Nano Sensors

PhD/ MTech/ BTech
Course No.: EEL7450
L-T-P [C]: 3–0–0 [3]

Prof. AJAY AGARWAL
ELECTRICAL ENGINEERING
IIT JODHPUR

Lecture 18 dated 18th Feb. 2025

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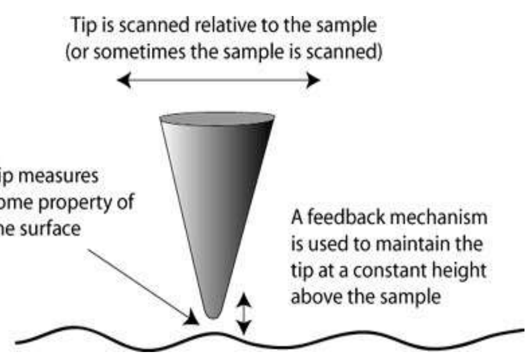
Scanning Probe Microscopy (SPM)

- Scanning Probe Microscopy is used to image surfaces at the nanometer scale.
- A family of microscopy forms where a sharp probe is scanned across a surface and some tip/ sample interactions are monitored.

- Basic idea of scanned probe techniques:
  - Monitor the interactions between a probe and a sample surface
- Two Types of SPM Microscopes:
  - ➢ Scanning Tunneling Microscope STM
  - ➢ Atomic Force Microscope AFM



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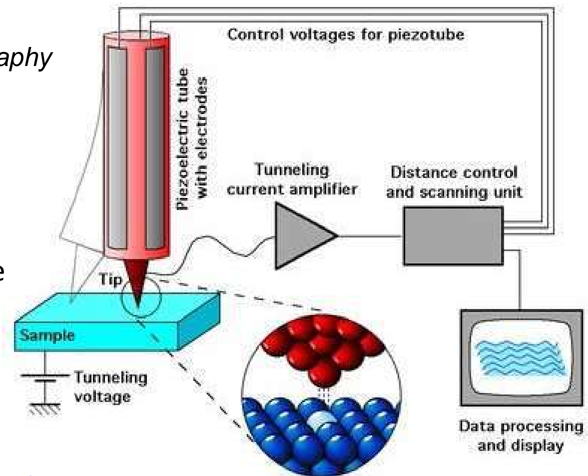
Scanning Tunneling Microscope (STM)

➢ ***Technique that allows the topographic information(image) of conducting surfaces down to the atomic scale.***

How It Works?

- *It allow us to see the image of Surface topography*

- The STM works by scanning a very sharp metal wire tip over a surface.
- By bringing the tip very close to the surface, and by applying an electrical voltage to the tip or sample, we can image the surface at an extremely small scale – down to resolving individual atoms.
- This is done by measuring tunneling current.



Typical distance between tip and sample- 0.2-0.6 nm

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What is Tunneling?

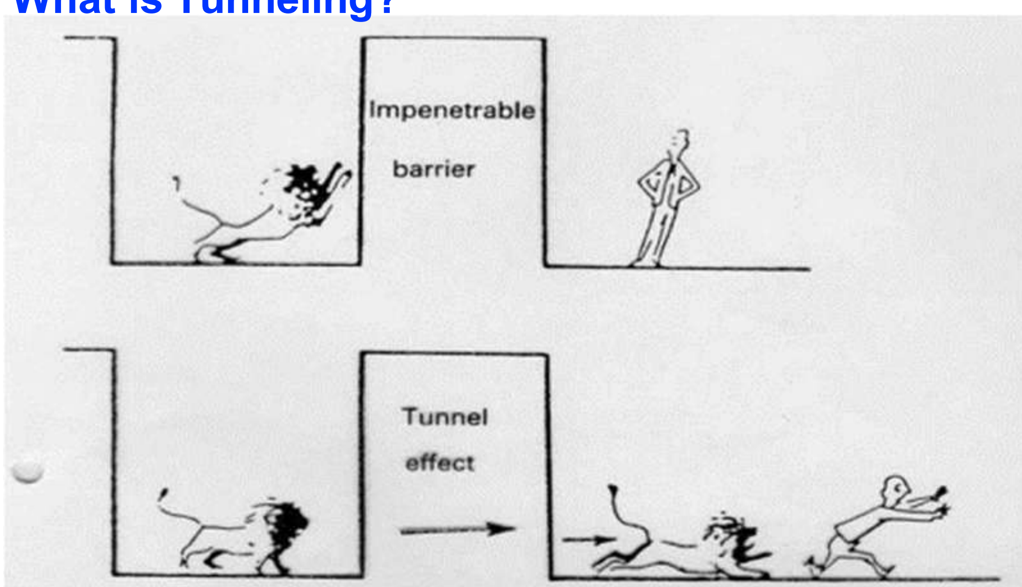

Fig. 1.1. The difference between classical theory and quantum theory, illustrating tunneling through a potential barrier (Bleaney, 1984).

