Scanning Probe Microscope :

STM / AFM / SNOM

Nanophysics – Characterisation of Nanomaterials by SPM (AFM/STM/SNOM)

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A Family of Microscopes

SPM (air, liquid, vacuum)

AFM

Contact Modes
- Topography
- Lithography

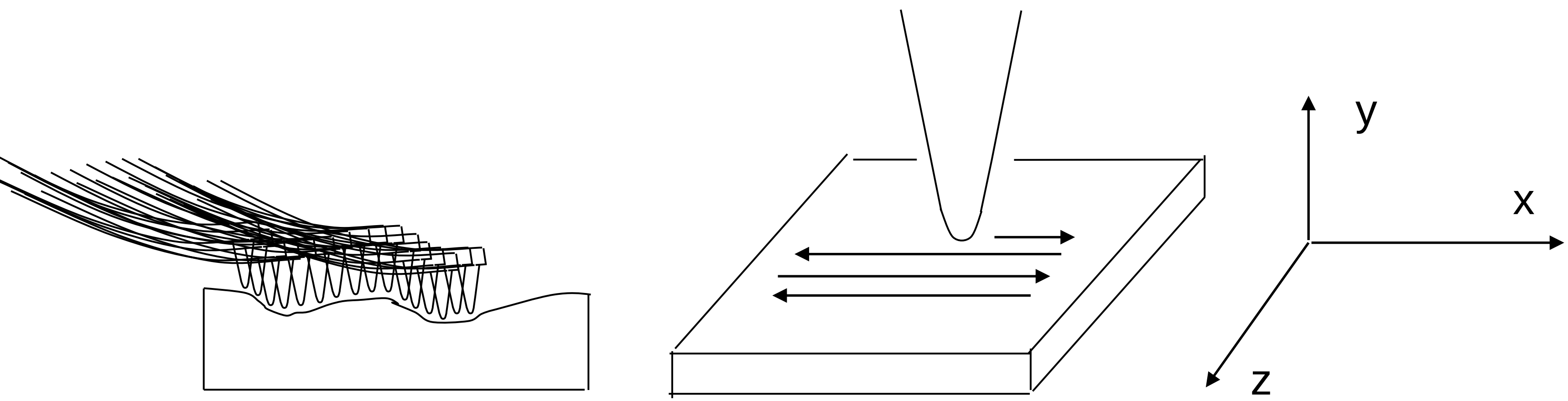
Non-contact (true and tapping)
- Topography
- MFM, EFM
- SKPM
- Others

STM
- Topography
- Spectroscopy

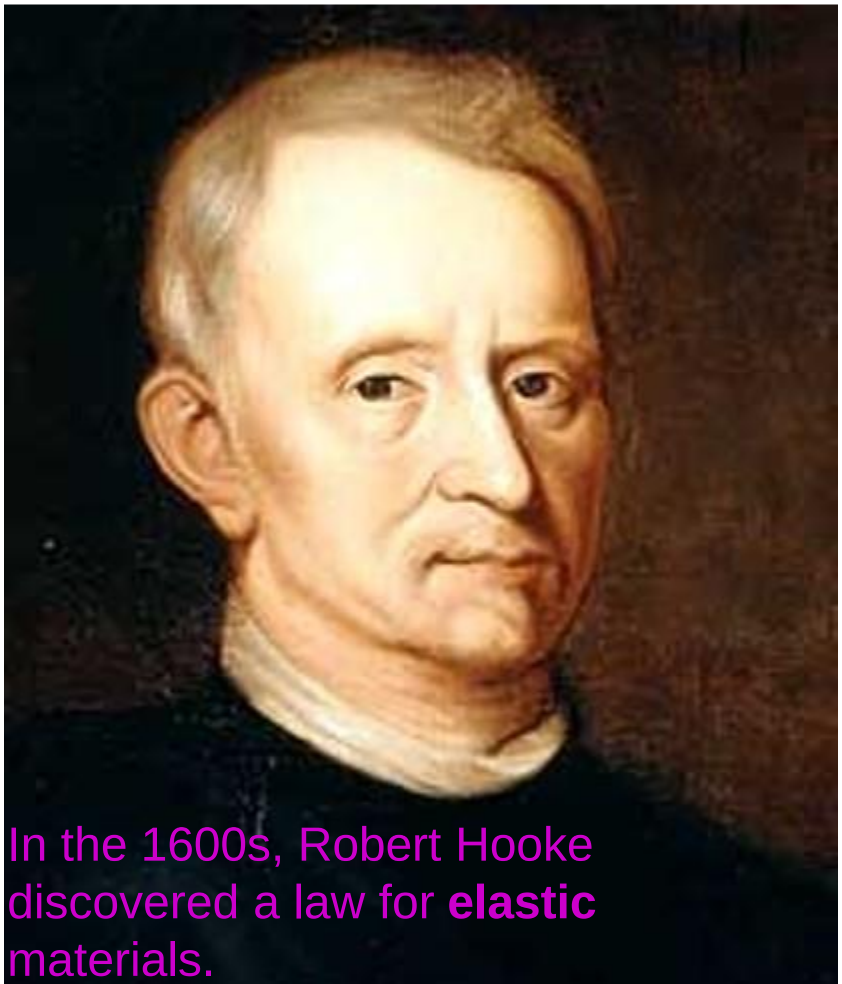
SNOM(NSOM)
- Aperture
- Aperatureless
- Reflection
- Transmission

What is SPM ?

- SPM is a mechanical imaging instrument in which a small probe is rastered over a surface. By monitoring the motion of the probe (deflected due to various forces of interaction), the surface topography and/or images of surface physical properties are measured.



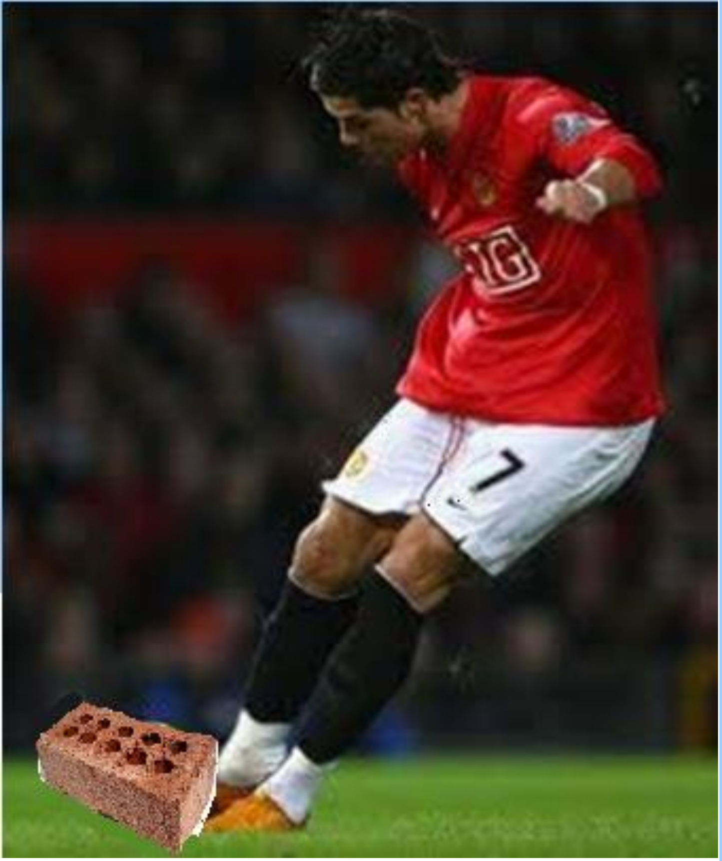
Hooke's Law

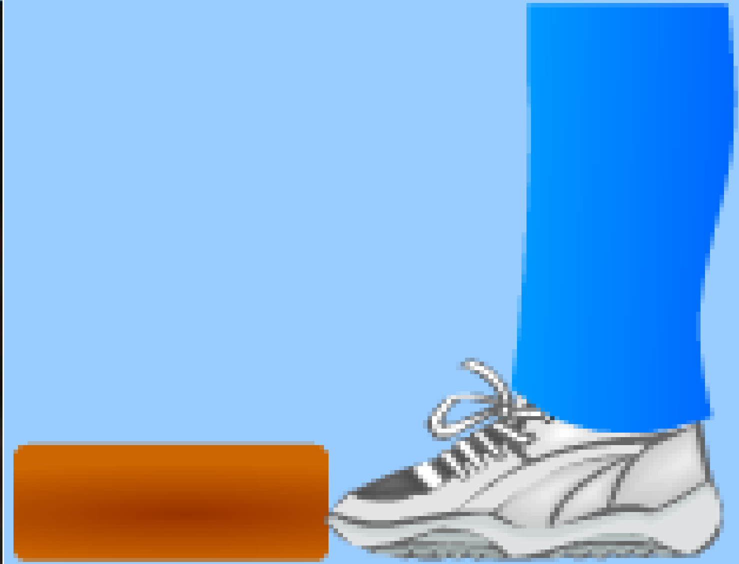

In the 1600s, Robert Hooke
discovered a law for **elastic**
materials.


The ball squashes giving a longer collision time

Andy O’Brien

his nose showing elastic behaviour





The brick doesn't squash giving a shorter collision time.

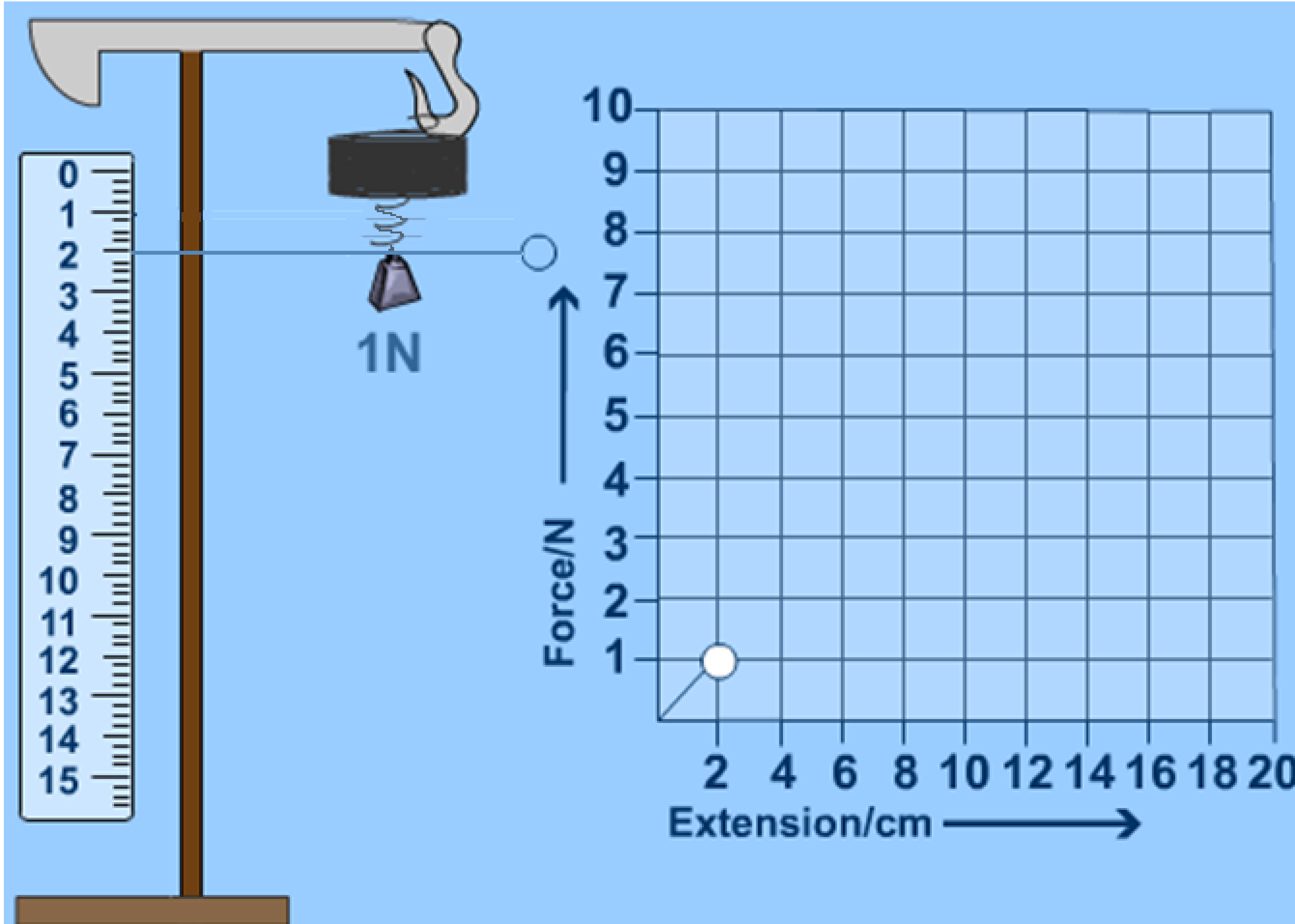
Hooke's Law, elastic and plastic behaviour

- If a material returns to its original size and shape when you remove the forces stretching or deforming it (reversible deformation), we say that the material is demonstrating **elastic behaviour.**

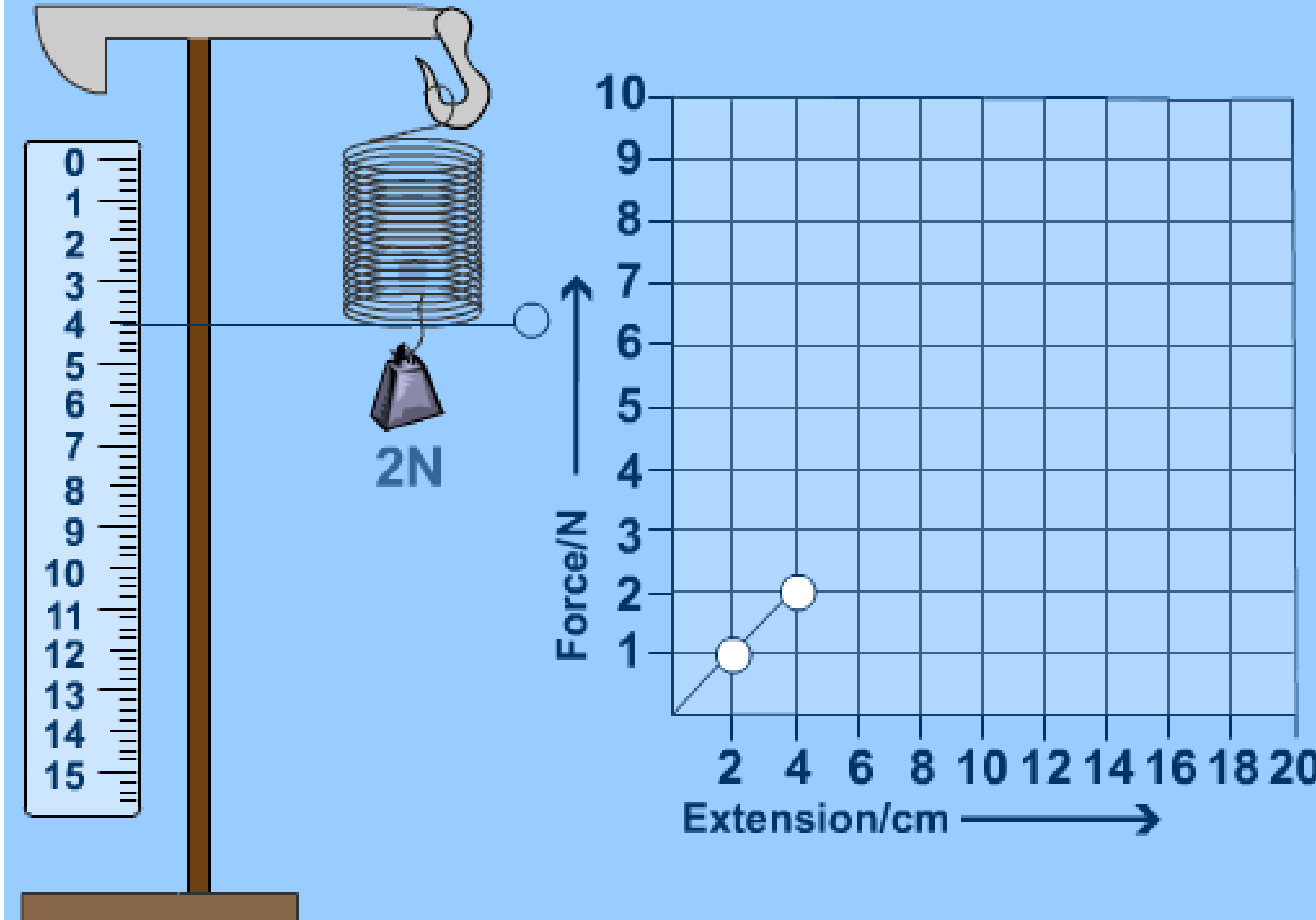
- A **plastic** (or **inelastic**) material is one that stays deformed after you have taken the force away. If deformation remains (irreversible deformation) after the forces are removed then it is a sign of **plastic behaviour.**

- If you apply too big force a material will lose its elasticity.

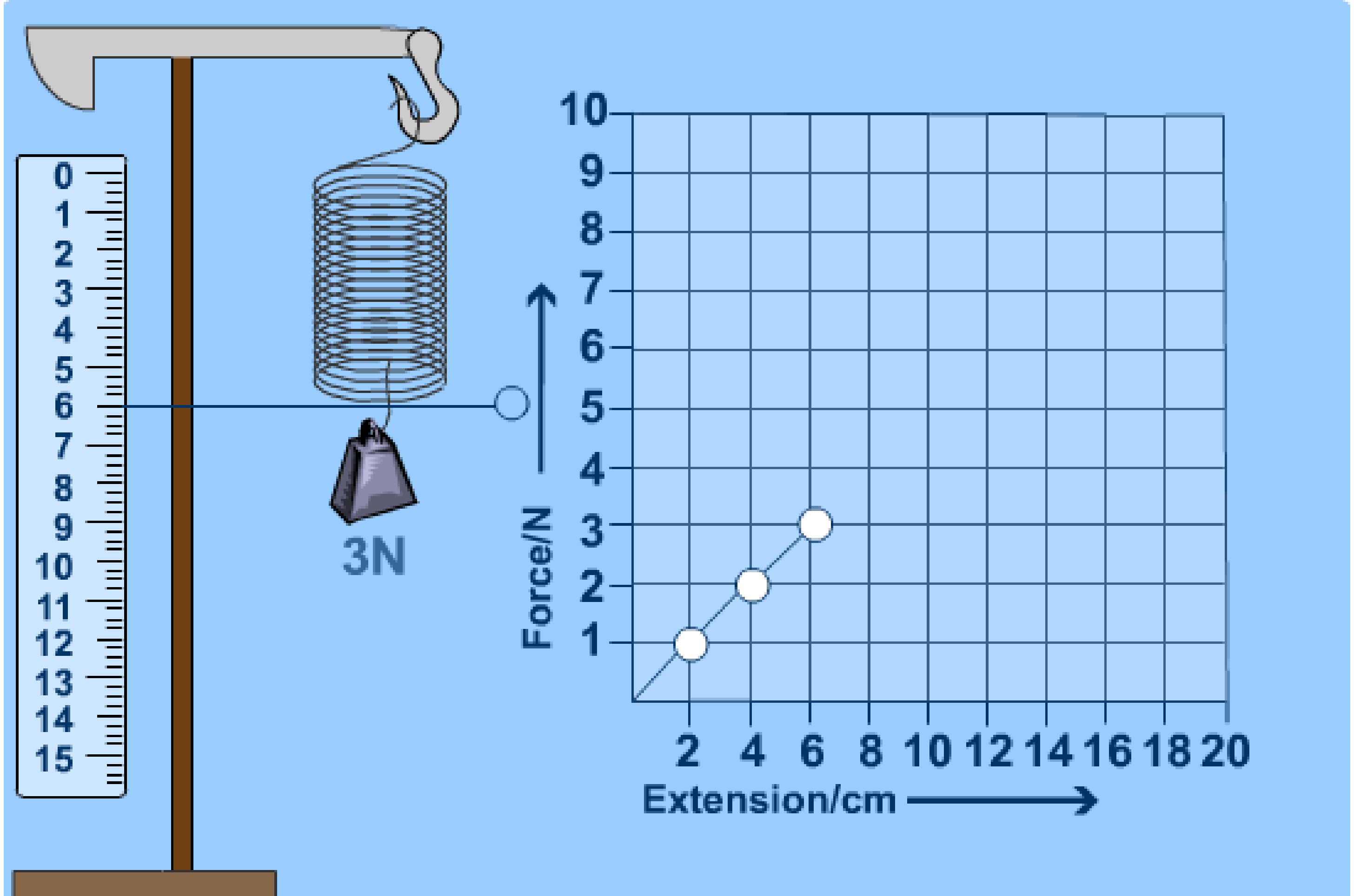
- Hooke discovered that **the amount a spring stretches is proportional to the amount of force applied to it.** This means if you double the force its extension will double, if you triple the force the extension will triple and so on.



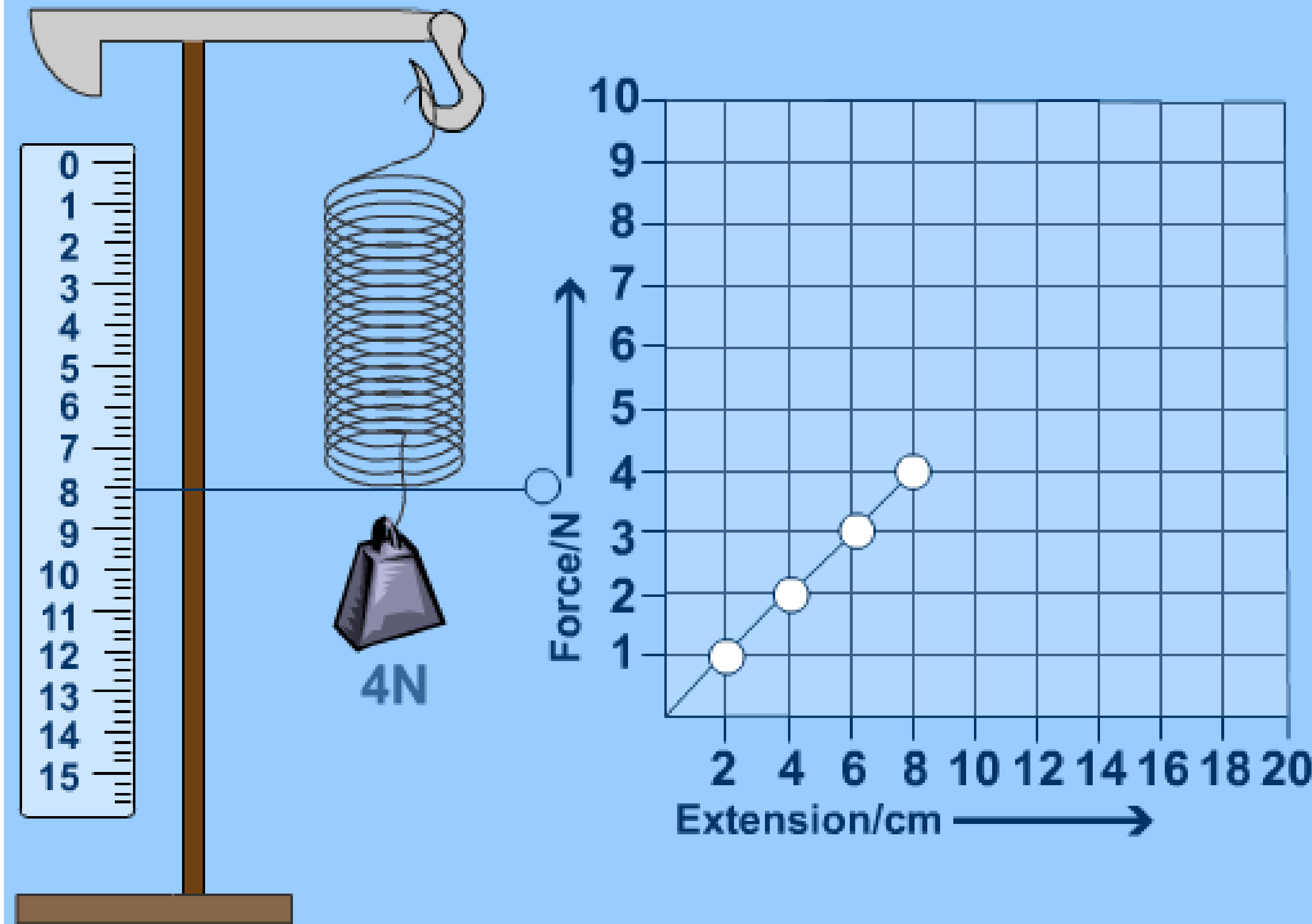
Weight in Newtons	1N	2N	3N	4N	5N	6N
Extension cm	2					



Weight in Newtons	1N	2N	3N	4N	5N	6N
Extension cm	2	4				

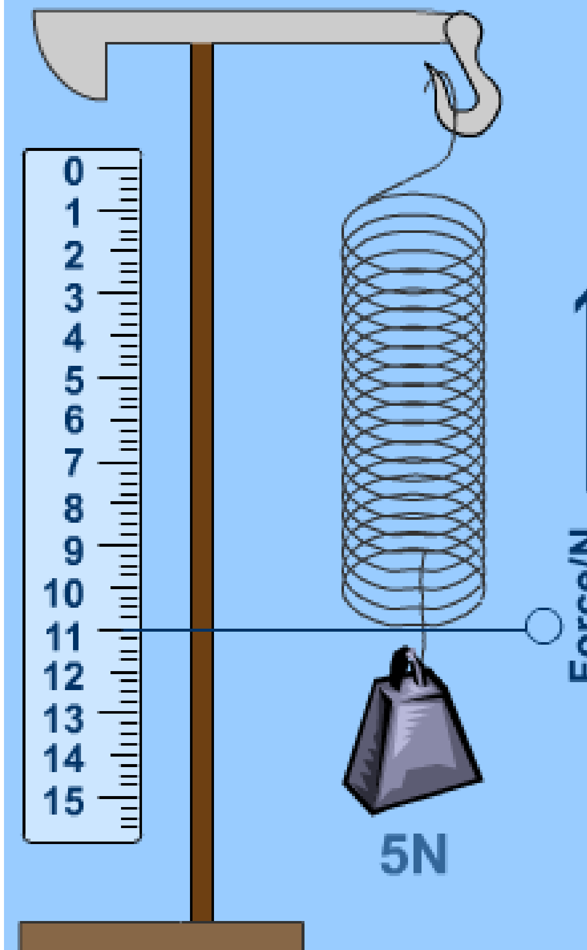


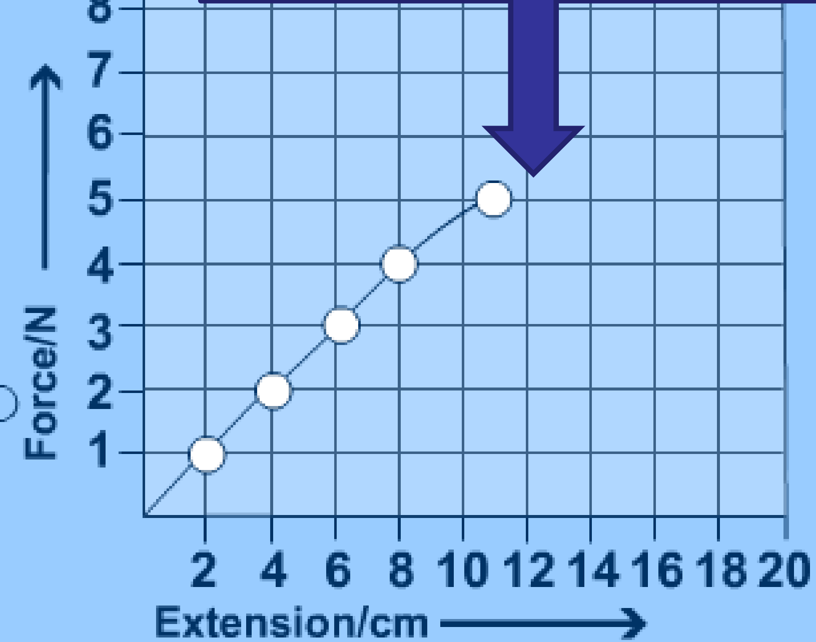
Weight in Newtons	1N	2N	3N	4N	5N	6N
Extension cm	2	4	6			



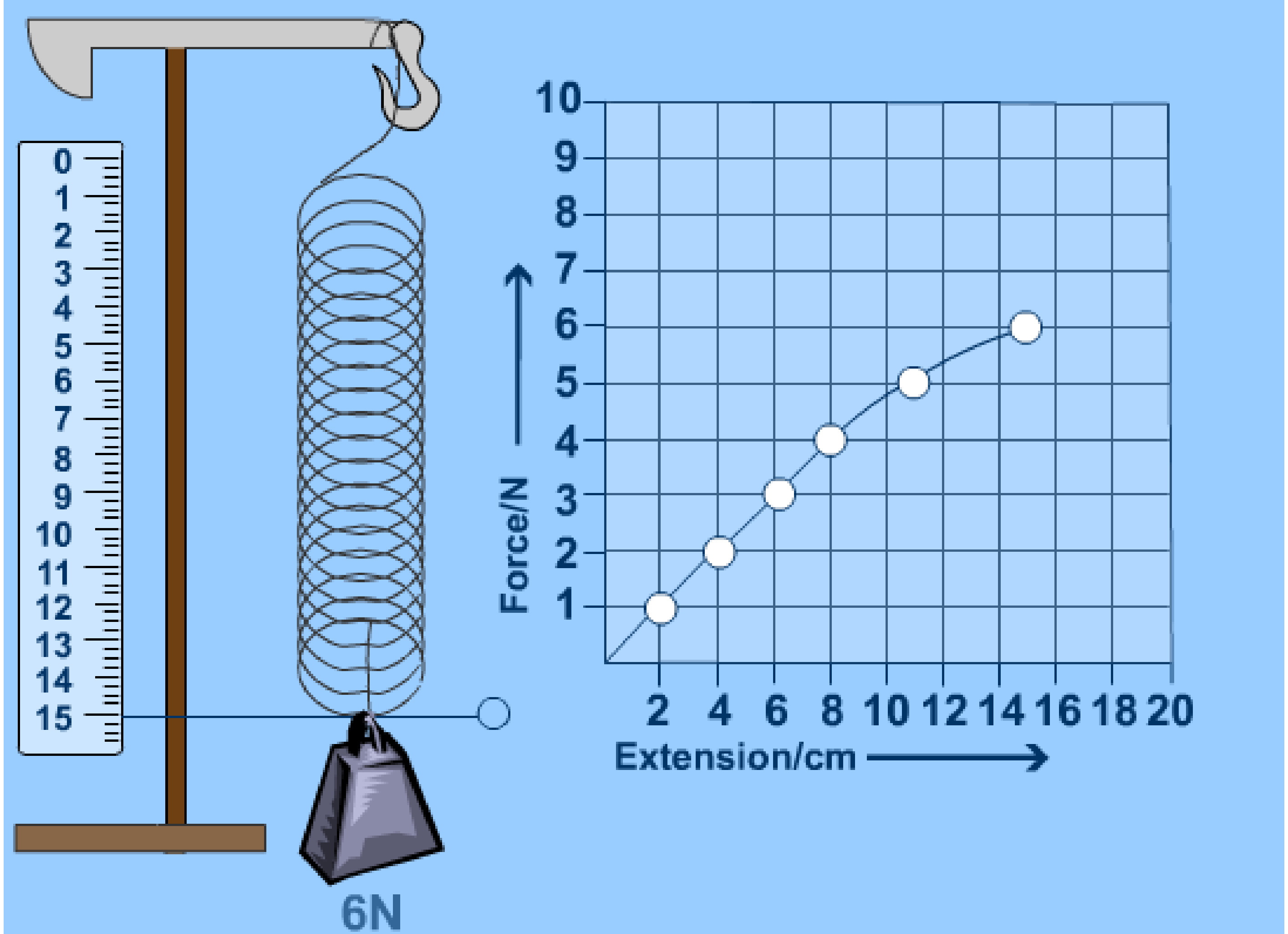
Weight in Newtons	1N	2N	3N	4N	5N	6N
Extension cm	2	4	6	8		

The **elastic limit** can be seen on the graph. This is where it stops obeying Hookes law.





Weight in Newtons	1N	2N	3N	4N	5N	6N
Extension cm	2	4	6	8	11	



Weight in Newtons	1N	2N	3N	4N	5N	6N
Extension cm	2	4	6	8	11	15

You can write Hooke's law as an equation:

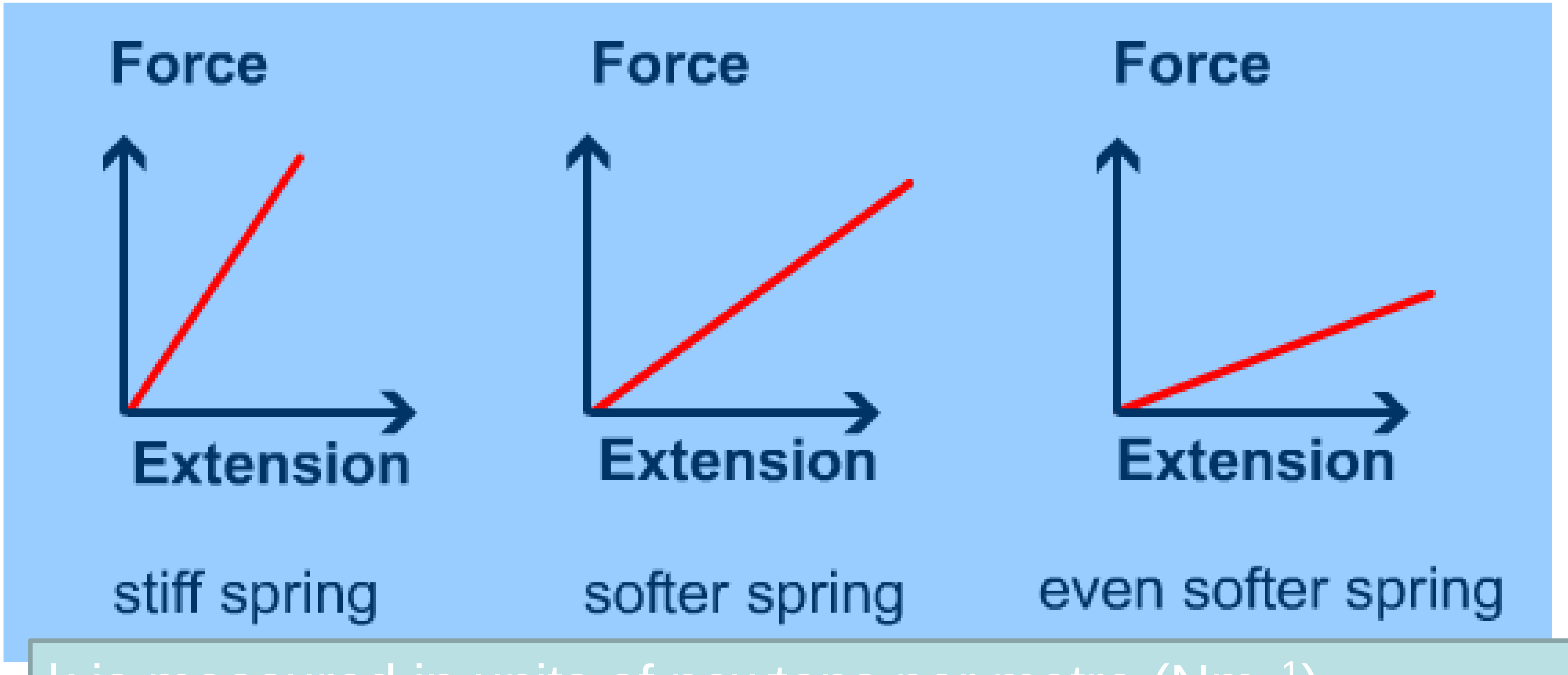
$$F = k \Delta x$$

Where:

- F is the applied force (in newtons, N),
- x is the extension (in metres, m) and
- k is the spring constant (in N/m).
- The extension Δx (delta-x) is sometimes written e or Δl. You find the extension from:
  - Δx = stretched length – original length.

Hold on a minute, K?
Spring Constant?!

- The spring constant measures how stiff the spring is.
- The larger the spring constant the stiffer the spring.
- You may be able to see this by looking at the graphs below:



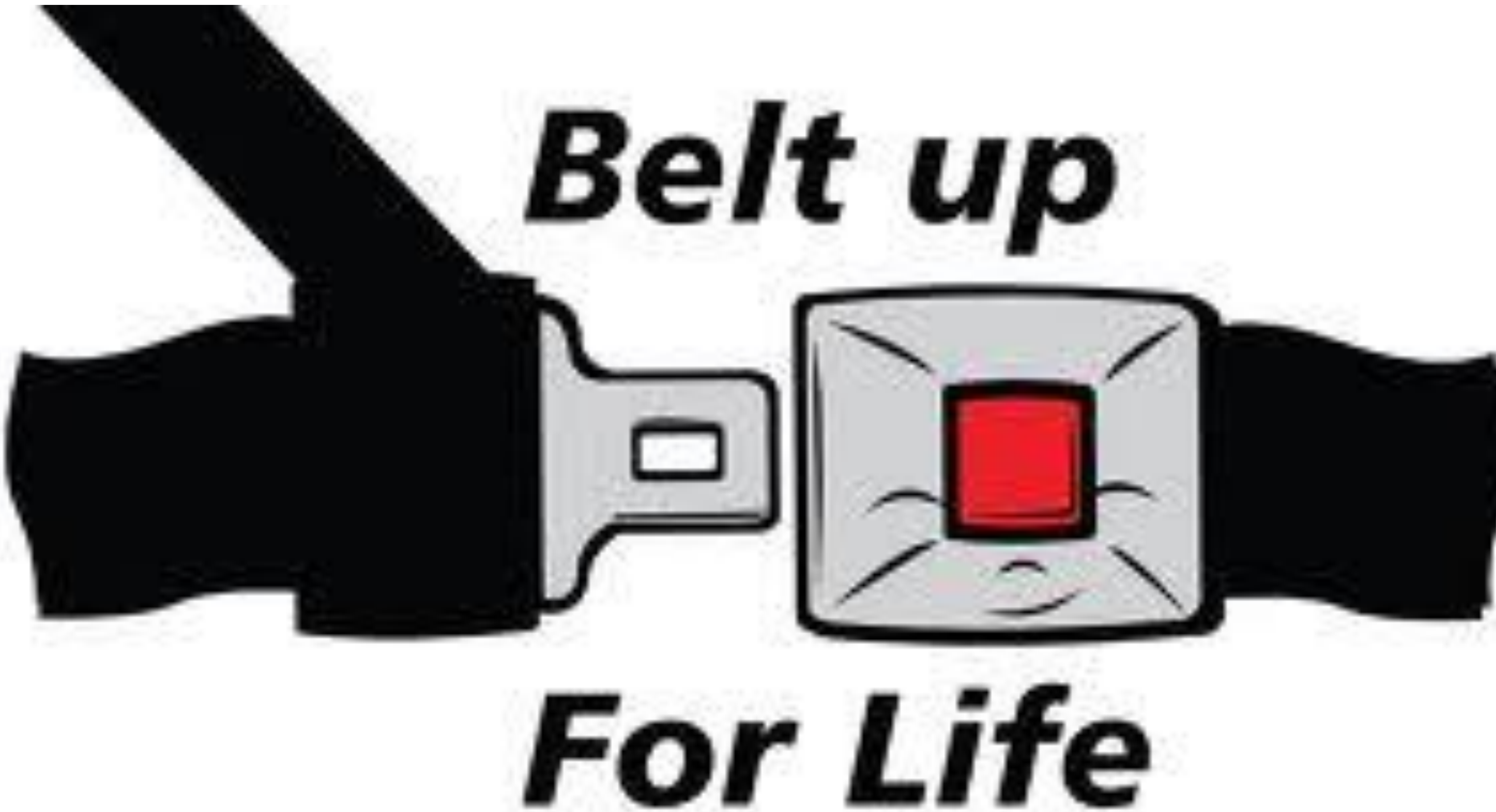
k is measured in units of newtons per metre (Nm^{-1}).

