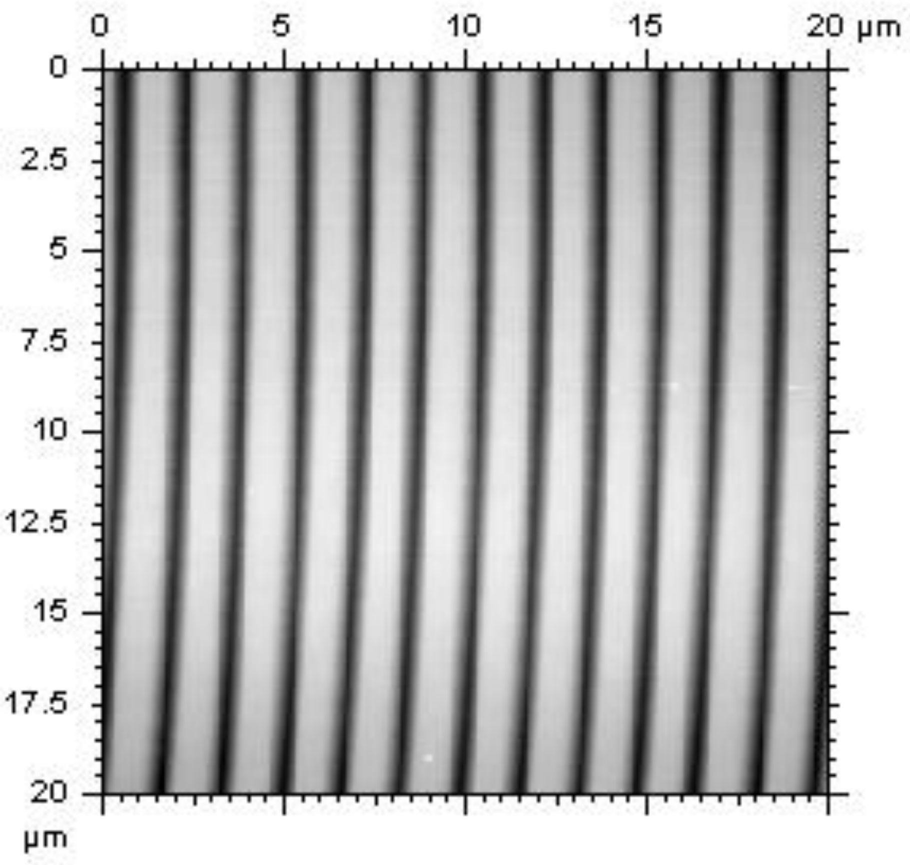


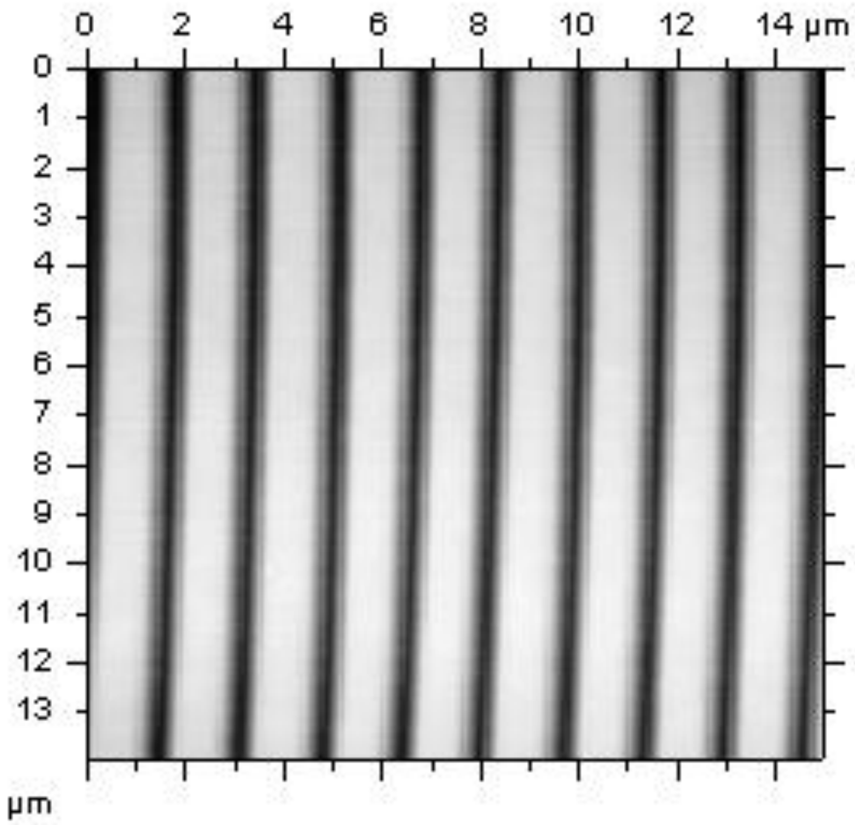
Can you think of any limitation of this image?

Any information that is missing?

Anything you are missing out in this pic?

Are these two images of the same sample? Different Magnification?





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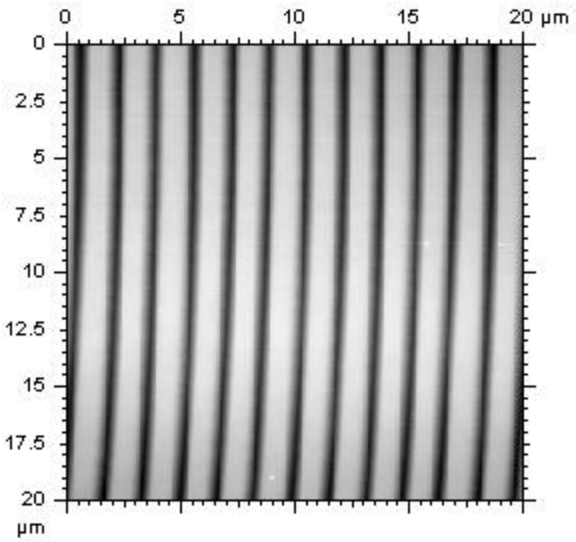
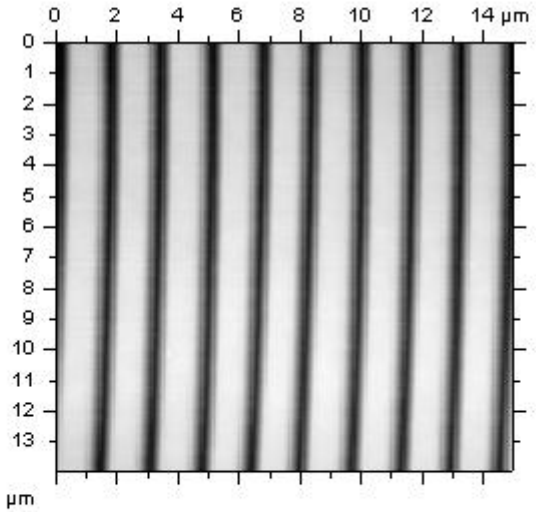
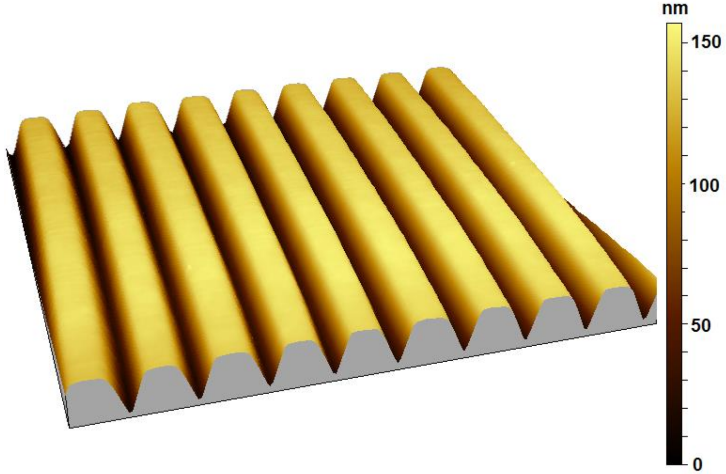
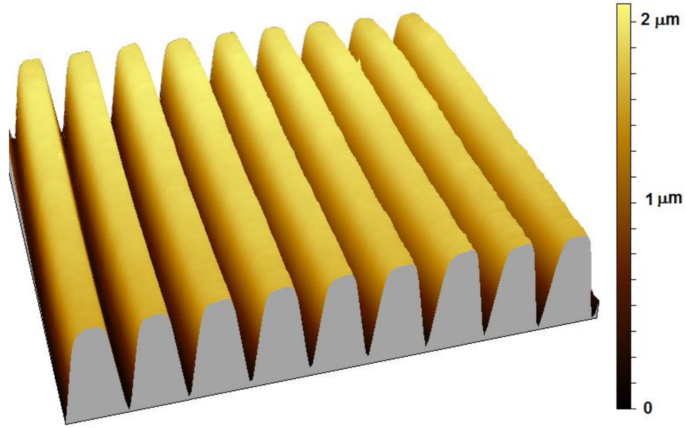




This height information AFM gives.





ANSWER IS NO!

In the early 1980's scanning probe microscopes (SPMs) produced the first real-space images of a surface.

Now, SPMs are used in a wide variety of disciplines, including fundamental surface science, routine surface roughness analysis, and spectacular three-dimensional imaging—from atoms of silicon to micron-sized protrusions on the surface of a living cell.

The scanning probe microscope is an imaging tool with a vast dynamic range, spanning the realms of optical and electron microscopes. It's also a profiler with unprecedented 3-D resolution. In some cases, scanning probe microscopes can measure physical properties such as surface conductivity, static charge distribution, localized friction, magnetic fields, and elastic moduli. As a result, applications of SPMs are very diverse.

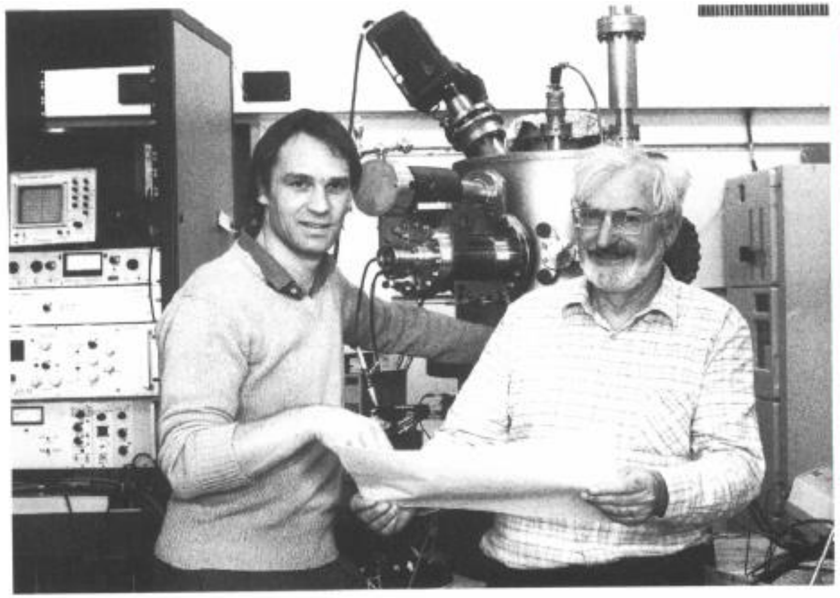
AFM was discovered in 1986.

Gerhard Binning and Co - workers IBM Corporation

Physical Review Letters, 56,930 (1986).

(An extension cum derivative of the STM)

Gerd Binning and Heinrich Rohrer were awarded Nobel Prize in 1986 for their Discovery of STM in 1982.



Gerd Binnig (left) and Heinrich Rohrer (right) who were awarded the Nobel Prize for their invention of the scanning tunneling microscope.

SCANNING TUNNELING MICROSCOPY - FROM BIRTH TO ADOLESCENCE

Nobel lecture, December 8, 1986

by

GERD BINNIG AND HEINRICH ROHRER

IBM Research Division, Zurich Research Laboratory, 8803 Rüschlikon, Switzerland

Short History of AFM http://hansmalab.physics.ucsb.edu/afmback.html

The Scanning Probe Microscope (SPM) family

SPM

STM (Tunneling)

STS,STP,Scanning Electrochemical Microscope

SFM (Force)

(AFM)

Lateral Force (LFM)

Magnetic Force (MFM)

Electrostatic Force (EFM)

DPN

Scanning Thermal Microscope (Local Temperature)

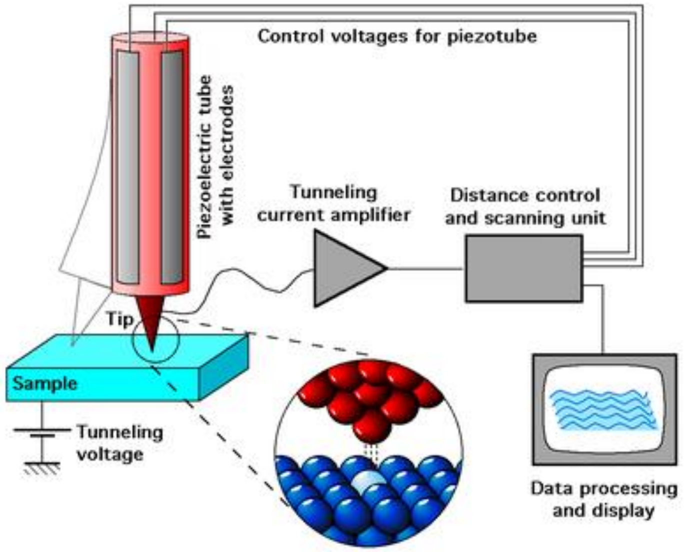
Scanning Near Field Optical Microscope (Optical imaging)

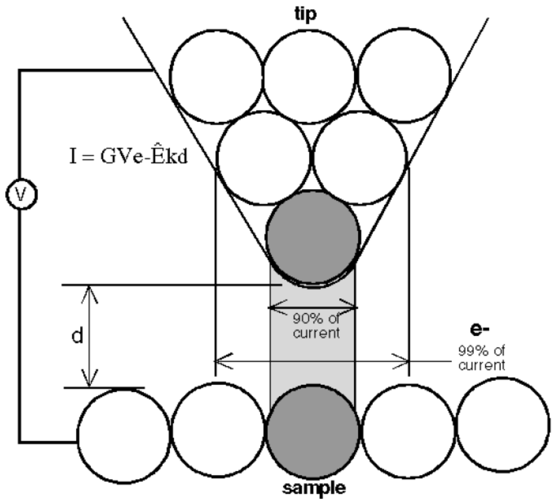
Scanning Tunneling Microscope (STM)

The resulting *tunneling current* is a function of tip position, applied voltage, and the local density of states (LDOS) of the sample.