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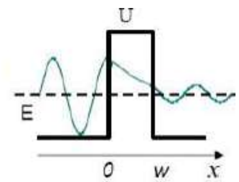
Electron Tunneling Through a barrier



The wave equation is $-\frac{\hbar^2}{2m}\frac{d^2\psi}{dx^2} + U(x)\psi = \varepsilon\psi$

In the region x<0, before barrier, $U = 0$, the eigenfunction is a linear combination of plane waves traveling to the right and to the left with energy $\varepsilon = \frac{\hbar^2 K^2}{2m}$

$\psi_1 = Ae^{iKx} + Be^{-iKx}$

In the region 0 < x < w, within the barrier, the solution is

$\psi_2 = Ce^{Qx} + De^{-Qx} = \psi_1(0)e^{-kx}$; where $U_0 - \varepsilon = \frac{\hbar^2 Q^2}{2m}$ and $\kappa = \frac{\sqrt{2m(U\text{-}E)}}{\hbar}$

In the region x>w, behind the barrier, the solution is

$\psi_3 = Fe^{iKx} + Ge^{-iKx}$

Probability of finding electrons on the other side of the barrier, i.e. ***tunneling current***

$$\boxed{|\psi_1(0)|^2 e^{-2\frac{\sqrt{2m(U-E)}}{\hbar^2}w}}$$

Tunneling current scales exponentially with the barrier width

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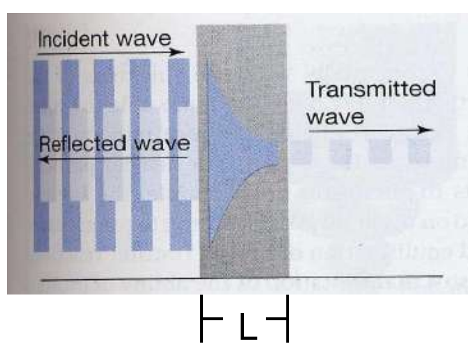
Quantum Mechanical Tunneling



- 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 = ?

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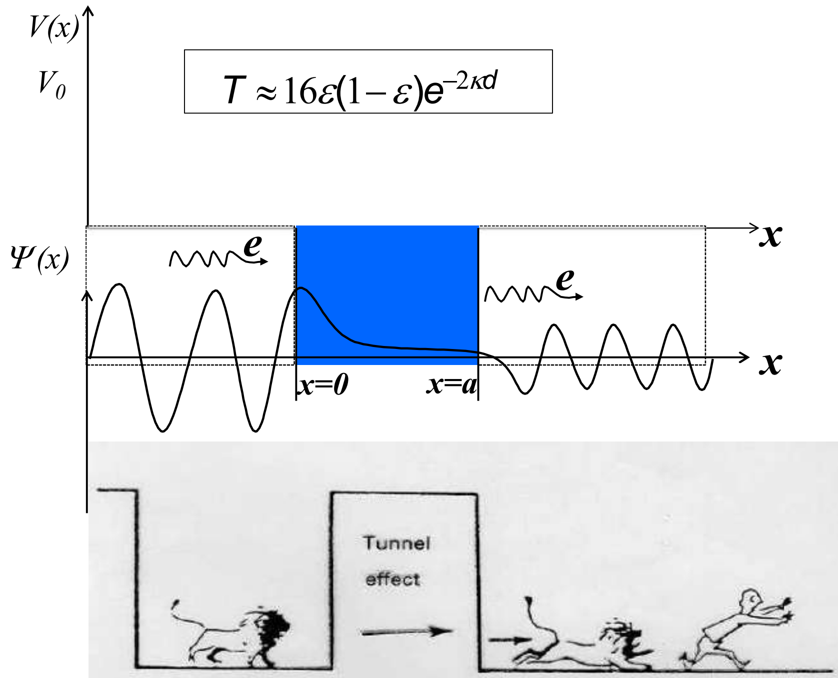