Elastic behaviour – Car Safety

- **Elastic behaviour** is very important in car safety, as car seatbelts are made from elastic materials. However, after a crash they must be **replaced** as they will ***go past their elastic limit.***

- **Why have seat belts that are elastic?**
- **Why not just have very rigid seatbelts that would keep you firmly in place?**
- The reason for this, is that it would be very dangerous and cause large injuries. This is because it would slow your body down too quickly. The quicker a collision, the bigger the force that is produced.

Elastic behaviour – Car Safety


Belt up
For Life

A Family of Microscopes

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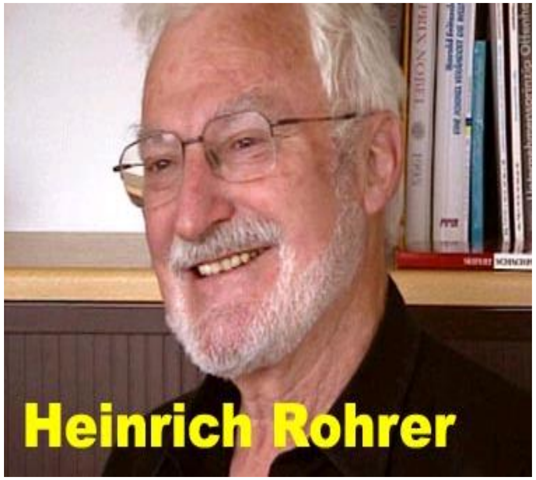
Scanning Tunneling Microscopy (STM)

Introduction

- Invented by Binnig and Rohrer at IBM Zurich Research Laboratory in 1981 (Nobel Prize in Physics in 1986).



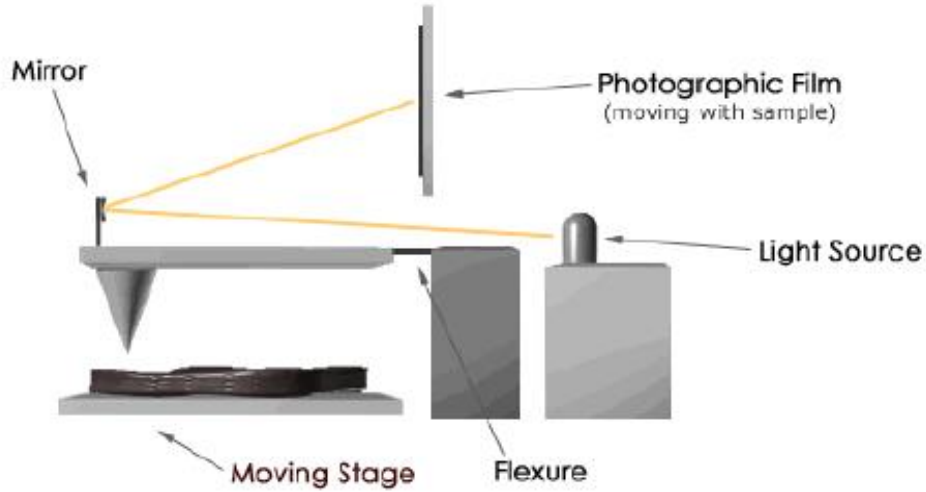
- Binnig also invented the Atomic Force Microscope(AFM) at Stanford University in 1986.

- Allows for the imaging of the surfaces of metals and semiconductors at the atomic level.

Scanning Tunneling Microscopy?

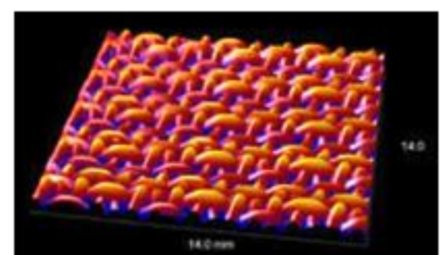
STM has a fathered host of new atomic probe techniques: **Atomic Force Microscopy, Magnetic Force Microscopy etc..**

Stylus Profiler (1929 –Schmalz)



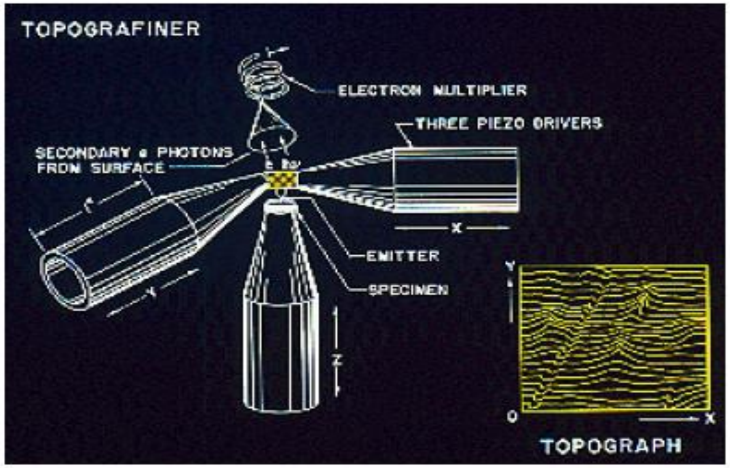
- Generated profile images up to ~1000X magnification
- Problems with large features (bent probes)

US quarter dollar,
11mm x 18mm scan.



Polyester mesh,
12mm x 12mm scan.

Topographiner (1971 –Young)



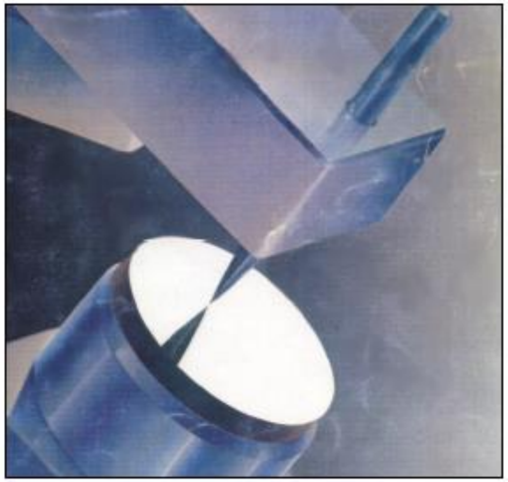
• The x and y piezo drivers scan the tip over and slightly above the specimen surface.
• The z piezo is controlled by a servo system to maintain a constant voltage, and hence a constant vertical separation between the tip and the surface.
• An electron multiplier detects the tiny fraction of the tunneling current which is scattered by the specimen surface. **Reference:** R.D. Young, Physics Today **24**, 42 (Nov. 1971).

FIRST SCANNING PROBE IMAGE.
Topographic map of a 180-line-per-mm diffraction-grating replica, obtained with the Topografiner, a non-contacting field-emission probe developed at NBS.

Reference: R. Young, J. Ward, and F. Scire, Rev. Sci. Instrum. **43**, 999 (1972).

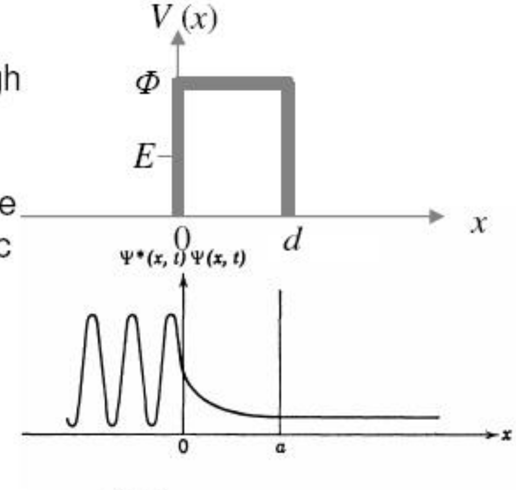
Was operated in field emission!

STM

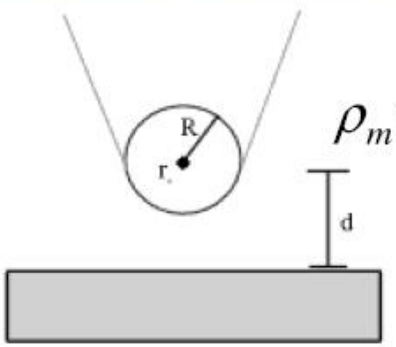


The STM based in electrical interaction between two metallic electrodes.

Small electronic current (~1 nA) crosses through a the small vacuum separation (~1 nA) when a potential difference (V=E/e ~1 V) is applied between the electrods. The transport follows the quantum mechanics principle that the electronic wave-function can penetrate into a energy barrier, i.e., the tunnel effect.



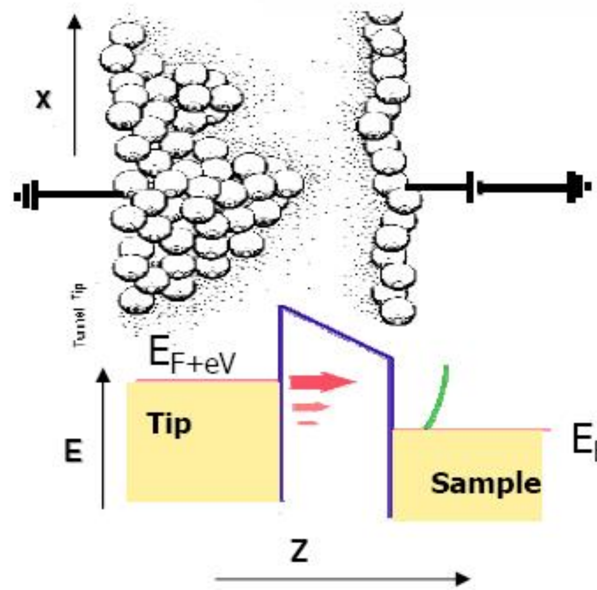
$$T(E) \propto \exp\left(-C \times a \times \sqrt{\Phi - E}\right)$$



$$\rho_m(E_F, r_0)$$

$$I \propto \sum_{\nu} \left|\psi_{\nu}(r_0)\right|^2 \delta(E_{\nu} - E_F)$$

For V small: The current is proportional to density of states.

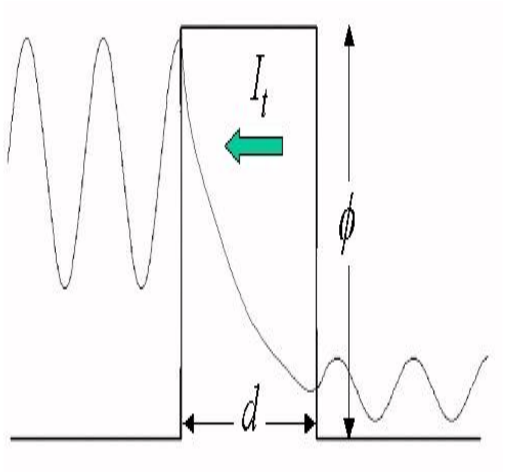


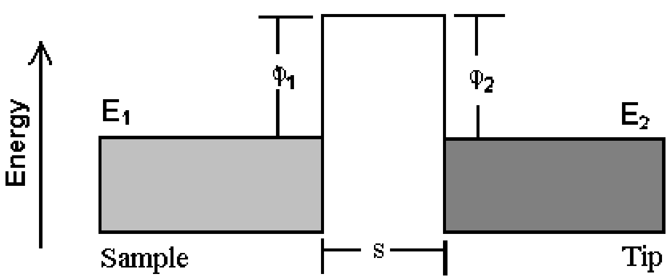
- Atomic resolution, several orders of magnitude better than the best electron microscope
- Quantum mechanical tunnel-effect of electron
- In-situ: capable of localized, non-destructive measurements or modifications
- material science, physics, semiconductor science, metallurgy, electrochemistry, and molecular biology

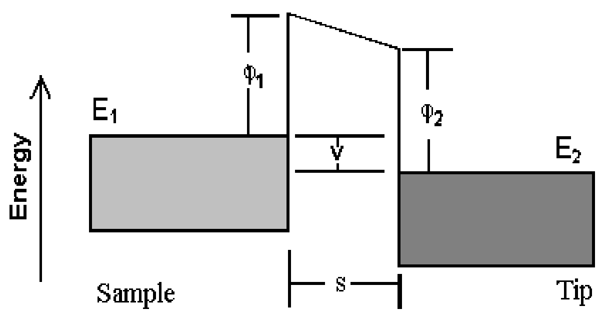
Theory and Principle

Tunneling Current

- A sharp conductive tip is brought to within a few Angstroms of the surface of a conductor (sample).
- The surface is applied a bias voltage, Fermi levels shift
- The wave functions of the electrons in the tip overlap those of the sample surface
- Electrons tunnel from one surface to the other of lower potential.

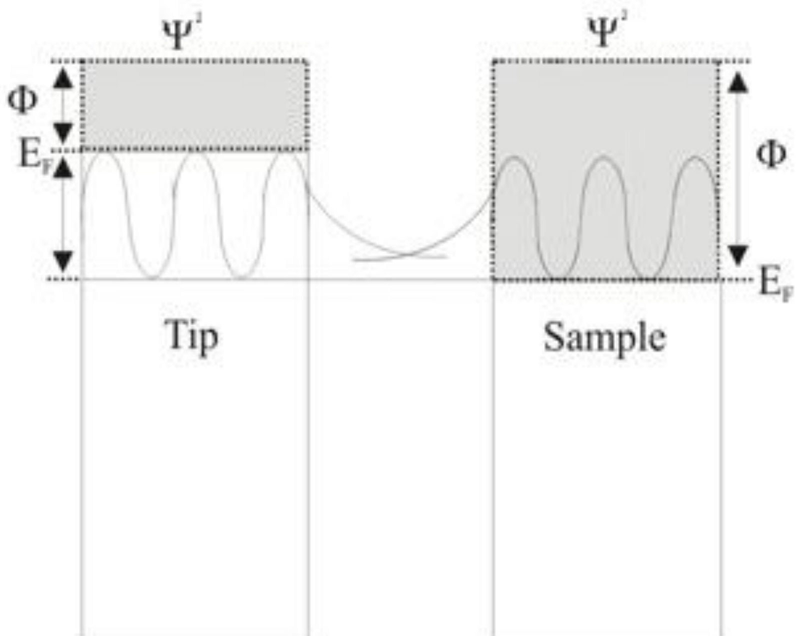






Theory and Principle

- The tunneling system can be described as the model of quantum mechanical electron tunneling between two infinite, parallel, plane metal surfaces



- E_F is the Fermi level
- ψ is the wave function of the electron
- Φ is the work function of the metal.
- Electrons tunnel through a rectangular barrier.

Theory and Principle

- The tunneling current can be calculated from Schrödinger equation (under some further simplifications of the model).

$$i\frac{\partial}{\partial t}\Psi(\vec{z},t) = \mathrm{H}\Psi(\vec{z},t) \quad \longrightarrow \; \ldots\ldots \longrightarrow \qquad I_t \propto V \exp\left[-\left(\phi_{av}\right)^{1/2} d\right]$$

  - I_t is the tunneling current; V is the sample bias
  - Φ_{av} is the average work function (barrier height), about 4 eV above the Fermi energy for a clean metal surface
  - d is the separation distance

- Tunneling current exhibits an exponentially decay with an increase of the separation distance!

- Exponential dependence leads to fantastic resolutions. Order of 10^{-12} m in the perpendicular direction and $\sim 10^{-10}$ m in the parallel directions

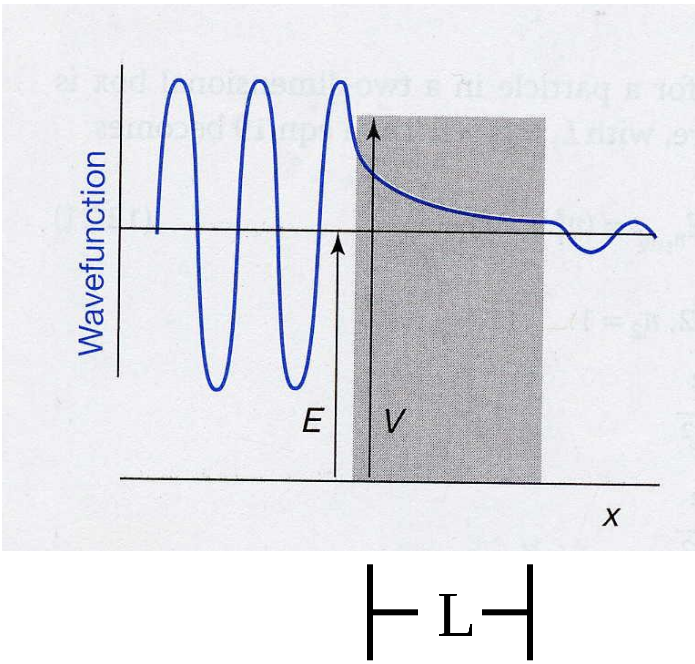
Theory and Principle

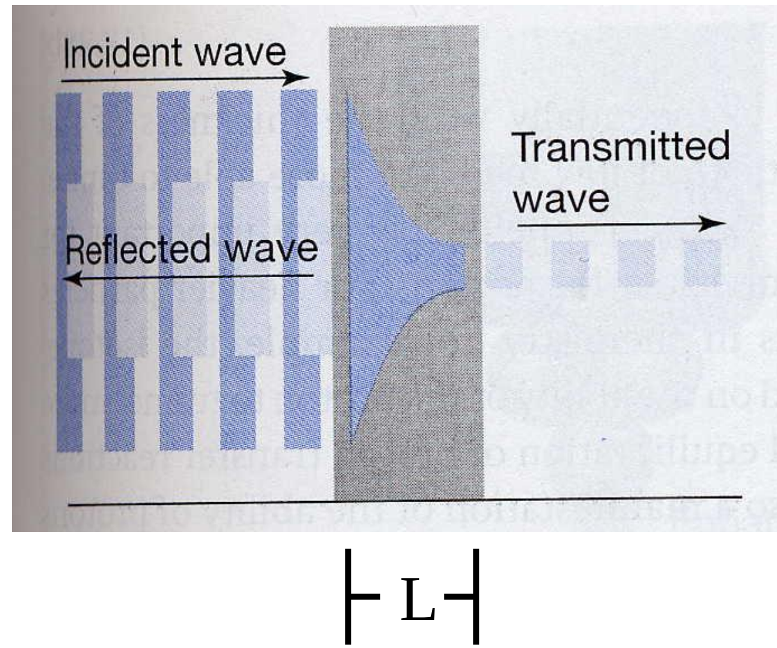
How tunneling works ?  Simple answer

- In classical physics e flows are not possible without a direct connection by a wire between two surfaces
- On an atomic scale a quantum mechanical particle behaves in its wave function.
- There is a finite probability that an electron will “jump” from one surface to the other of lower potential.

"... I think I can safely say that nobody understands Quantum Mechanics"
Richard P. Feynman

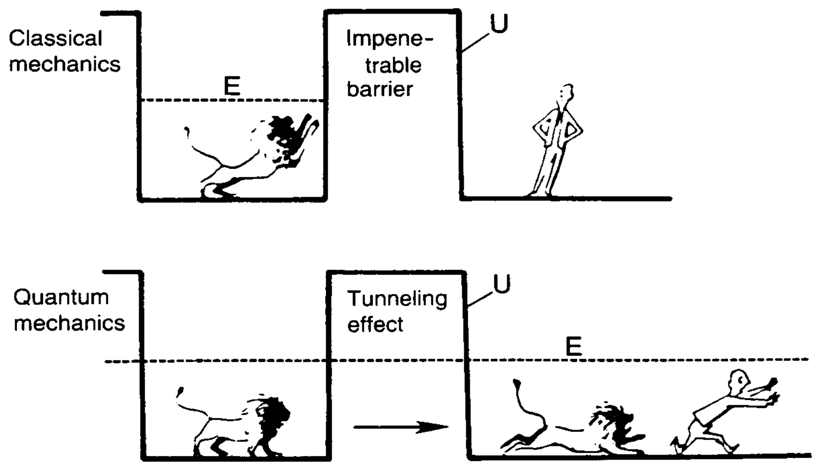
Quantum Mechanical Tunneling





- Quantum mechanics allows a small particle, such as an electron, to overcome a potential barrier larger than its kinetic energy.
- Tunneling is possible because of the wave-like properties of matter.
- Transmission Probability: $T \approx 16\varepsilon(1 - \varepsilon)e^{-2\kappa L}$

The Tunneling Phenomenon



Chen, C.J. In *Introduction to Scanning Tunneling Microscopy*; Oxford University Press: New York, 1993; p 3.

In classical mechanics, the energy of an electron moving in a potential U(x) can be shown by $\frac{p_x^2}{2m} + U(x) = E$

The electron has nonzero momentum when E > U(x), but when E<U(x) the area is forbidden.

The quantum mechanical description of the same electron is $\hat{H}\psi(x) + U_x\psi(x) = E\psi(x)$

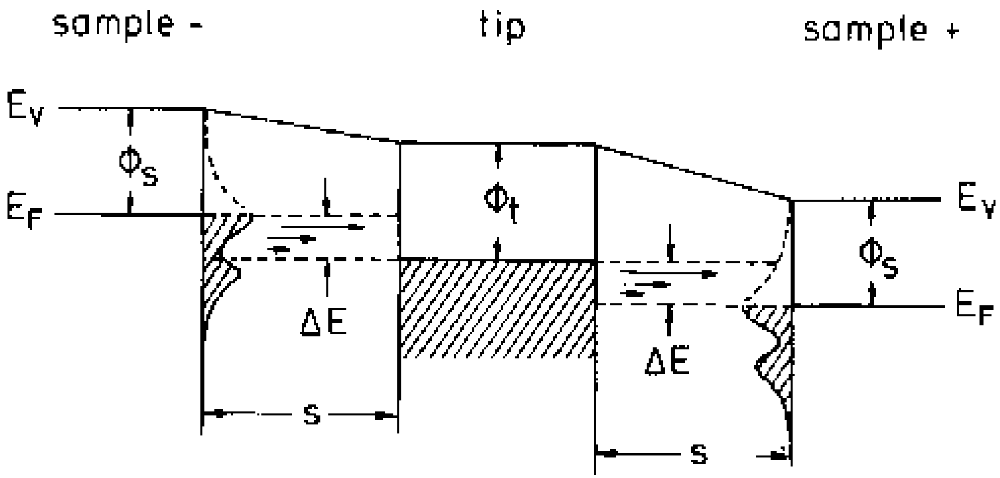
In the classically allowed region (E>U), there are two solutions, $\psi(x) = \psi(0)e^{\pm ikx}$, where $k = \frac{\sqrt{2m(E-U)}}{\hbar}$

These give the same result as the classical case. However, in the classically forbidden region (E<U) the solution is

$$\psi(x) = \psi(0)e^{-\kappa x}, \text{ where } \kappa = \frac{\sqrt{2m(U-E)}}{\hbar}$$

κ is a decay constant, so the solution dictates that the wave function decays in the +x direction, and the probability of finding an electron in the barrier is non-zero.

Tunneling Energy Diagram



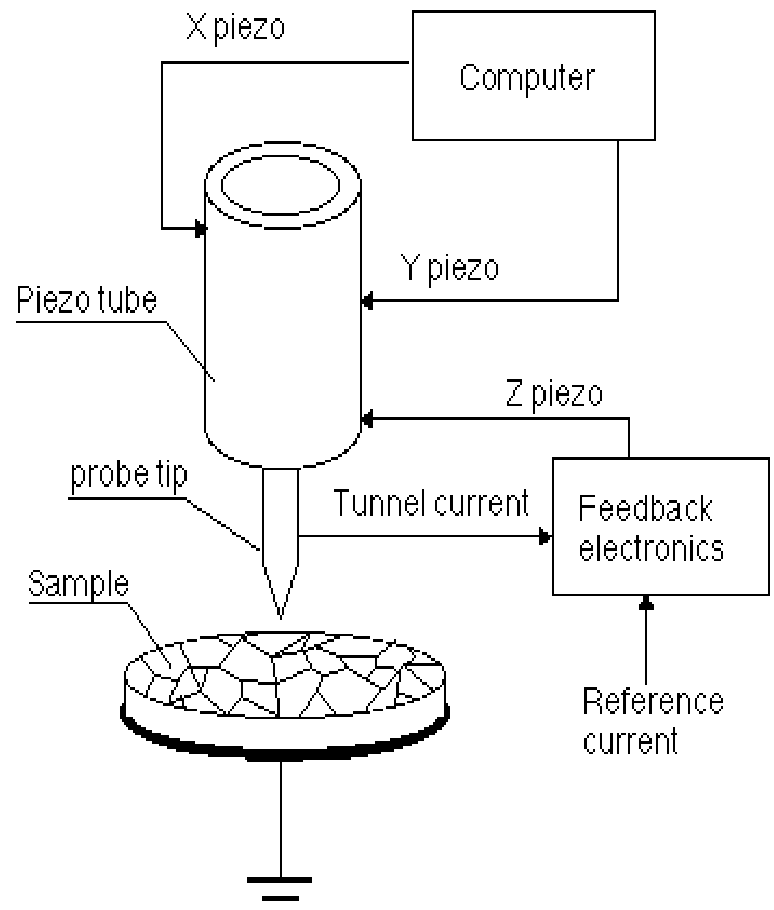
Behm, R.J.; Hosler, W. In *Chemistry and Physics of Surfaces VI*; Vanselow, R., Howe, R., Eds.; Springer: Berlin, 1986; p 361.

This diagram shows the bias dependence on tunneling. E_v is the vacuum level, or the reference energy level. E_F is the Fermi level, which is the highest occupied level in a metal. ϕ_s is the work function of the sample. The work function is defined as the amount of energy needed to remove an electron from the bulk to the vacuum level. The work function of the tip is labeled as ϕ_t. If the **sample bias is positive, the Fermi level of the sample is less than that of the tip, so electrons flow towards the sample. When the sample bias is negative, the Fermi level of the sample is at a higher level than that of the tip, so the electrons travel towards the tip.**

Experimental methods

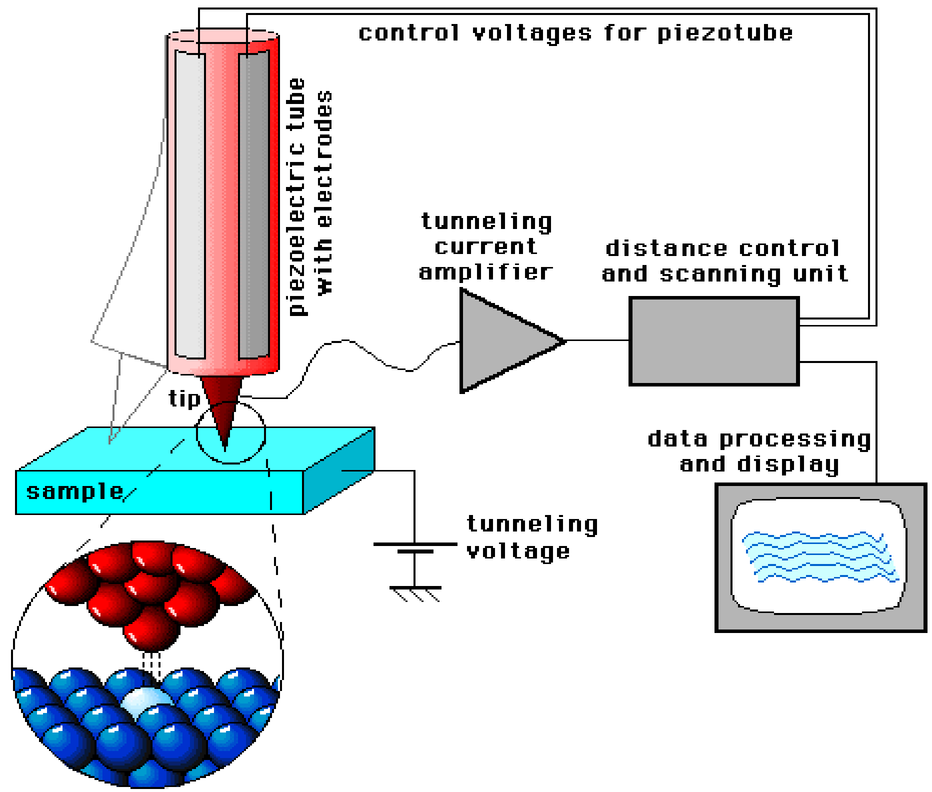
Basic Set-up

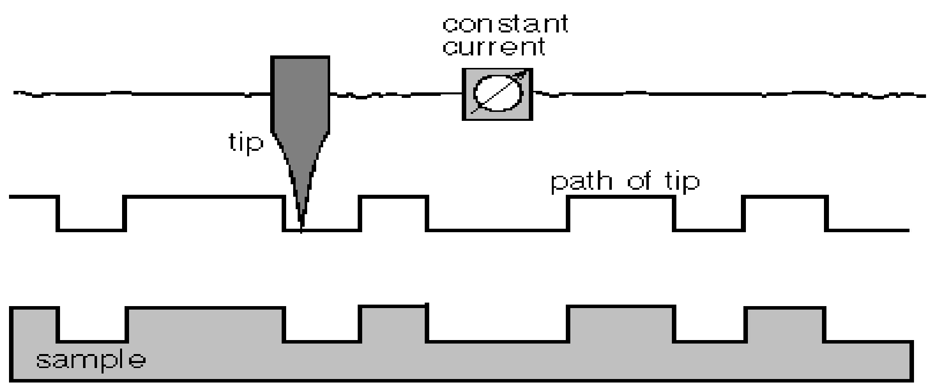
- the *sample* you want to study
- a sharp *tip* mounted on a piezoelectric crystal tube to be placed in very close proximity to the sample
- a mechanism to control the location of the tip in the x-y plane parallel to the sample surface
- a *feedback* loop to control the height of the tip above the sample (the z-axis)



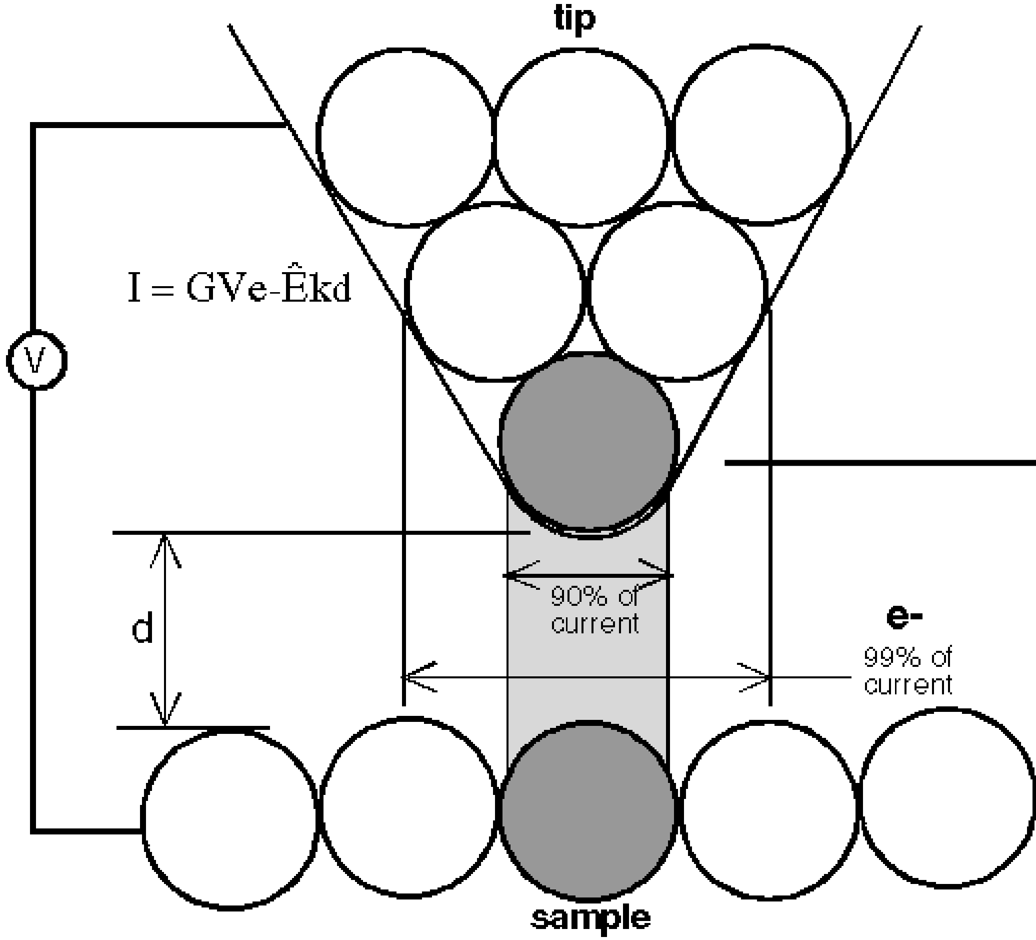
How to operate?

- Raster the tip across the surface, and using the current as a feedback signal.
- The tip-surface separation is controlled to be constant by keeping the tunneling current at a constant value.
- The voltage necessary to keep the tip at a constant separation is used to produce a computer image of the surface.





Basic Principles of STM



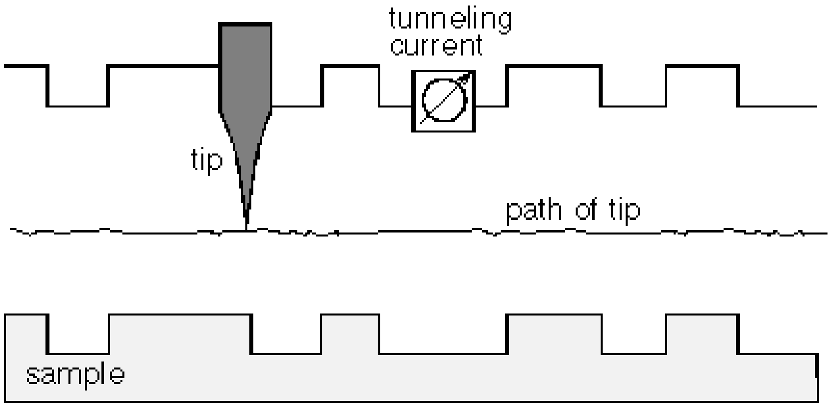
d ~ 6 Å

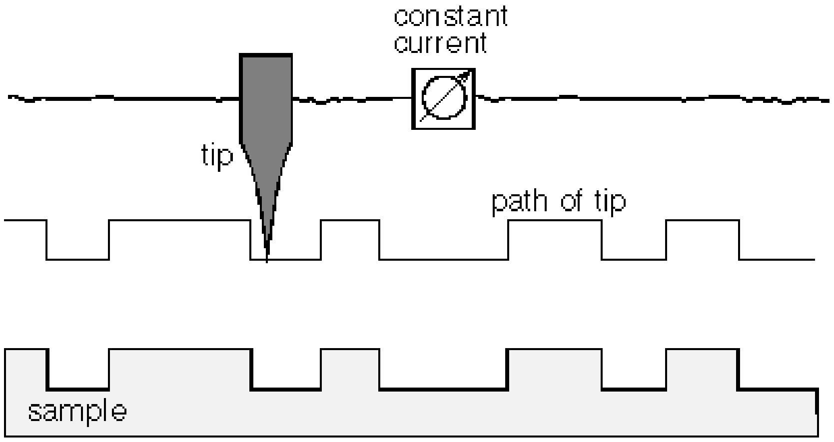
Bias voltage: mV – V range

- Electrons tunnel between the tip and sample, a small current I is generated (10 pA to 1 nA).
- I proportional to $e^{-2\kappa d}$, I decreases by a factor of 10 when d is increased by 1 Å.

Two Modes of Scanning

Constant Height Mode



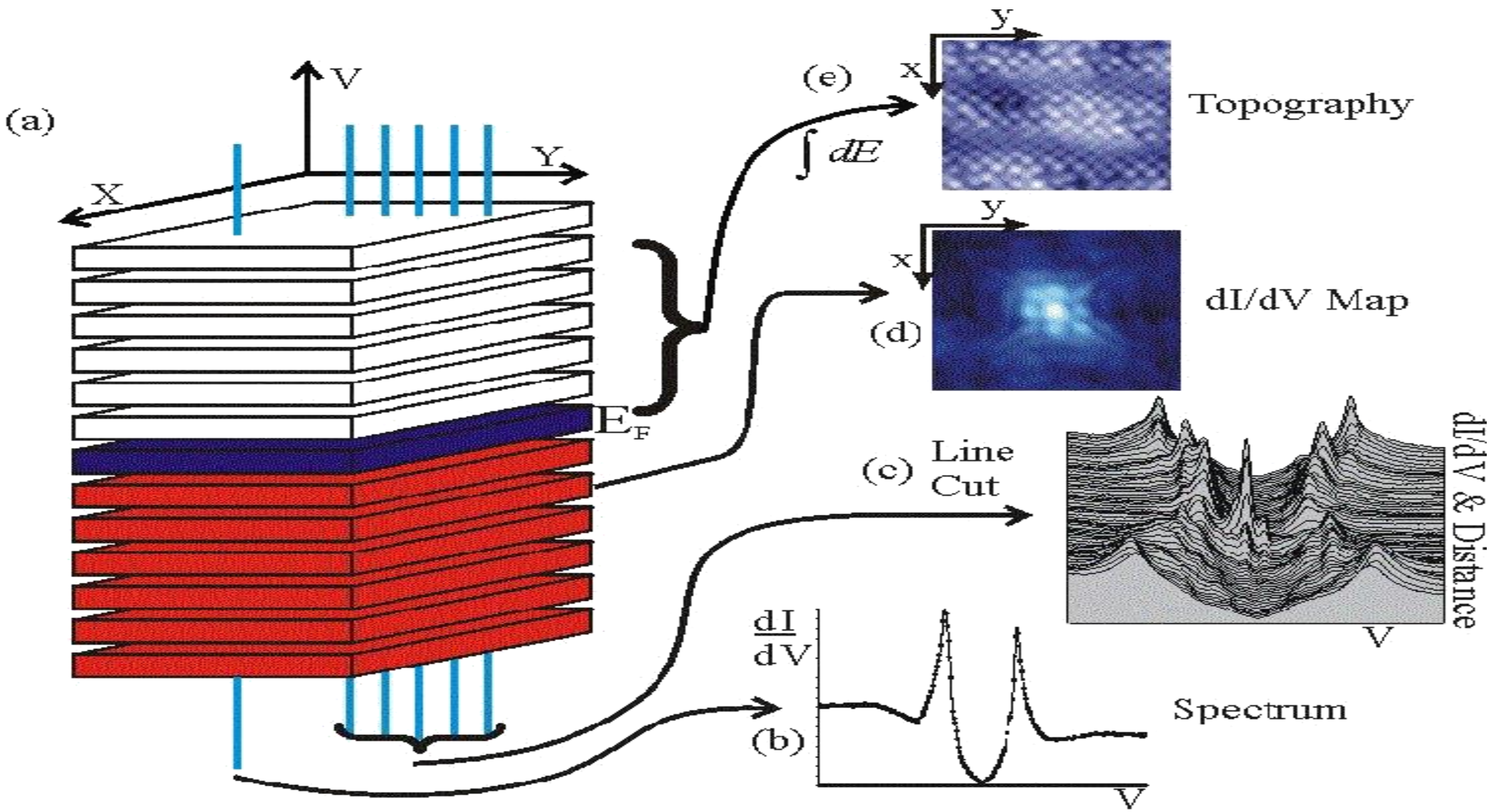


Constant Current Mode

- Usually, constant current mode is superior.