Information is acquired by monitoring the current as the tip's position scans across the surface, and is usually displayed in image form.

STM can be a challenging technique, as it can require extremely clean and stable surfaces, sharp tips, excellent vibration control, and sophisticated electronics.



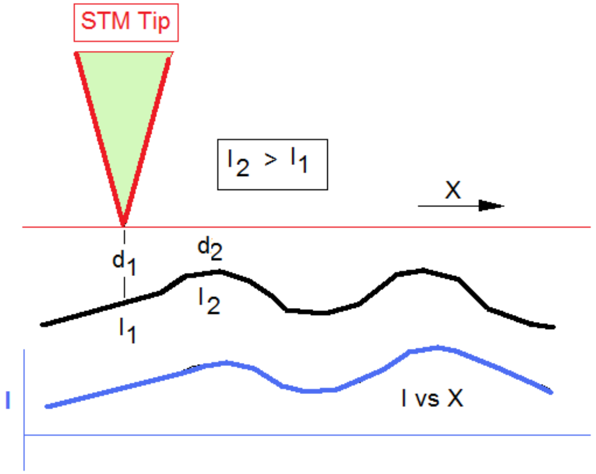


Constant Height Mode STM

STMs can be designed to scan a sample in either of two modes: ***constant-height*** or ***constant-current*** mode.

In constant-height mode, the tip travels in a horizontal plane above the sample and the tunneling current varies depending on topography and the local surface electronic properties of the sample.

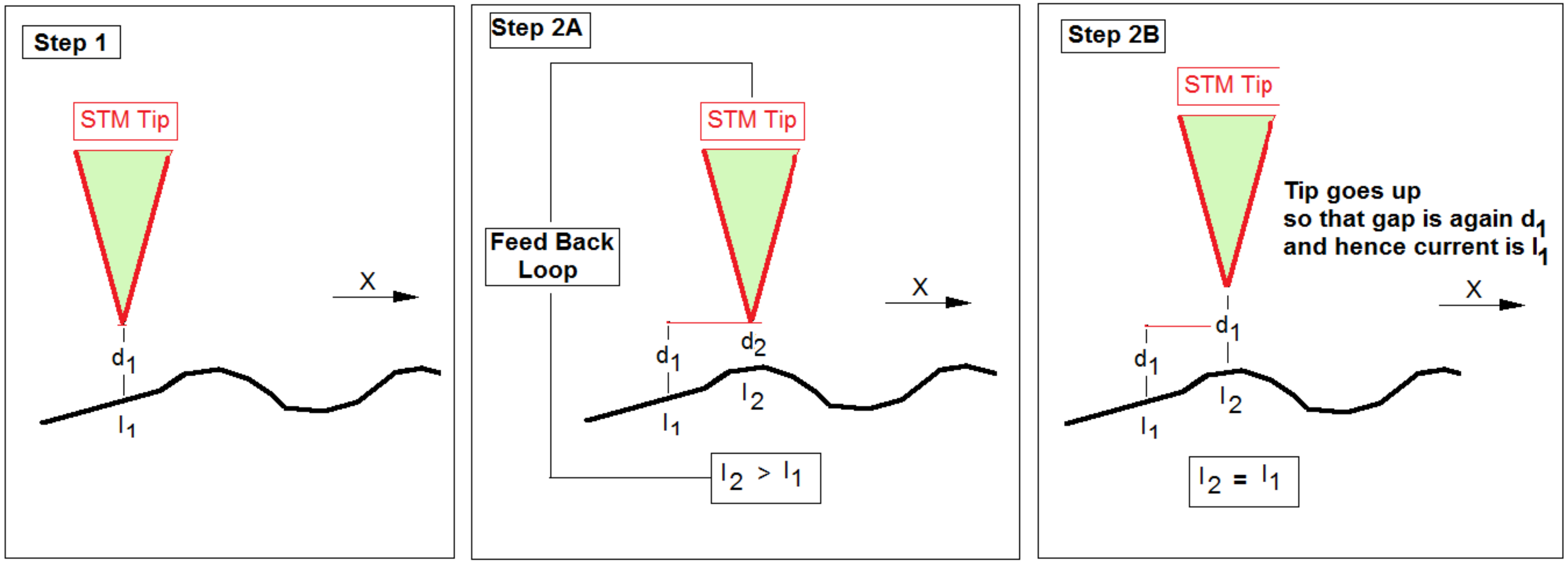
The tunneling current measured at each location on the sample surface constitute the data set, the topographic image.



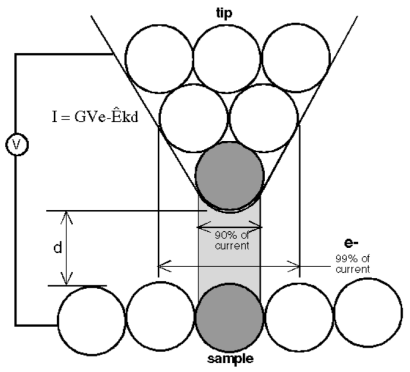
Constant Current Mode STM

In constant-current mode, STM use feedback to keep the tunneling current constant by adjusting the height of the scanner at each measurement point.

For example, when the system detects an increase in tunneling current, it adjusts the voltage applied to the piezoelectric scanner to increase the distance between the tip and the sample.



Scanning Tunneling Microscope (STM)



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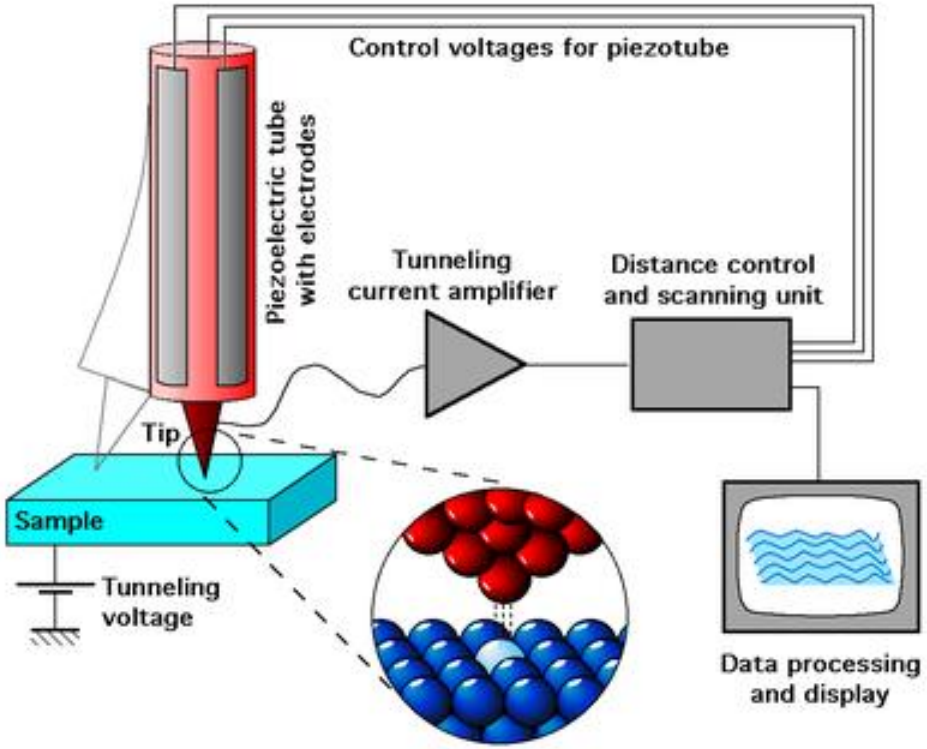
Limited to Metallic Surfaces ONLY

STM (Tunneling)

The STM is based on the concept of quantum tunneling.

When a conducting tip is brought very near to the surface to be examined, a bias (voltage difference) applied between the two can allow electrons to tunnel through the vacuum between them.



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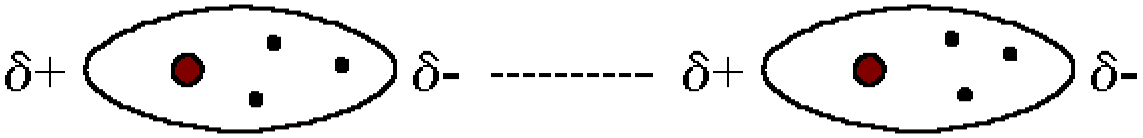
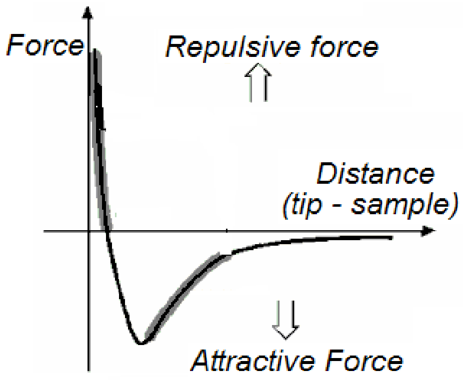
Information is acquired by monitoring the current as the tip's position scans across the surface, and is usually displayed in image form.

STM can be a challenging technique, as it can require extremely clean and stable surfaces, sharp tips, excellent vibration control, and sophisticated electronics.

What “indicator” should be used to replace “tunneling current”?

We need to identify something that is extremely generic.





- For a charge neutral sample scanned in air, the interaction force between the sample and the tip originates due to attractive vander Waal’s interaction.
- Between two molecules (or atoms), the nature of van der Waal’s force is always attractive and scales as **$1/r^6$**.
- The signature of non retarded van der Waal’s interaction can be felt over ~ 10 nm separation distance.
- Between two surfaces, the scaling or decay shows a functionality of $1/r^2$.
- The signature of the interaction extends to ~ 80 nm to 100 nm.
- This force is in the range of inter-atomic forces ~ 10^{-13} – 10^{-06} N.
- There can be host of other type of interaction forces acting between the tip and the sample like mechanical contact force, van der Waals forces, capillary forces, chemical bonding, electrostatic forces, magnetic forces Casimir forces, solvation forces etc.

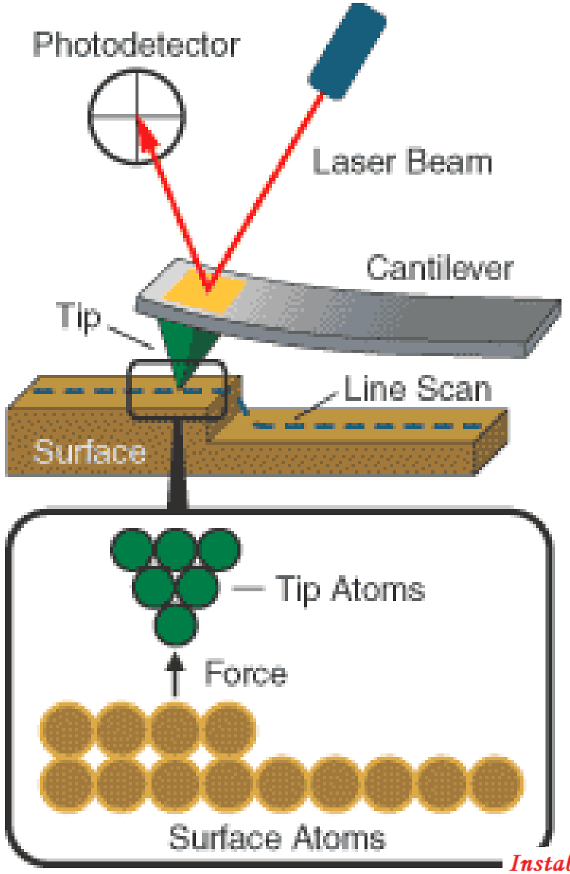
What is an AFM

• In an Atomic Force Microscope the imaging (or more accurately, the information about the topography) of a surface is done based on the modulation of interaction forces between two atoms (or molecules).

• In reality, the instrument operates based on the interaction between two surfaces, the sample and a sharp tip.

• The probing tip is attached to a cantilever and the force acting on the tip causes a small deflection of the cantilever.

• This deflection is detected and mapped as the tip scans the surface to obtain the image of the surface.