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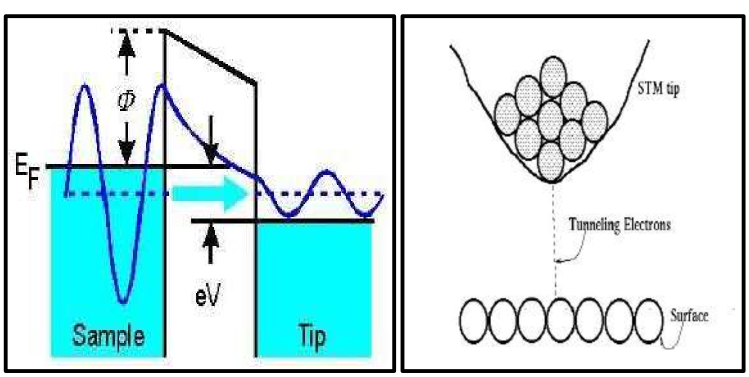
Principle of operation:

- *The STM is based on principle of quantum mechanical tunneling effect*
- *Monitor the tunneling current between a probe tip and a sample surface*

Electron Tunneling:

In **scanning tunneling microscopy** a small bias voltage V is applied so that due to high electric field between tip and sample, the tunneling of electrons results in a tunneling current I.



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Principle of operation:

- *Tunnelling current depend on thickness of the barrier whereby Tip–Sample distance (<1nm)*
- *By monitoring the current through the gap, we have very good control of the **tip-sample distance.***

Piezoelectric Tube scanner:

- *Piezoelectric tube scanner: Piezoelectric materials are used to create a tube scanner.*
- *These can be used to manipulate an object in three dimensions under electronic control*
- *Piezoelectric Effect Certain materials exhibit what is called the piezoelectric effect. This is an effect whereby applying a voltage across a piezoelectric crystal, it will elongate or compress (the size of the object changes)*
- *Piezoelectric Effect PZT: Lead zirconium titanate is one of the most common piezoelectric material used.*

Feedback loop attached with a Display:

- *Lastly, a feedback loop is required, which monitors the tunneling current and coordinates the current and the positioning of the tip.*

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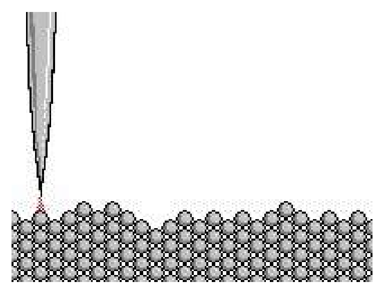
Two Operating modes of STM:

- **Constant Current mode**
- **Constant Height mode**



Constant Current Mode :

If the **tunneling current** is kept **constant** the Z position of the tip must be moved up and down. If this movement is recorded then the topography of the specimen can be imaged.

Constant Height Mode :

Alternatively, if the **Z position of the tip is kept constant** the tunneling current will change as it moves across the surface. If the changes in current are recorded, then the topography of the specimen can be imaged.

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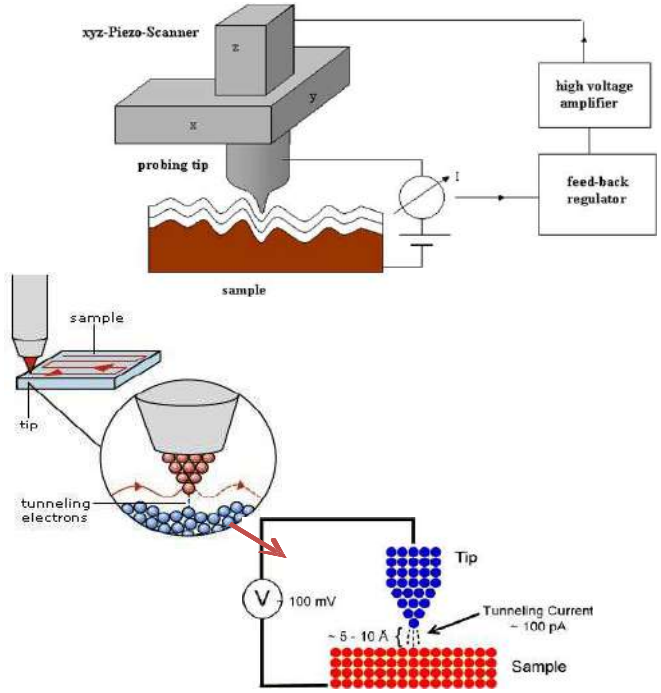
Two Operating modes of STM:

Constant Current Mode:

- **In STM, a conductive tip is positioned above the surface of the sample.**
- **When the tip moves back and forth across the sample surface at very small intervals, the height of the tip is continually adjusted to keep the tunneling current constant.**
- **The height of the tip attached to XYZ piezo scanner is adjusted with the help of feedback loop that monitors the tunneling current continuously.**
- **The tip positions (Z movement) are used to construct a topographic map of the surface.**



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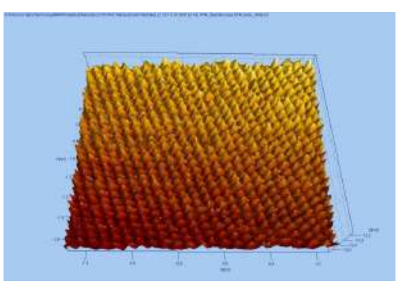
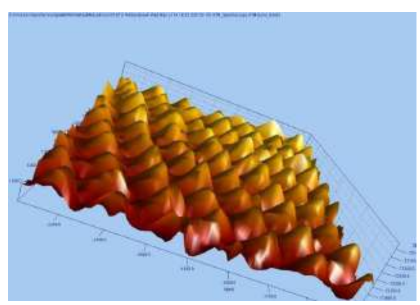
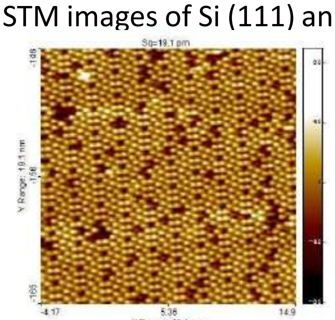
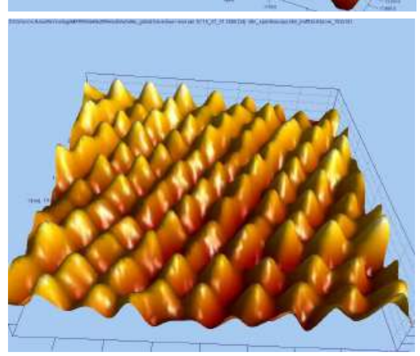
STM Topography of HOPG





STM images of Si (111) and Au





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STM Advantages & Disadvantages

Advantages

- Used to image only conductors and semiconductors
- Able to obtain very high-resolution images

(Scanning resolution is ~ 0.01nm in XY directions and 0.002 nm in Z directions, offering true atomic resolution three-dimensional image)

- Probe tips can be made out of conducting wire.

Disadvantages

- STM does not work with insulating materials

(If there are insulating materials present on the sample you can crash the tip)

- Often need to be used under vacuum

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Questions / Discussions

https://www.youtube.com/watch?v=FQzUrbKTLVU

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Prof. AJAY AGARWAL
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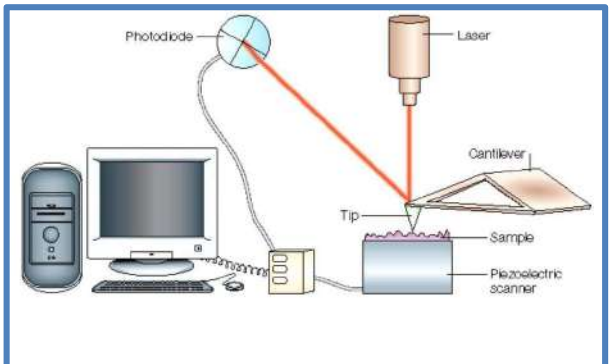
Atomic Force Microscope AFM

- An atomic force microscope (AFM) creates a highly magnified three-dimensional image of a surface.
- *AFM has the advantage of imaging almost any type of surface, including polymers, ceramics, composites, glass, & biological samples such as proteins and DNA.*
- In an AFM, the **force** between the sample and tip is detected, rather than the tunneling current.
- **Determine the roughness of a sample surface or to measure the thickness of a thin layers**

https://www.youtube.com/watch?v=s6KqJS1GZNE



Schematic representation of the components of an atomic force microscope (AFM).