What an STM measures?------local density of states

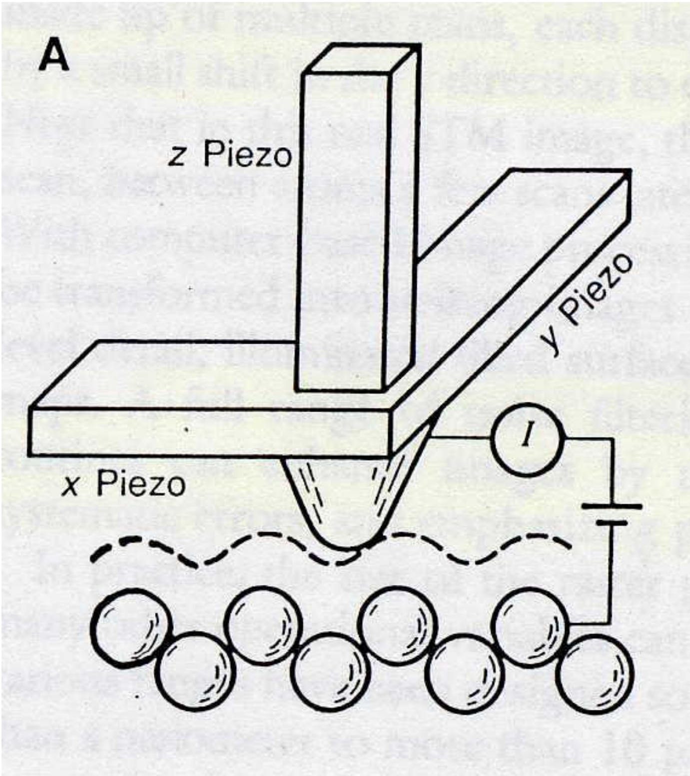
- Each plane represents a different value of the tip-sample bias V, and the lateral position on the plane gives the x,y position of the tip. Filled states are given in red. The plane at the Fermi energy (V=0) is shown in blue.



Experimental Optimization

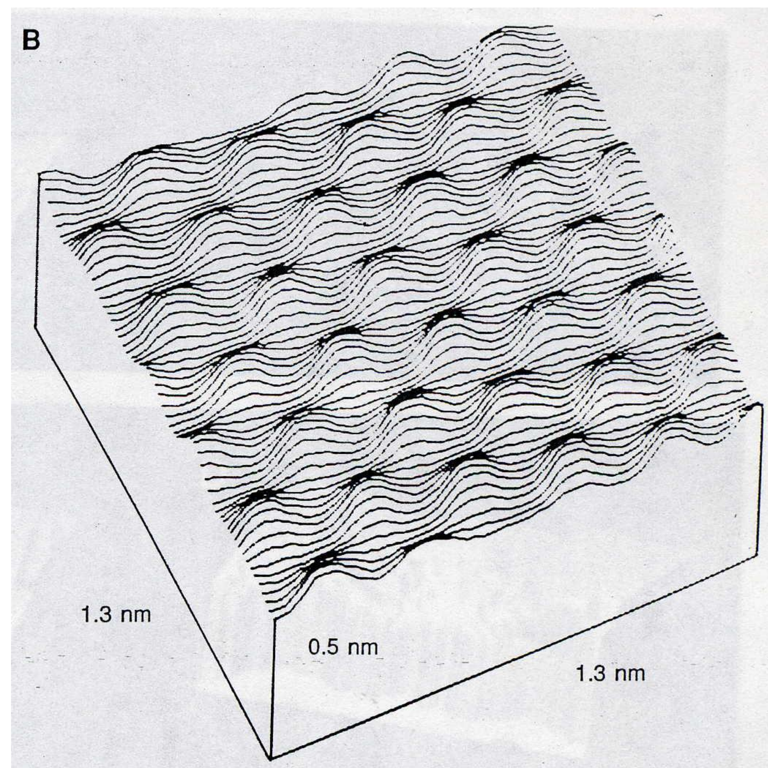
- Control of environment vibration:
  - building the instrument with sufficient mechanical rigidity;
  - hung on a double bungee cord sling to manage vibration;
  - vibration isolation systems have also been made with springs and frames;
  - ***operate at night with everything silent***.
- Ultrahigh vacuum (UHV): to avoid contamination of the samples from the surrounding medium. (The STM itself does not need vacuum to operate; it works in air)
- Using an atomically sharp tip.

Instrumental Design: Controlling the Tip



- Precise tip control is achieved with Piezoelectrics
- Displacement accurate to ± .05 Å

Raster scanning →

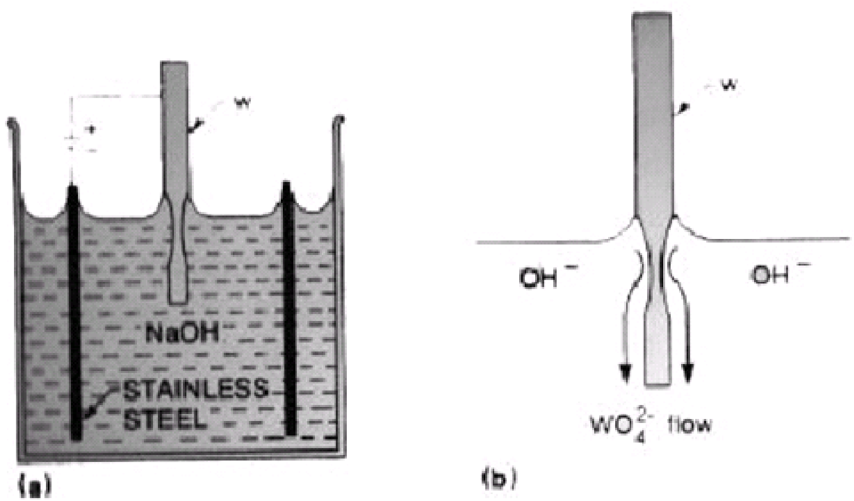


Tip details

- STM tip: atomically sharp needle and terminates in a single atom
- Pure metals (W, Au)
- Alloys (Pt-Rh, Pt-Ir)
- Chemically modified conductor (W/S, Pt-Rh/S, W/C…)

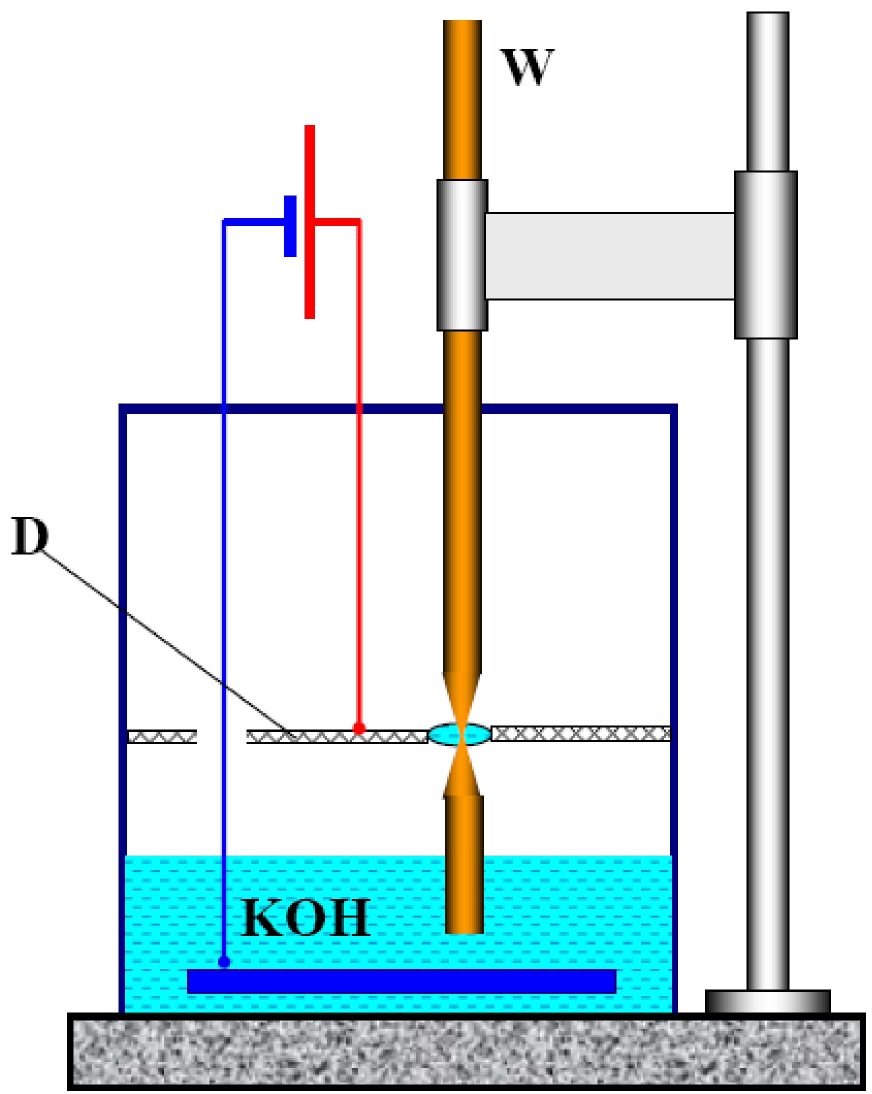
- Preparation of tips: cut by a wire cutter and used as is

  cut followed by electrochemical etching

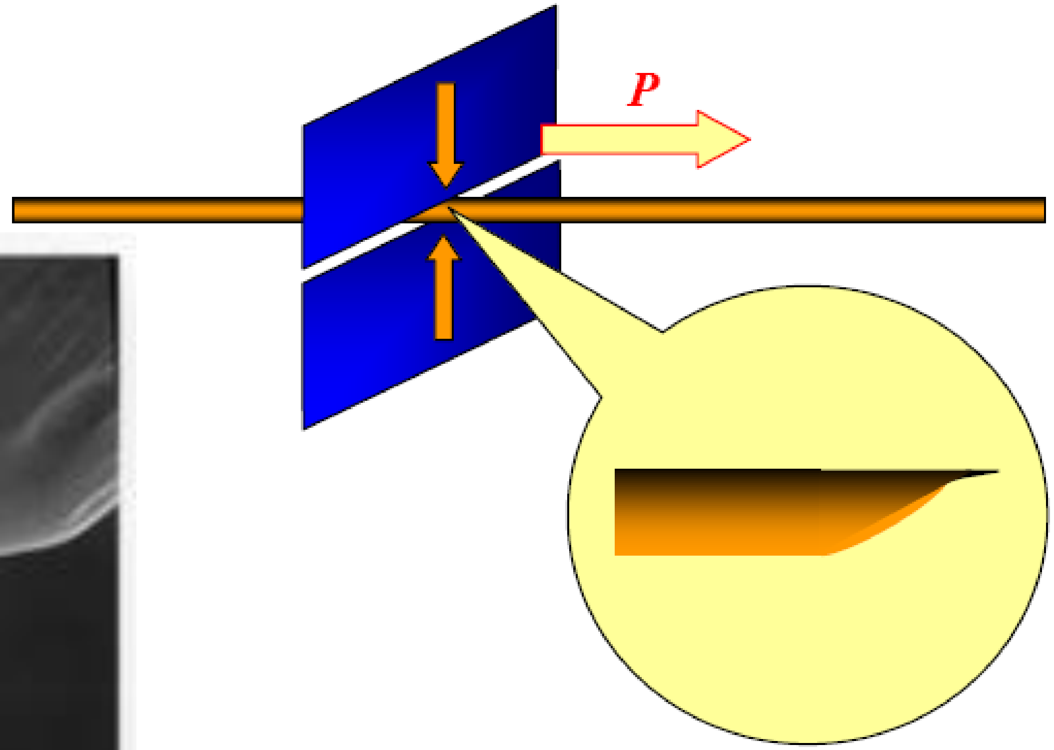


- Electrochemical etching of tungsten tips. A tungsten wire, typically 0.25 mm in diameter, is vertically inserted in a solution of 2M NaOH. A counter electrode, usually a piece of platinum or stainless steel, is kept at a negative potential relative to the tungsten wire.
- The etching takes a few minutes. When the neck of the wire near the interface becomes thin enough, the weight of the wire in electrolyte fractures the neck. The lower half of the wire drops off.

The STM tips manufacturing scheme from a tungsten wire by electrochemical etching



Schematical image of process of STM apex formation during cutting of a PtIr alloy wire



Advantages

- No damage to the sample
- Vertical resolution superior to SEM
- Spectroscopy of individual atoms
- Relatively Low Cost

Disadvantages

- Samples limited to conductors and semiconductors
- Limited Biological Applications: AFM
- Generally a difficult technique to perform

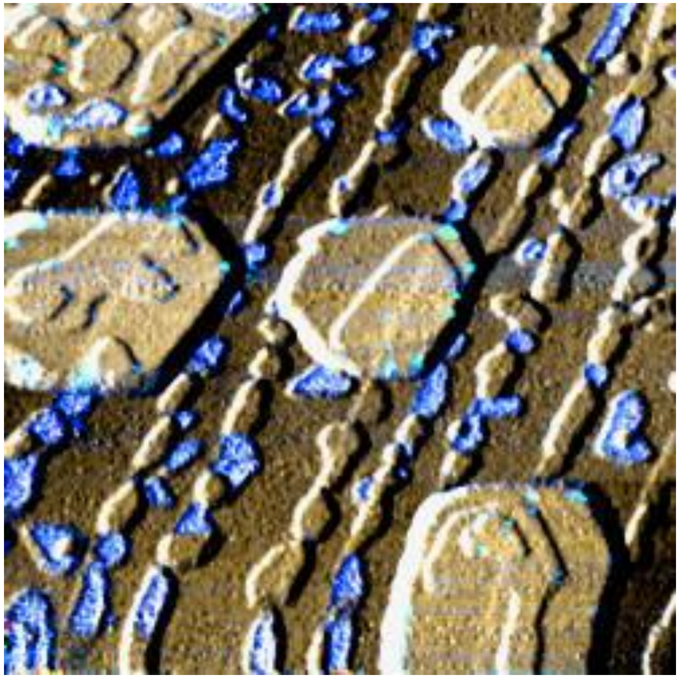
Figures of Merit

- Maximum Vertical Resolution: .1 Å
- Maximum Lateral Resolution: 1 Å
- Maximum Field of View: 100 µm

Applications of STM

- Surface Structure: Compare to bulk structure
- Stuff Physicists Do: Semiconductor surface structure, Nanotechnology, Superconductors, etc.
- Metal-catalyzed reactions
- Spectroscopy of single atoms

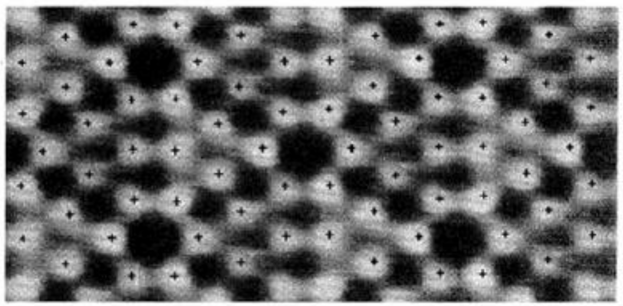
Interpreting STM Images


Hydrogen on Gadolinium

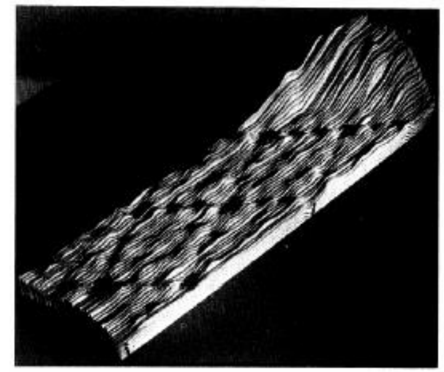
- “Topography” model good for large scale images, but not for the atomic level.
- Electron charge density model more accurate for atomic level images.
- Best model requires complex quantum mechanical considerations

Scanning Tunneling Spectroscopy

Since you are measuring the electronic states, images of the same surface can vary!

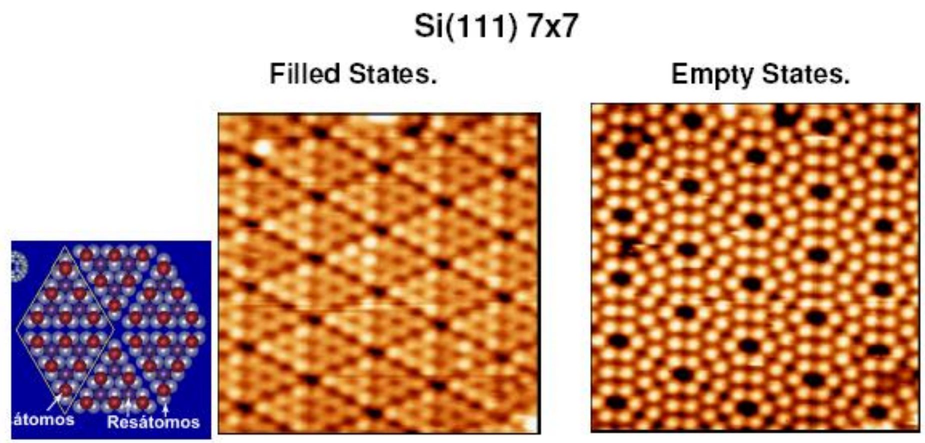


7 x 7 unit cells of a Si Surface at 300 K



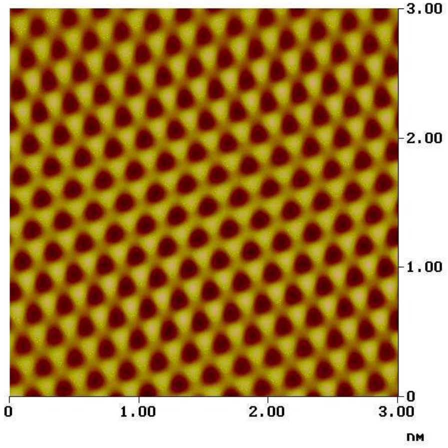
First images were of the Si (111) reconstruction

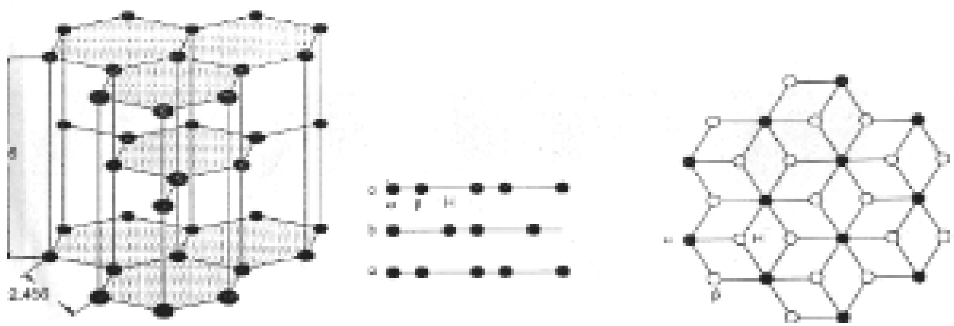
The images vary depending on the electronic state of the material/tip.



Graphite is a good example!

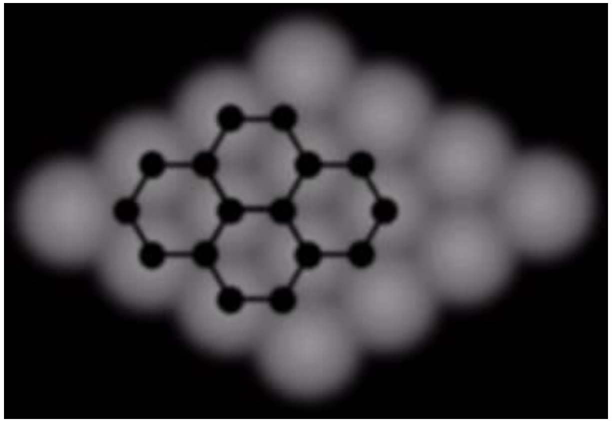
- STM images of graphite



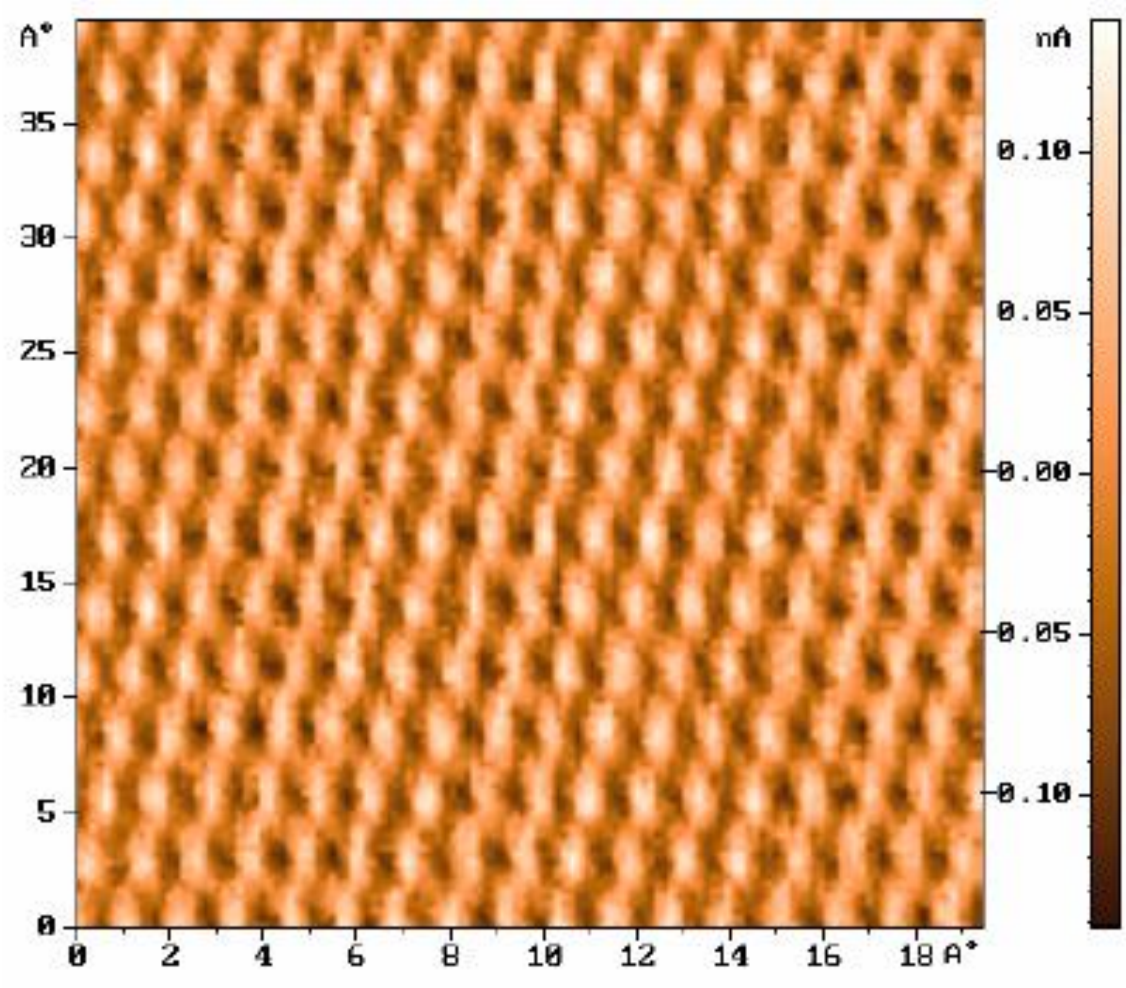


Structure of graphite

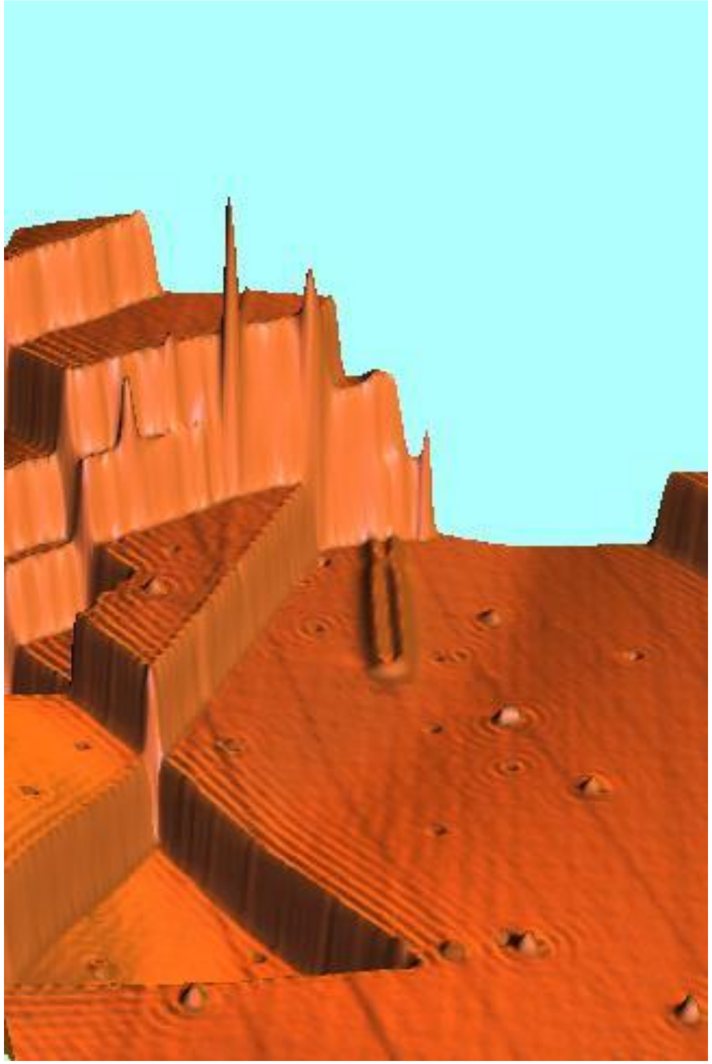
- Overlay of structure shows only every other atom is imaged



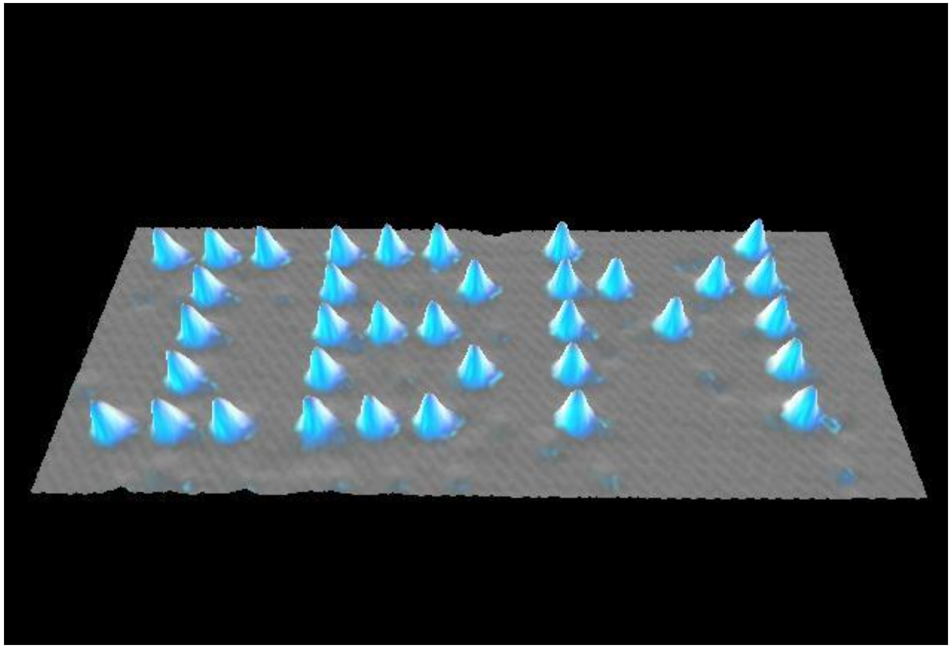
The STM image of an atomic structure of a surface of pyrolitic graphite



Interesting Images with STM



Copper Surface



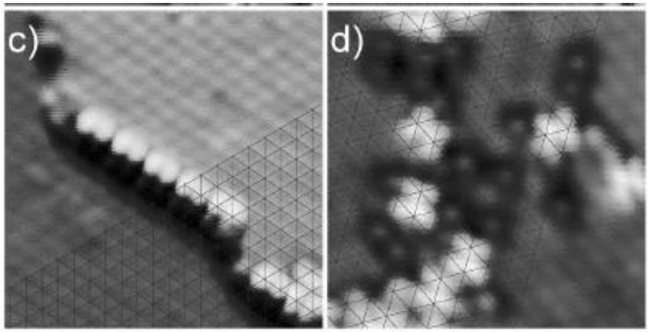
Xenon on Nickel

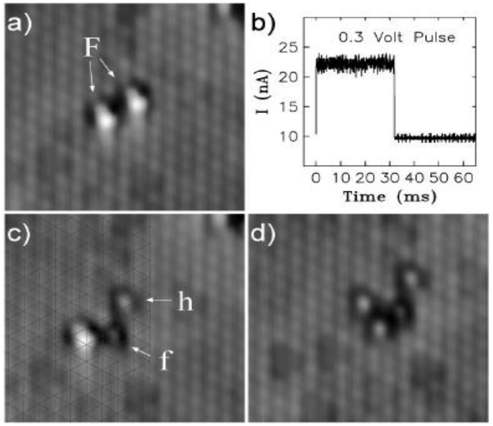
Single atom lithography

Catalytic Processes

- **Tunneling current can be used to dissociate single O_2 Molecules on Pt(111) surfaces.**
- **After dissociation O atoms are ~ 1-3 lattice sites apart.**

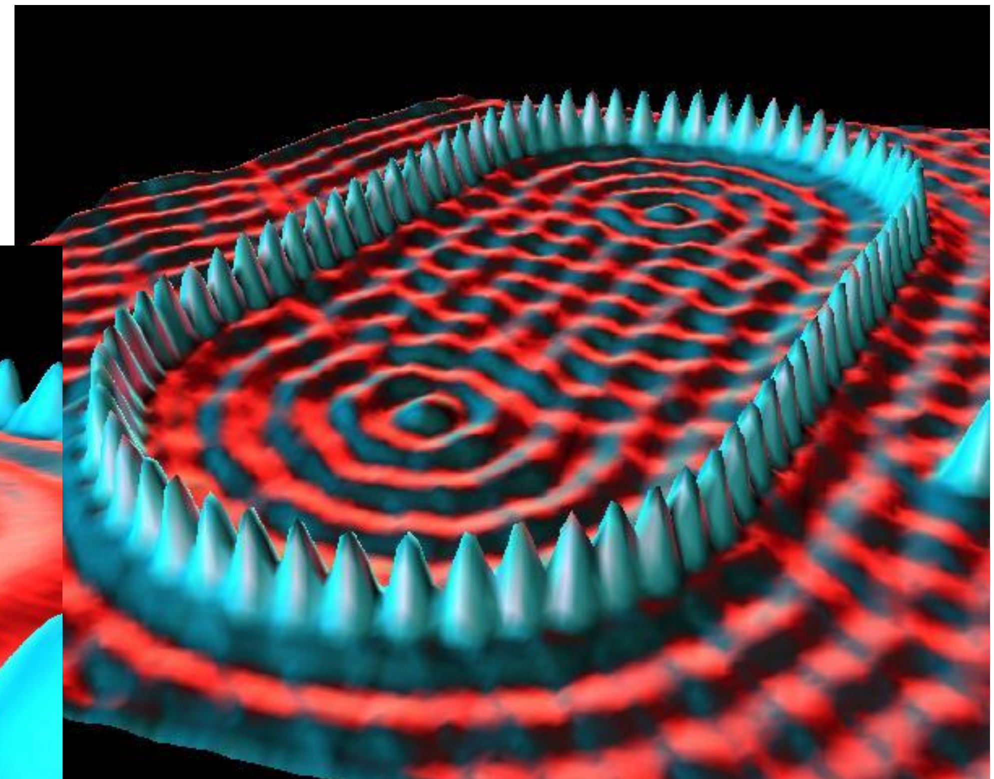
Stipe et al, PRL 78 (1997) 4410.

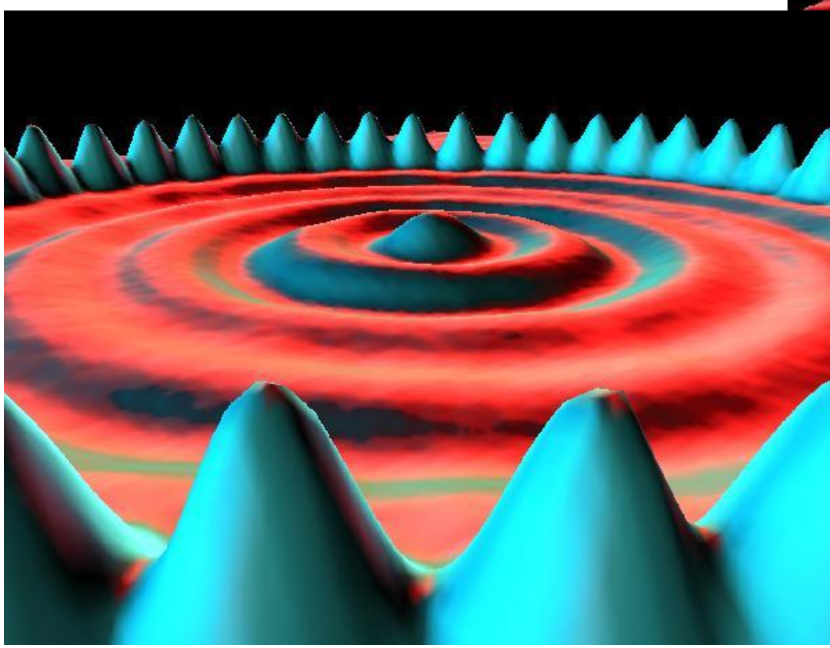




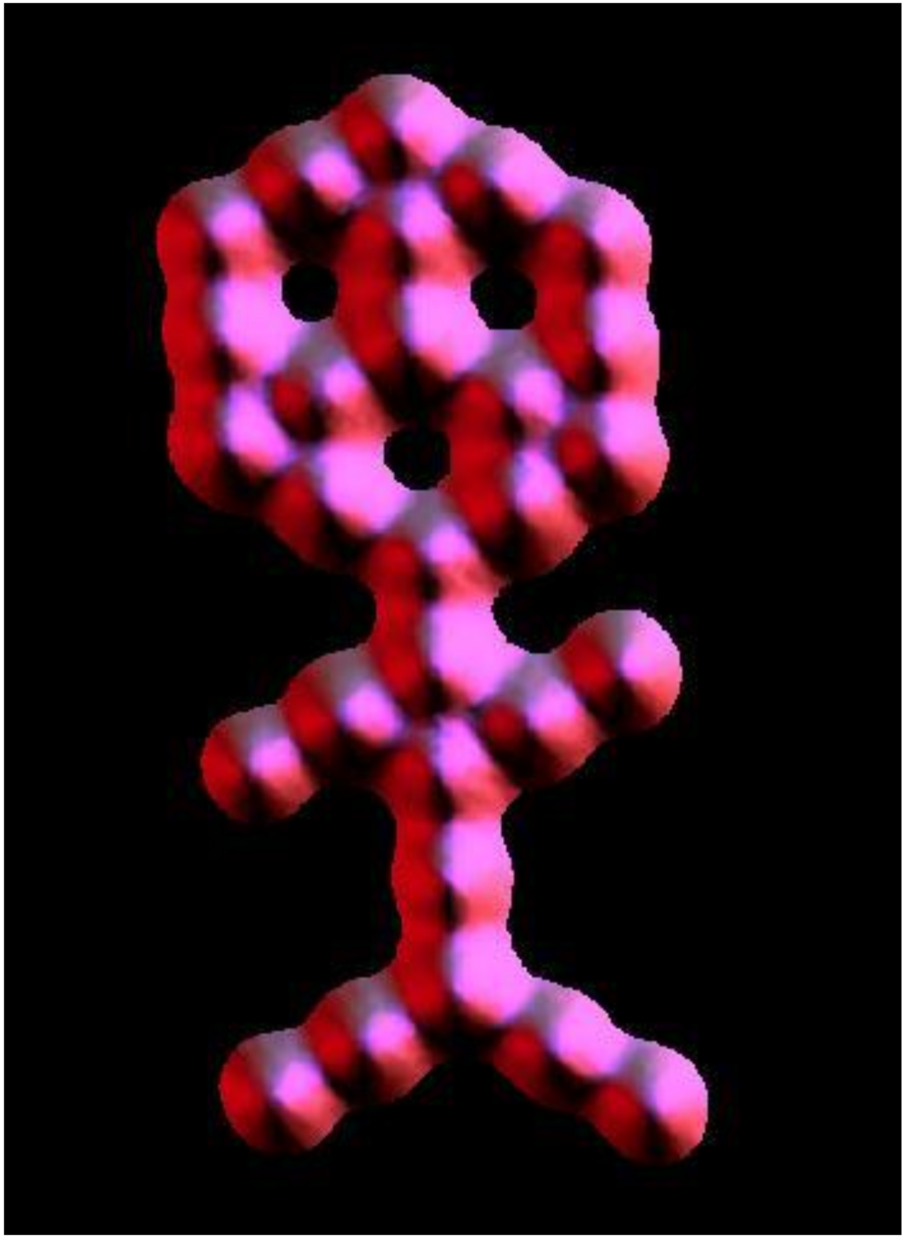
Quantum Corrals

Imaging the standing wave created by interaction of species

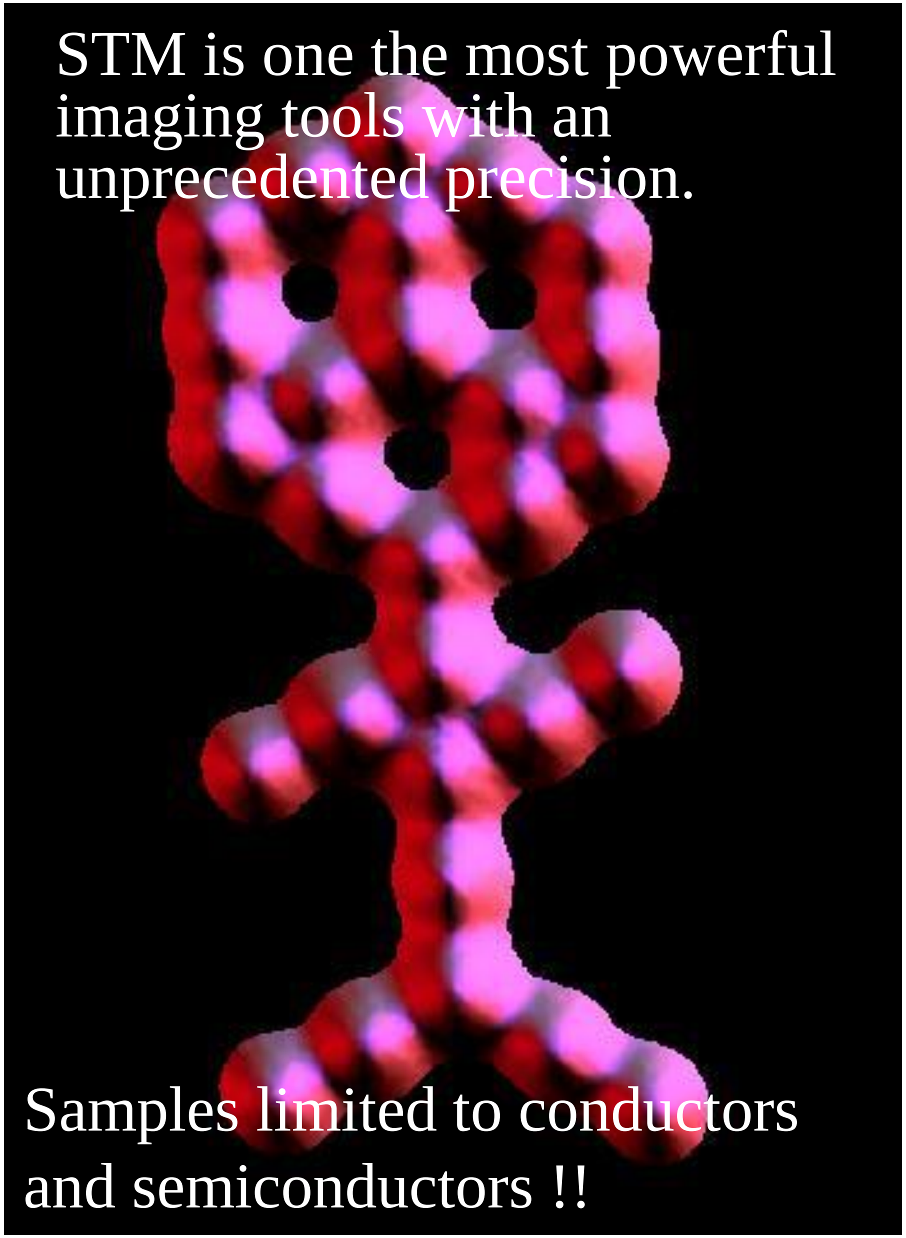




Iron on Copper



Carbon Monoxide Man: CO on Platinum



Carbon Monoxide Man: CO on Platinum

Sources

Stroscio, Joseph A.; Kaiser, William J. Scanning Tunneling Microscopy. 1993. Academic Pres Inc. San Diego.