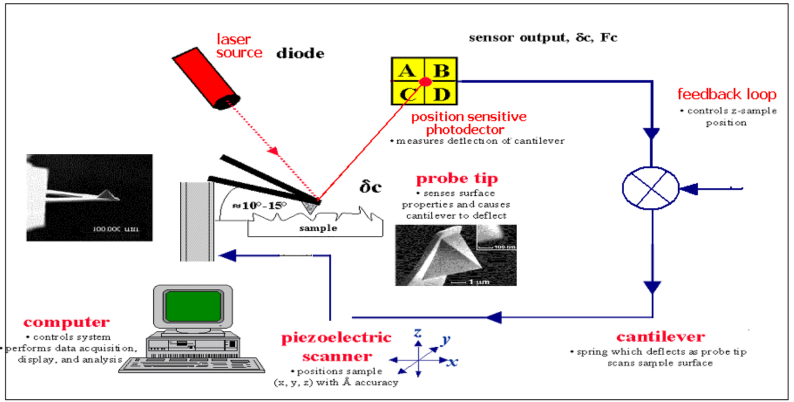


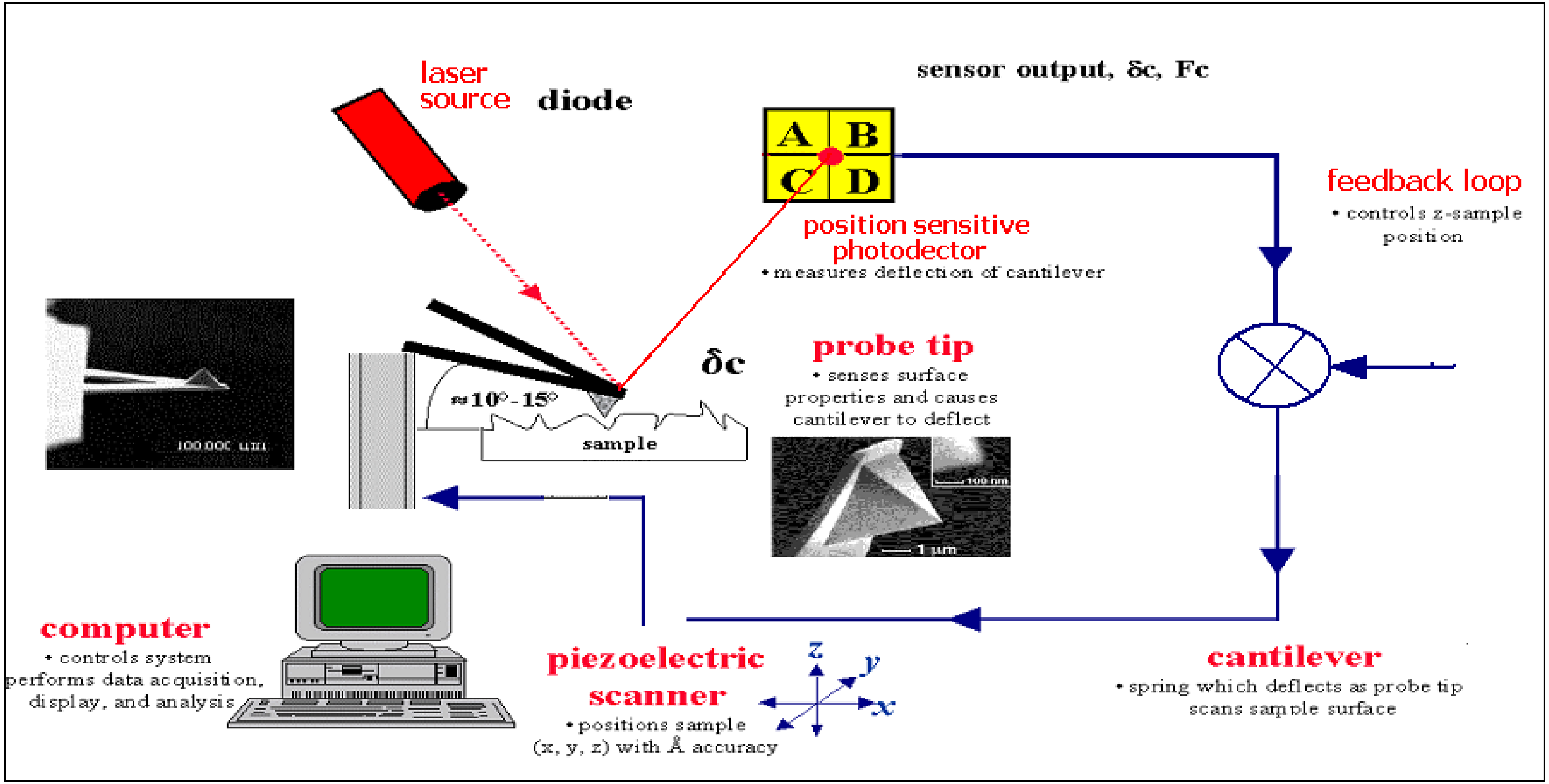
Major Components of an AFM:

- **Probes or Tips** and Cantilever for mounting the tips
- Photo detector (with laser Source)
- The piezo
- Feedback Control Module

Operational Aspects

- Alignment (Operational Aspect)
- Approach
- Sample stage and Raster Scanning
- Scanning Modes
- Data Rendering





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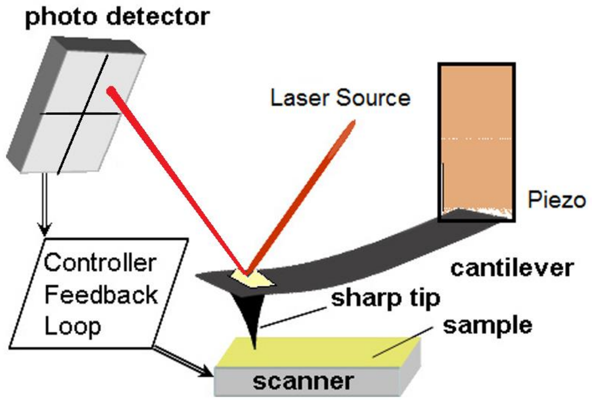
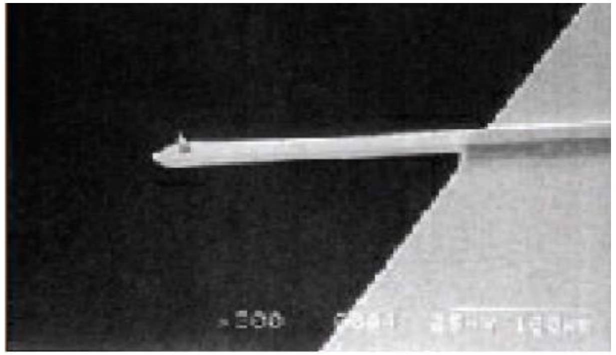
AFM Probes or Tips



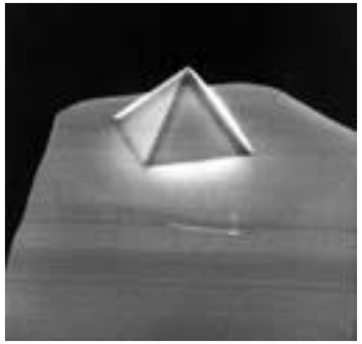
- *Tips are some sort of a sharp probe that is used to "Scan" the surface.*
- *This is the closest approximation of a single molecule* probing the surface.
- *Tip diameters are typically ~ 15 – 25 nm (Hundreds of* molecules)
- *Resolution is a major function of tip Size and Geometry*

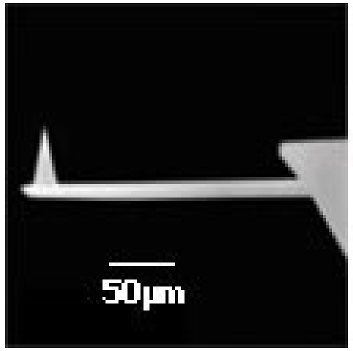


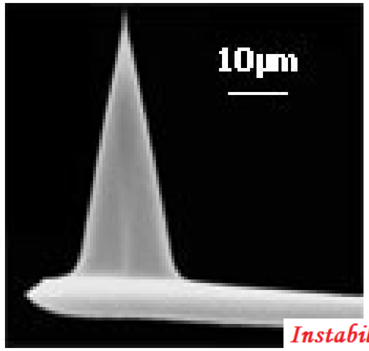
The cantilever behaves like a spring

Its stiffness is critical

Stiffness determines the spring constant







QPD (Quadrant Photo Diode)

SPD (Split Photo Diode)

PSD (Position Sensitive Photo Diode)

When a Reverse Biased P – N junction diode is illuminated with light, the reverse diode current varies linearly with the light flux. Such a P – N junction diode is called as photo diode.

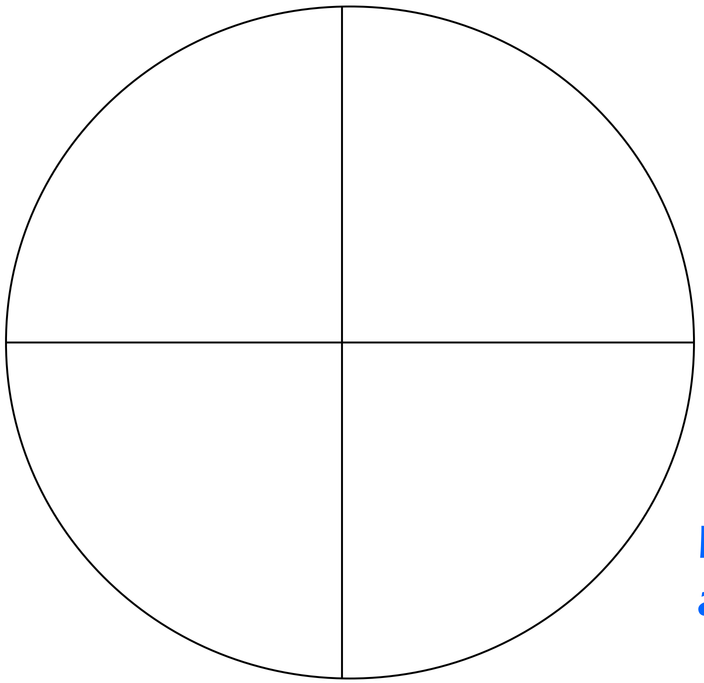


Photo Diode:

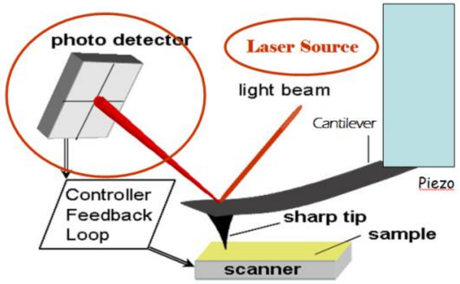
When light (of a certain wavelngth) falls on it, a voltage is generated.

Here, the voltage generated is a function of the position at which the light falls.

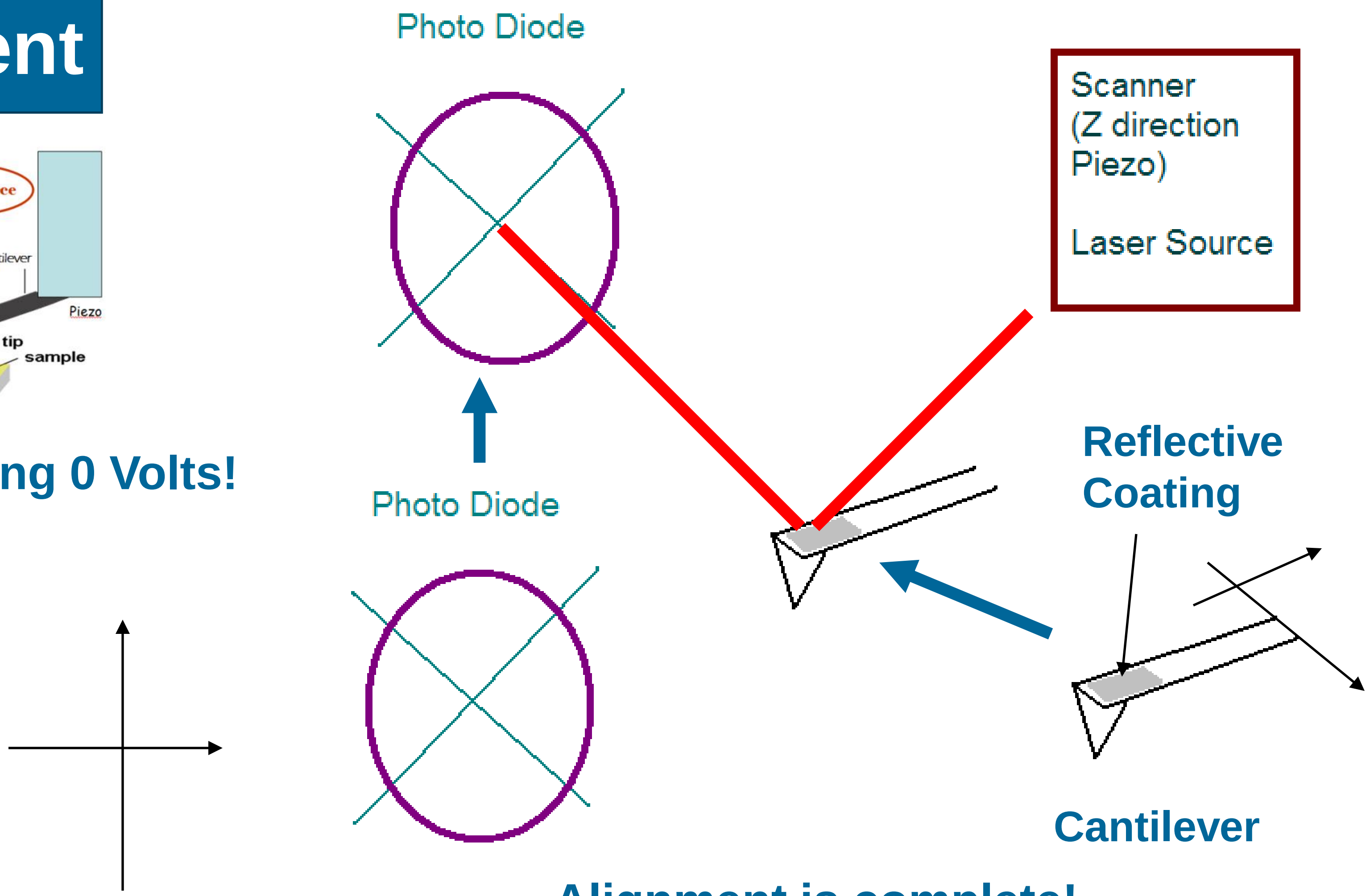
Best way to understand is to consider it as a graph paper

For most commercial AFMs, the range of the voltage is 0 -10 mV with centre being 0 mV.

Alignment



Reading 0 Volts!



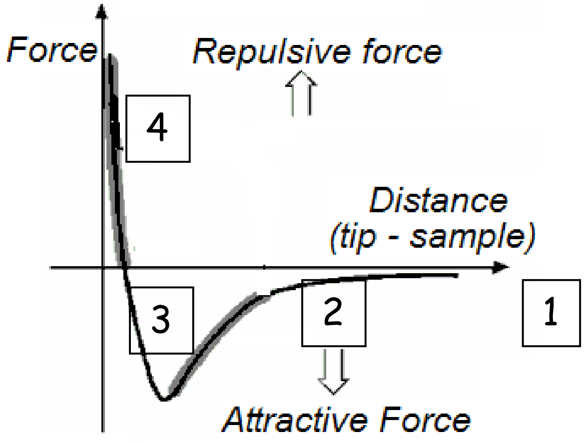
Alignment is complete!