Tip radius ~ 2 ... 50 nm
Force ~ 0.01 nN ... 1 nN
Sample: nearly any sample

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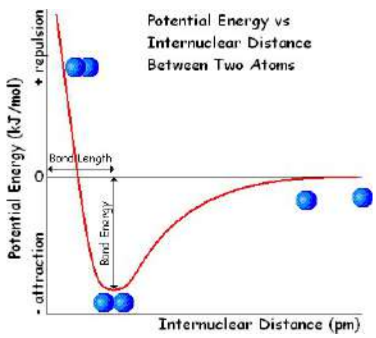
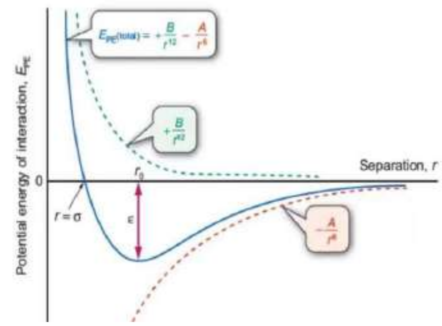
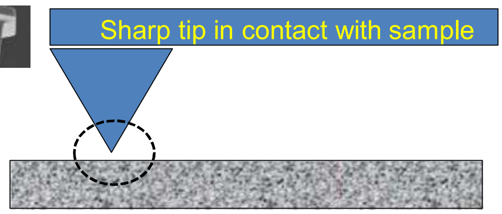
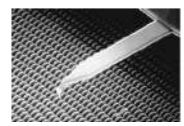
Lennard-jones potential









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Invented in 1986.
Binnig, Quate & Gerber.

Digital Instrument's
AFM Set-up

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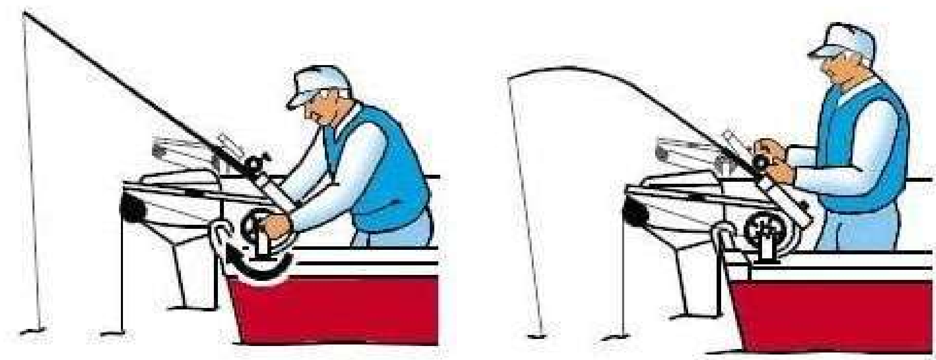
Cantilever



Fishing Spool inserted into water.

Depending upon the weight of fish caught….. the spool bends.

AFM Cantilever

Depending upon the interaction forces, the cantilever bends!!!

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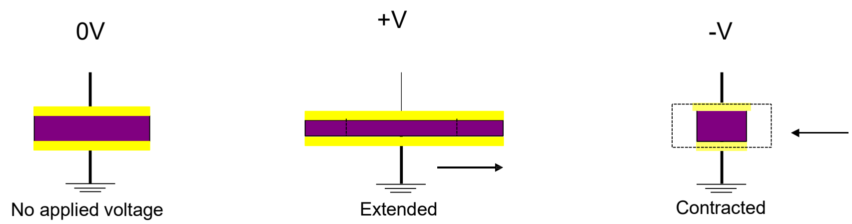
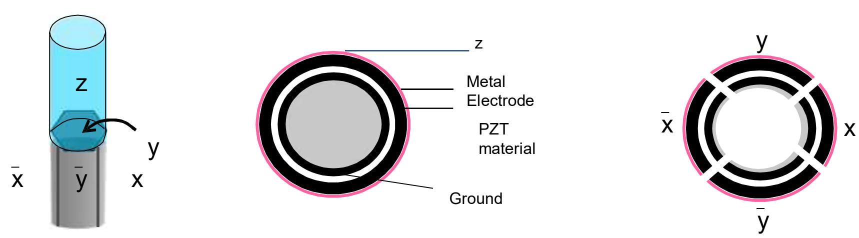
Instrumentation Overview