Golovchenko, JA. *Science*. 232, p. 48 – 53.

Pool, Robert. *Science*. 247, p. 634 – 636.

Hansma, PK; Elings, VB; Marti, O; Bracker, CE. *Science*. 14 October 1988, p. 209 – 215.

STM Image Gallery. IBM Corporation 1995. http://www. almaden.ibm.com/vis/stm/gallery .html

"A Practical Guide to Scanning Probe Microscopy." Veeco Metrology Group. http://www. topometrix.com/spmguide/contents.htm

Preuss, Paul. "A Close Look: Exploring the Mystery of the Surface." *Science Beat*. April 12, 1999. http://www. lbl.gov/Science-Articles/Archive/STM-under-pressure.html

"Scanning Tunneling Microscopy." National Center for Photovoltaics at the National Renewable Energy Laboratory. http://nrel.gov/measurements/tunnel.html

"Scanning Tunneling Microscopy." http://www. physnet.uni-hamburg. de/home/vms/ pascal/stm.htm

"The Nobel Prize in Physics 1986." Nobel e Museum. http://www. nobel.se/ physics/laureates/1986/index.html

A Family of Microscopes

SPM (air, liquid, vacuum)

AFM

Contact Modes

- **Topography**
- **Lithography**

Non-contact (true and tapping)

- **Topography**
- **MFM, EFM**
- **SKPM**
- **Others**

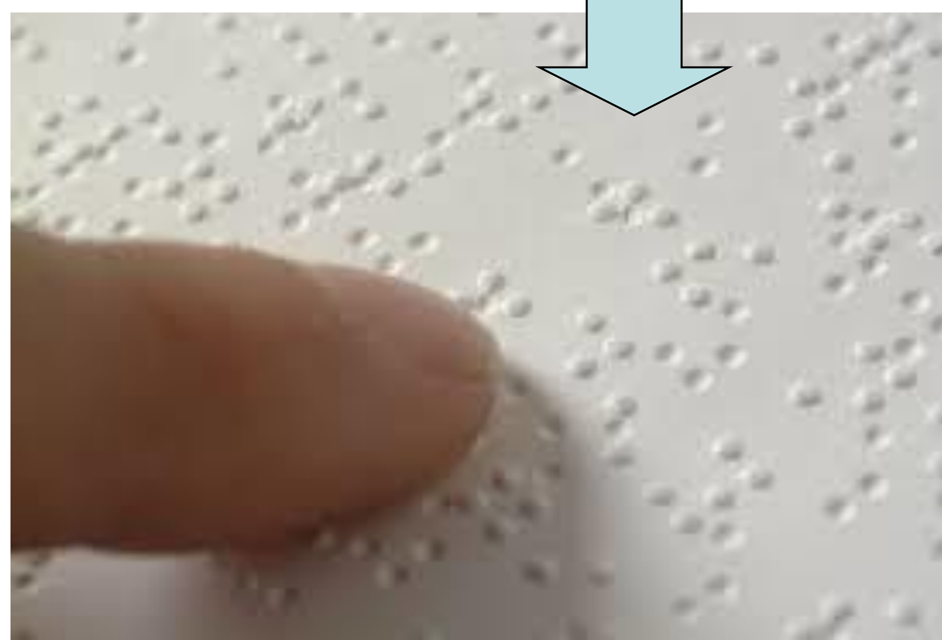
STM

- **Topography**
- **Spectroscopy**

SNOM(NSOM)

- **Aperture**
- **Aperatureless**
- **Reflection**
- **Transmission**

How AFM works



How AFM works

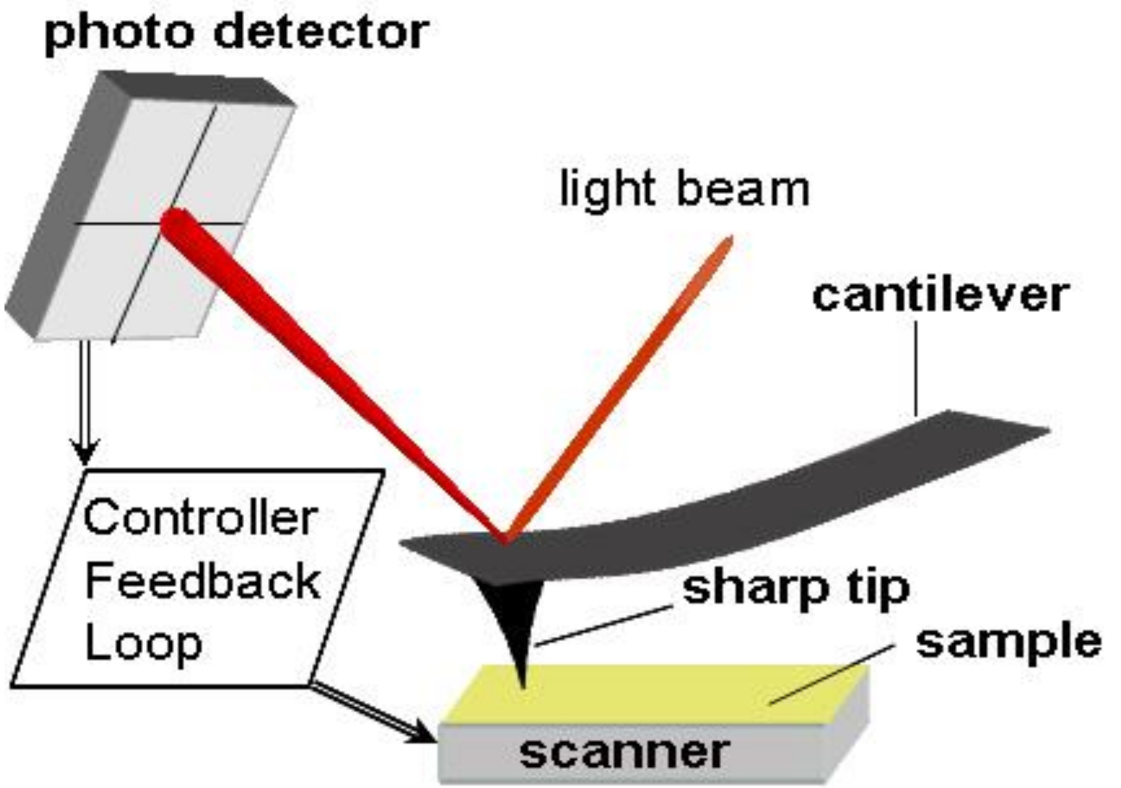
- Direct mechanical contact between the probe and the sampler surface
  - Essential difference from traditional microscopy
- How AFM “feels” the surface topography?
  - Optical level detection

Click for the Next

How does an AFM work?

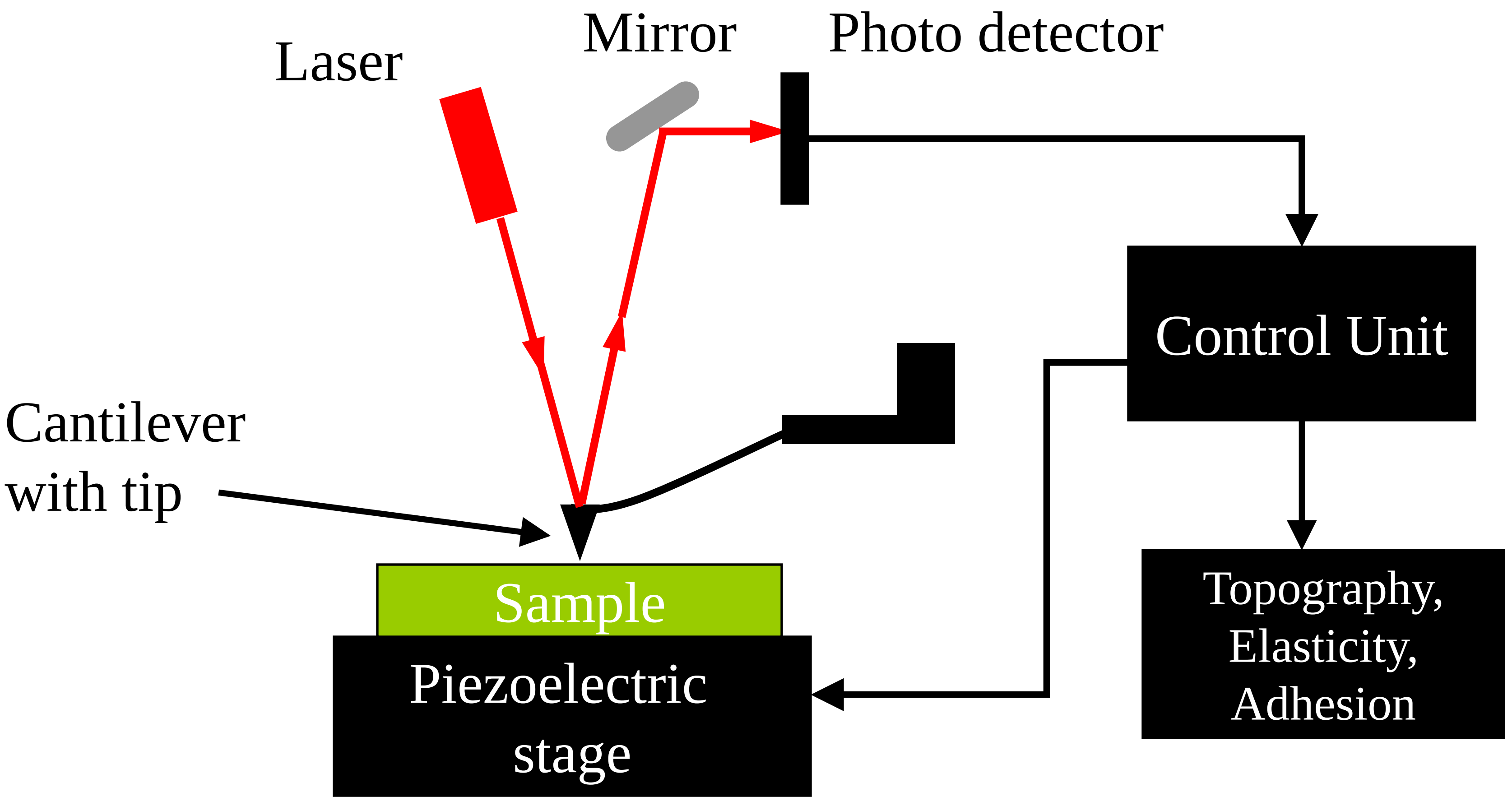
- Measures attractive or repulsive forces between tip and sample
- Detection apparatus measures the vertical deflection for height
- Can achieve resolution of up to 10 pm
- Unlike electron microscopes, can measure samples in air and liquid

How does an AFM work?

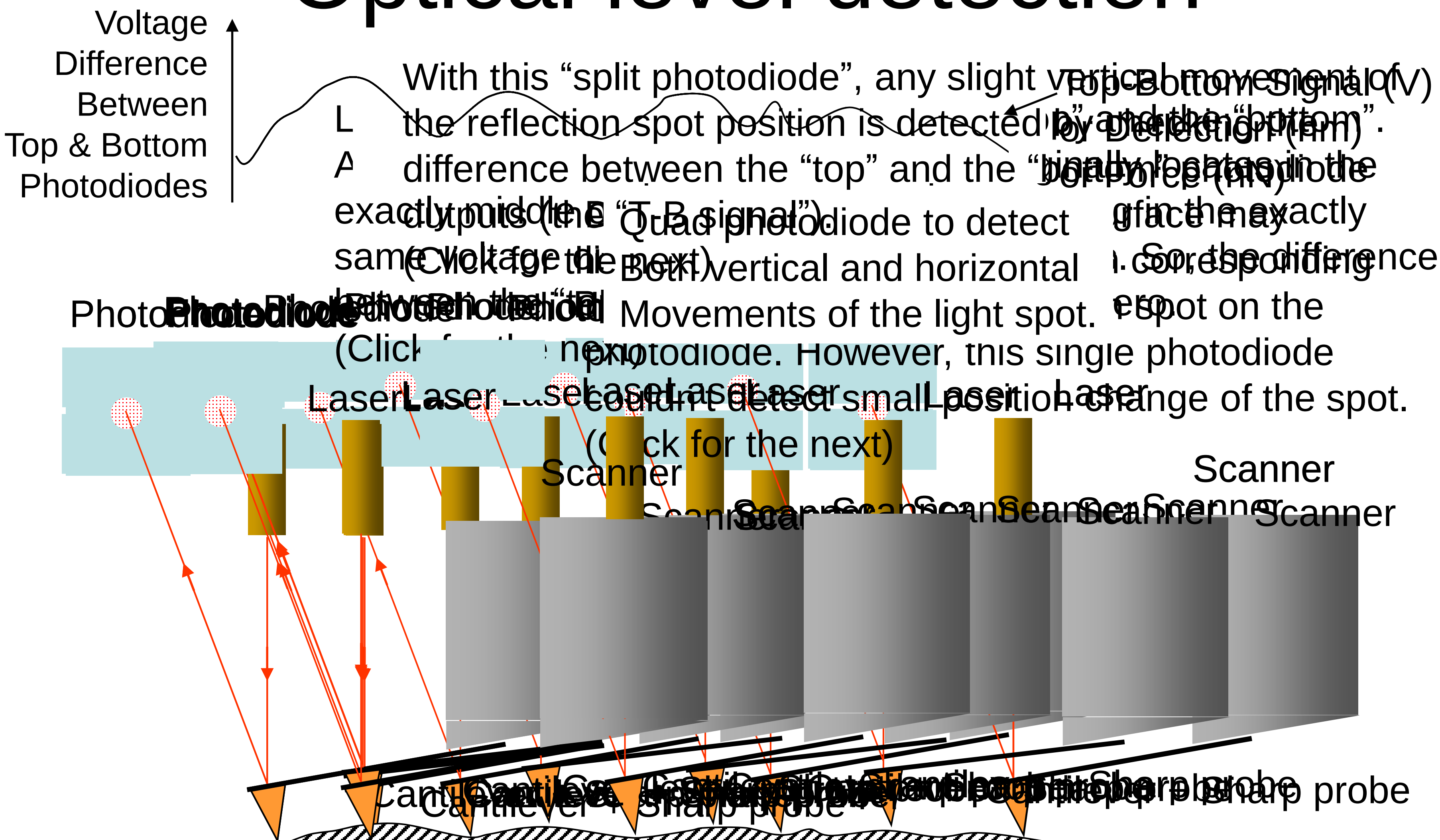


AFM capabilities include measuring topography, surface energy, and elasticity of samples at the nanometer, even molecular scale.

Makeup of the AFM



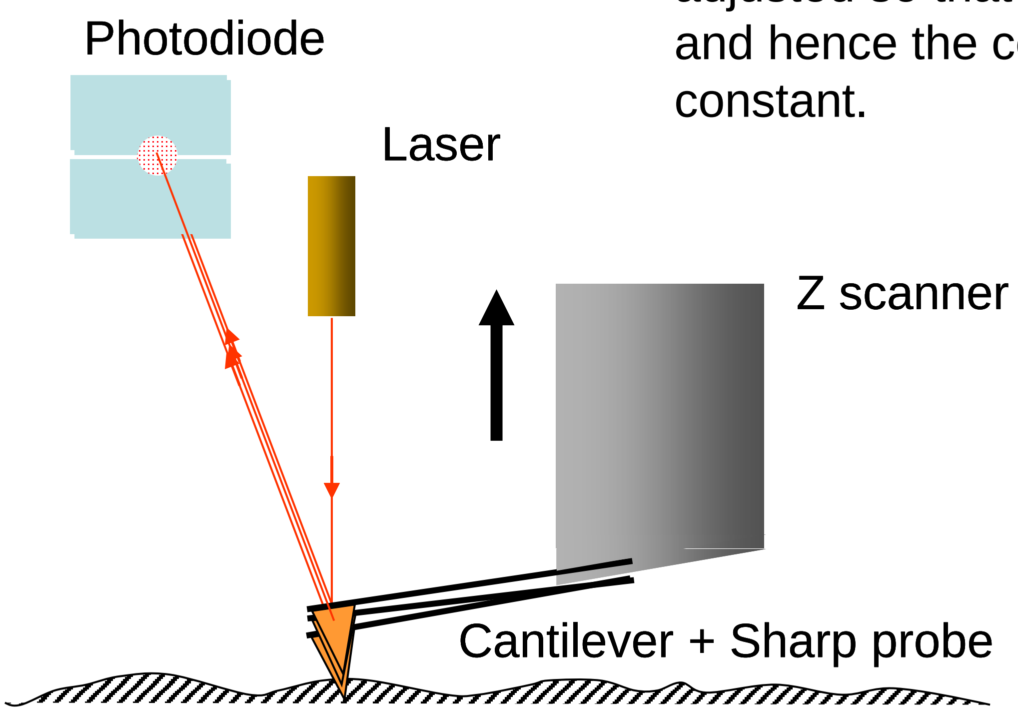
Optical level detection



Click for the Next

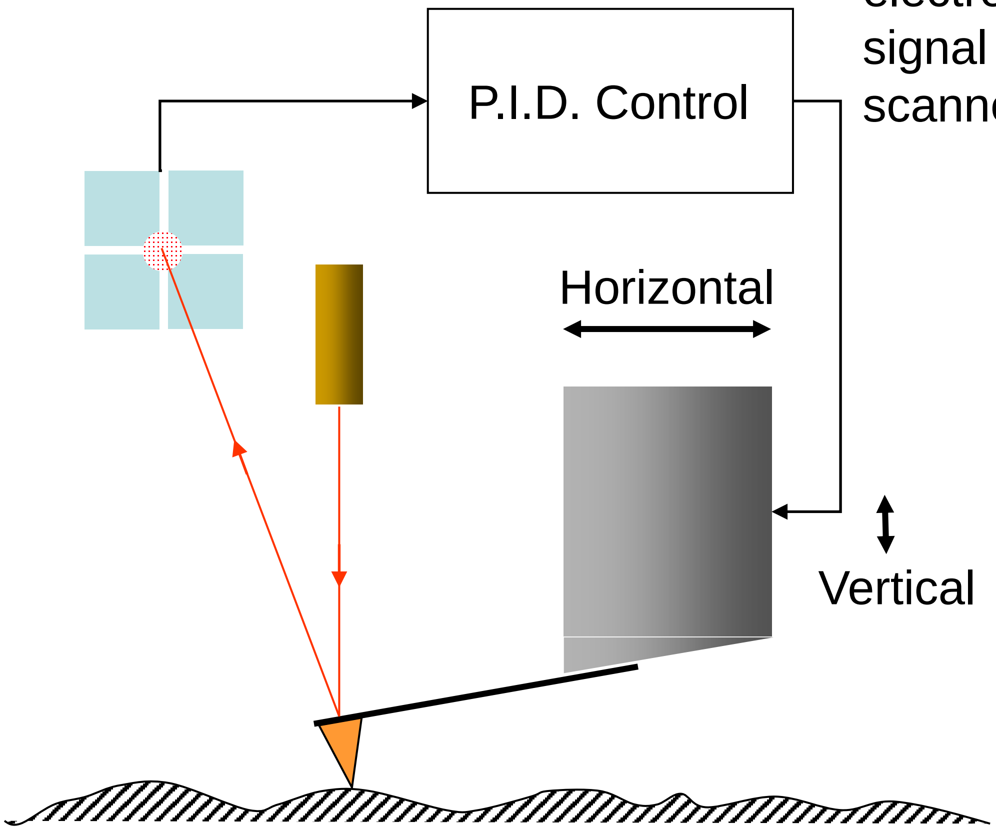
Optical level detection in constant-force mode

In constant-force mode, whenever the sample surface topography would result in the cantilever deflection change, the other end of cantilever would be accordingly adjusted so that the cantilever deflection angle, and hence the contact force, would keep constant.



Click for the Next

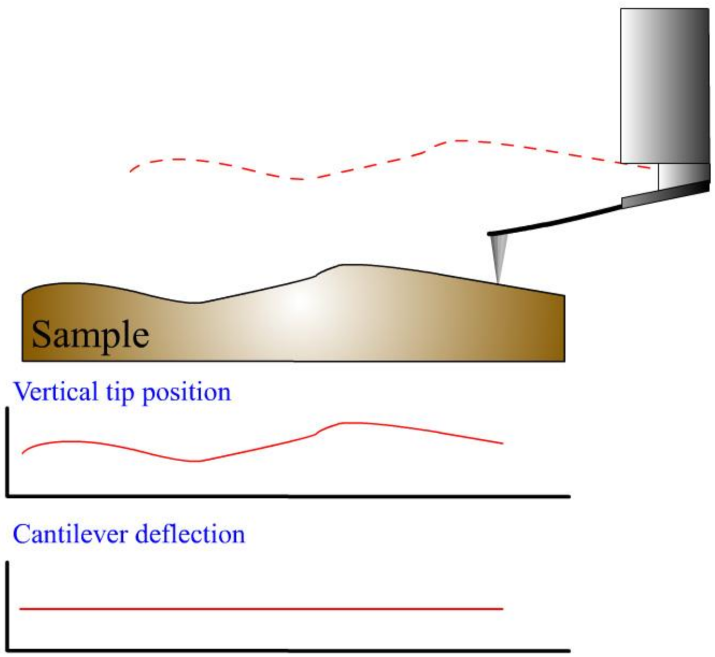
Feedback control in constant-force mode



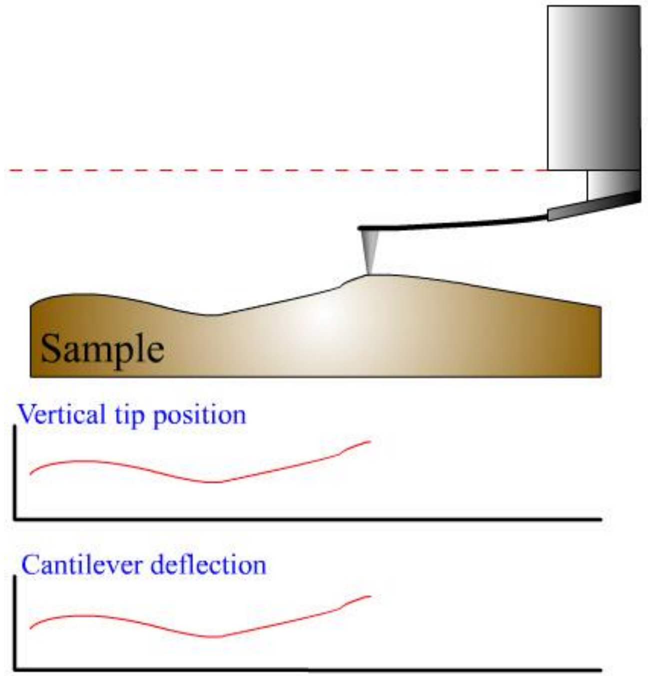
In constant-force mode, the cantilever's vertical position is adjusted by an electronic feedback loop, with the T-B signal as the input and the vertical scanner voltage as the output.

Click for the Next

Constant-force scan vs. constant-height scan



Constant-force mode



Constant-height mode

Click for the Next

Click on graph to play animation (internet connection required)

www.ntmdt.com

Constant-force scan vs. constant-height scan

Constant-force

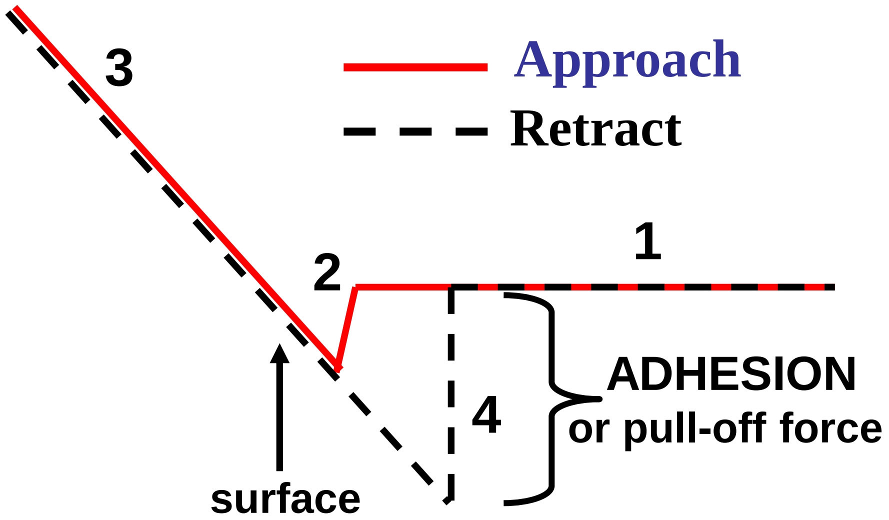
- Advantages:
  - Large vertical range
  - Constant force (can be optimized to the minimum)
- Disadvantages:
  - Requires feedback control
  - Slow response

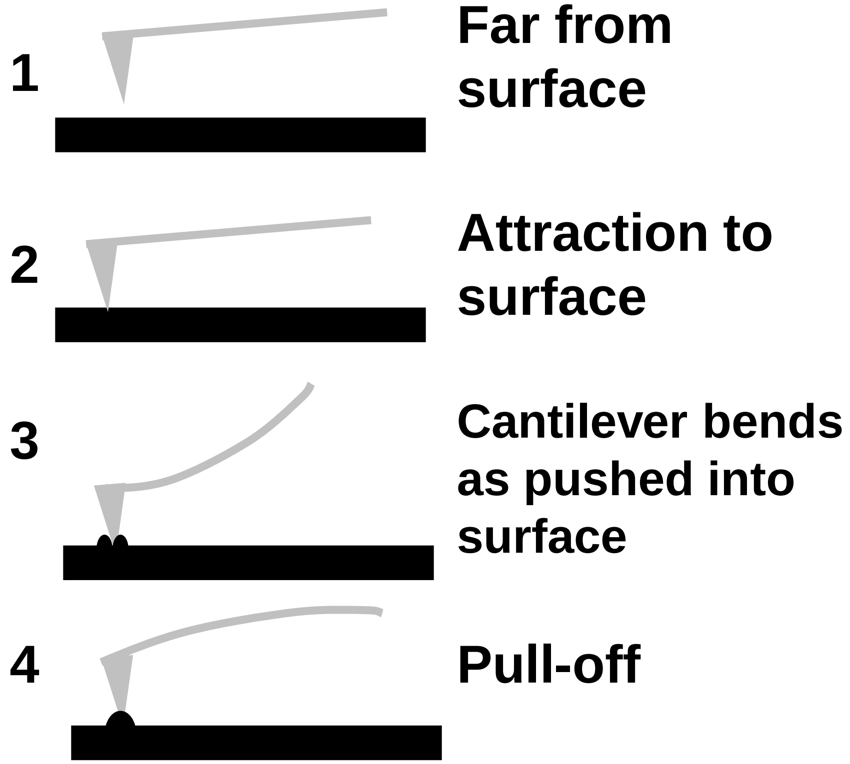
Constant-height

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

Click for the Next

Typical AFM Force Curves

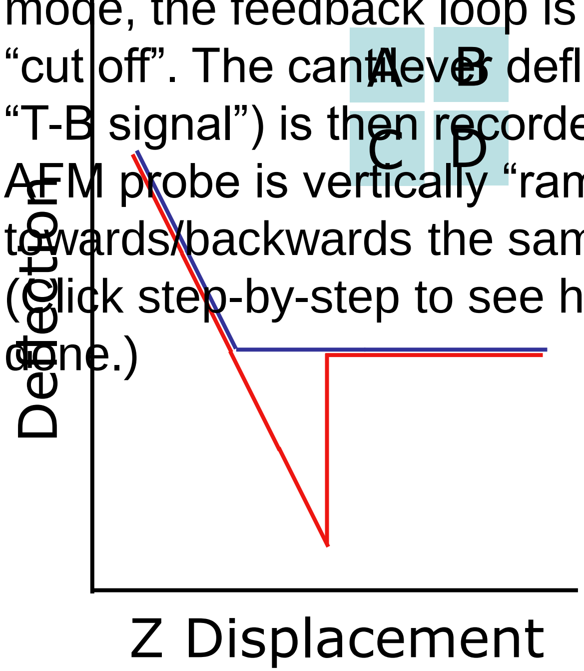


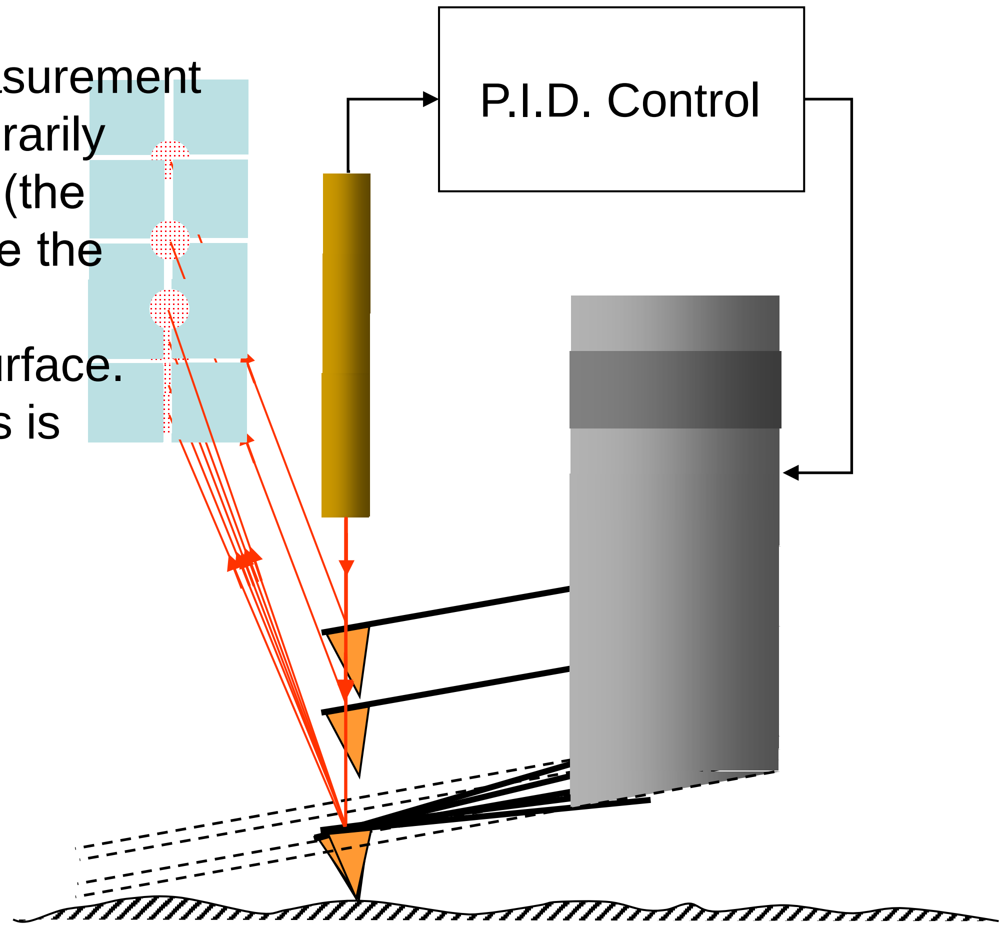


Force measurements with AFM

When an AFM works in force measurement mode, the feedback loop is temporarily "cut off". The cantilever deflection (the "T-B signal") is then recorded while the AFM probe is vertically "ramped" towards/backwards the sample surface. (Click step-by-step to see how this is done.)

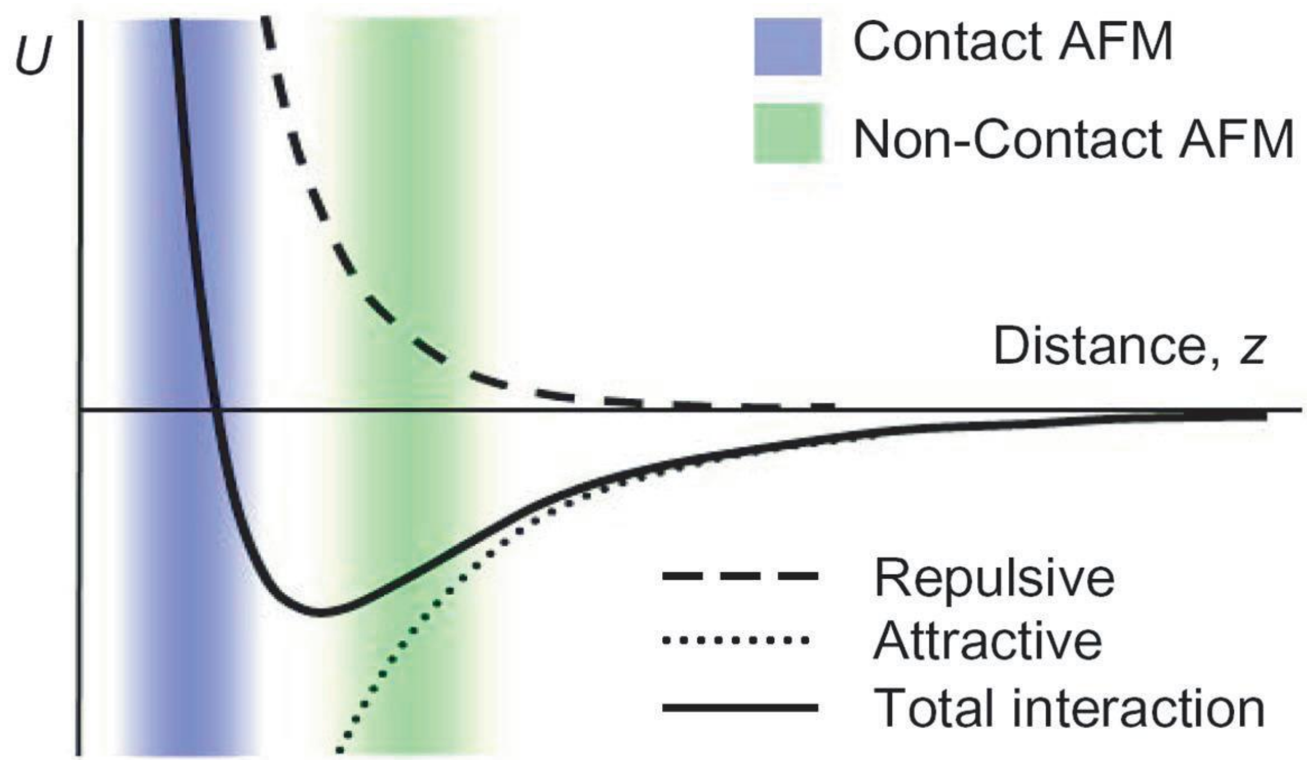
$$\text{Defl} = \frac{(A+B)-(C+D)}{A+B+C+D}$$