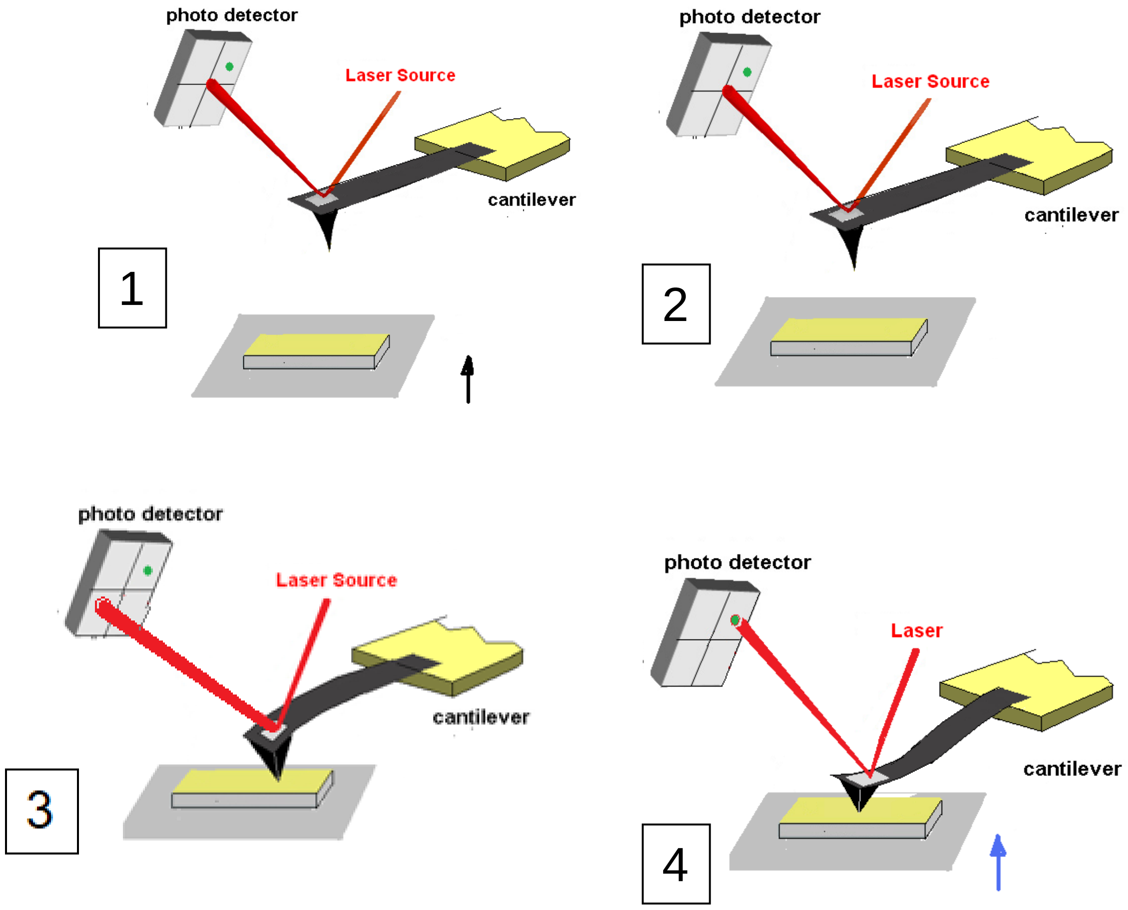
- Tip is far away (few microns to ~ mm) from sample surface.

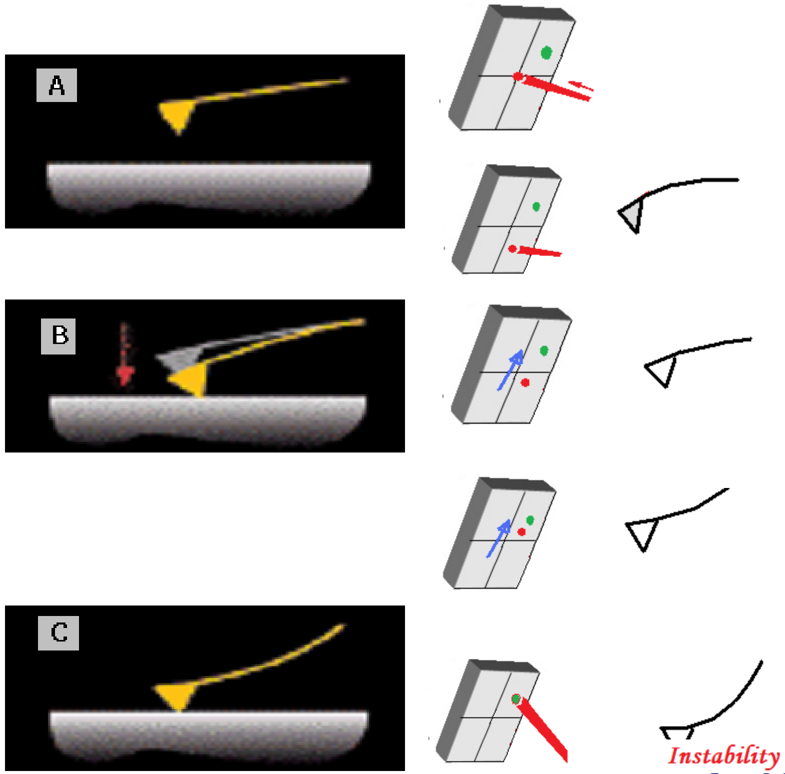
Approach

Set a Set Point

Where you want your Stepper motor to stop



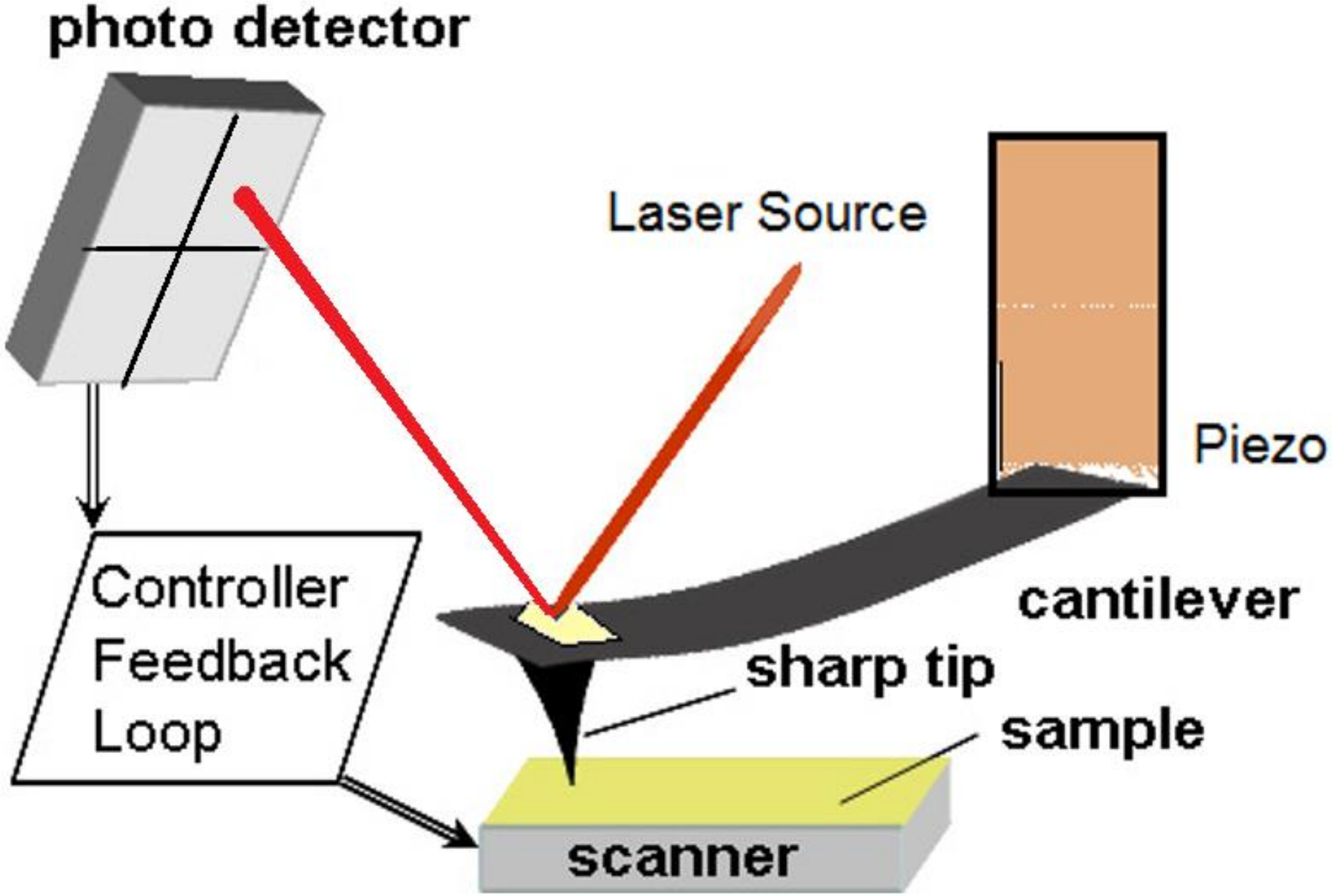


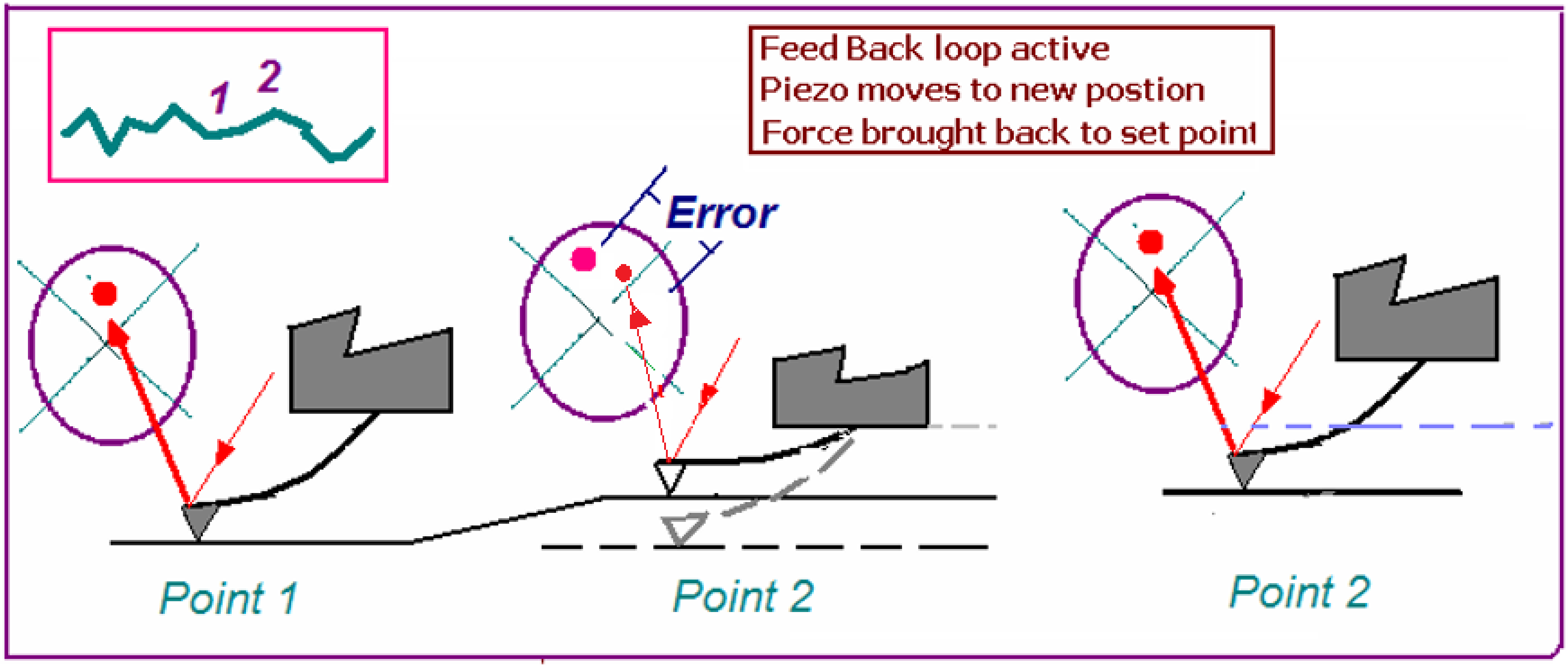


Once approach is complete the Piezo elements get activated

There are typically 3 piezos, 1 each for X and Y and 1 for Z.

Key Components





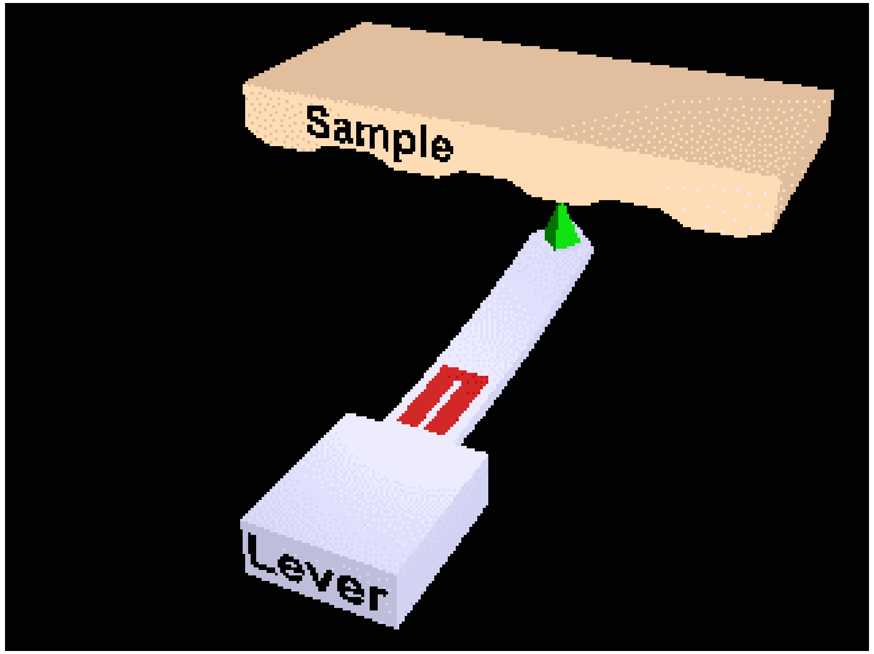
When sample has an elevation (less separation distance)

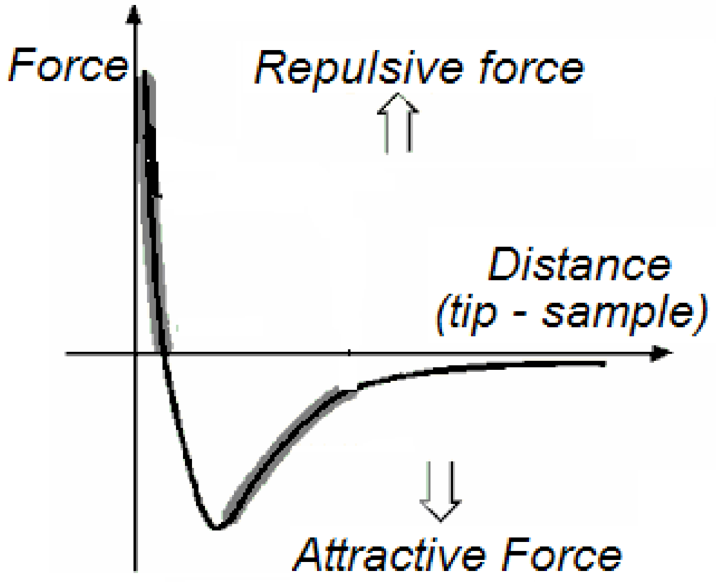
- ✓ More force exerted on the cantilever. Piezo goes up.