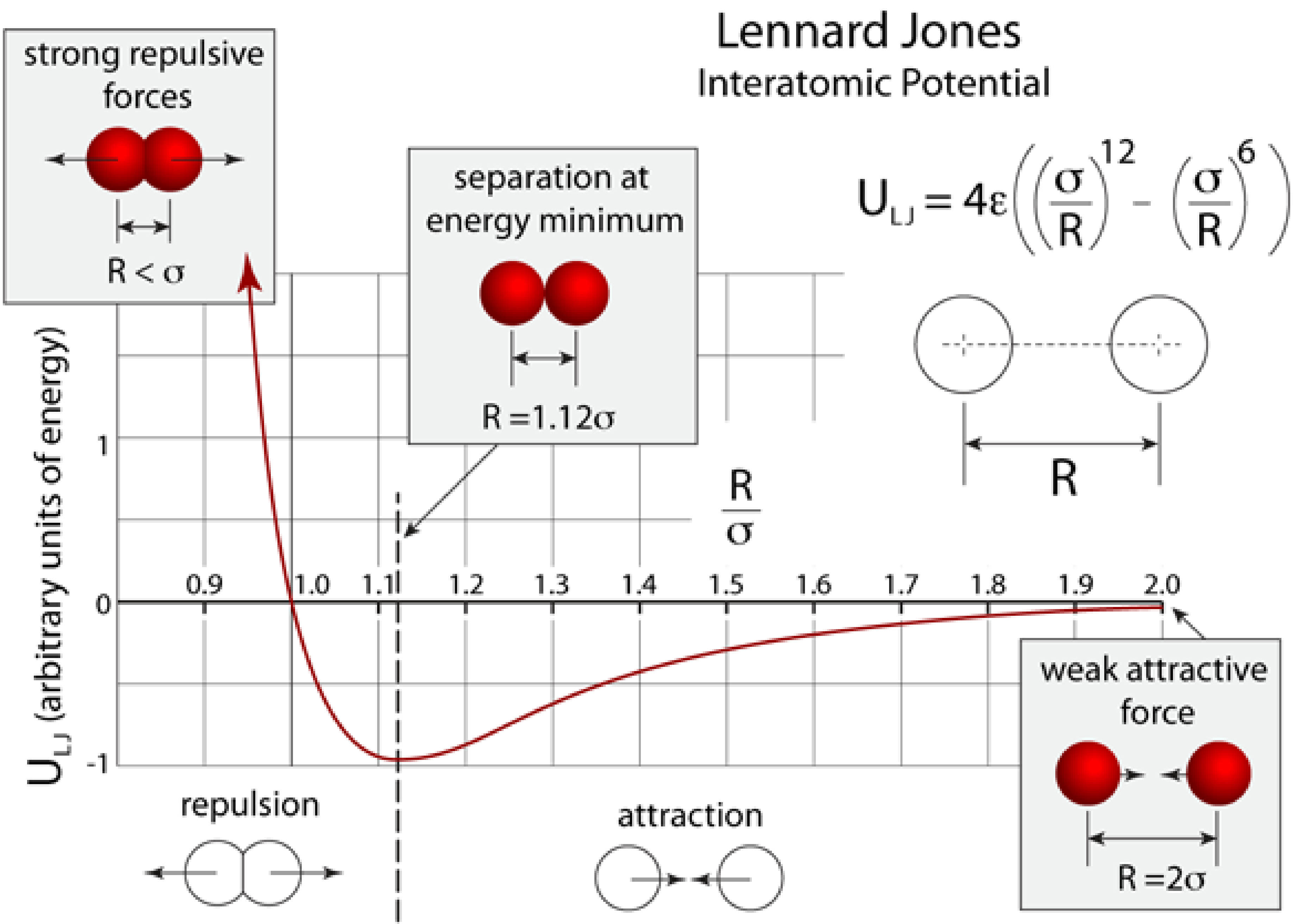




Click for the Next

Typical AFM Force Curves

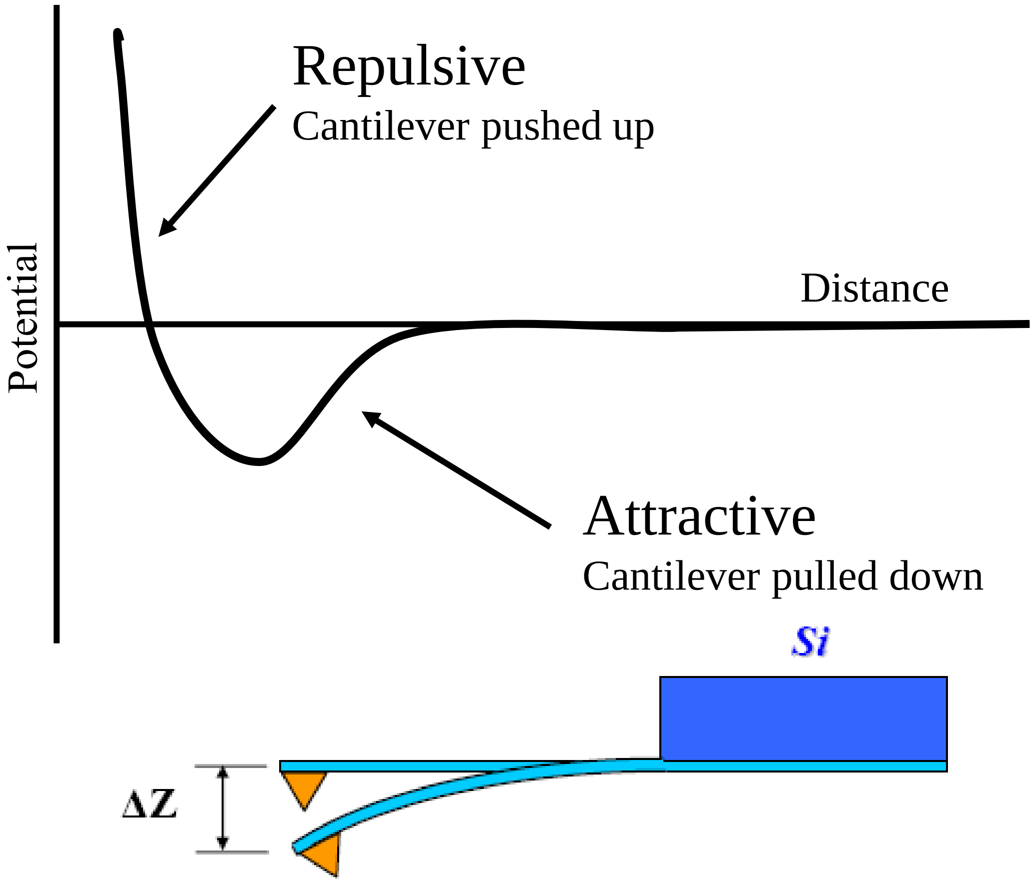




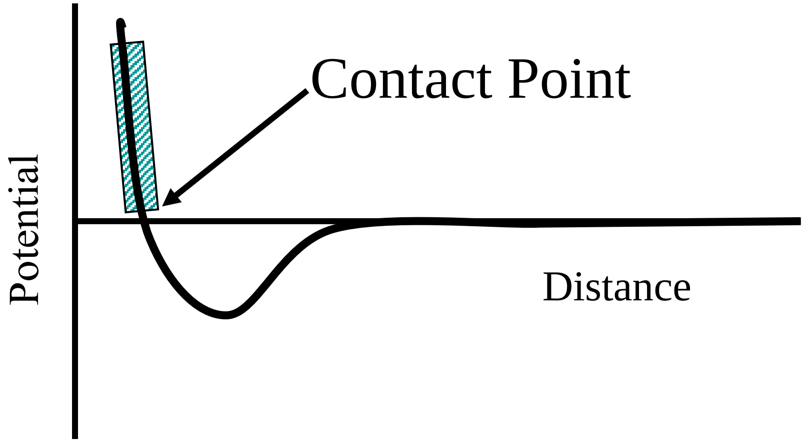
Lennard Jones
Interatomic Potential
$U_{LJ} = 4\varepsilon\left(\left(\frac{\sigma}{R}\right)^{12} - \left(\frac{\sigma}{R}\right)^{6}\right)$
strong repulsive forces
$R < \sigma$
separation at energy minimum
$R = 1.12\sigma$
R
$\frac{R}{\sigma}$
0.9
1.0
1.1
1.2
1.3
1.4
1.5
1.6
1.7
1.8
1.9
2.0
1
0
-1
U_{LJ} (arbitrary units of energy)
repulsion
attraction
weak attractive force
$R = 2\sigma$

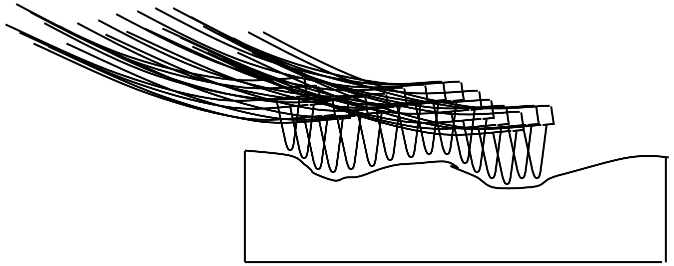
Typical AFM modes

Potential Diagram

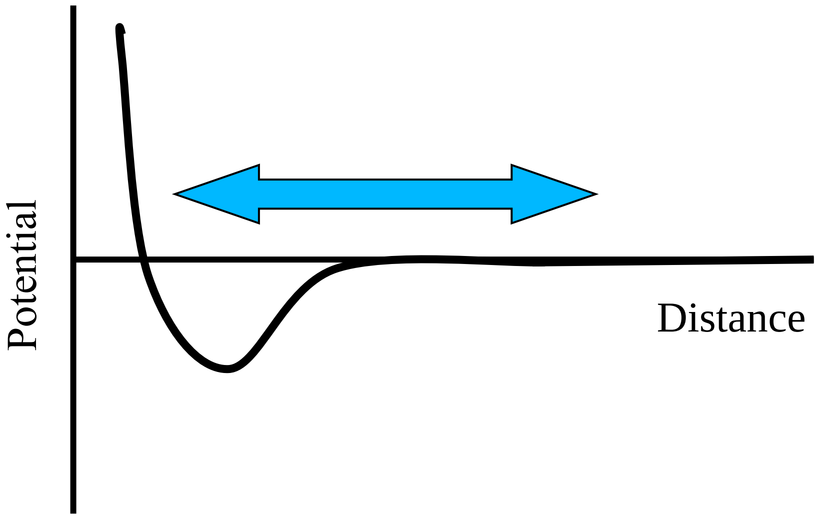


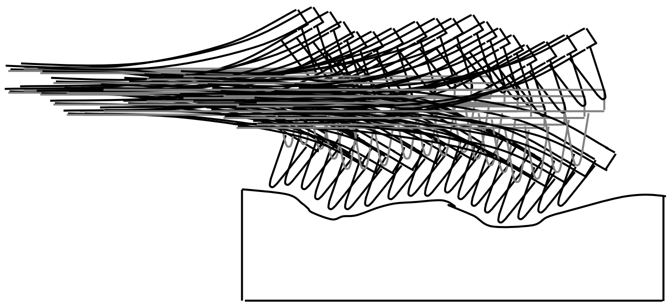
Continuous - Contact





Vibrating Mode



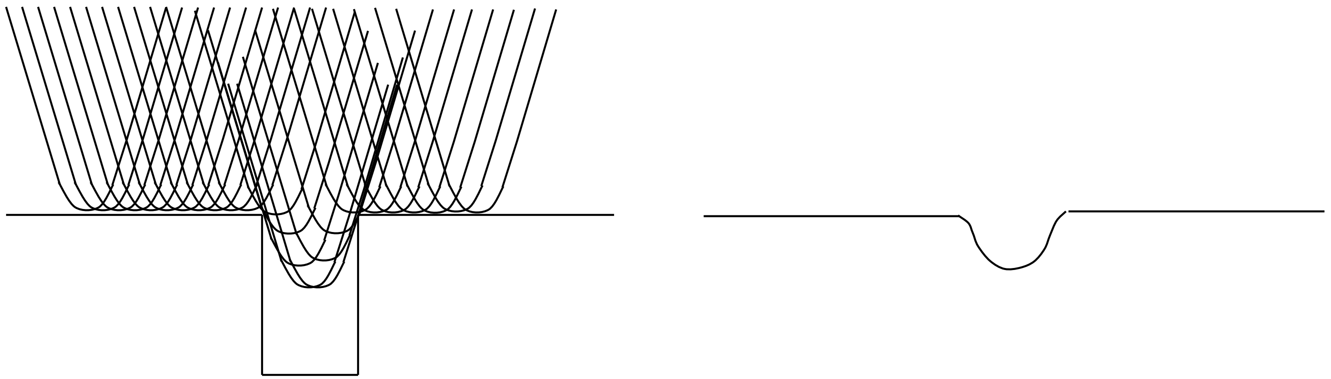


-The tip is vibrated in and out of the potential associated with the surface.

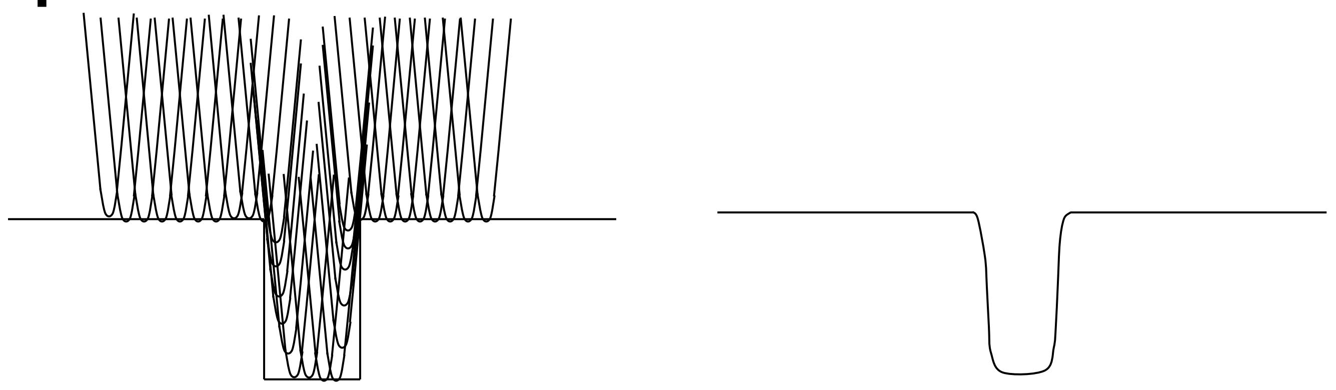
- Large or small amplitudes
- Advantages are: low forces, reduced lateral forces

Probe Geometry

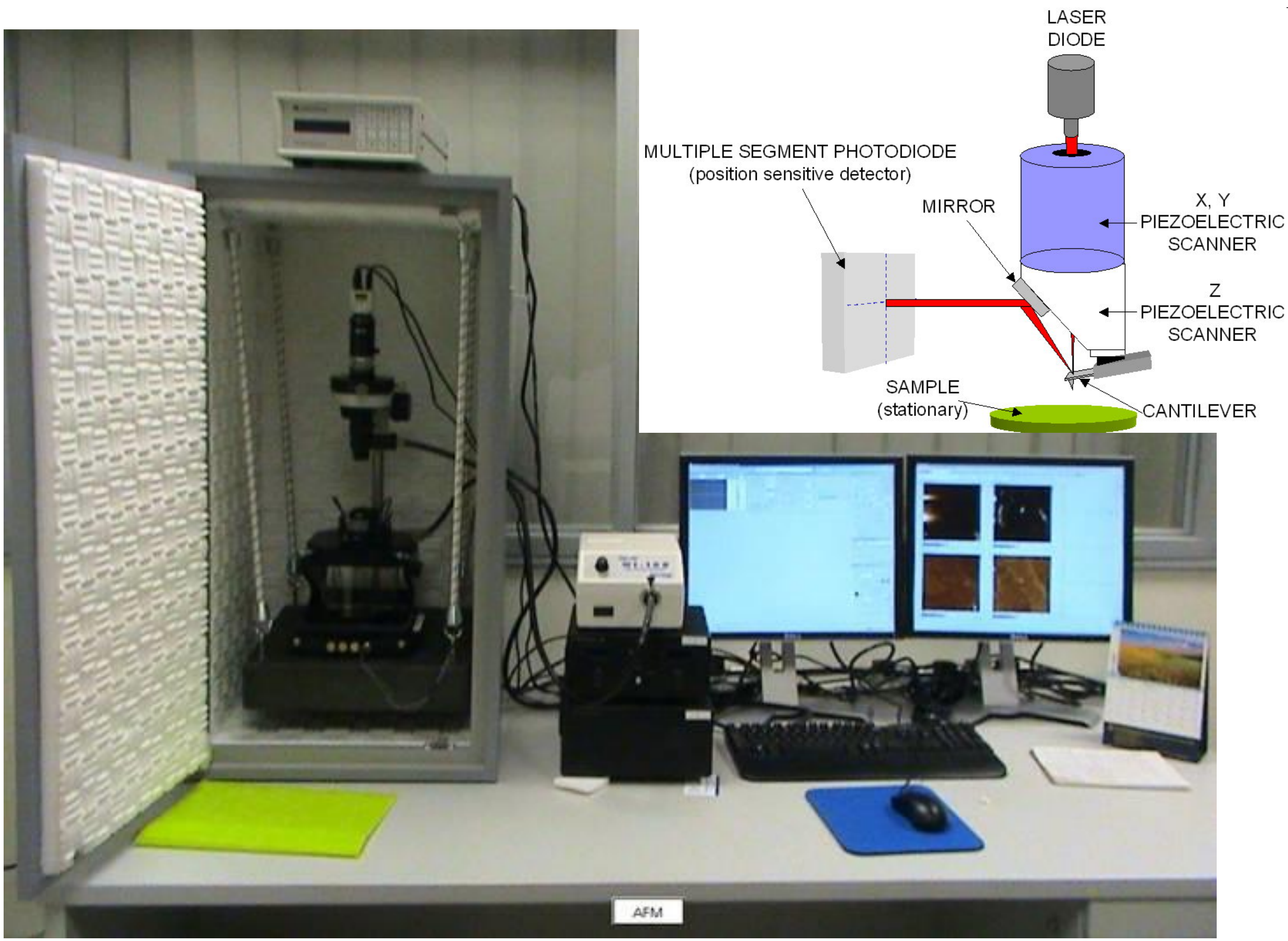
Dull Probe



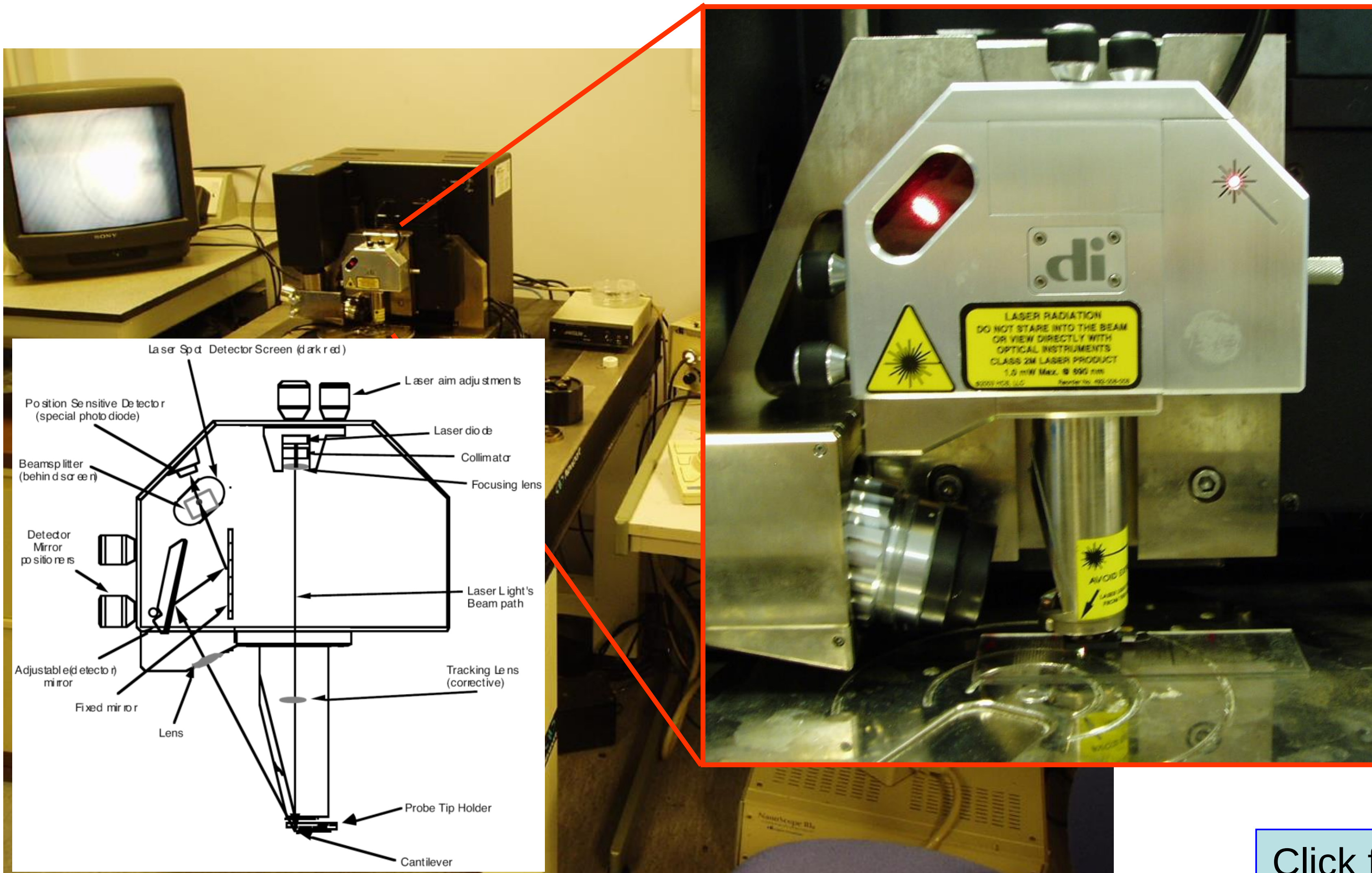
Sharp Probe



AFM Facility at Bharathidasan University - Agilent 5500



Dimension AFM

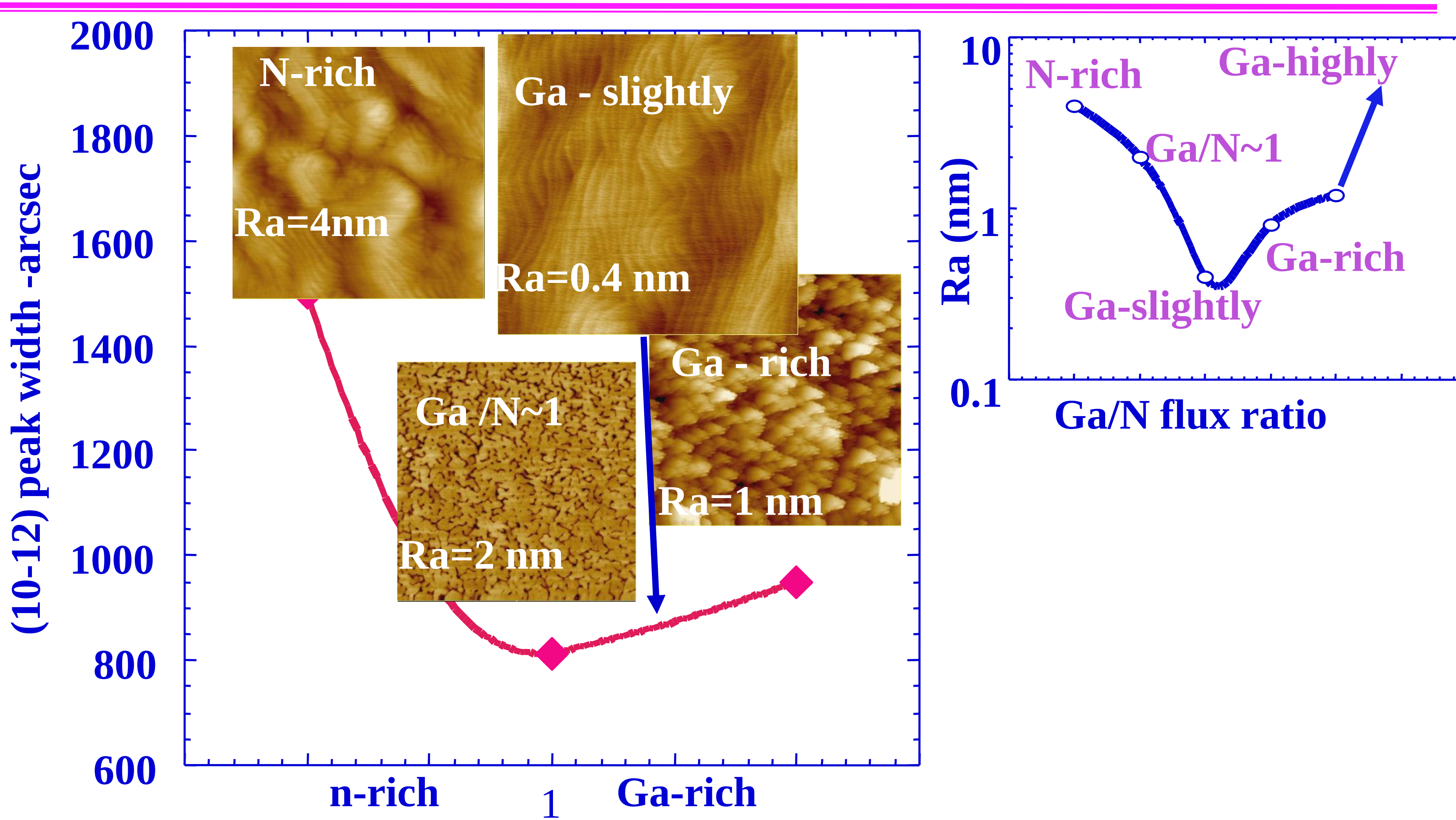


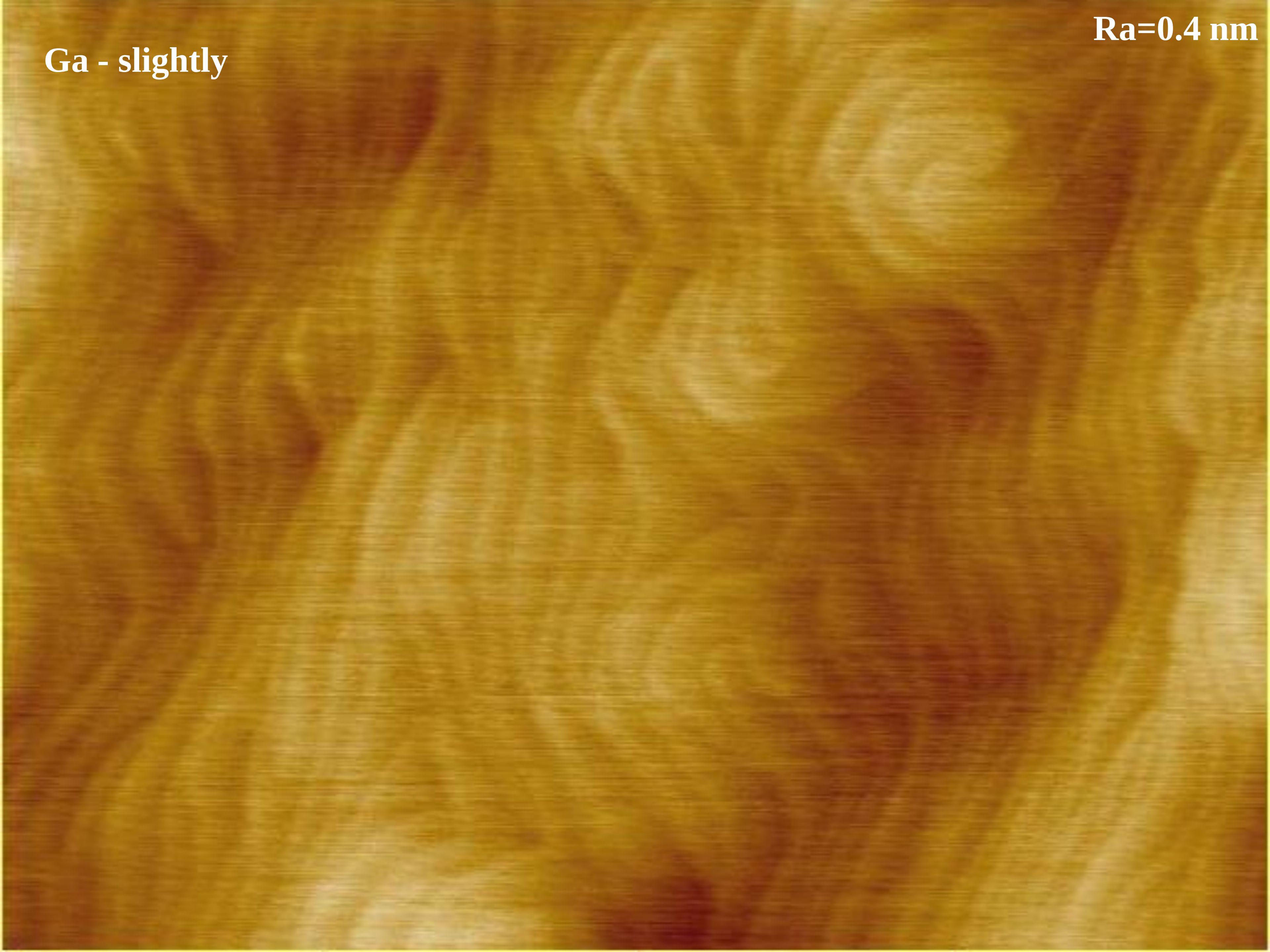
Click for the Next

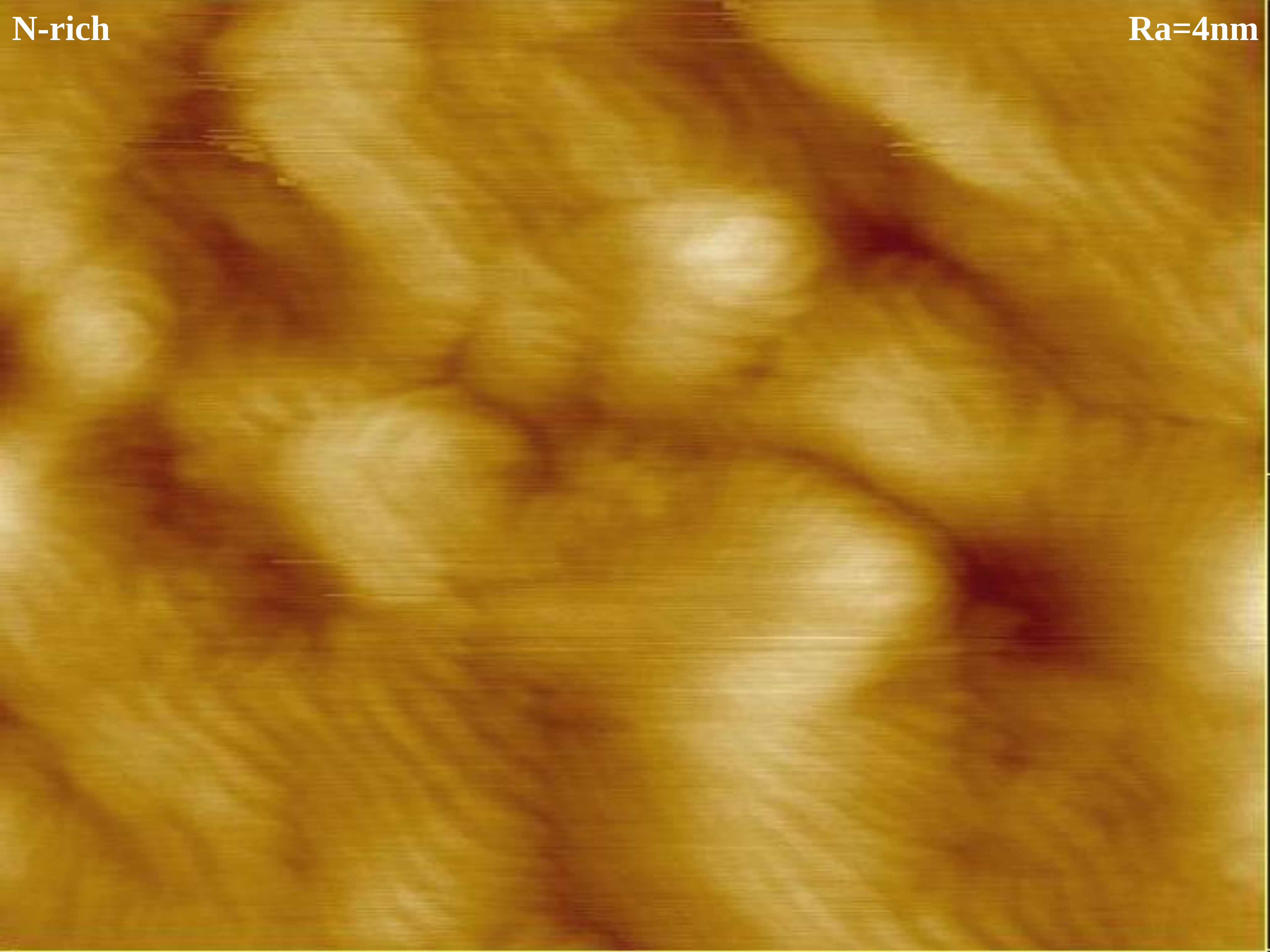
MultiMode AFM

Click for the Next

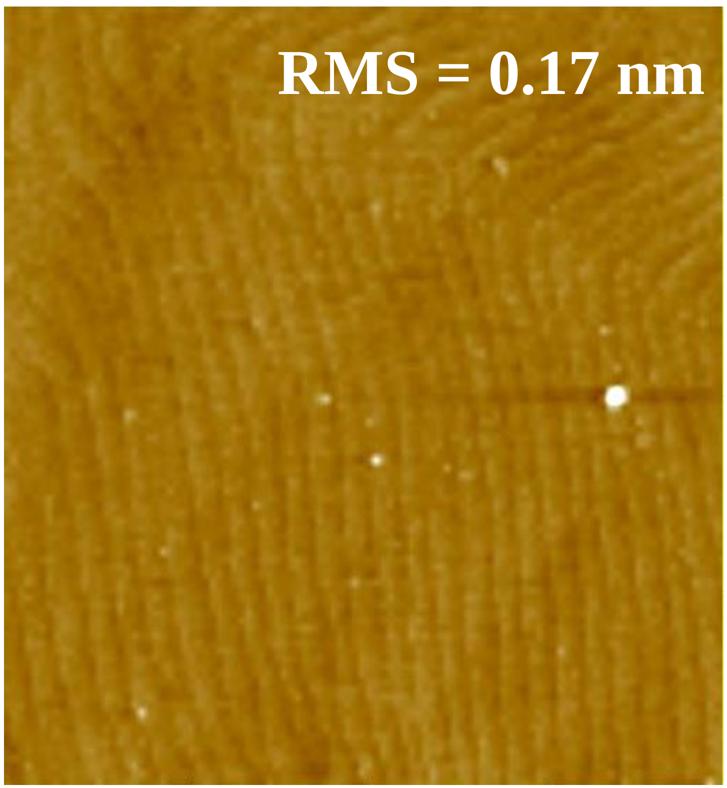
Dislocation vs. Ga flux

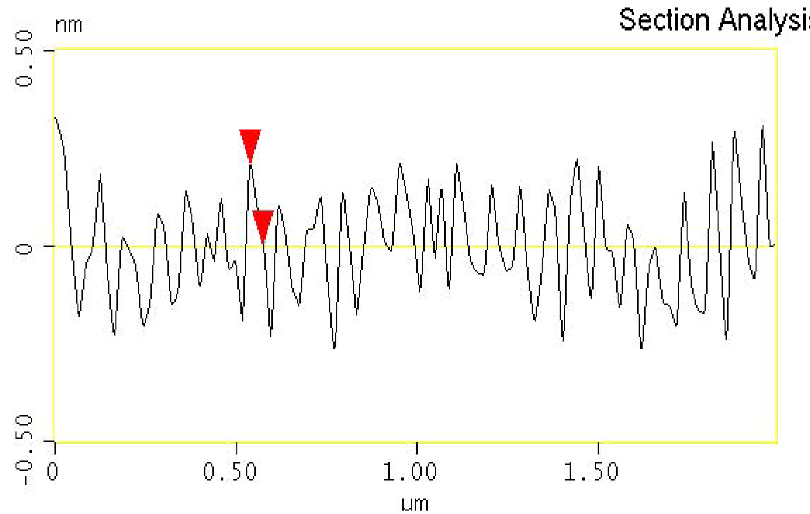



Ga - slightly
Ra=0.4 nm


N-rich
Ra=4nm

AFM image of GaN





30-nm-$In_xAl_{1-x}N$

1-nm-AlN

500-nm-GaN MBE

3-µm-MOCVD GaN

AFM -Cantilevers

- Choice depends on application, k- spring constant, ω -resonant frequency

- **Simplest geometry L-shape (bent wire and etching),**
- **ideal for non-contact applications and friction measurements because of strong tangential forces**

- **Single V-shape and double V-shape are ideal for contact measurements**

- **Resonance frequency (ω) should be higher then data acquisition and noise level (kHz range)**

$$\omega = \sqrt{\frac{E}{\rho}}\frac{r}{l^2}$$

- **To increase ω wire should be stiff, thin, and short**

AFM -Cantilevers

- **Soft cantilever (small k) increase sensitivity, problem of crashing**

- **In non-contact soft cantilever will require long distance, reduce resolution**

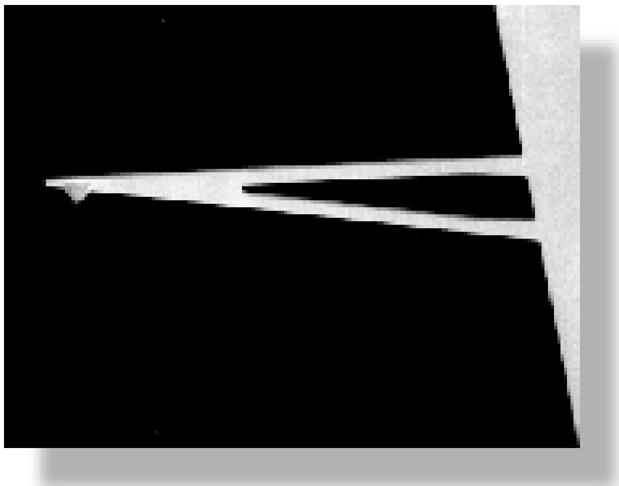
- **For triangular cantilevers:**

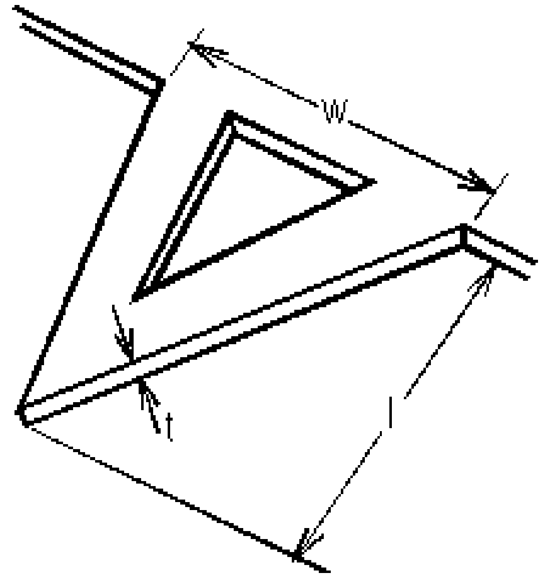
$$k \sim \frac{Er^4}{l^3} \qquad \omega = \sqrt{\frac{E}{\rho}}\frac{r}{l^2}$$

- **Compromise between small k and large ω**

- **Determination of k and ω**

AFM – Triangular Cantilevers





$$k = \frac{Ewt^3}{4l^3},$$

E - Young Module

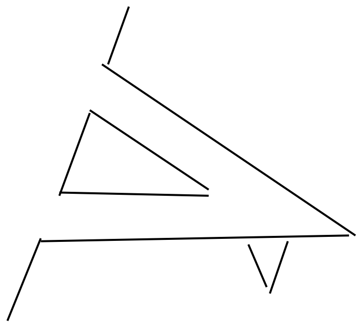
w – width, l - lenght, t - thickness

Comercial cantiliver s - SiO_2 and Si_3N_4

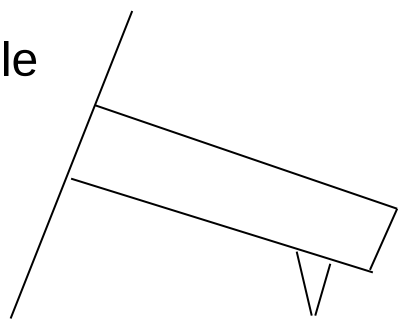
Tip radii - 300 $\mathring{A}$

Cantilever/Probe

Triangle



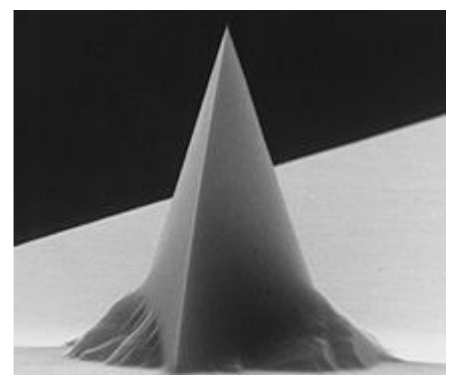
Rectangle



-Cantilevers are either triangular or rectangular shaped.

-Material can be Silicon on Silicon Nitride (plastic in development)

- Probes shapes can be pyramidal or conical and may be sharpened.



AFM -Cantilever Fabrication

- **Si is etched through a window in the mask to twice the thickness of cantilever using 37.5 wt%**

KOH. (ER <111>: <100>: <110>=1:300:600).

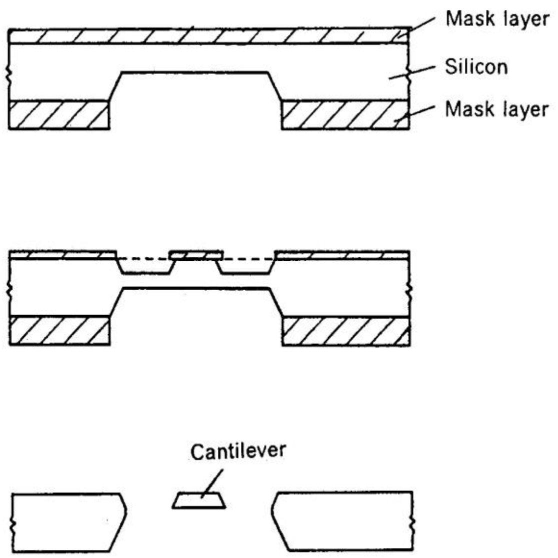


FIG. 2. Fabrication of silicon cantilever. above: The silicon wafer is etched through a window in the mask layer from the backside until the thickness equals twice the desired thickness of the cantilever. middle: The cantilever is patterned by lithography on the front side and consecutively etched into the silicon until below: both etch fronts meet and the cantilever is released.

- **Cantilever is defined by lithography,**

  and then released by etching from both sides

- **Shape of cantilever facets depend on**

  orientation of mask relative to crystal orientatio

AFM -Tip Fabrication

- **Using a circular mask Si is etched away in the surrounding of the mask. Then the mask is completely under etched.**

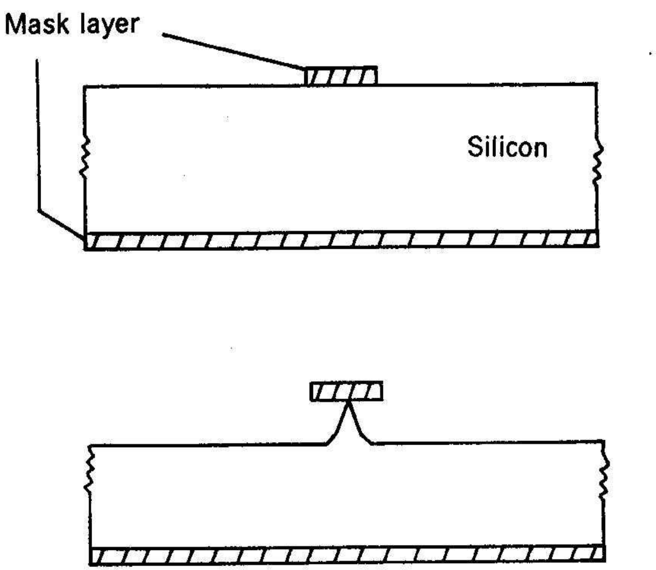
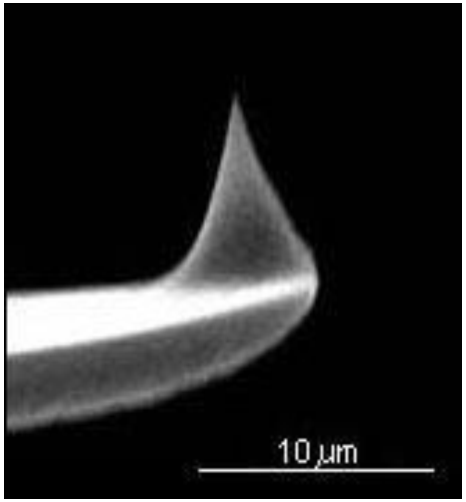
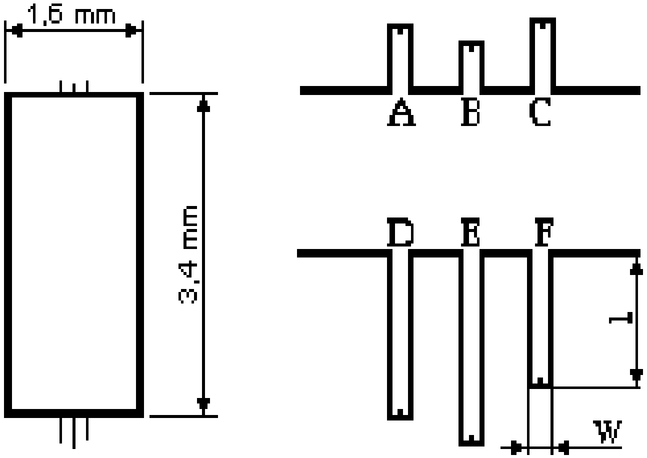


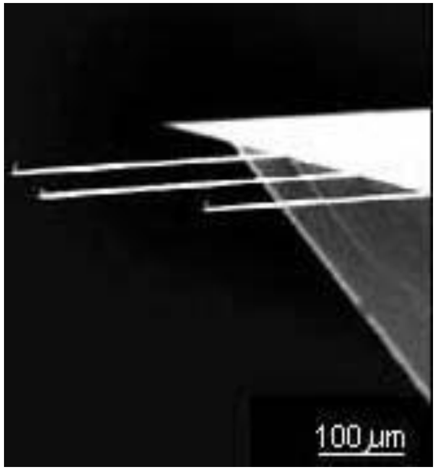
FIG. 4. Fabrication of ⟨100⟩ oriented silicon tips. above: Starting with a circular mask on a ⟨100⟩ oriented wafer the silicon is etched away in the surroundings of the mask (KOH solution). below: Simultaneously the mask is undercut until it is completely under etched. The etching is stopped and a silicon cone remains.

- **Tips should be mechanically robust for contact**
- **The surface force (~ 1 nN) with 10 nm radius tip creates a pressure of ~ 100 atm !**
- **Tips, cantilever, and holder are made of one piece**
- **single crystalline parts yield good mechanical properties**

AFM -Tips



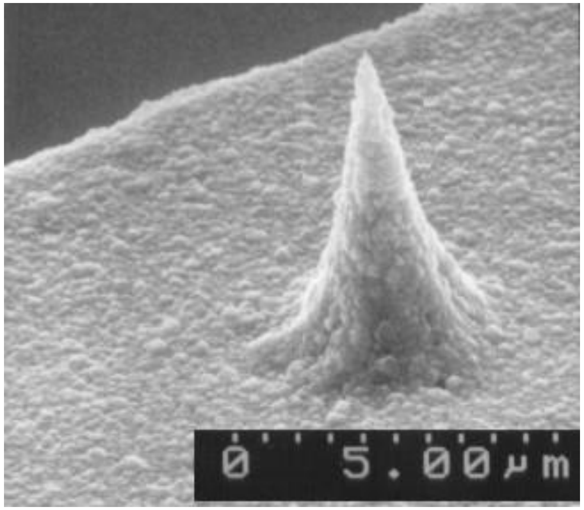




AFM -Tips

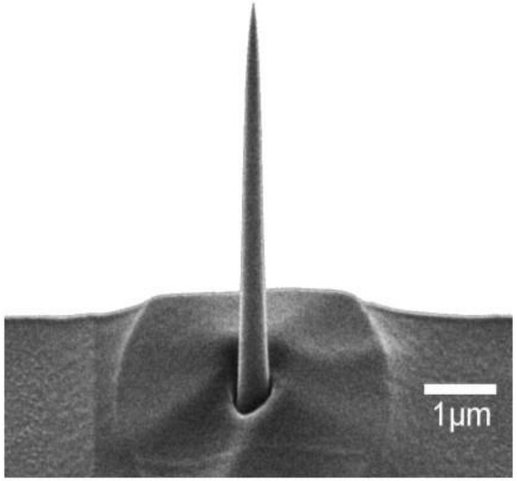
Diamond-coated AFM tip

Sharp Tip

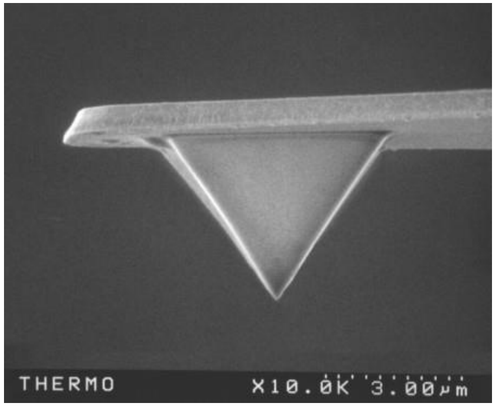


- **Pyramidal, tetrahedral, or conical tips are the most common tip shapes**

FIB



Gold-coated Si_3N_4 Tip

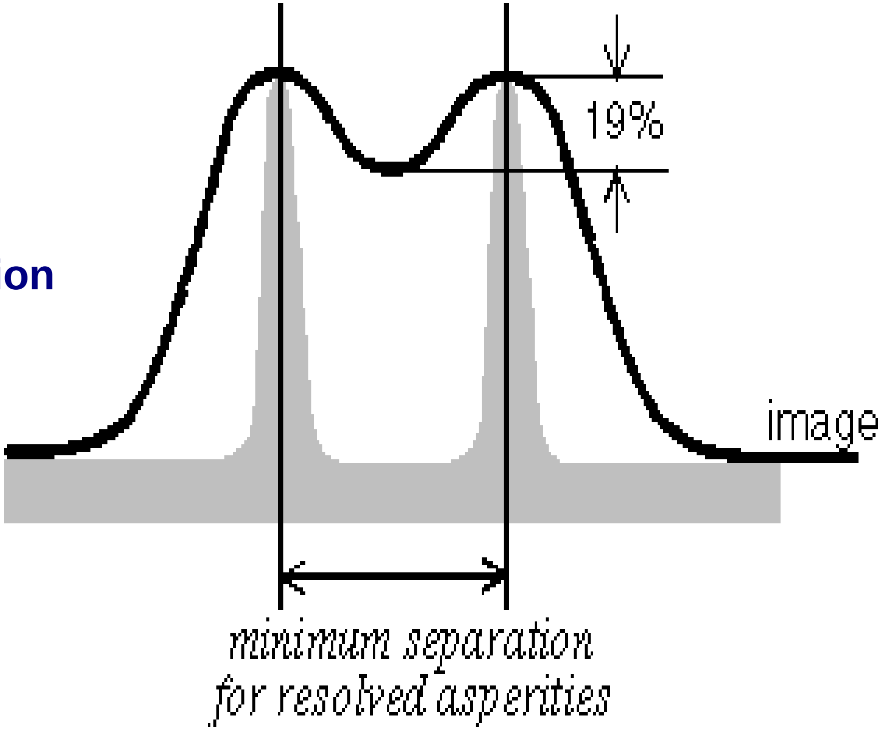


AFM - Resolution

Lateral resolution is determined by step size and tip radius

The sharpest tips are with r ~ 50 Å

Resolution criterion, determining resolution reference samples

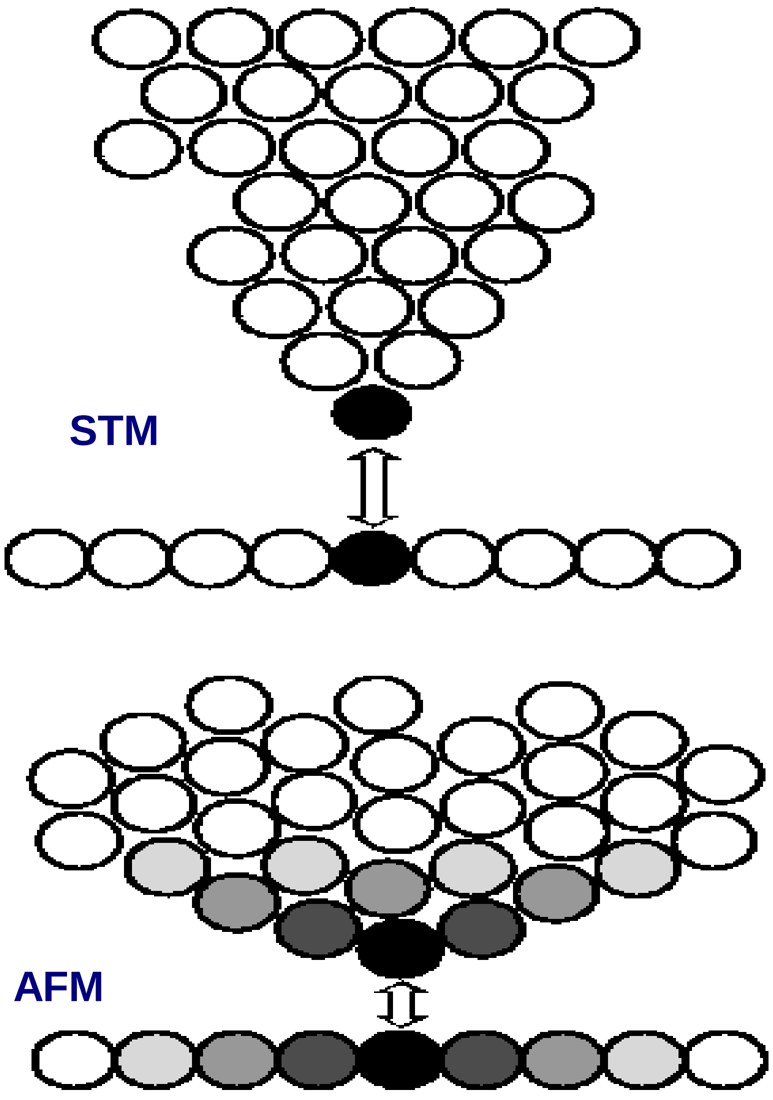


AFM -Resolution

- STM-single atom interaction

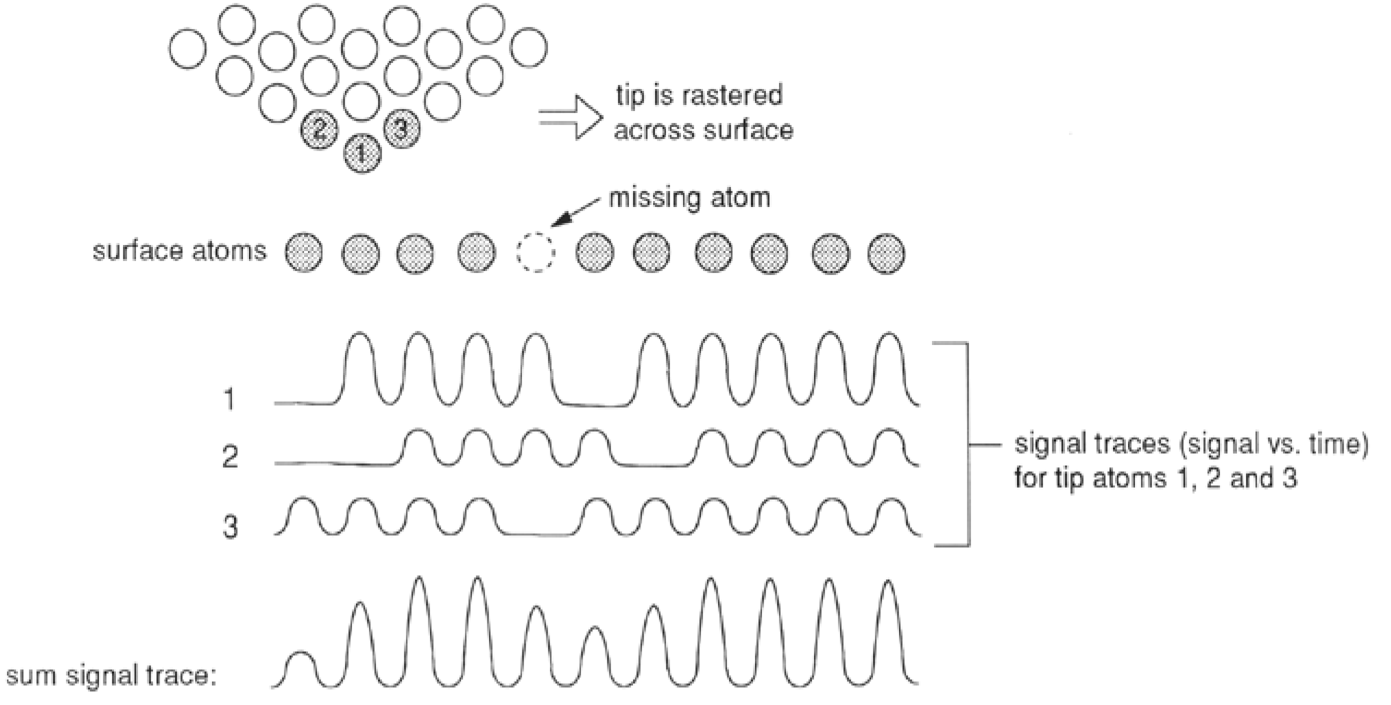
- AFM-several atoms on tip interact with several atoms on surface

- In contact, not necessarily a single atom contact, radius of contact ~$(Rd)^{1/2}$ (d-penetration depth, R-radius of tip)

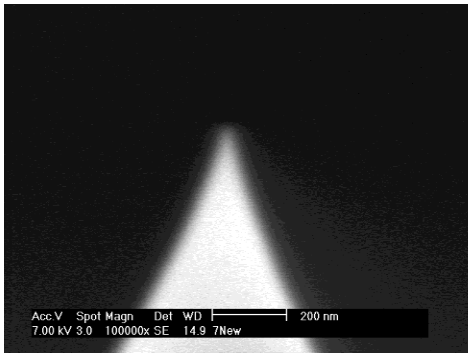


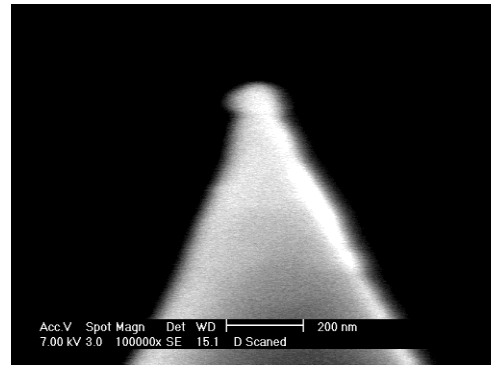
AFM -Resolution

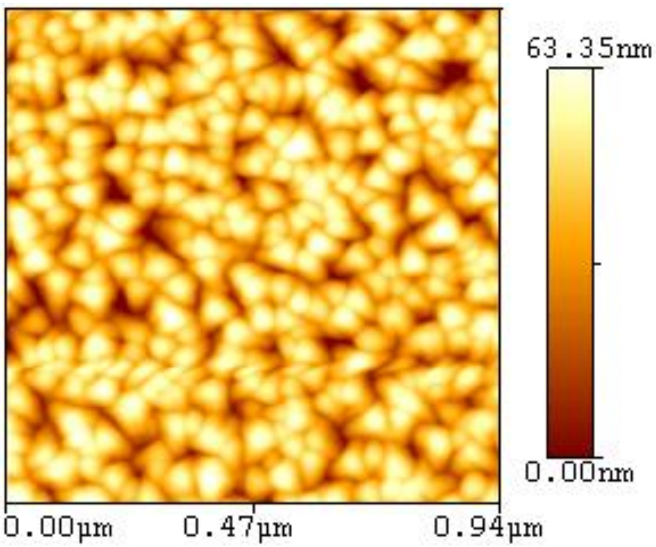
- **Interaction of atom 1 : t = 0 different from interaction of atom 3,2**
- **Each tip atom produces a signals with offset to each other**
- **Periodicity reproduced but no true atomic resolution**

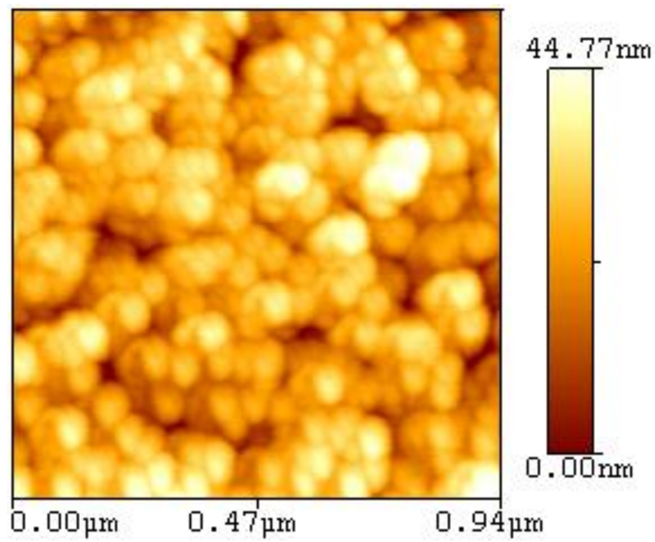


Good/Bad Tip Approach

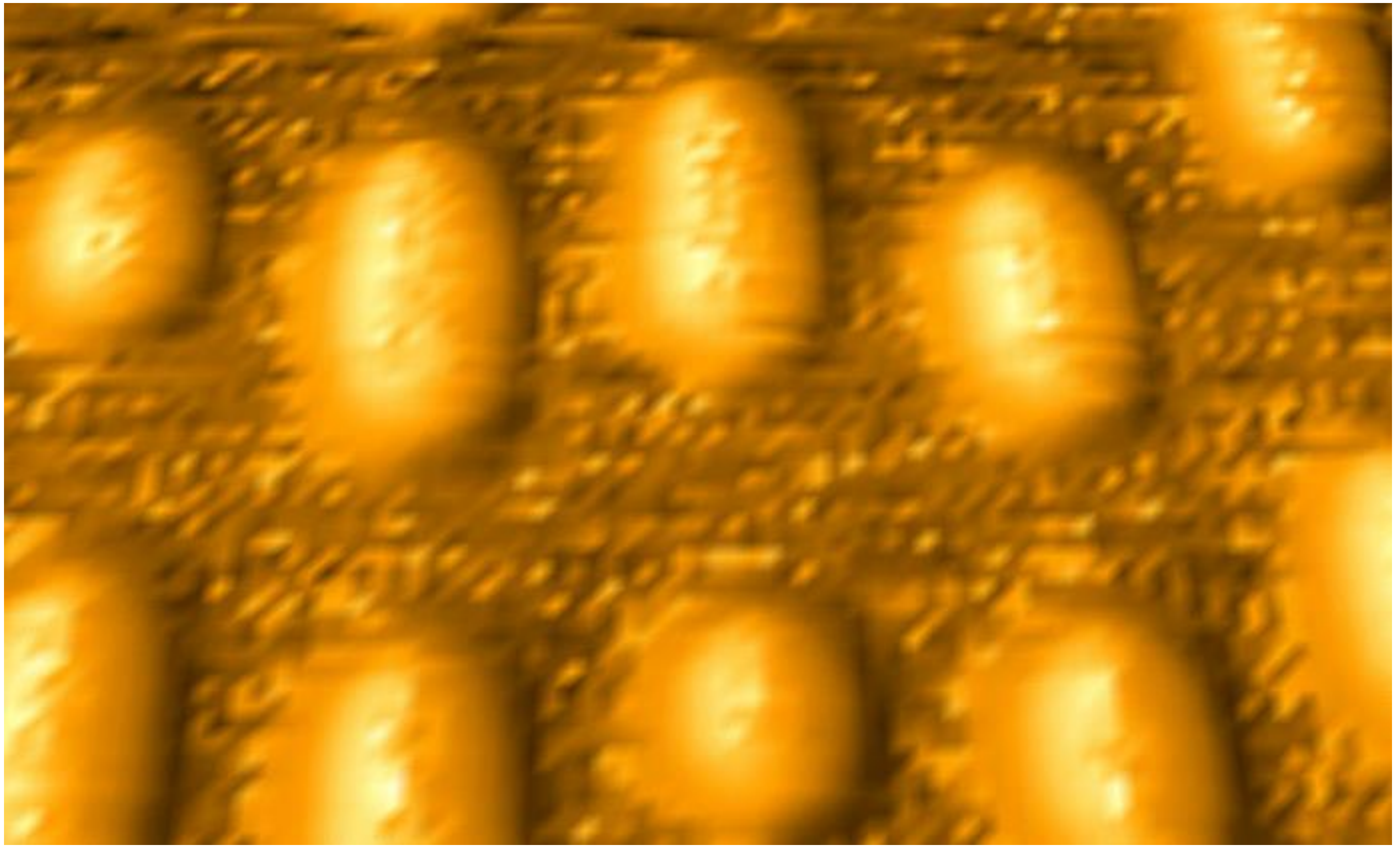




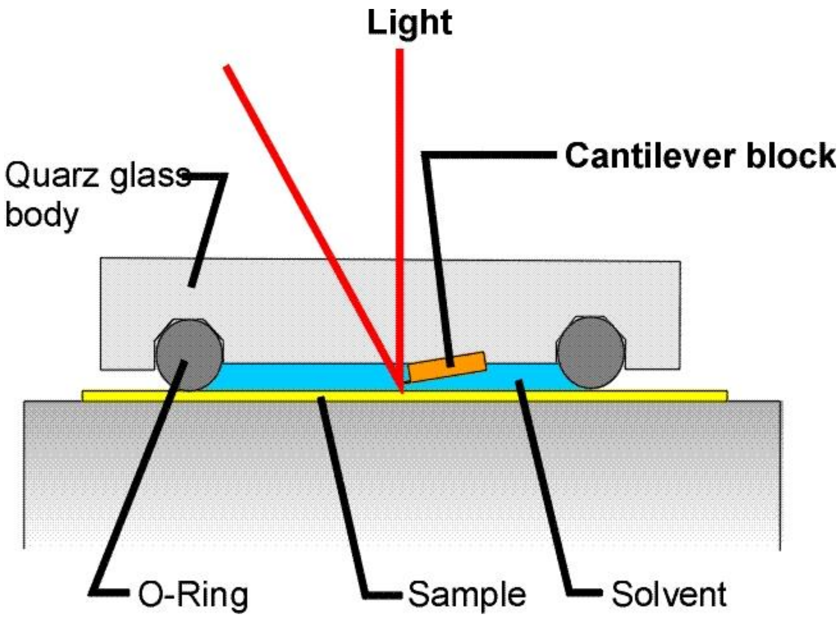




Display



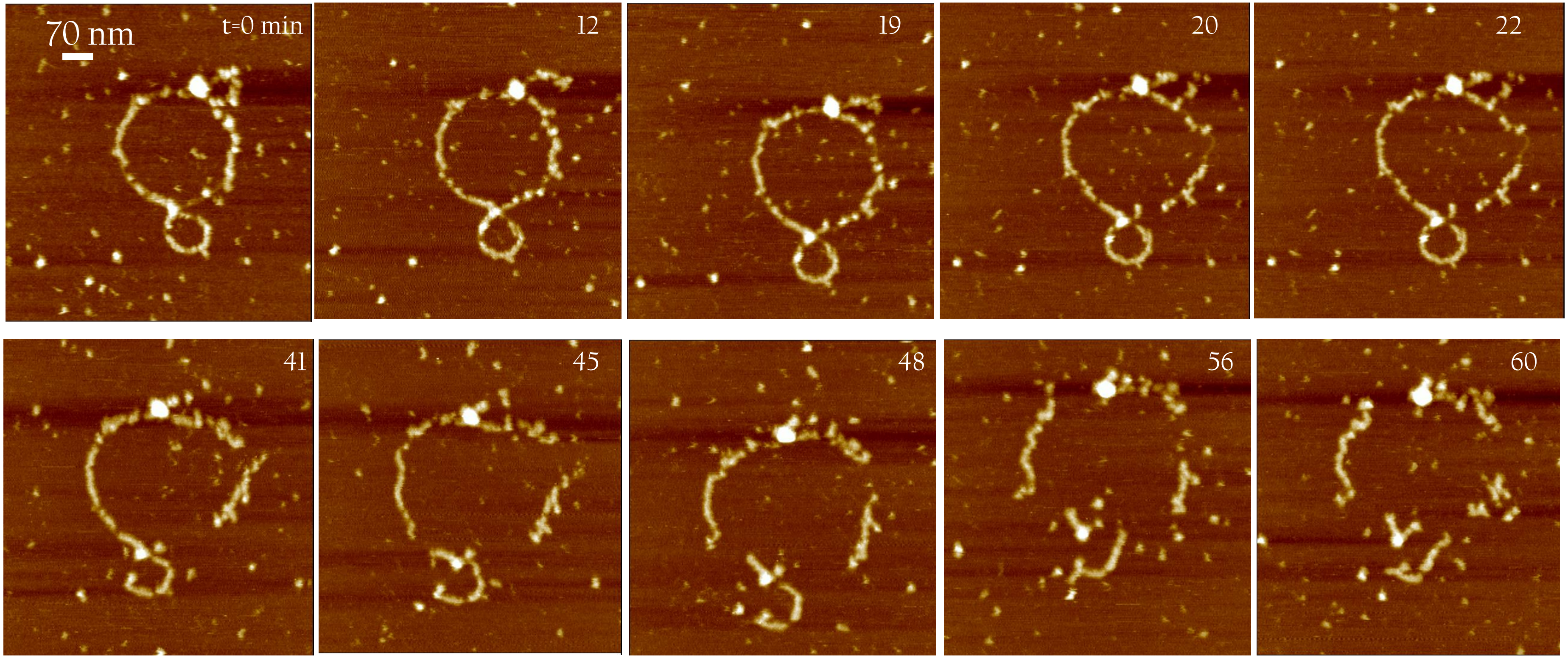
AFM in liquid environment



One extraordinary feature of AFM is to work in liquid environment. A key point for liquid AFM is a transparent solid (usually glass) surface, which, together with the solid sample surface, retains the liquid environment whilst maintains stable optical paths for the laser beams. An optional O-ring can be used to form a sealed liquid cell. Otherwise, the system can also work in an "open cell" fashion.

Click for the Next

Liquid AFM Images



Effect of DNase I enzyme on G4-DNA (0.5:1) complex, the complex was immediately adsorbed onto mica and imaged until stable images were obtained, then the DNase I was introduced.

Click for the Next

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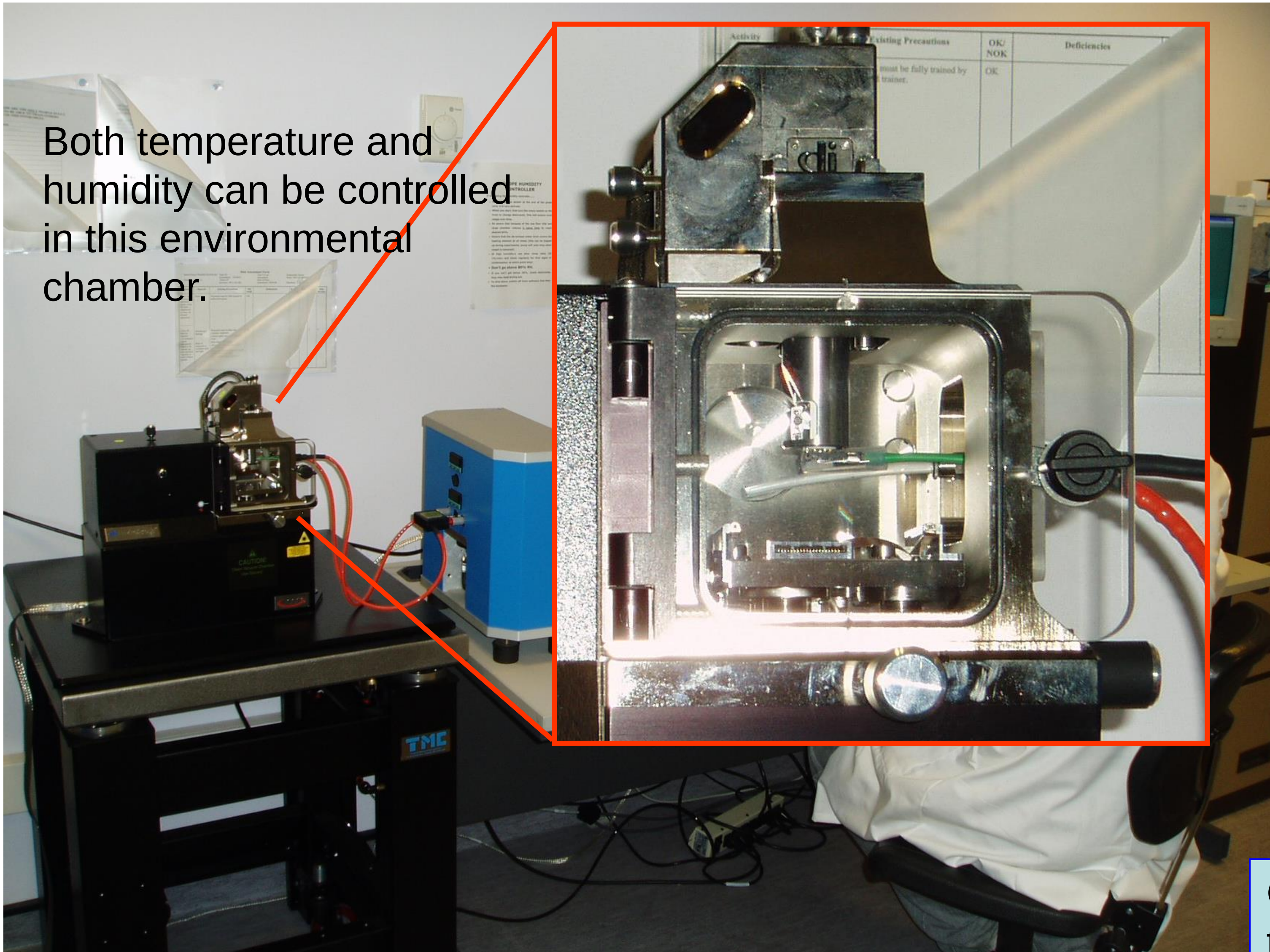
Environmental AFM



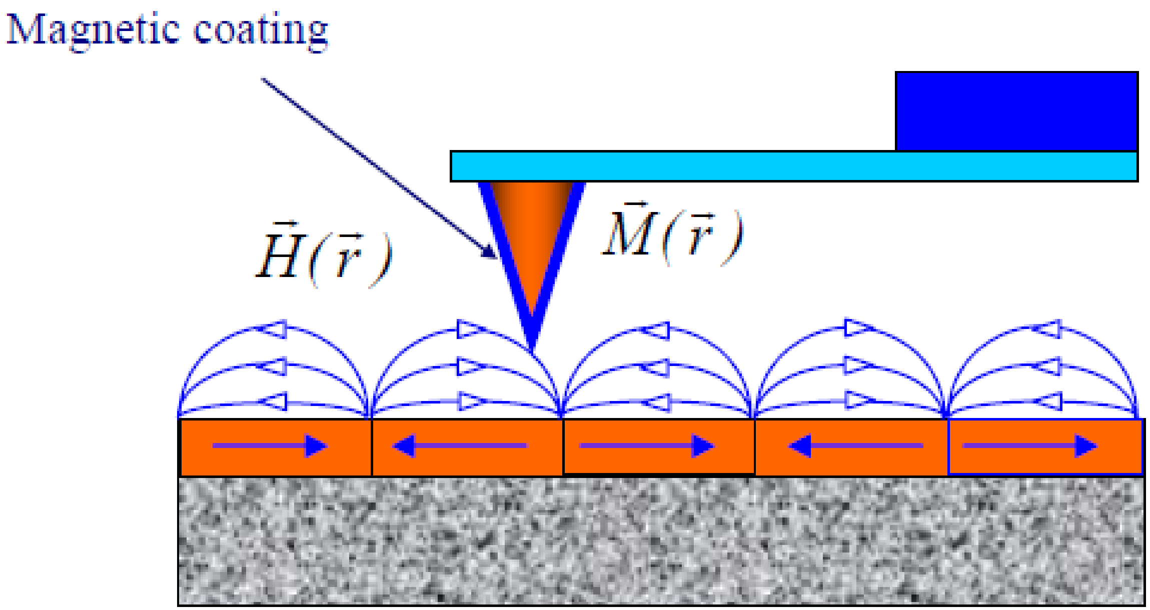
Both temperature and humidity can be controlled in this environmental chamber.

Click for the Next

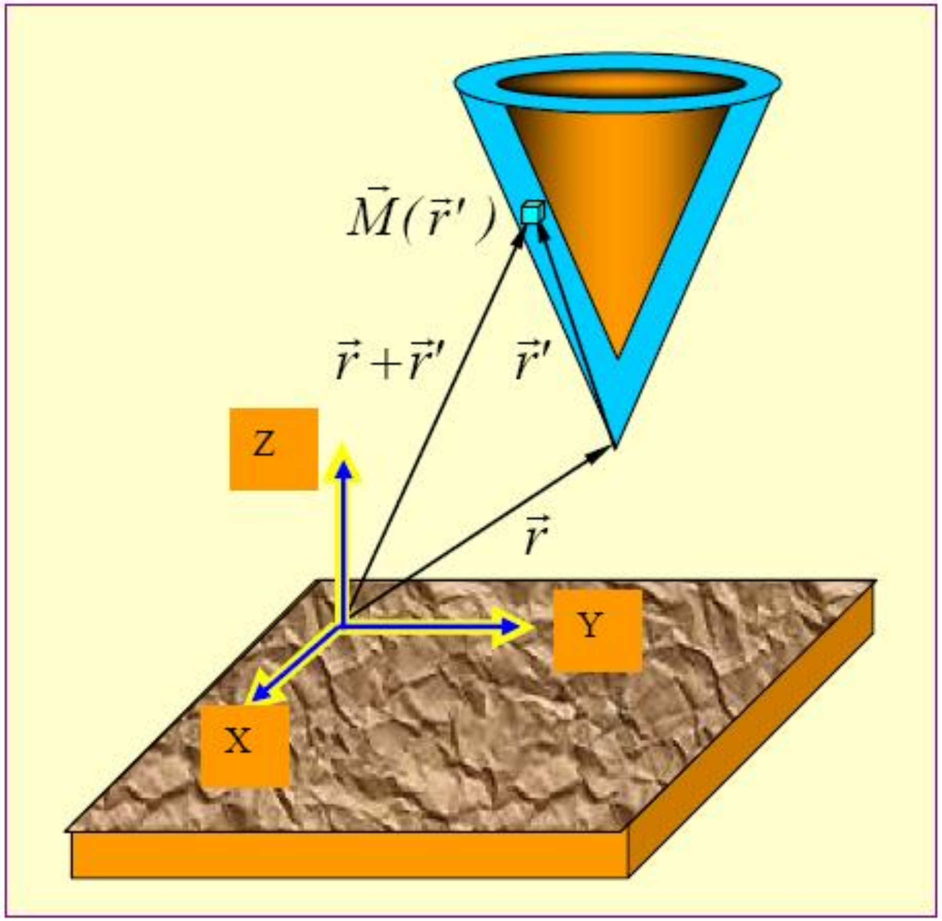
Magnetic force microscope (MFM)

- Magnetic force microscope (MFM) has been invented by **I. Martin and K. Vikramasinghe** in 1987 for research of local magnetic properties of samples. This device represents an atomic force microscope, the tip of which is covered with a layer of a ferromagnetic material with specific magnetization $.\vec{M}(\vec{r})$

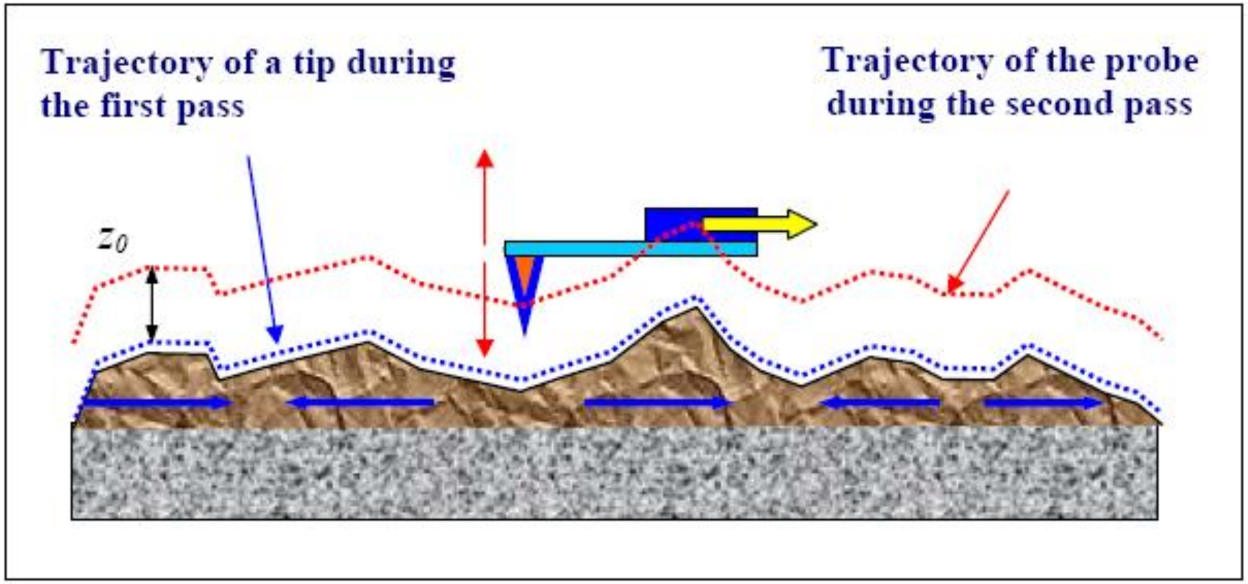
The MFM tip in a magnetic field of a sample



Interaction of a MFM tip with a magnetic field of a sample



Two-pass technique of the MFM acquisition



On the first pass the AMF image of a topography in a contact or "semi-contact" mode is obtained.