- Sample has a depression (separation distance more)
  - ✓ Less force exerted on the cantilever. Piezo comes down.
- Feedback loop works on the error in the Force signal
  - The deflection is converted into change in voltage and the controller, register in the feedback loop controls the force or distance and records the topography.

Contact Mode

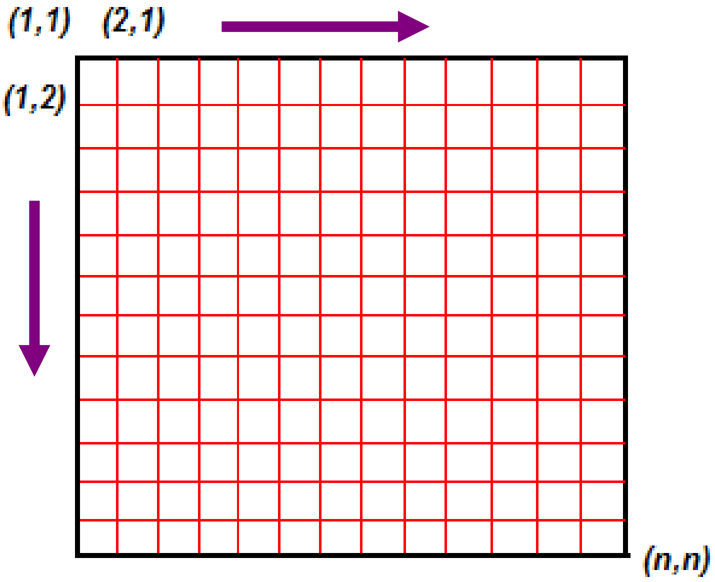
- The set point is in terms of the force of the cantilever.
- Cantilever deflects under Hooke's law: $F = -kx$, where k is cantilever spring constant.
- The scanner moves along the surface (always in contact)
- Scanning is done in the repulsive interaction regime.
- Along with the surface profile (topography) the force on the cantilever will change
- Feed back loop activated due to error in force set point



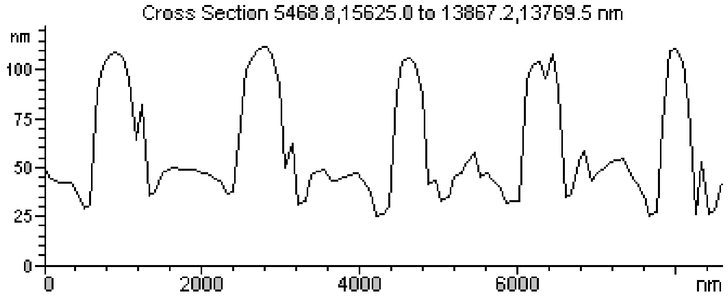


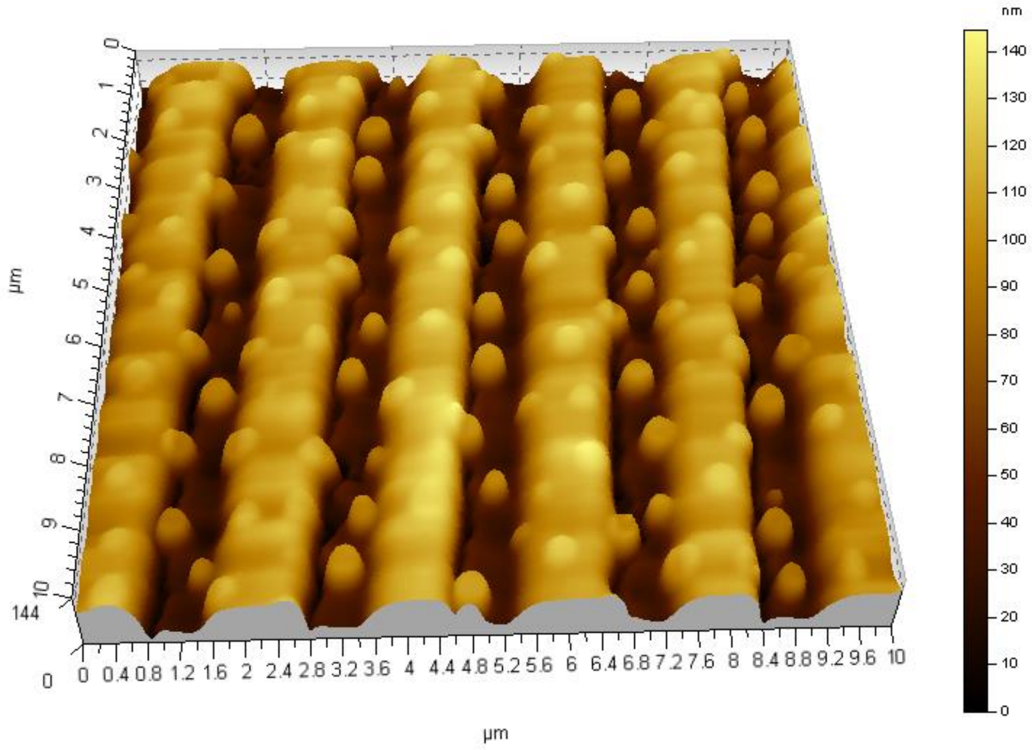
Ruster Scan and Data Rendering

- Normalize with respect to the total range
- Create a contrast ….

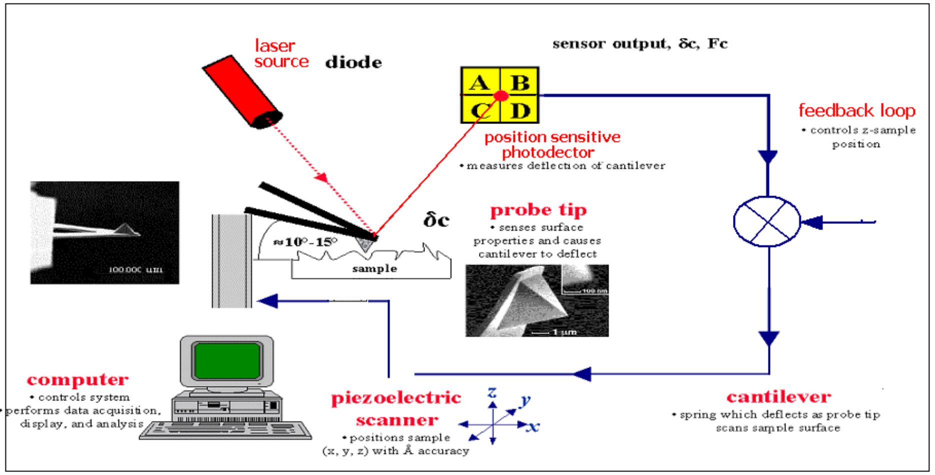


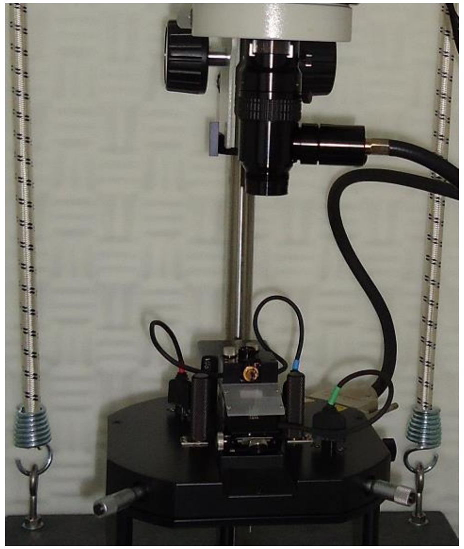
x	y	z
1	1	0.213
2	1	- 0.947
3	1	0.776
4	1	0.312
….	….	….
1	2	- 0.398
…	….	…..
n	n	0.012

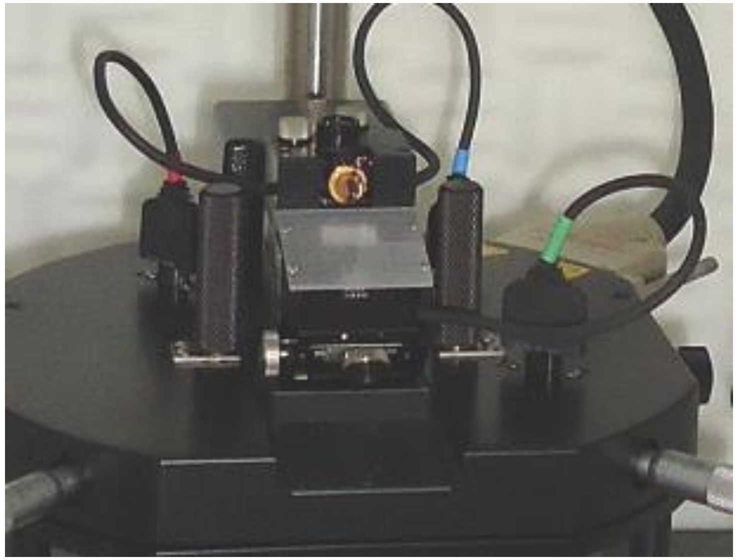




In AFM images, colors are always artificial


laser source
diode
sensor output, δc, Fc
A B
C D
feedback loop
• controls z-sample position
position sensitive photodetector
• measures deflection of cantilever
probe tip
• senses surface properties and causes cantilever to deflect
δc
≈10°-15°
sample
computer
• controls system
• performs data acquisition, display, and analysis
piezoelectric scanner
• positions sample (x, y, z) with Å accuracy
z
y
x
cantilever
• spring which deflects as probe tip scans sample surface





Tapping modes: frequency

Resonant oscillation frequency:

$$\boxed{\omega = \sqrt{\frac{k_{eff}}{m}}}$$

k_{eff}: effective force constant
m: cantilever mass

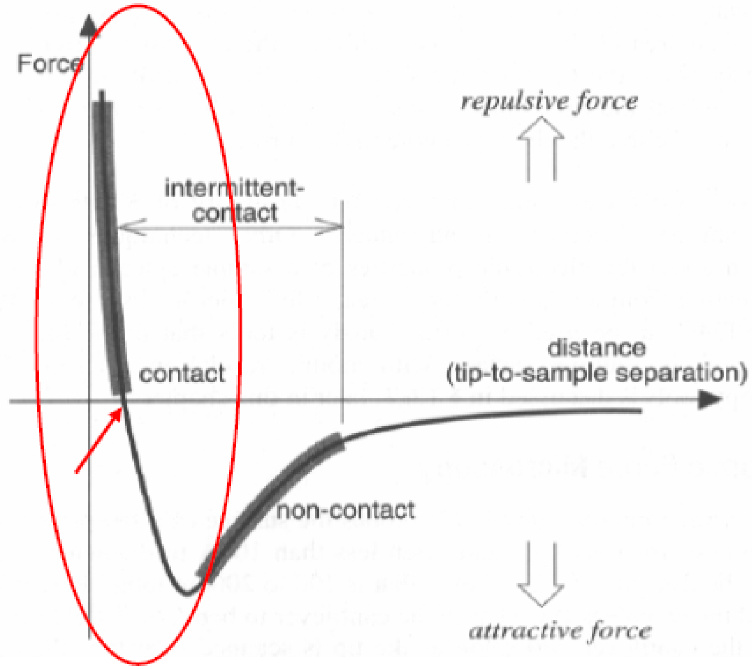
$$\boxed{k_{eff} = k - \Delta F}$$

k: force constant of cantilever
ΔF: change in the external force

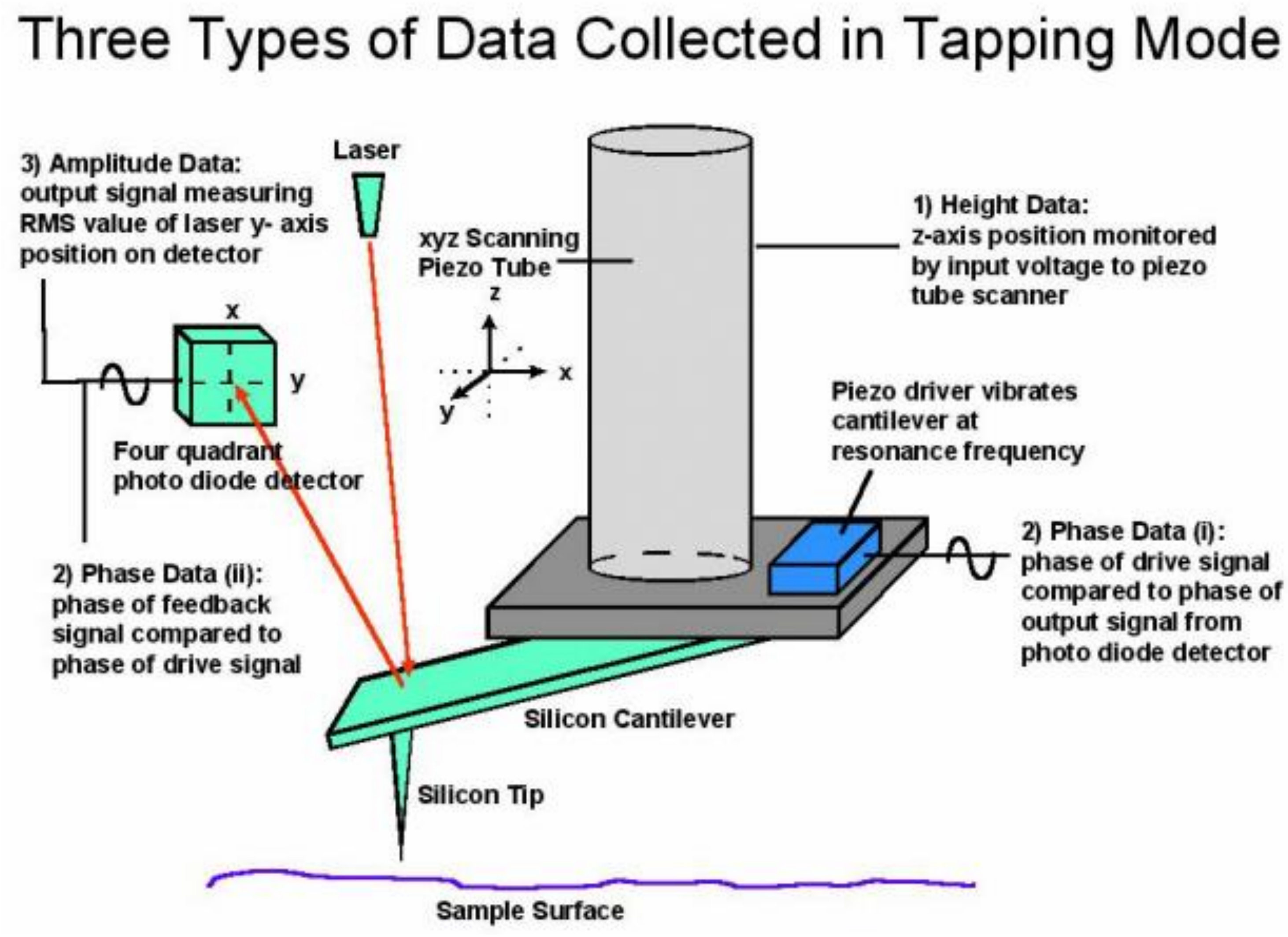
movement

$\rightarrow$ $\Delta F < 0$, k_{eff} increases, ω increases

$\leftarrow$ $\Delta F > 0$, k_{eff} decreases, ω decreases

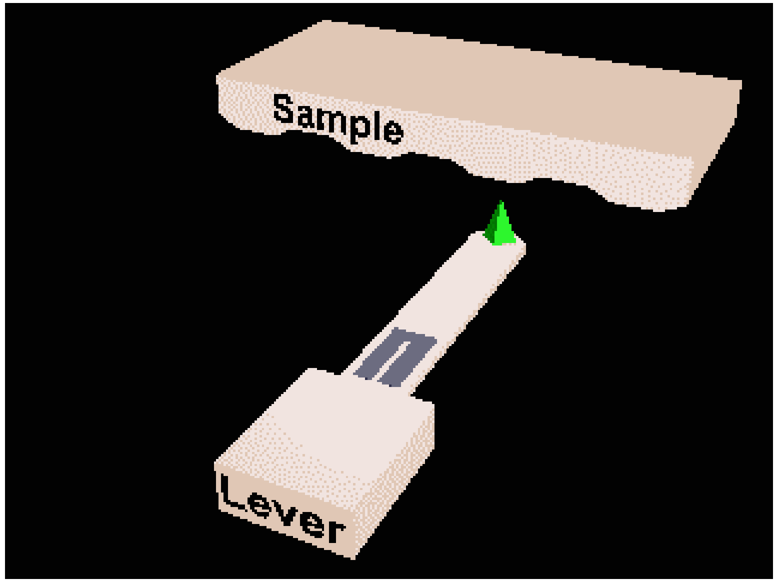


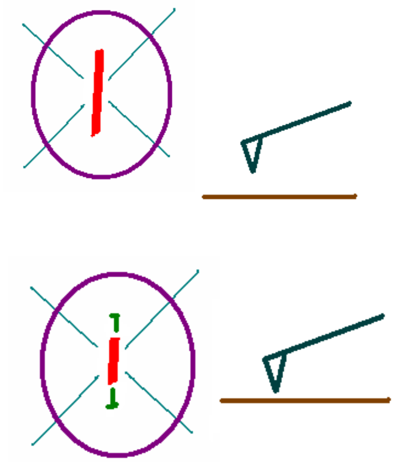
Tapping mode



Tapping Mode or Intermittent Contact Mode

- The set point is in terms of the Amplitude of the cantilever.
- The scanner moves along the surface, at a fixed amplitude
- Scanning is done in the attractive interaction regime.
- Along with the surface profile (topography) the Amplitude of the cantilever will change
- Feed back loop activated due to error in Amplitude set point

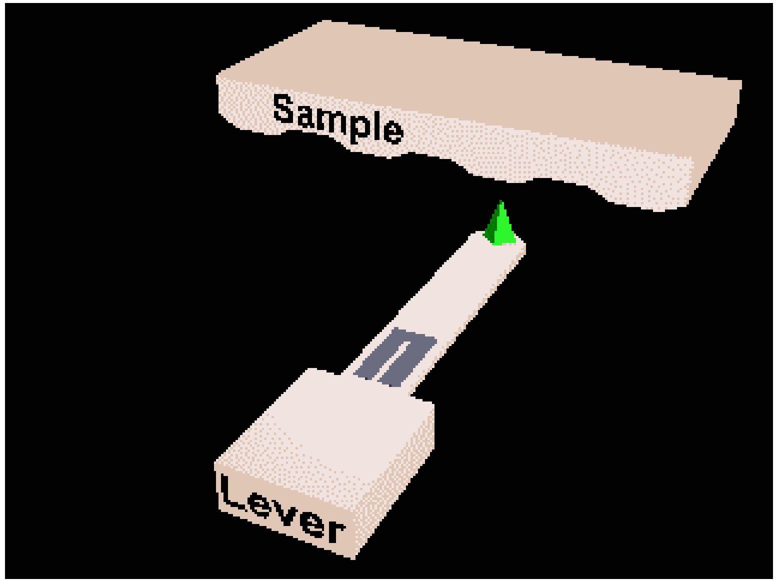




Martin, Y., et al., J. Appl. Phys. (1987) 61 (10), 4723

Tapping Mode

- When sample has an elevation (less separation distance)
  - Interaction between tip and Surface increases
    - Amplitude Decreases.
- When sample has an depression (more separation distance)
  - Interaction between tip and Surface decreases
    - Amplitude Increases



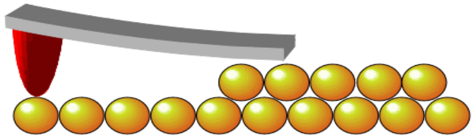
In amplitude modulation, changes in the oscillation amplitude yield topographic information about the sample.

A stiff cantilever is oscillated closer to the sample than in non-contact mode.

Part of the oscillation extends into the repulsive regime, so the tip intermittently touches or "taps" the surface.

AFM modes

contact mode



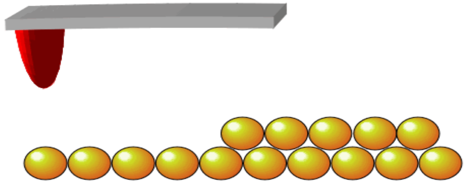
Tip angstroms from surface (repelled)

Constant force

Highest resolution

May damage surface

non-contact mode



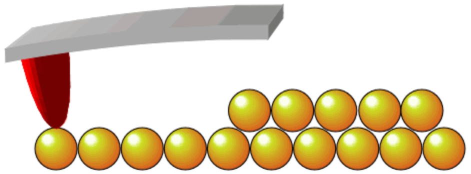
Tip hundreds of angstroms from surface (attracted)

Variable force measured

Lowest resolution

Non-destructive

tapping mode



Intermittent tip contact

Variable force measured

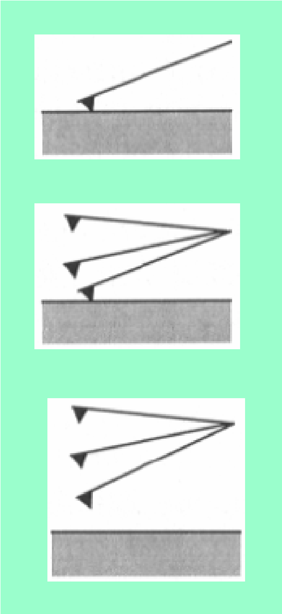
Improved resolution

Non-destructive

Courtesy of F. Ernst

Different Modes

AFM operating modes



contact mode
C

Laser beam measures the deflection of the tip
Feedback to a piezoelectroc scanner keeps force (cantilever deflection) constant.

tapping mode
IC

Tip oscillates with the amplitude of several nm
Typical frequency 50 – 400 kHz
Touches the surface at the max. amplitude
Sample is moved up/down, so that amplitude is const.

non-contact mode
NC

Tip oscillates with the amplitude of several nm
Typica frequency 50 – 400 kHz
Remains 5-10 nm from the surface
Sample is moved up/down, so that amplitude is const.
Good for ”soft” materials

This is a very difficult mode to operate in ambient conditions with the AFM. The thin layer of water contamination which exists on the surface on the sample will invariably form a small capillary bridge between the tip and the sample and cause the tip to "jump-to-contact".

Contact Mode

Advantages

- High scan speed
- Atomic resolution can be obtained on a hard surface
- Can image fairly rough samples

Disadvantages

- Capillary bridge formation very likely in ambient conditions.
- Chances of damage to the scanning surface in case of soft samples (Polymer and Bio-logical samples particularly!)
- Chances of damage to the tip for hard rough samples

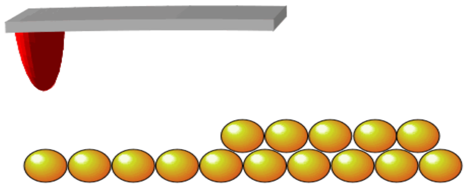
Tapping Mode - Advantages

- High lateral resolution
- No damage to sample and the cantilever tip
- Can image fairly rough samples
- Ideal for sampling in liquid cells.

Disadvantages

- Resolution a shed poorer than contact mode.
- Lesser scan speed than contact mode
- Micro ripples can be generated while scanning under liquid.

Non Contact mode



- Tip hundreds of angstroms from surface (attracted)
- Variable force measured
- Lowest resolution
- Non-destructive
- Rarely used as a stand alone imaging mode.

Courtesy of F. Ernst

A Good Cantilever .. .. ..

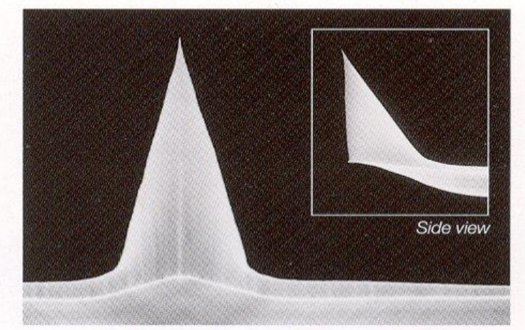
One of the most important factors influencing the resolution which may be achieved with an AFM is the sharpness of the scanning tip.

- In order to measure small (10^{-12} – 10^{-5} N), the spring constant should be as small as possible. A stiff cantilever will not respond (show no deflection) to very small forces.
- The cantilever's resonance frequency (f) (~10-800 kHz) should be higher than the instrument's data acquisition rate.
- The best tips may have a radius of curvature of only around 5nm.
- Mode of operation.

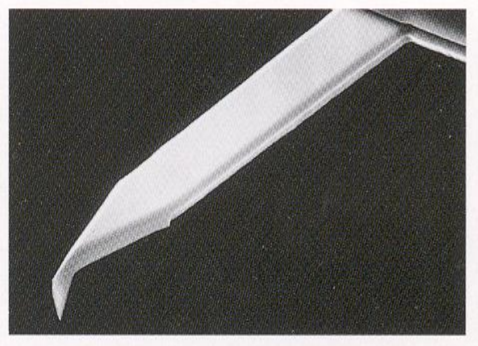
    a) Contact mode: low force constant

    b) Non contact mode: high force constant.

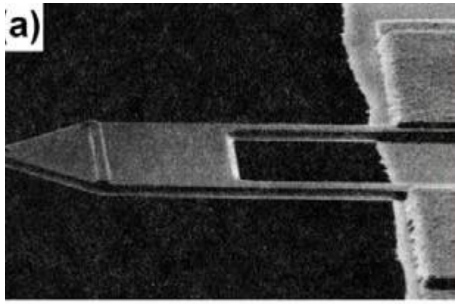
AFM Probes: Some Typical Examples



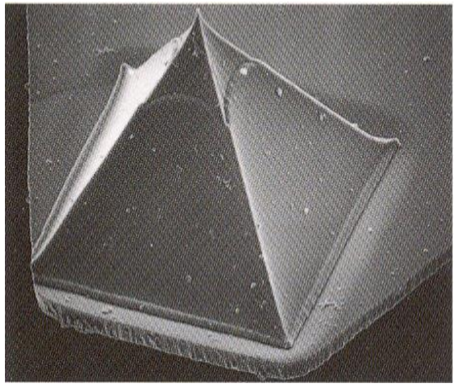
Tip Shape: Tetrahedral
Height: 10mm
Radius: <10nm
Material: Si



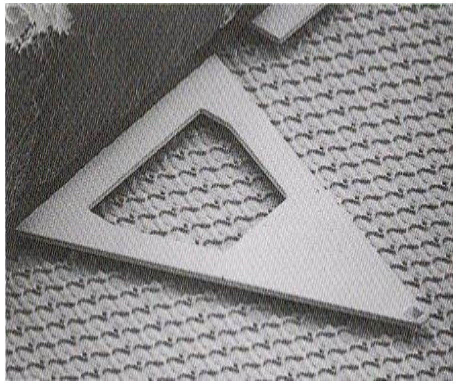
Cantilever Shape: Rectangular
Length:120mm
Thickness: 2.8mm
k : 20N/m; f = 300kHz; Material: Si



Piezo resistive cantilever (amplitude can be adjusted)



Tip Shape: Pyramidal
Height: 2.9mm
Radius: <20nm
Material: SiN



Cantilever Shape:Triangular
Length:200mm
Thickness: 0.8mm
k : 0.18N/m, f = 27 kHz; Material: SiN

The first tips used by the inventors of the AFM were made by gluing diamond onto pieces of aluminum foil.

AFM Probes or Tips

- Contact Mode : Silicon Nitride
- Tapping Mode : Etched Silicon
- Spring Constant : 0.10 ~ 1.0 N/m
- Freq (Tapping) : 50 – 400 KHz
- Tip Radius : 5 to 20 nm
- Cantilever Length : 100 ~ 200 μm
- Commercial probe tip smallest ~ 6 nm.
- Now Carbon Nano Tube (CNT) is being used as an AFM tip.

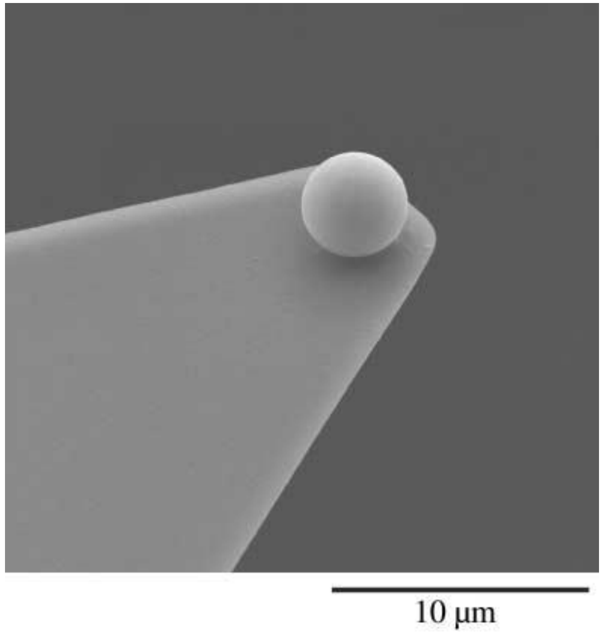
- **Tip senses the Force across the sample.**
- **Cantilever responds to this force by deforming.**
- **Next step is to track this deflection of the cantilever (spring)**

Colloidal Probe

A colloidal particle is attached to the front of the cantelever, unlike a sharp probe.

These tips are specifically used for measuring forces between tip and sample surfaces.

Adhesion force measurements

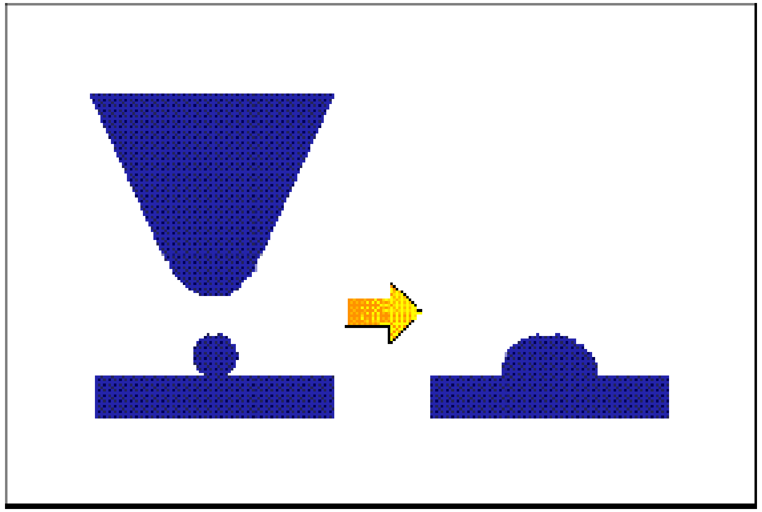


Tip Convolution

One of the most important factors influencing the resolution which may be achieved with an AFM is the sharpness of the scanning tip. best tips may have a radius of curvature of only around 5nm. The need for sharp tips is normally explained in terms of ***tip convolution***.

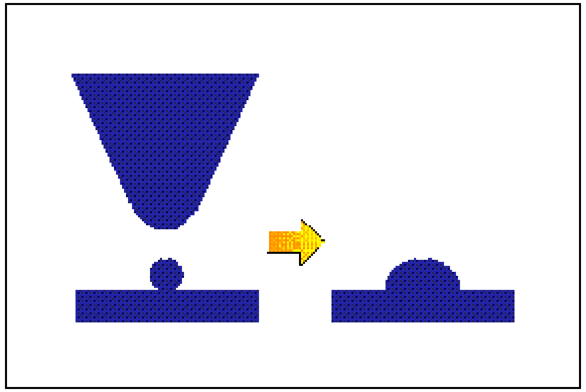
Tip broadening arises when the radius of curvature of the tip is comparable with, or greater than, the size of the feature to be imaged.

The diagram illustrates this problem; as the tip scans over the specimen, the sides of the tip make contact before the apex, and the microscope begins to respond to the feature. This is what we may call tip convolution.

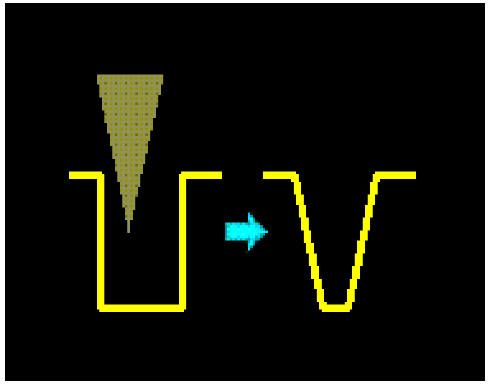


Tip Effects

- The need for sharp tips is normally explained in terms of *tip convolution*.
- This term is often used (slightly incorrectly) to group together any influence which the tip has on the image.
- The main influences are
- broadening (*r* tip is comparable to feature size)
- compression
- interaction forces
- Aspect ratio



Broadening



Aspect Ratio

Instability and Soft Patterning Laboratory
भारतीय प्रौद्योगिकी संस्थान खड़गपुर
Indian Institute of Technology
Kharagpur

AFM: Beyond Topographic Imaging

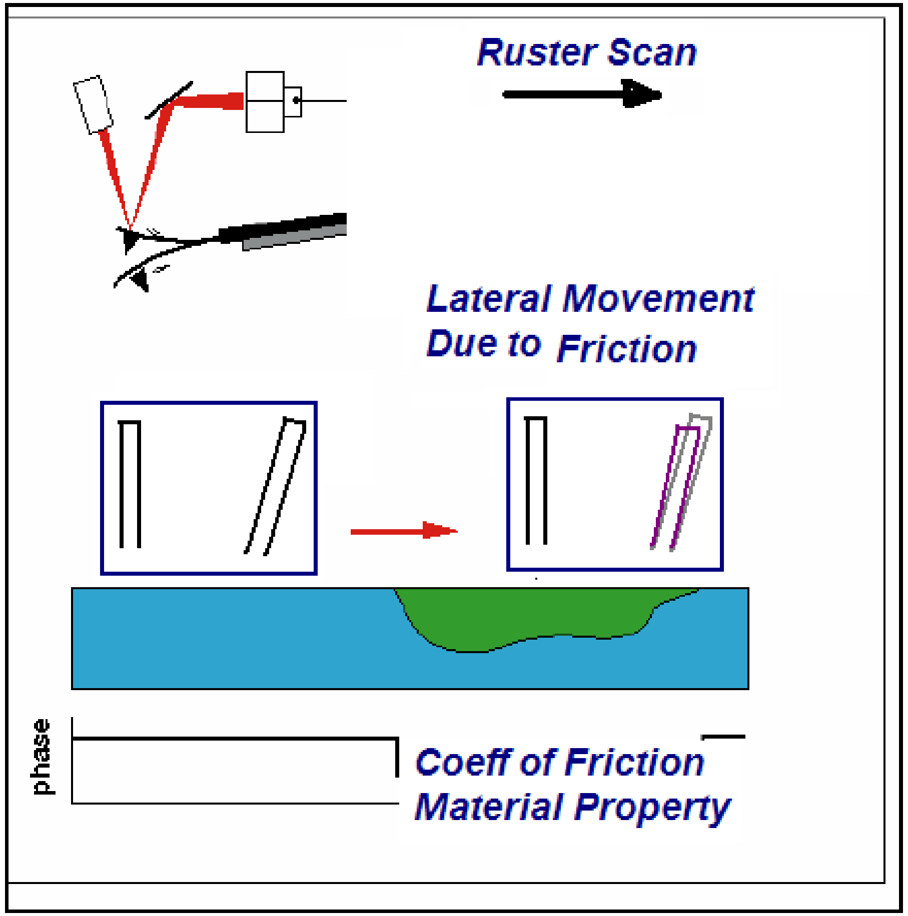
"The Art of SPM: Scanning Probe Microscopy in Material Science"

J. Loos, Advanced Materials, 17, 1821, 2005

AFM: Beyond Topographic Imaging

Mode	*Function*
Lateral Force Microscopy (LFM):	Friction
Phase Imaging:	Phase
Pulse Force Mode	Stiffness, Adhesion
Magnetic Force Microscopy (MFM):	Magnetic Domains
Electric Force Microscopy (EFM):	Electric Field, Charge
Thermal Resistance Microscopy	
Nanoindentation /Scratching:	Mechanical Properties
Dip pen, fountain pen Lithography:	Patterning
Spectroscopy:	Force Measurement
Atomic Manipulation	(more common with STM)

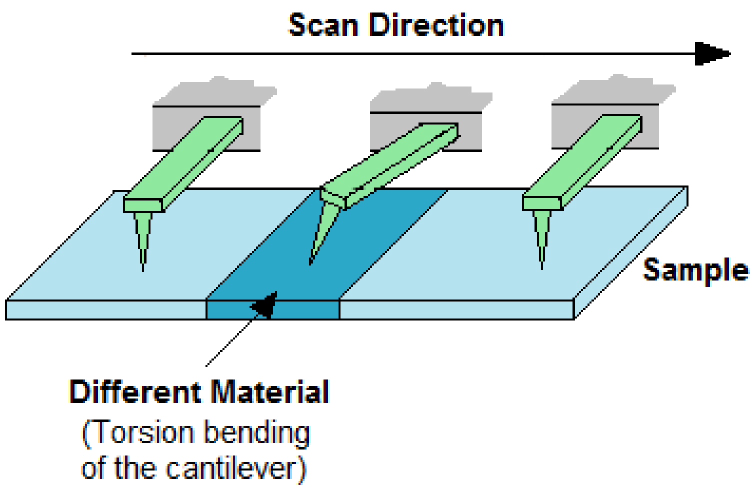
Lateral Force Microscopy (LFM)



- Powerful extension of Contact Mode AFM.
- Goes beyond Topographical imaging to detect:
  - Friction
  - Adhesion
  - Mapping of different components of composites, block co-polymers etc.
  - Mapping of different surface energy domains.
- It measures the torsional deformation of the probe, rather than its vertical deflection.

Lateral Force Microscopy (LFM)

- During scanning in contact mode the cantilever bends not only along normally to the surface but also the cantilever torsional deformation occurs. (Twisting)
- LFM measures the torsional deformation of the cantilever during scanning in contact mode.
- The lateral deformation depends on frictional force acting on tip.
- LFM studies are useful for imaging variations in surface friction that can arise from in homogeneity in surface.



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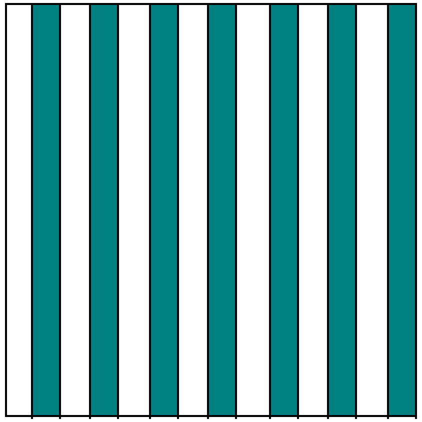
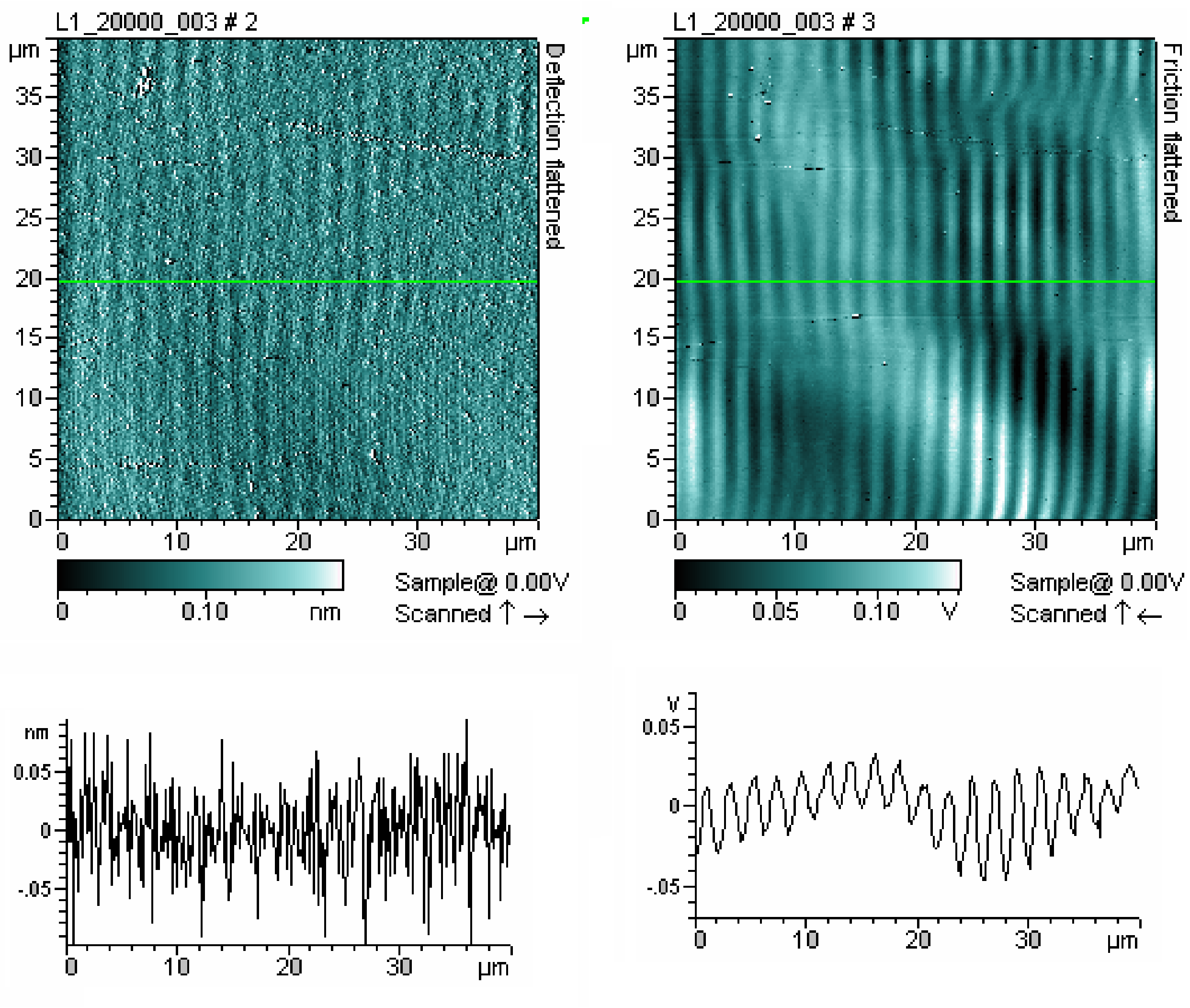
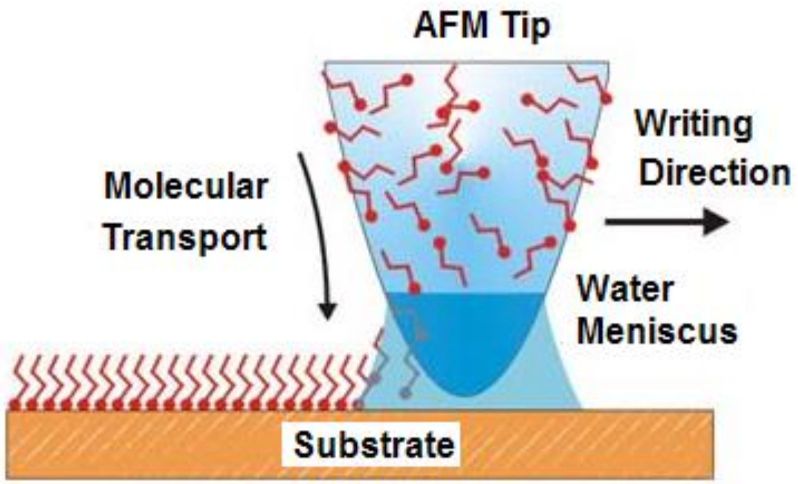
Lateral Force Microscopy (LFM)