Then the tip is retracted from a surface to a distance, and the scanning is repeated. The distance is selected so that the van der Waals force is less than the magnetic interaction force.

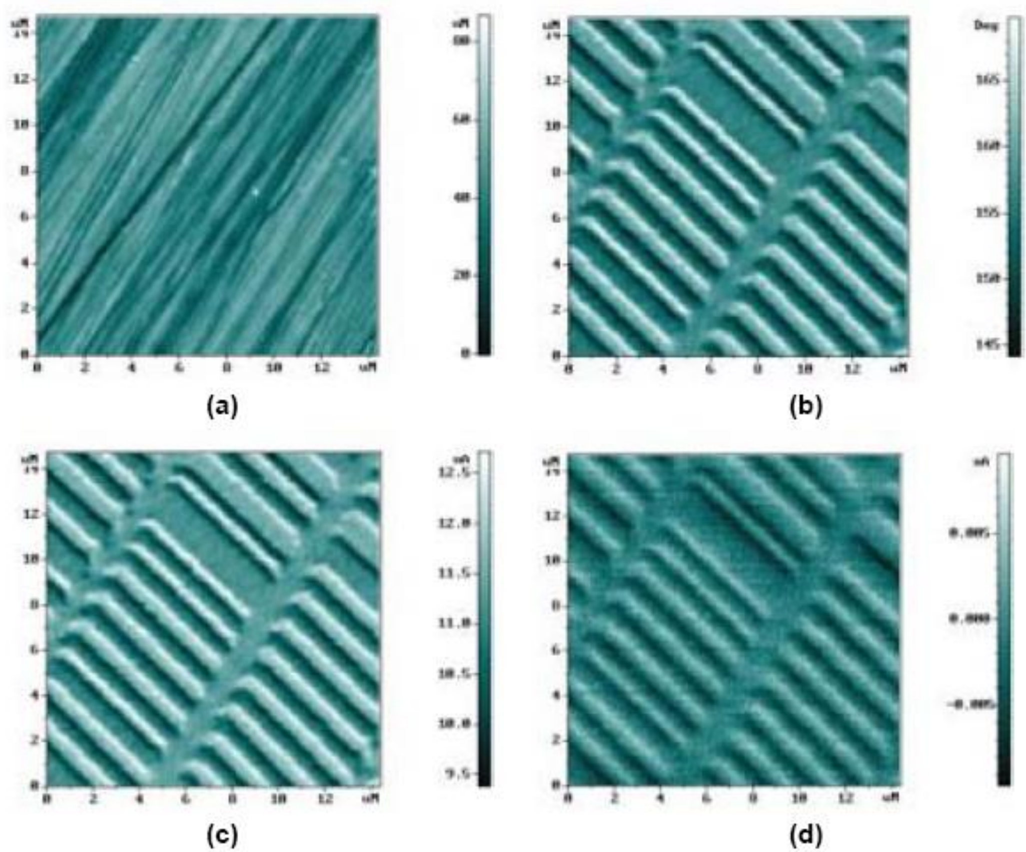
Two-pass technique of the MFM acquisition

During the second pass the probe moves above a surface with a trajectory repeating the topography of a sample.

Since the local distance between the tip and a surface in every point is constant in this case, changes of a cantilever bend during scanning are connected to the heterogeneity of the magnetic forces affecting the tip from a sample.

Thus, the final MFM frame represents bi-dimensional function F (x, y), describing the distribution of magnetic interaction force of a tip with a sample.

MFM research of a magnetic disk surface



(a) –AMF image of a surface topography;

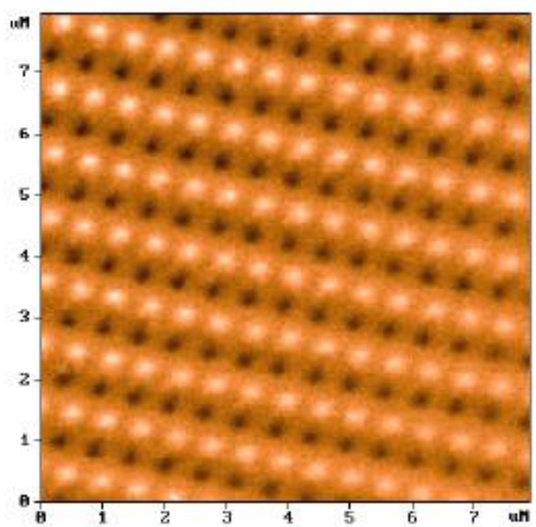
(b) – MFM image of a phase contrast;

(c) – MFM image of an amplitude contrast;

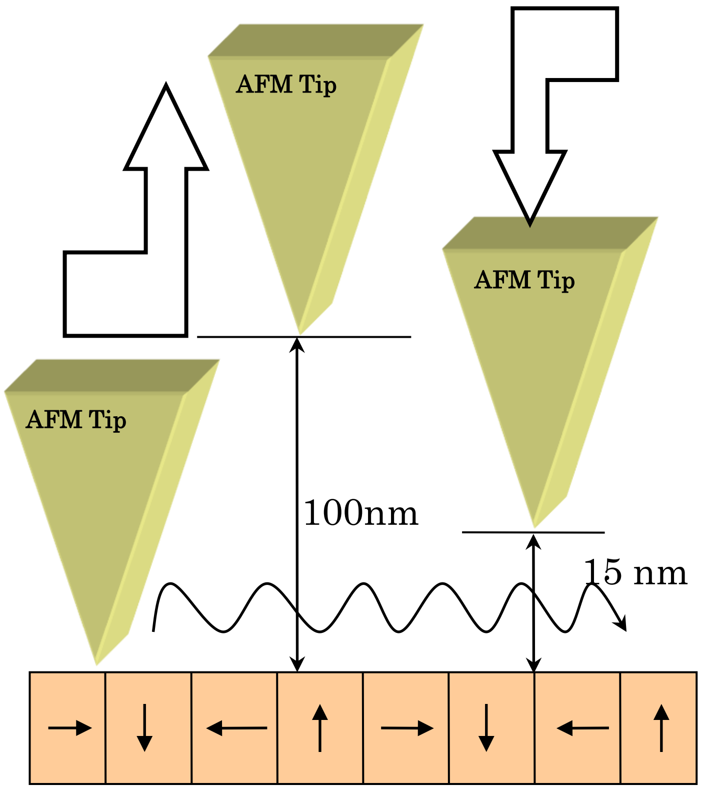
(d) – MFM image of distribution of tip-surface force interaction

MFM of thin films

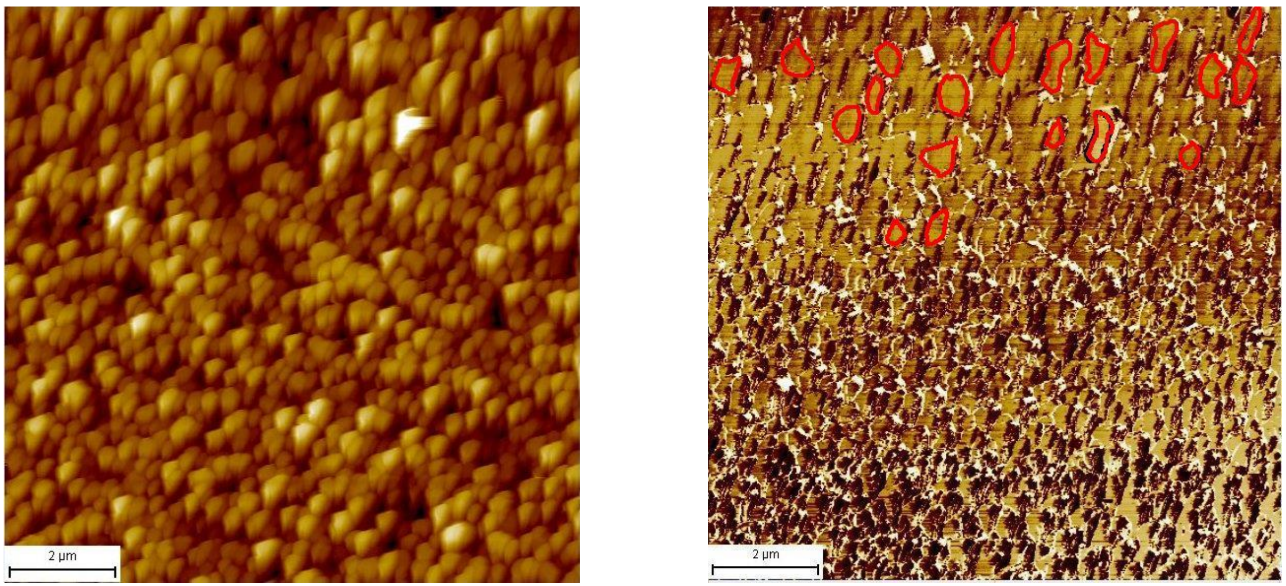
The MFM image of an array of magnetic nanoparticles, formed by the interferential laser annealing of Fe-Cr films method

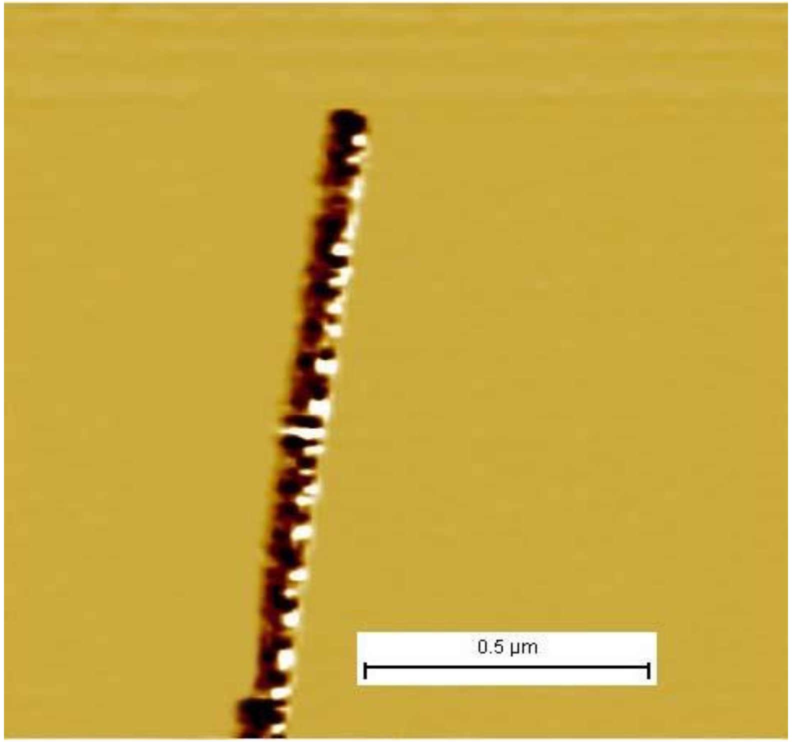


MFM – Nanowires



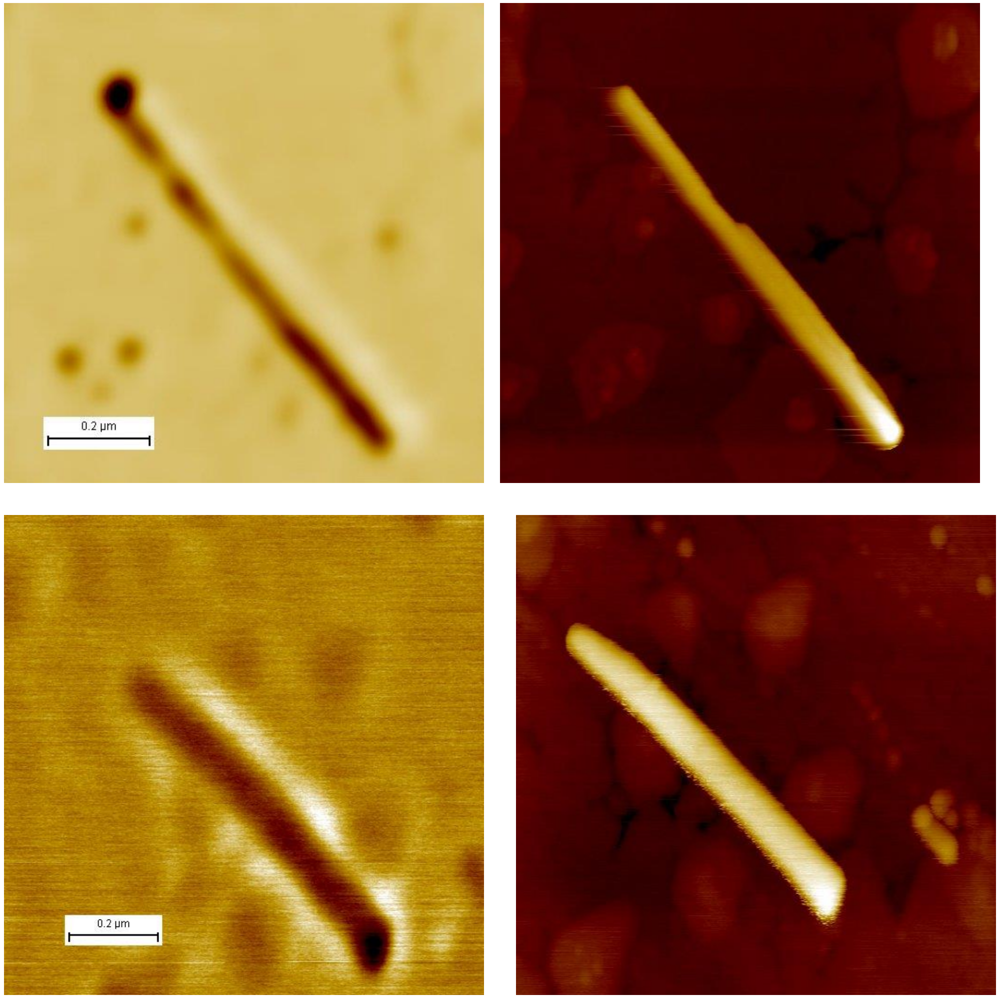
shows the schematic sketch of dual scan method employed for the MFM measurement associated with topographic image. In dual scan method first the tip reads the topographic signal, then moves to the height of several tens of nm and comes back to the desired height (15 nm) with respect to the surface of the sample and the second scan reads the magnetic signal. MFM images taken in the Phase buffer, since the MFM signal shows the phase shift between the tip oscillation and magnetic signal from the sample.


2 µm
2 µm
Interleave
Height: 50 nm
Interleave
Height: 30 nm
Interleave
Height: 20 nm
Interleave
Height: 10 nm



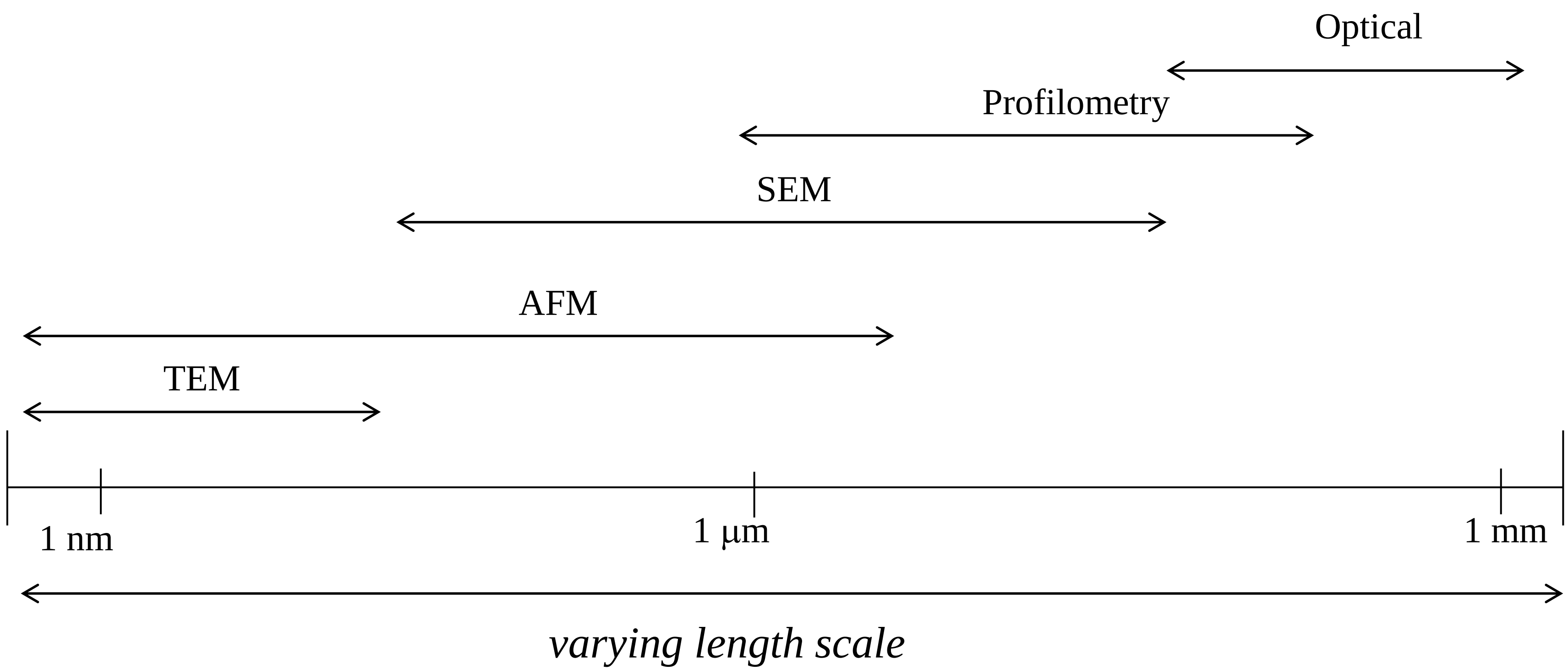
MFM images of 67 nm diameter single GaN Nanowire.

The nanomagnets of about 20-30 nm are clearly visible on the surface of the NW. The magnetic signal originates from the surface structural defects on the NWs


0.2 µm
0.2 µm

SNOM / NSOM

Hierarchy of Measurements

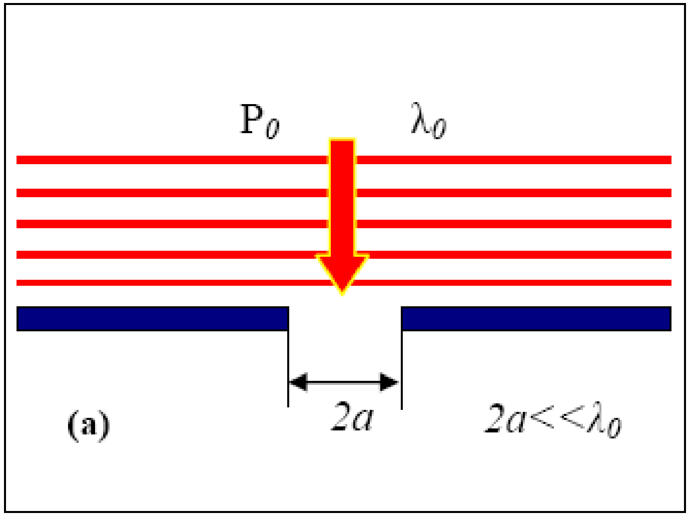


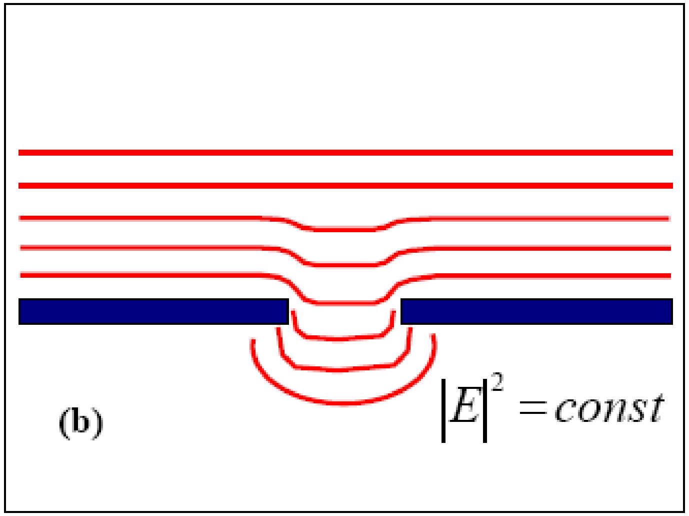
The resolution of an optical imaging system — a microscope , telescope , or camera — can be limited by factors such as imperfections in the lenses or misalignment. However, there is a fundamental maximum to the resolution of any optical system which is due to diffraction . An optical system with the ability to produce images with angular resolution as good as the instrument's theoretical limit is said to be diffraction limited

An optical microscopy is limited by the so-called Abbe diffraction limit, which means that the dimensions of a focusing spot are always about half of the wavelength used.

$$\Delta x \sim 0.61 \frac{\lambda}{2\,NA} \sim 250\ nm$$

A plane wave encounter a barrier with a hole smaller than the wavelength, the waves spread out in circular fashion form the small hole.





(a) - Passage of light through an orifice in the screen with the sub wave aperture,

(b) - Lines of constant intensity of optical radiation in sub wave aperture area

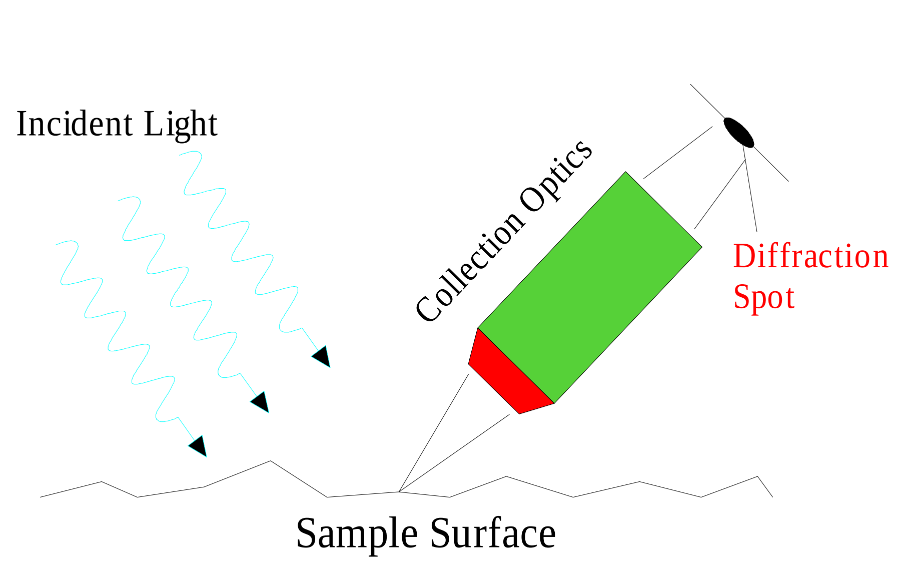
Mathematically, the diffraction pattern is characterized by the wavelength of light illuminating the circular aperture, and the aperture's size. The /appearance/ of the diffraction pattern is additionally characterized by the sensitivity of the eye or other detector used to observe the pattern. Owing to diffraction, the smallest point to which a lens or mirror can focus a beam of light is the size of the Airy disk. Even if one were able to make a perfect lens, there is still a limit to the resolution of an image created by this lens. An optical system in which the resolution is no longer limited by imperfections in the lenses but only by diffraction is said to be diffraction limited.

Airy disc and Airy pattern are descriptions of the best focused spot of light that a perfect lens with a circular aperture can make. The diffraction pattern resulting from a uniformly-illuminated circular aperture has a bright region in the center, known as the Airy disk which together with the series of concentric bright rings around is called the Airy pattern. --

Therefore, the resolving power with visible light illumination, far field microscopy, has an upper limit of several hundred nms. It is possible to use other microscopic techniques such as scanning probe microscopy or electron microscopy, but the information obtained by visible light can be a valuable tool in scientific studies. ***In 1984, Lewis's group and Pohl's group have developed near-field microscopy independently****. Since then, there have been many reports of different types of scanning near-field optical microscopes (SNOM). However, the resolving powers of their probes are not over those of atomic force microscopes (AFM) or scanning tunneling microscope.*

Far-Field Vs Near-Field Microscopy

Far-Field Microscopy

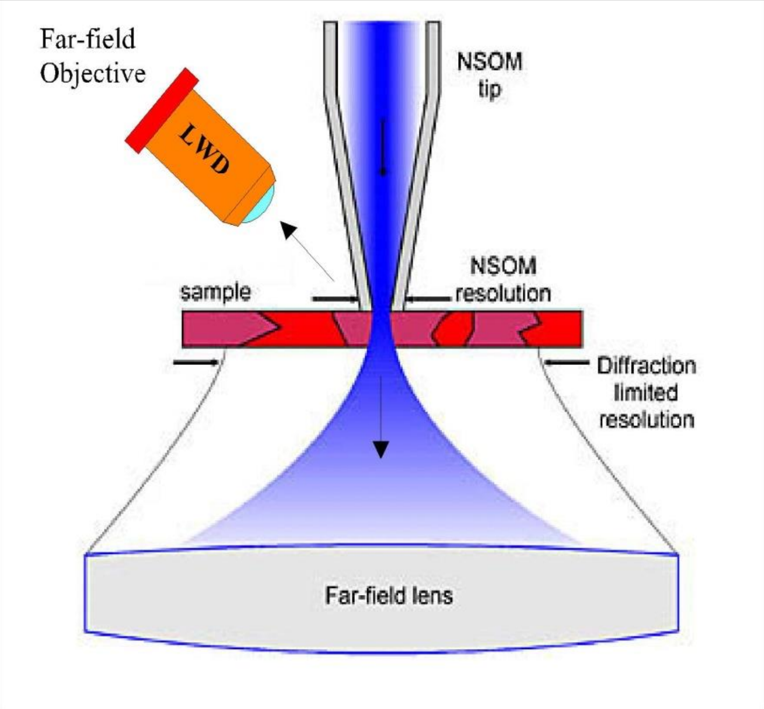


• Both the light source and the detector are placed at several wavelengths from the sample.

•The Lateral resolution is determined by the Abbe diffraction limit.

$$\Delta x \sim 0.61 \frac{\lambda}{2\,NA} \sim 250\ nm$$

Near-Field Microscopy



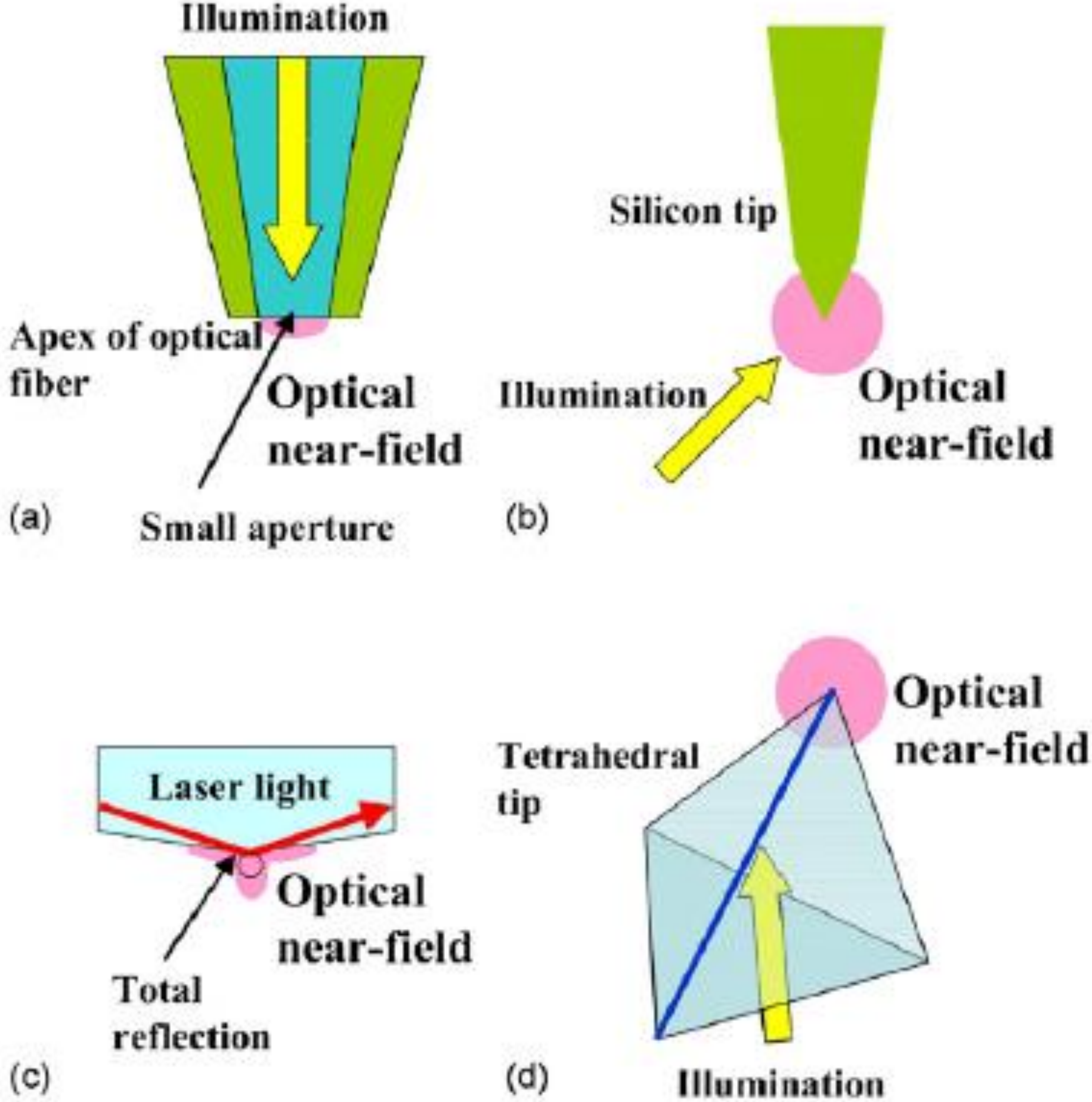
• The sample is illuminated by a nanoscopic light source located close to the surface (10 nm).

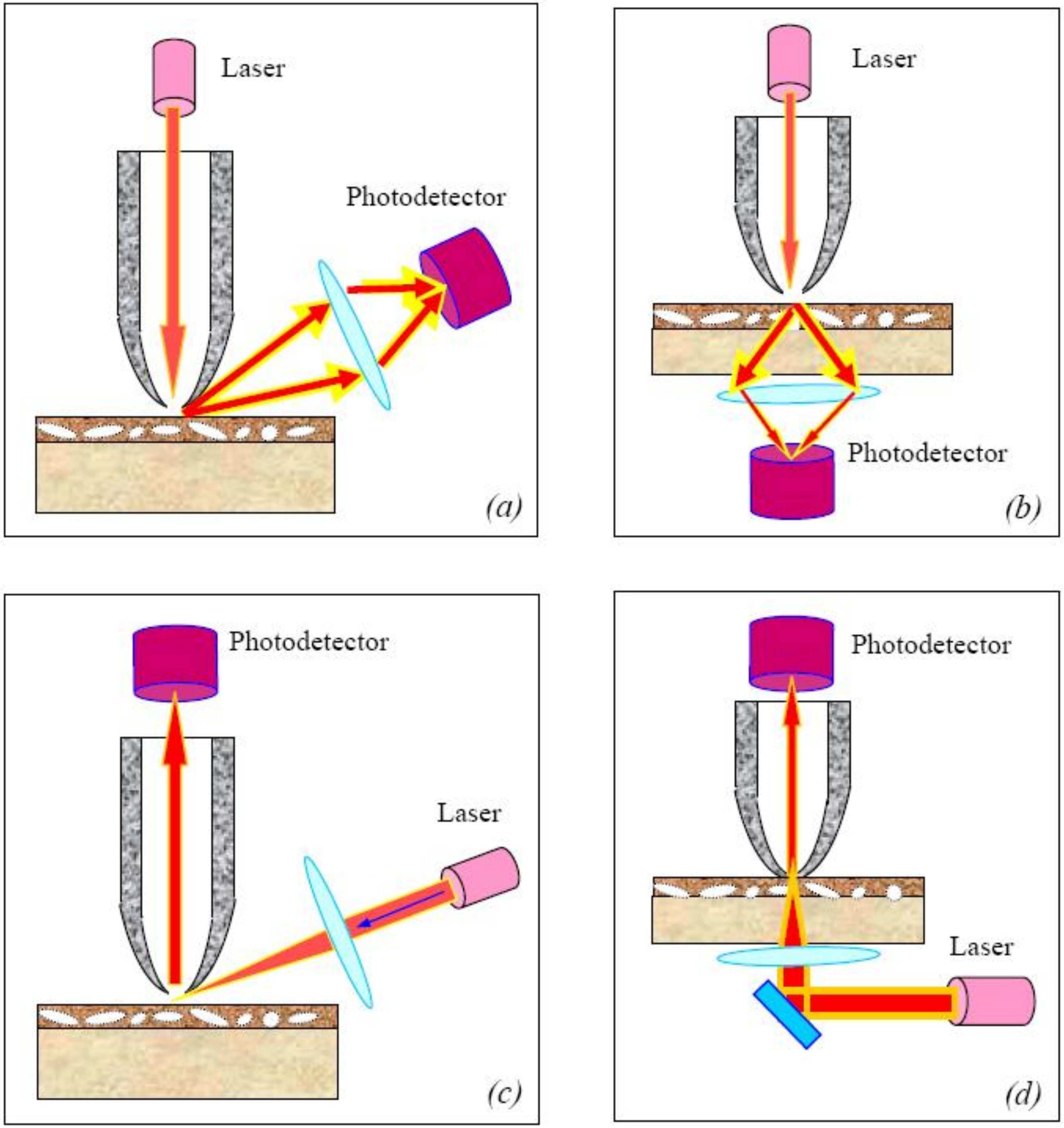
• The resolution is limited by the source diameter

$$\Delta x \sim a \leq 10\ nm$$

The most advanced SNOMs, which have lateral resolutions less than 10 nm, have accepted the following probes:

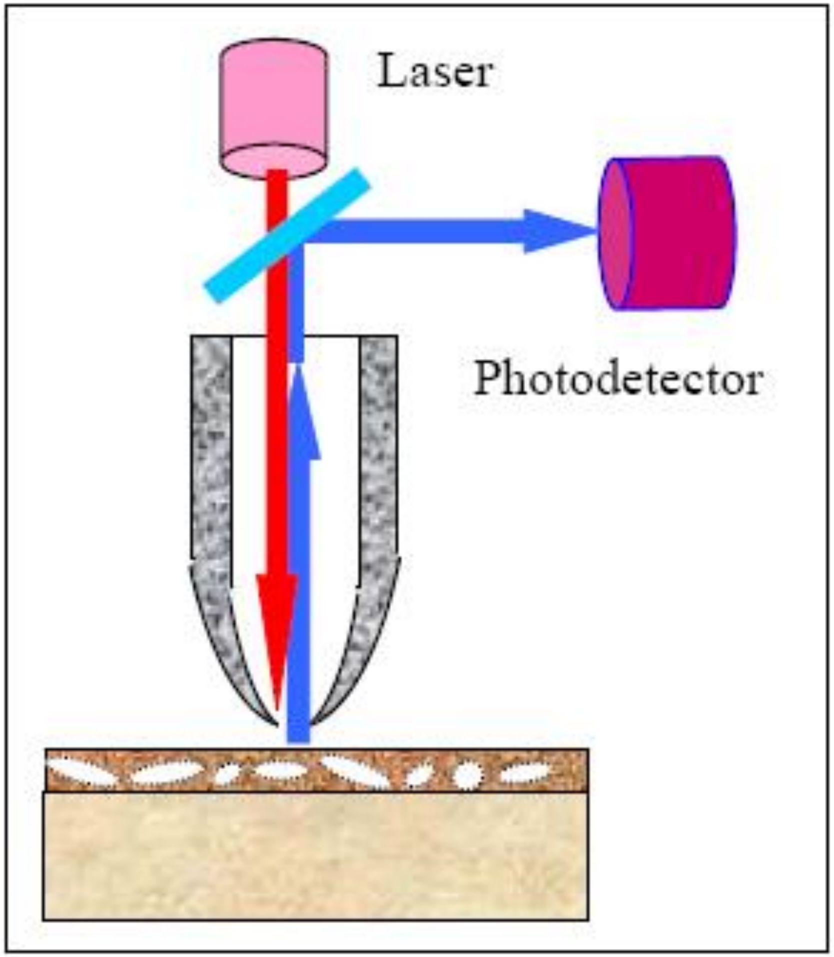
(1) optical fiber tip with a small aperture on the coated Al film
(2) Apertureless silicon tip with 2nm radius
(3) Polystyrene small sphere illuminated by evanescent light
(4) tetrahedral glass tip covered with gold thin film illuminated by surface plasmon


Illumination
Apex of optical fiber
Optical near-field
Small aperture
(a)
Silicon tip
Illumination
Optical near-field
(b)
Laser light
Optical near-field
Total reflection
(c)
Optical near-field
Tetrahedral tip
Illumination
(d)

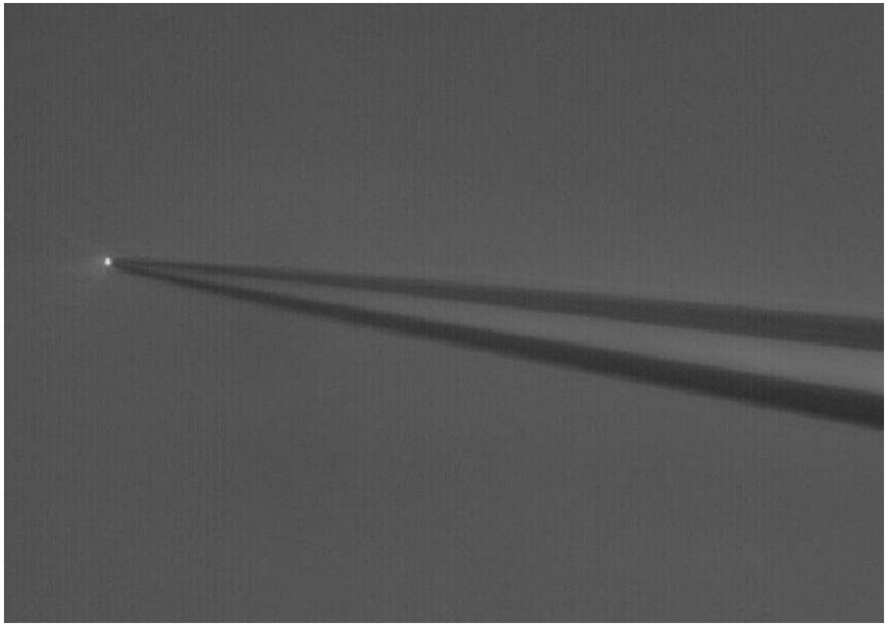


Laser
Photodetector
(a)
Laser
Photodetector
(b)
Photodetector
Laser
(c)
Photodetector
Laser
(d)

The SNOM scheme where light-striking of a sample and reception of emission are carried out by the same tip



Such combination of a near-field source with a near-field receiver is rather a promising method providing very high spatial resolution. However, in the emission passes twice in this scheme through a subwave aperture. As a result, the signal coming on a photodetector has a very low intensity, and hence high-sensitivity methods of its registration are required. Integration of the SNOM with an optical monochromator allows to carry out local spectroscopic researches of samples. The basic fields of application of near-field optical microscopes are the research of local optical and photoelectric properties of semi-conductor photosensitive structures, research of biological objects, nanotechnology.