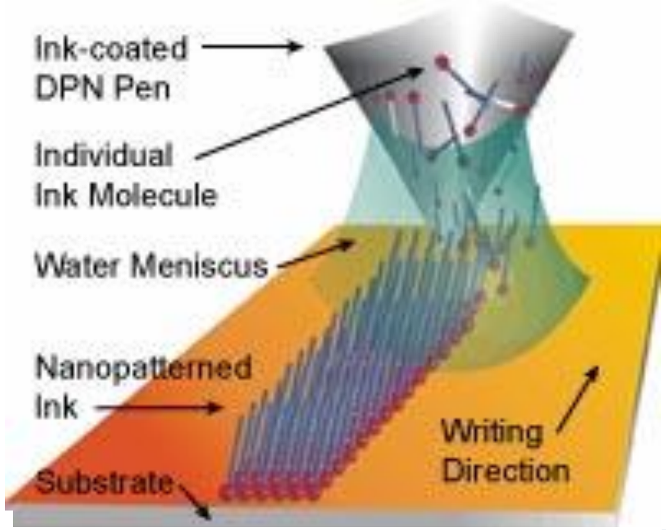


- Alternate array of low and high surface area domains
- Monolayer of thiol molecules
- Micro contact printing



Dip Pen Lithography

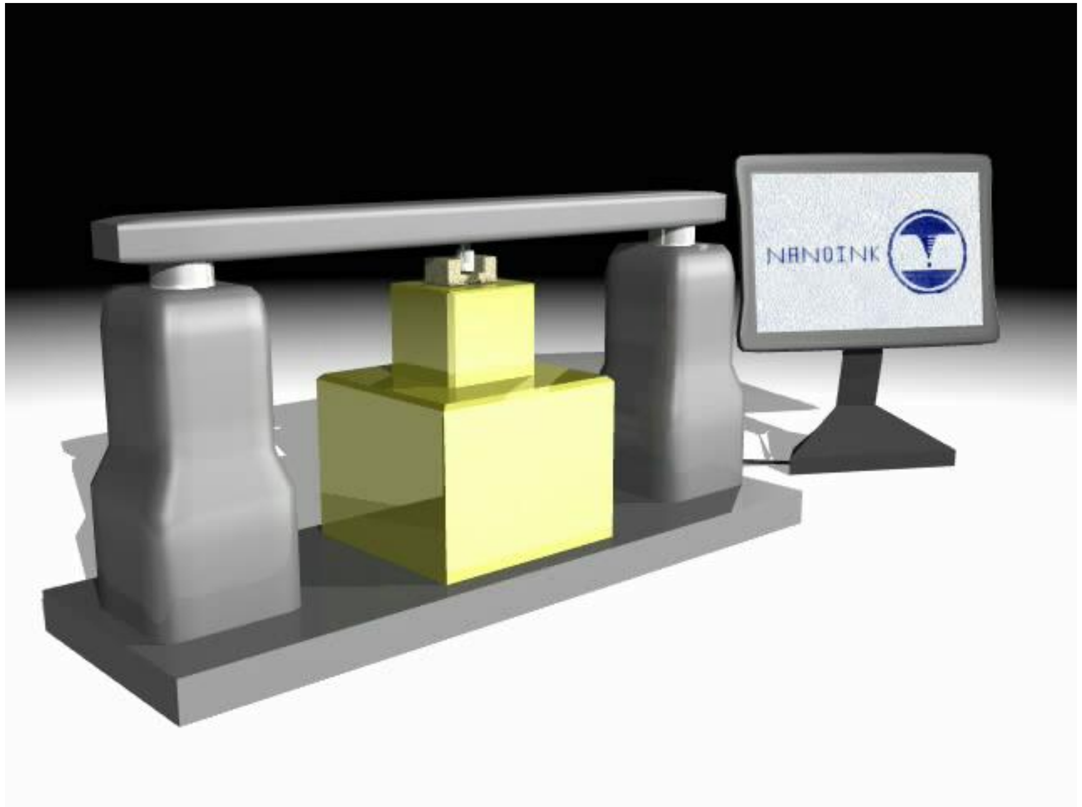




- Program your own Pattern!
- Direct and single step process.
- No requirement of Mask, Resist Layer.
- Tool: conventional atomic force microscope (AFM)
- Ultra-high resolution features with line widths as small as 10-15 nm with ~ 5 nm spatial resolution.
- Selective functionalization of surfaces with patterns of two or more components is possible.

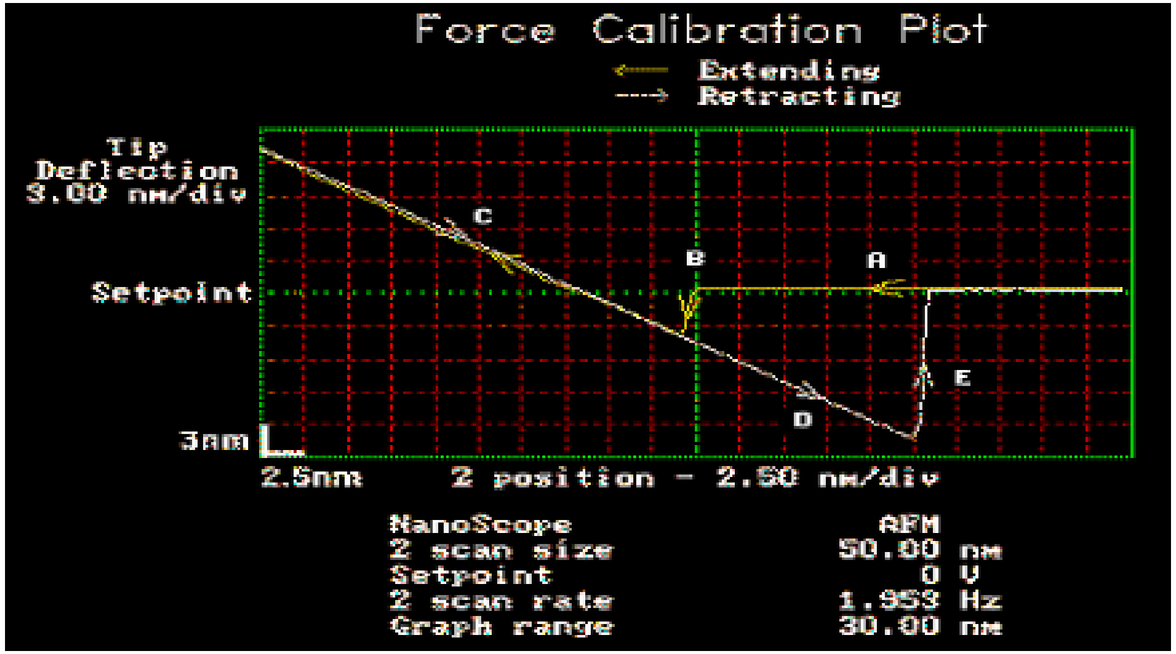
Chad Mirkin, http://www.chem.northwestern.edu/~mkngrp/dpn.htm

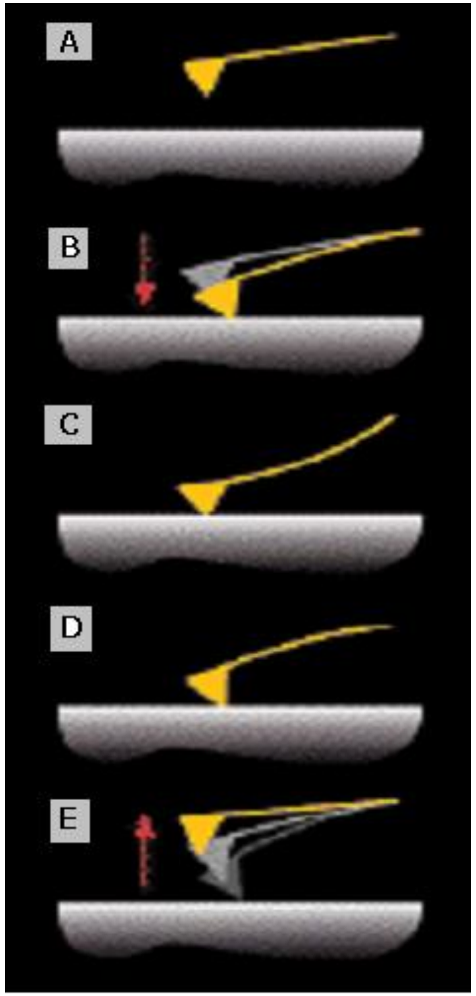
Dip Pen Lithography



Nano Writing

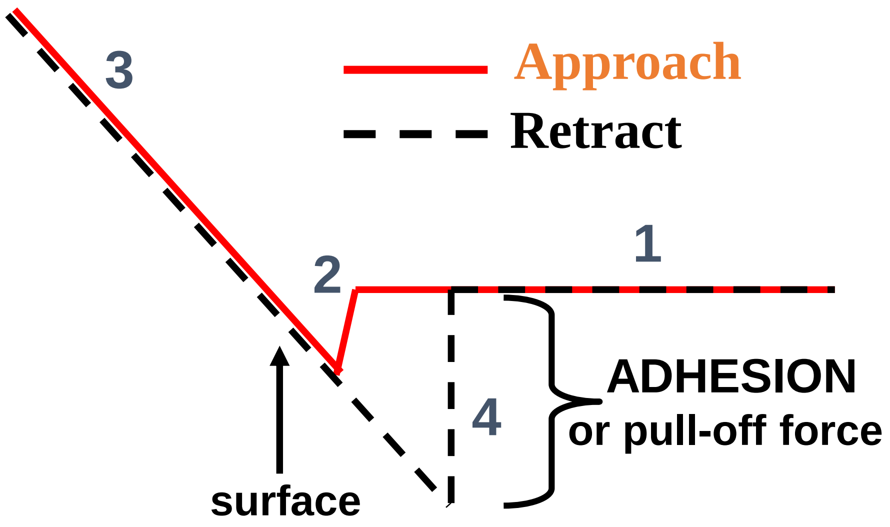
Force Measurement

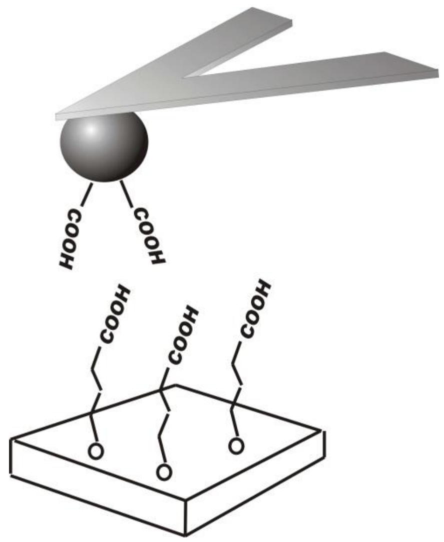




- The tip separation distance is slowly reduced (Approach) and then once the deflection in the cantilever reaches zone of constant compliance, it is slowly taken off.
- The jump to contact regime & pull off regime gives idea about the adhesion
- There might be repulsion all the way in presence of surfactants

Force Measurement





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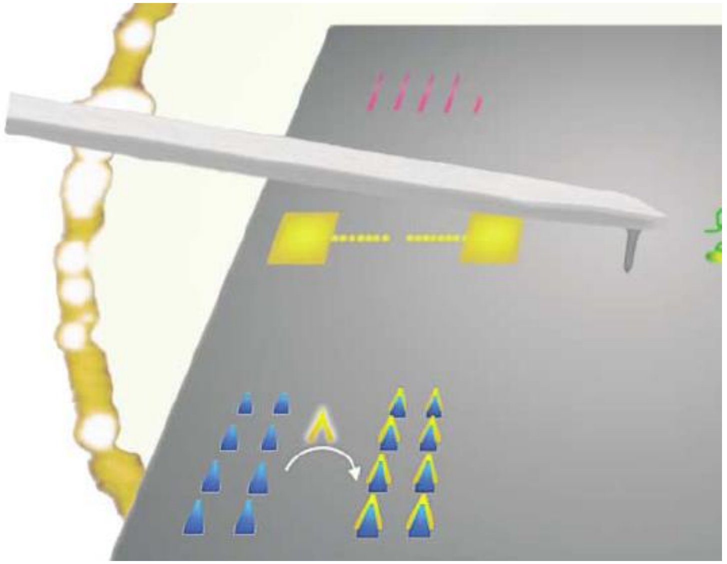
Advantages of AFM

- Lateral resolution allows imaging & measurements of features on the order of a few nano-meters, the vertical (height) resolution is ~ 1 Å.
- In-situ Scanning in Different environment including ambient, UHV, under liquid, high temperature
- This is a non destructive scanning procedure
- Material Properties characterization can be done using AFM including surface force measurement, Topography, Adhesion, Visco-elasticity, Hardness, friction, Magnetic Domains etc.
- Apart from imaging the surface, AFM can also be used to measure the extant of the interaction forces.
- Unlike STM, (which is the ancestor of the AFM) which can only image conducting surfaces, AFM can be used on any kind of surfaces.
- Unlike traditional microscopes, scanned-probe systems do not use lenses, so the size of the probe rather than diffraction effects generally limit their resolution.

Concluding Remarks:

- Imaging, fabrication, manipulation, property measurement, topography ….
- It is not only a characterization tool, but much beyond that.



Couple of good references:

1. Advanced Materials, 17, 1821, 2005.
2. Mechatronics, 14, 907, 2004.
3. Chem. Rev. Volume 97, Issue 4.
4. Angew. Chem. Int. Ed. 43, 248, 2004.

Thank You

Some Salient Points in Simple Words

- While you “see” in other microscopes, you actually “touch” the sample in an AFM!
- The simplest (and probably the biggest difference!): You get “DIRECT” information about the topography (X, Y and Z directions).
- Not only one gets to look at but one can “DO” things at the surface.