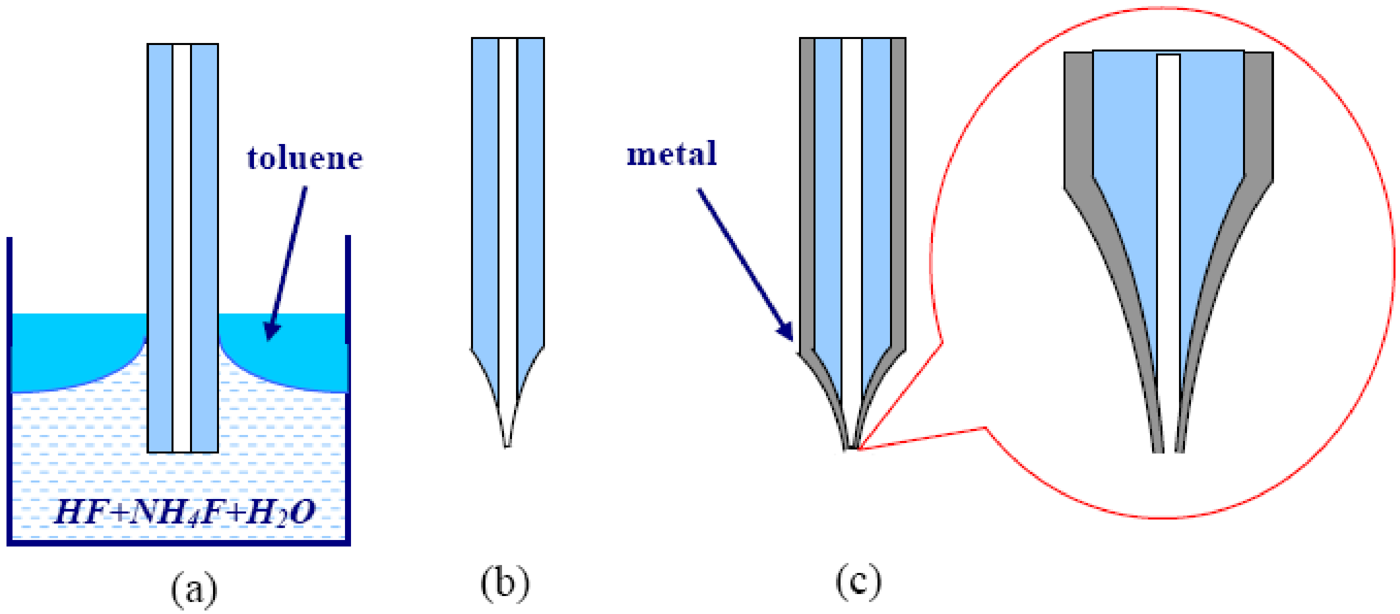
A fiber optical tip used as a light source. The tip end is placed very close to the sample surface.

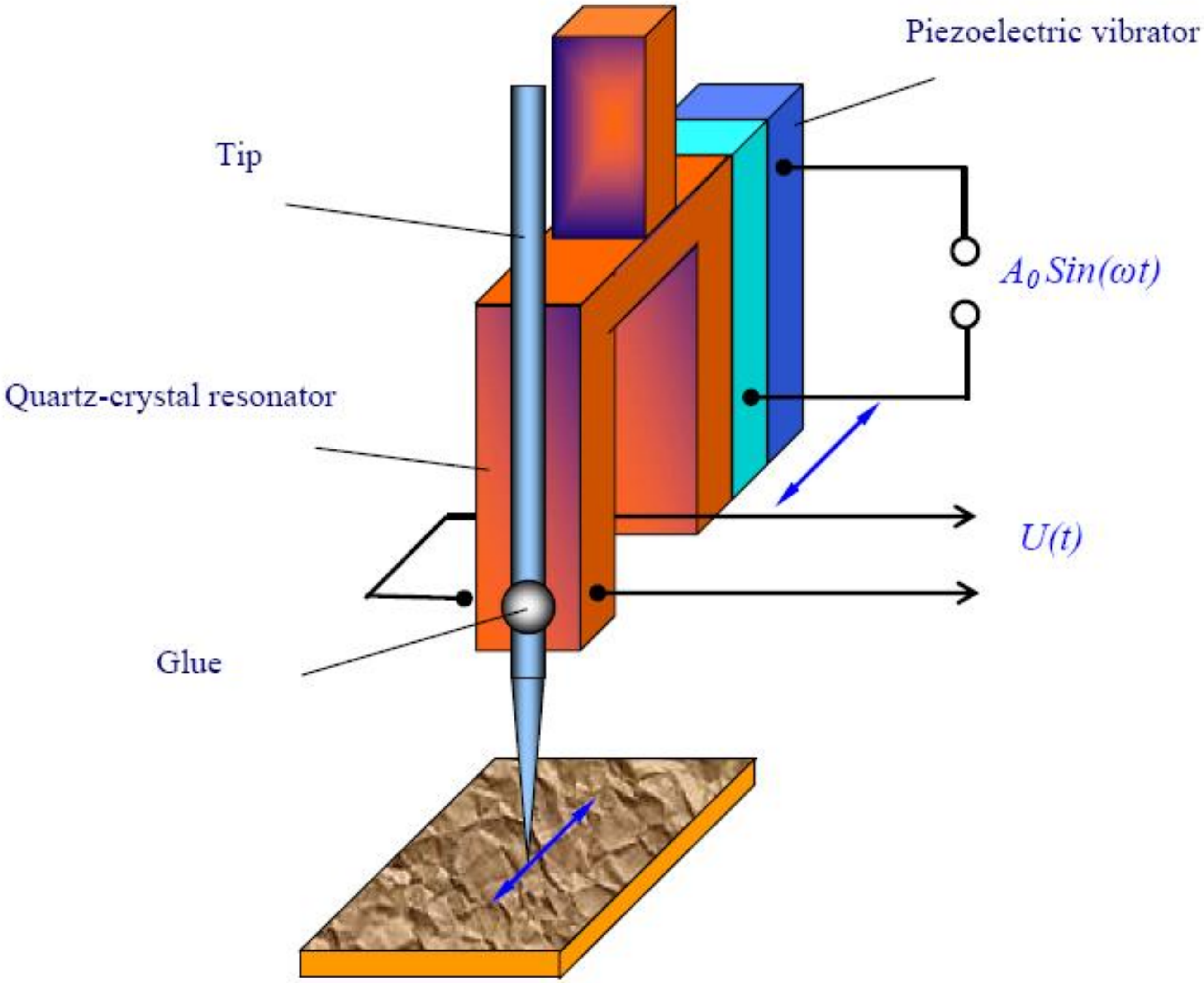
Manufacturing of SNOM tips on the basis of an optical fiber:

(a) - chemical etching of fiber;

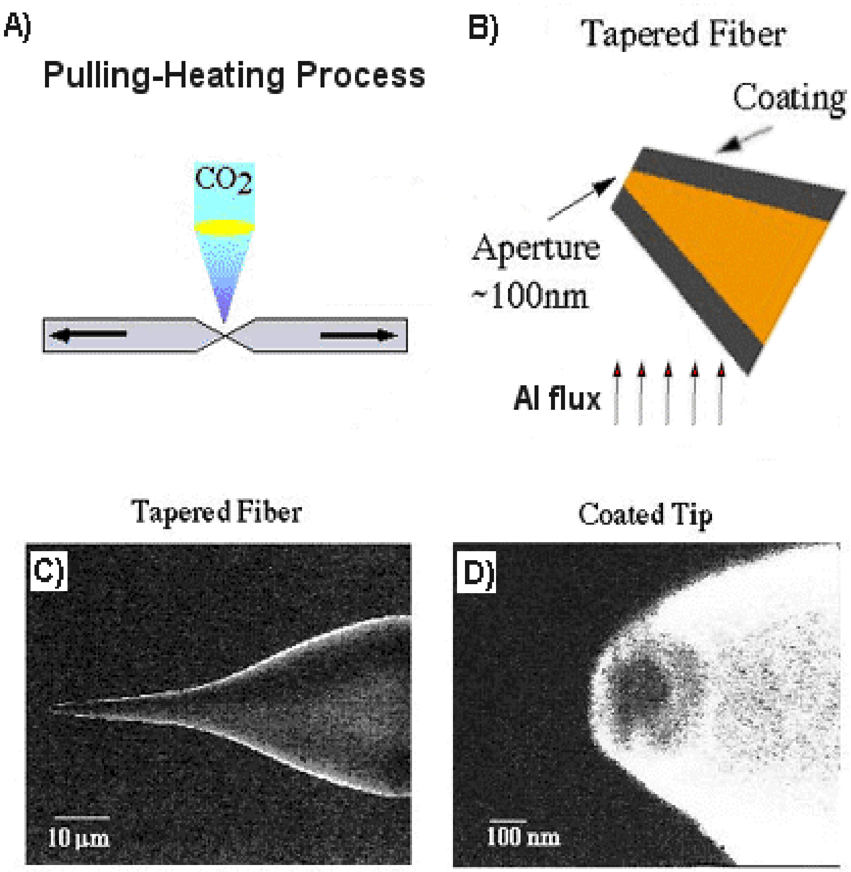
(b) – appearance of a fiber apex after etching;

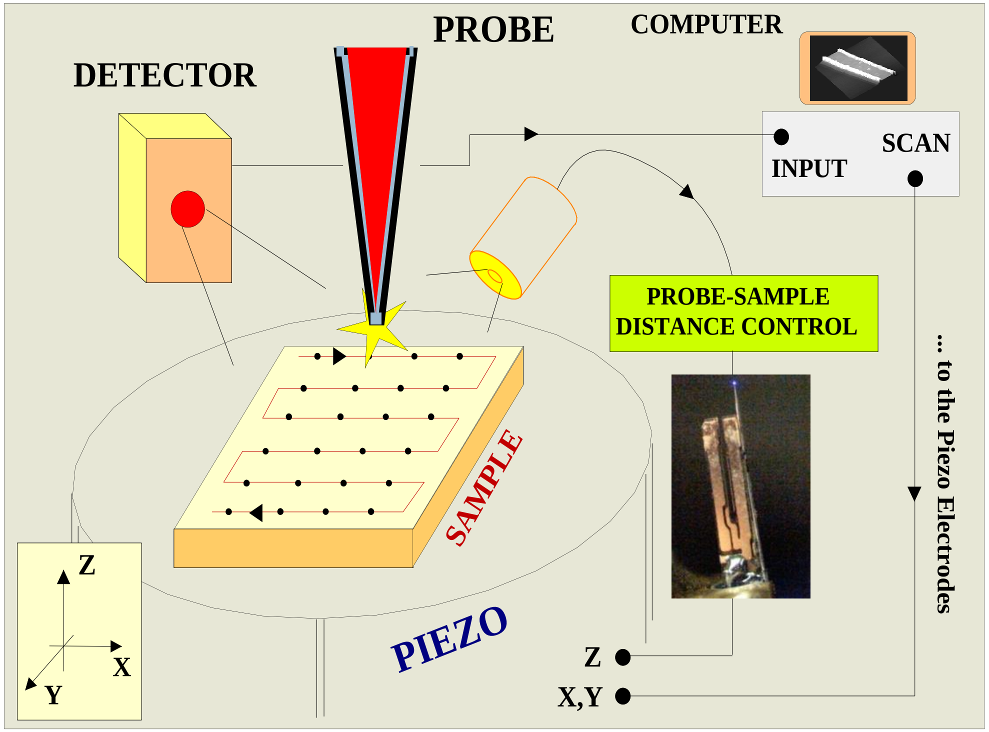
(c) –thin metal film evaporation.




Piezoelectric vibrator
Tip
$A_0 Sin(\omega t)$
Quartz-crystal resonator
$U(t)$
Glue

Schematic of the Experiment





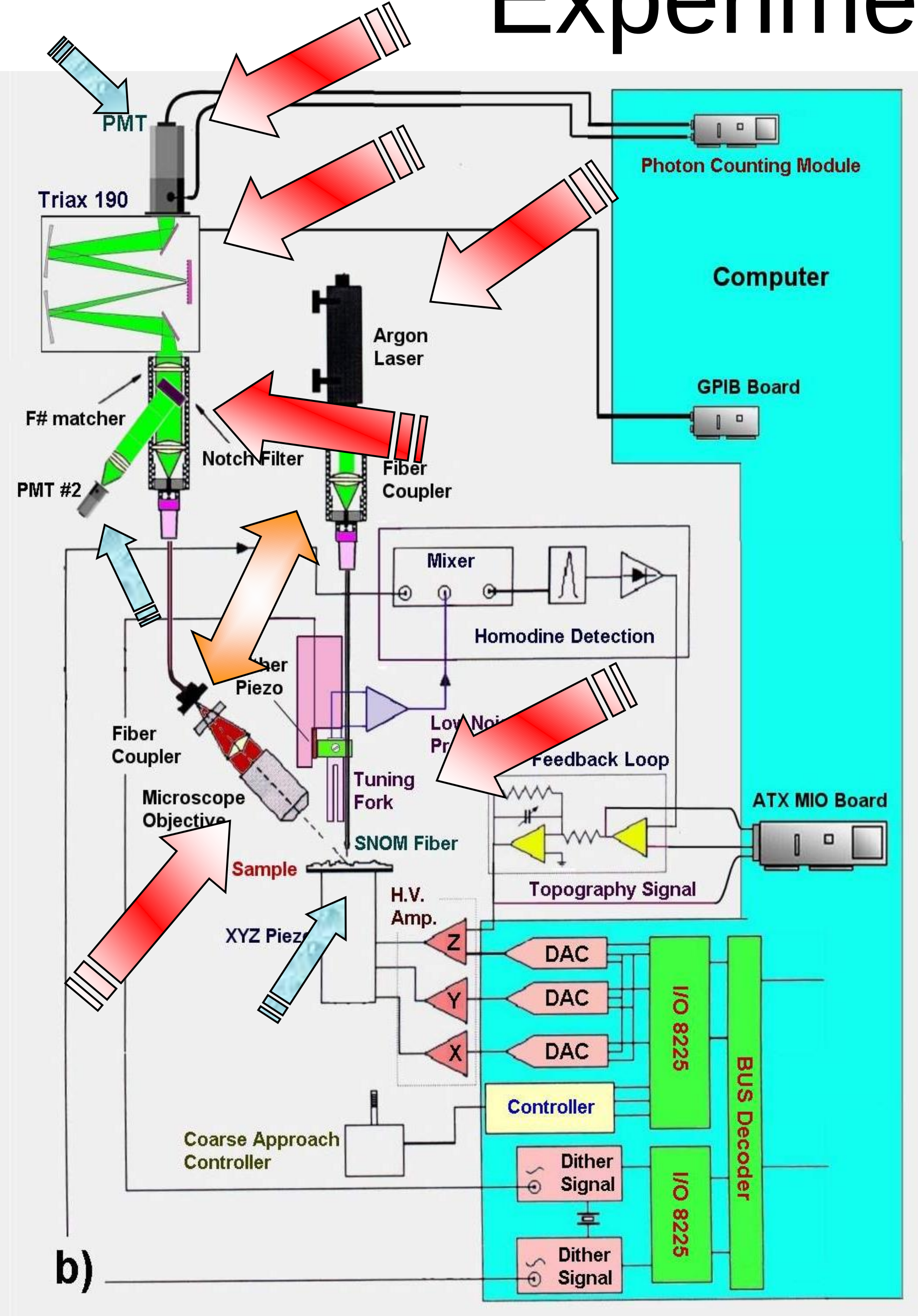
• SNOM probes: commercial single mode optical fibers, tapered and coated by a thin CrAl film.

• The optical aperture is ~ 100 nm

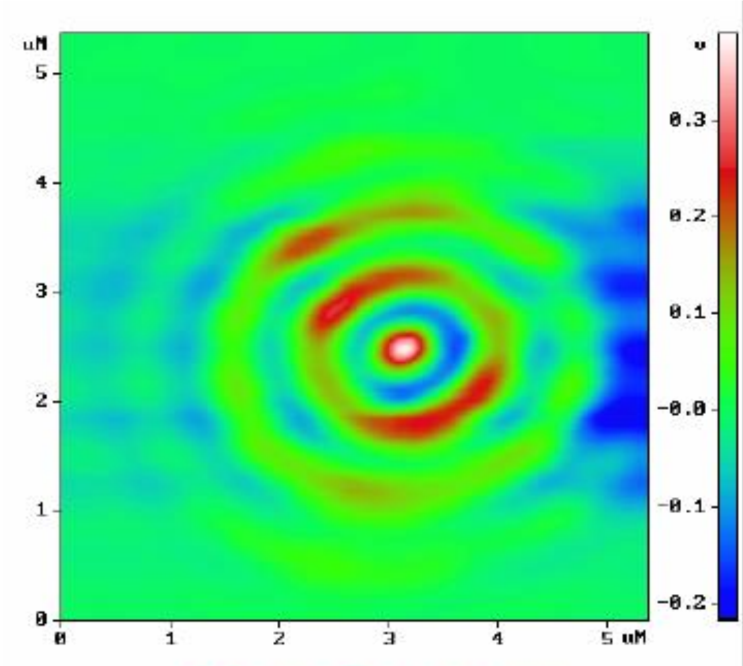
• The sample is raster scanned by means of a piezo-tube under the probe.

• Non-optical shear-force detection is accomplished for probe/sample distance stabilization, by means of a quartz tuning-fork.

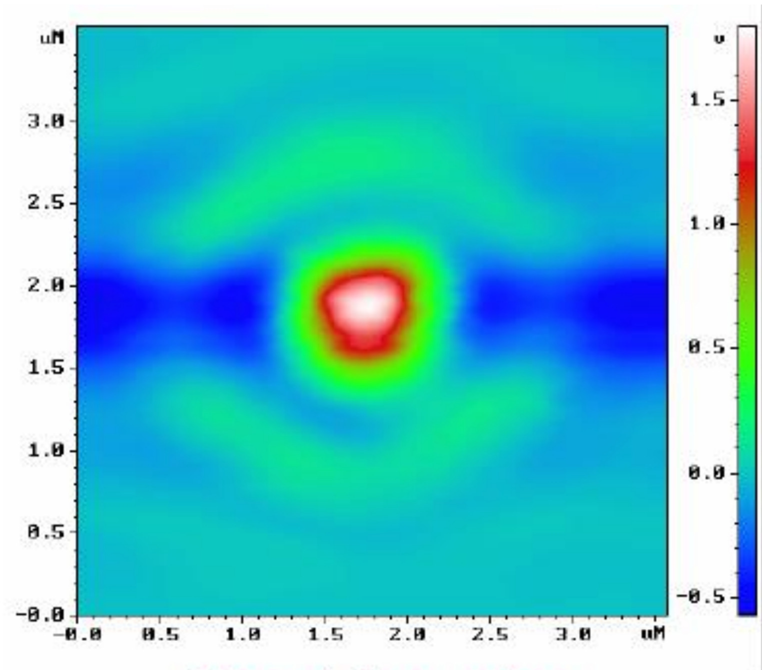
Experimental setup



- Excitation: Ar^{++} laser line 514.5 nm.
- Collection: Nikon 50X objective, NA 0.5, WD 10.6 mm.
- Notch Filter: Rejection Ratio ~ 10^{-6}.
- Spectrometer: Triax 190, single grating, 1200 lines/mm, 190 mm focal.
- Detector: PMT in photon counting regime, 200-300 dark cts/sec.
- Shear-Force: tuning-fork with etherodyne detection.
- Signals: Topography, Elastic, Raman.
- Modes: Illumination or Collection.



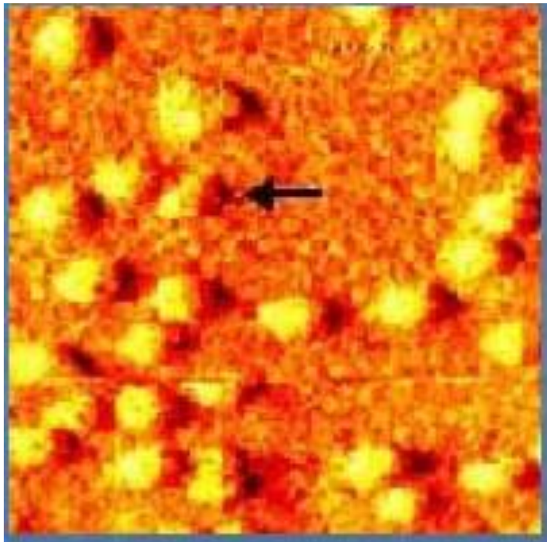
Poor focusing



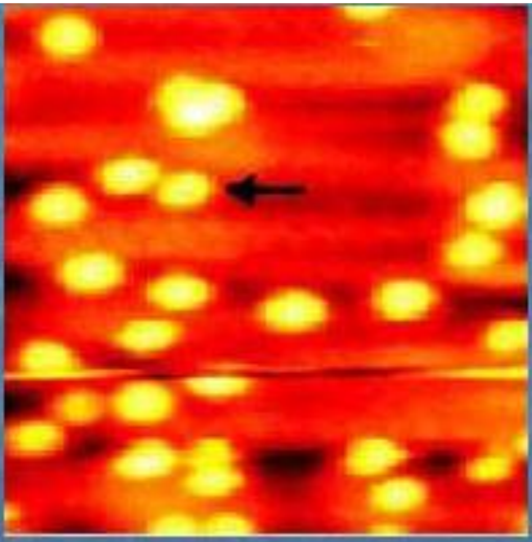
Good focusing

Laser beam focused by 100x micro objective lens shows distinct diffraction rings as seen by SNOM visualization. The best focusing (right picture) provides the energy distribution peak about 0.3 μm wide.

As the optical near-field is generating the image, the Abbe limit that restricts the resolution of a normal optical microscope to $\lambda/2$ is defeated.



SNOM image

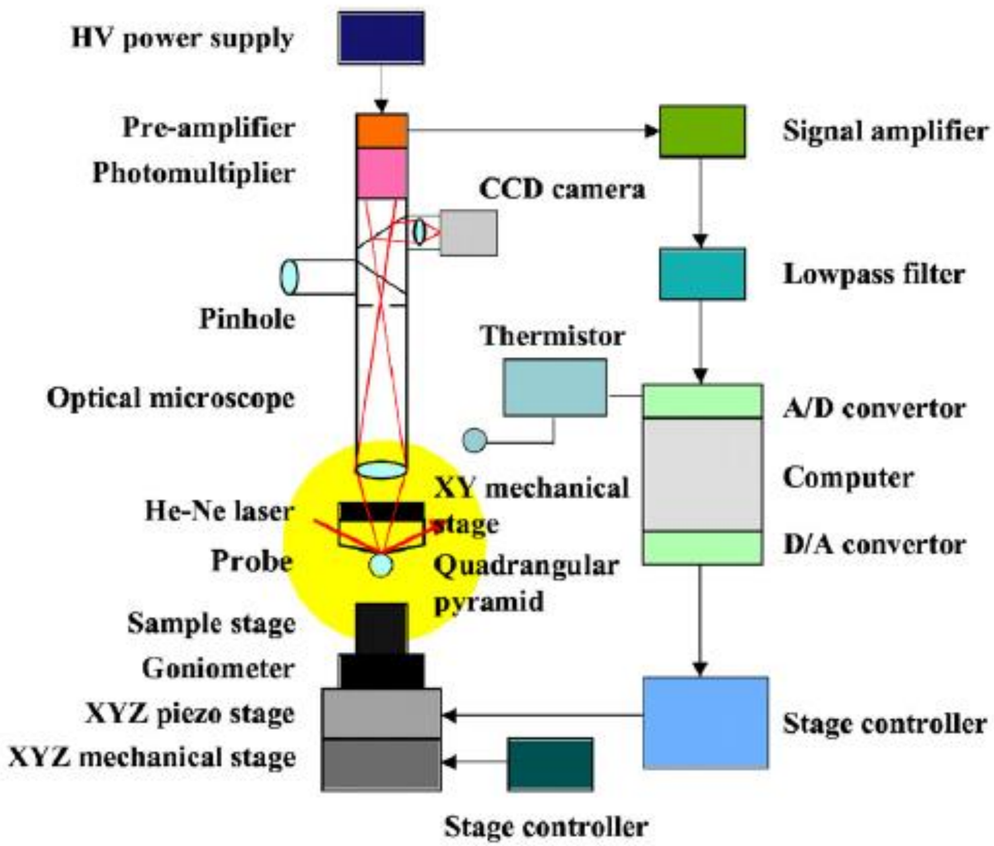


Corresponding AFM image

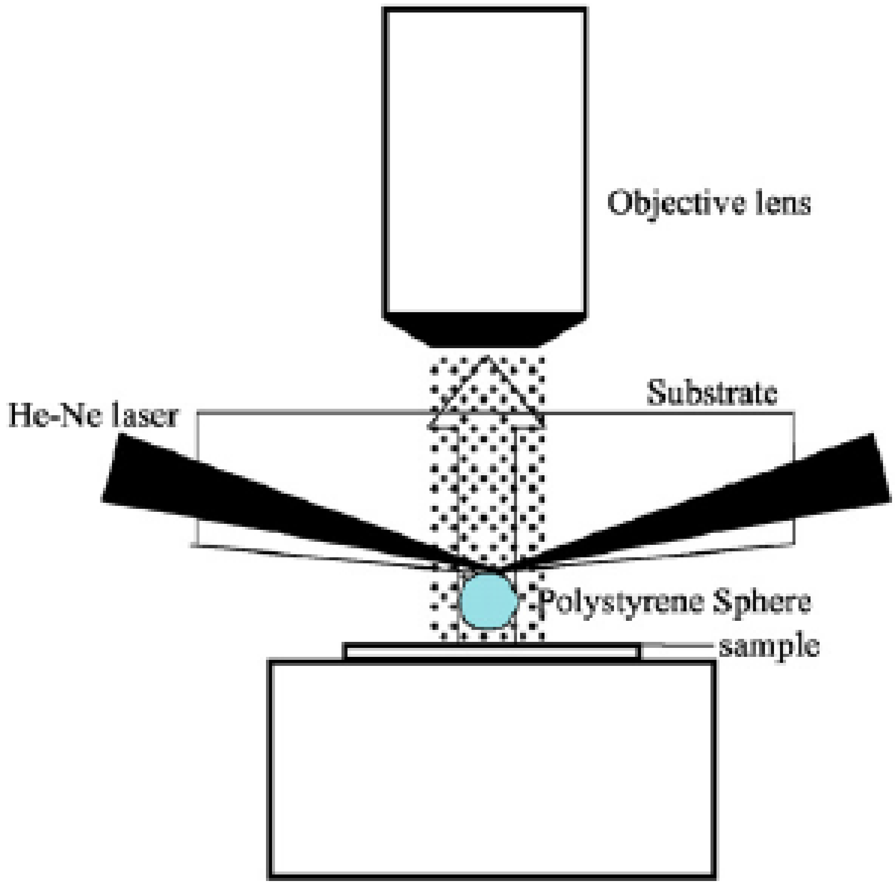
Comparison between AFM and SNOM images of 30 nm gold spheres.

(Nanonics Imaging Ltd)

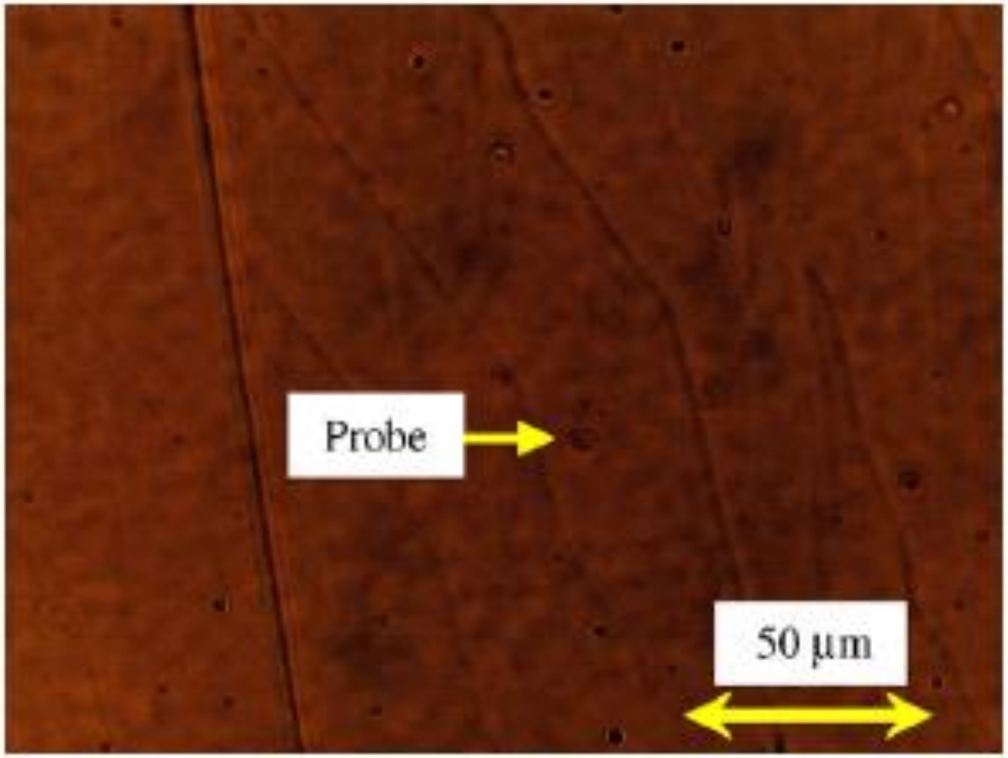
SNOM system with small sphere probe. In the scan phase, scattered light intensity from the probe is kept constant by monitoring PMT signal and controlling the height of Z piezo stage.



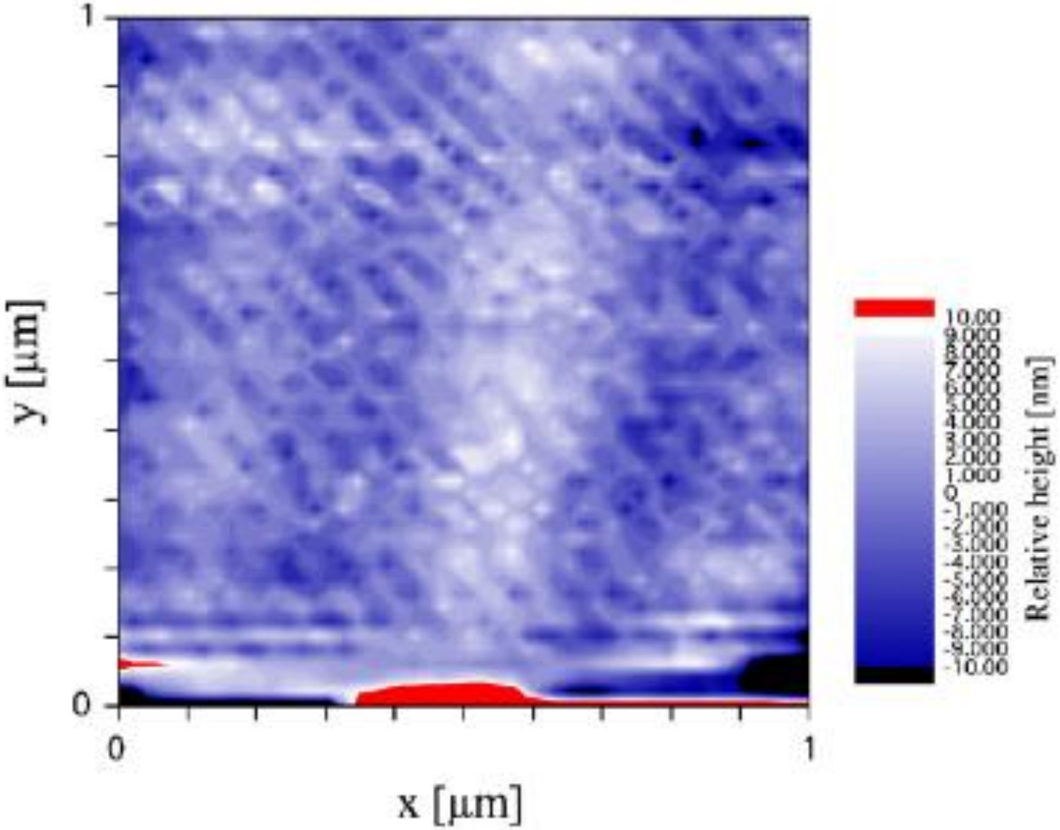
SNOM system with small sphere probe. In the scan phase, scattered light intensity from the probe is kept constant by monitoring PMT signal and controlling the height of Z piezo stage.



A polystyrene sphere with the diameter of 500nm is located on top of a quartz substrate with quadrangular pyramidal shape. Since the incident laser beam totally reflected at the internal surface of apex of the pyramid, the generated evanescent wave illuminates the polystyrene sphere. When the sphere approaches very closely to the sample, the near field around the sphere excites dipoles on the sample surface and the far-field light wave is emitted into an objective lens. The intensity of this emission drastically changes depending on the distance between the sphere and the sample surface.



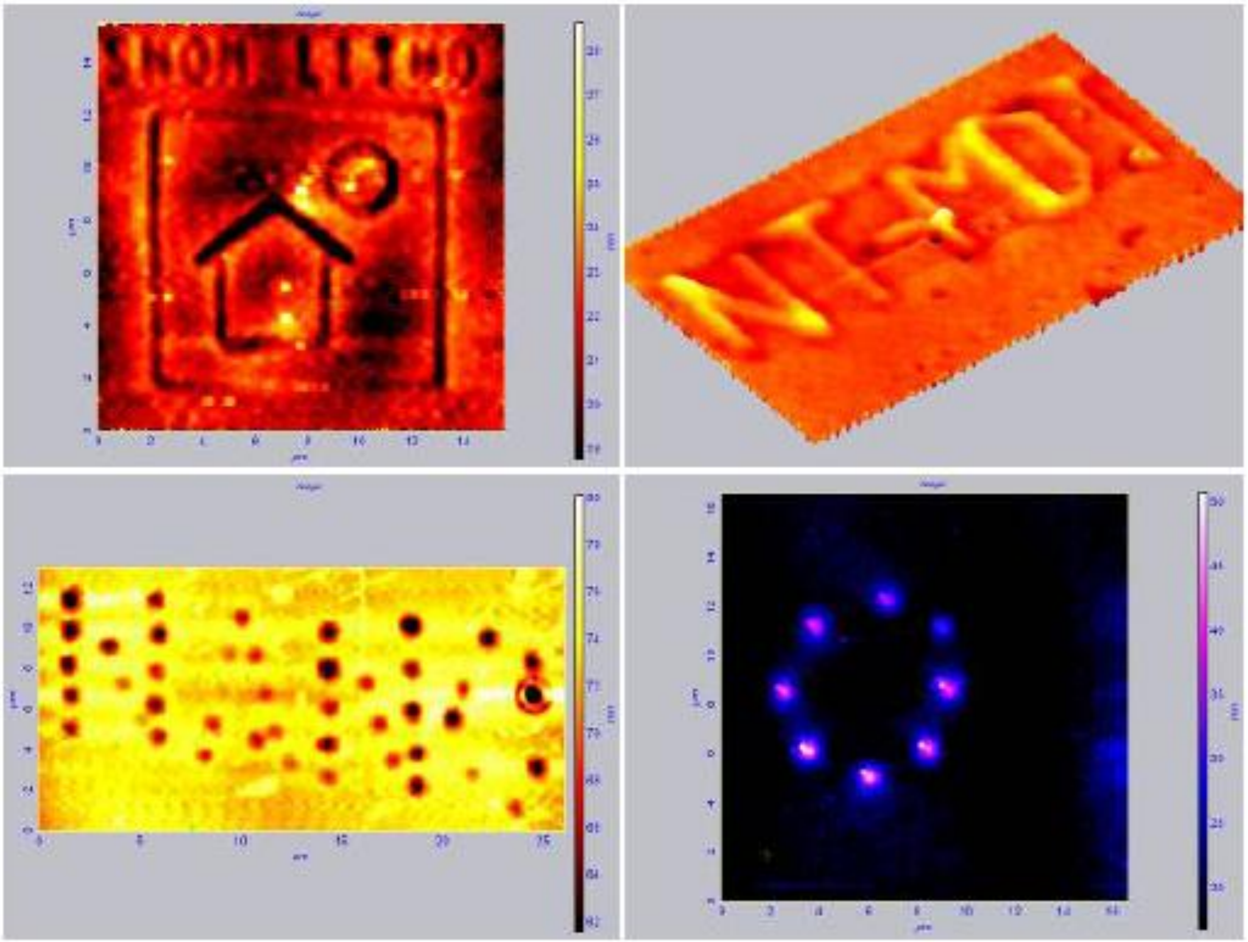
Microscopic appearance of the surface of KCl–KBr solid solution covered with 50nm thick gold film. Several “river patterns”



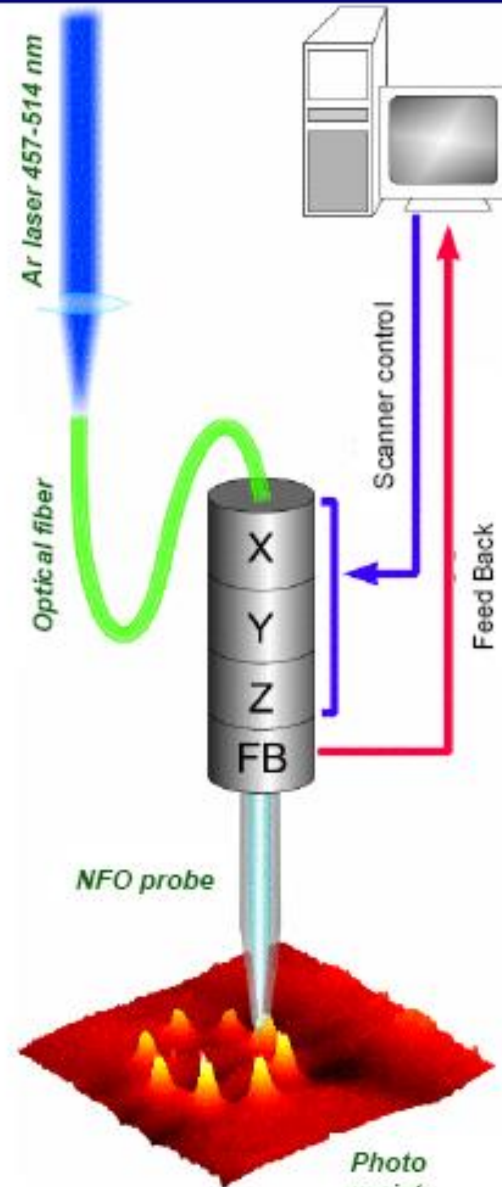
The magnified SNOM image of the small area in a river pattern existing on the cleaved surface of KCl–KBr solid-solution single crystal.

SNOM Lithography

Sample : Positive photoresist FP 380 on Si substrate



Shear Force image of SNOM lithography results.
Lithography made using Ar laser.



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* Students at Centre for Nanoscience and Nanotechnology, BDU

* Alexander von-Humboldt (AvH-Germany)

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