- Scanner
- Probe
- Photodetector
- Feedback

Scanners consist of Piezoelectric materials



Design of a scanner tube



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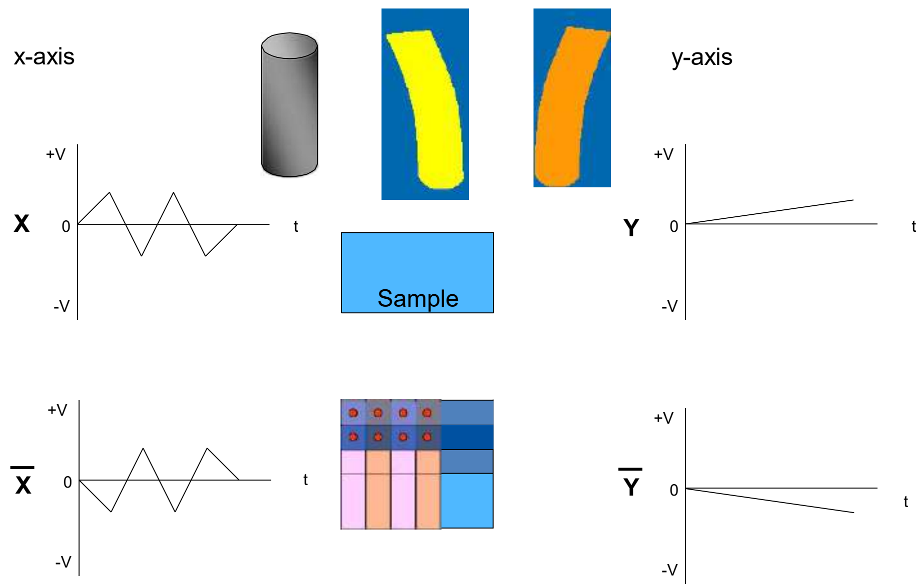
AC voltages applied at different segments of scanner tube



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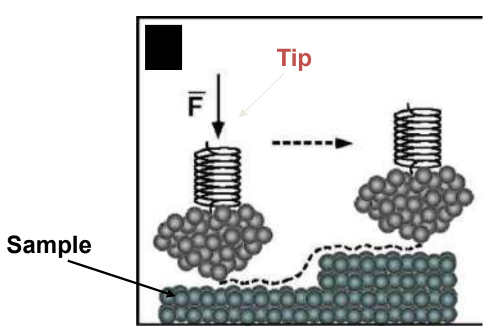
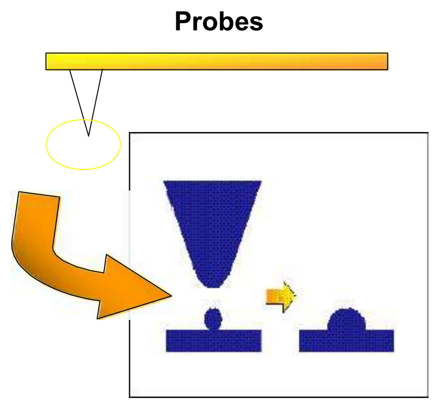
There is a reflective coating at the back side of the cantilever.

The cantilever moves on the sample surface. It follows the contours on the sample i.e. bumps or pits present on the surface.





Usually Si or Si_3N_4 tips are used.

The cantilevers obey Hooke’s law for small displacements. So, the interaction force between tip and sample can be found out.

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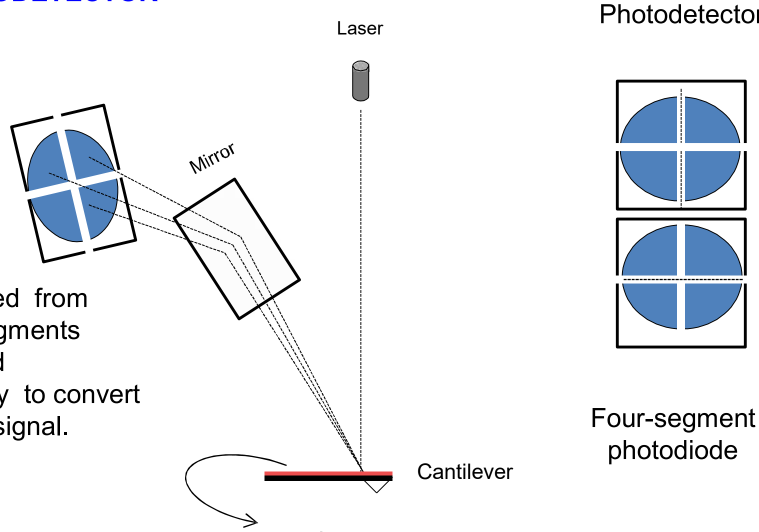
The PHOTODETECTOR



Light received from different segments is processed electronically to convert into height signal.

Reflective coating on the back-side of the cantilever

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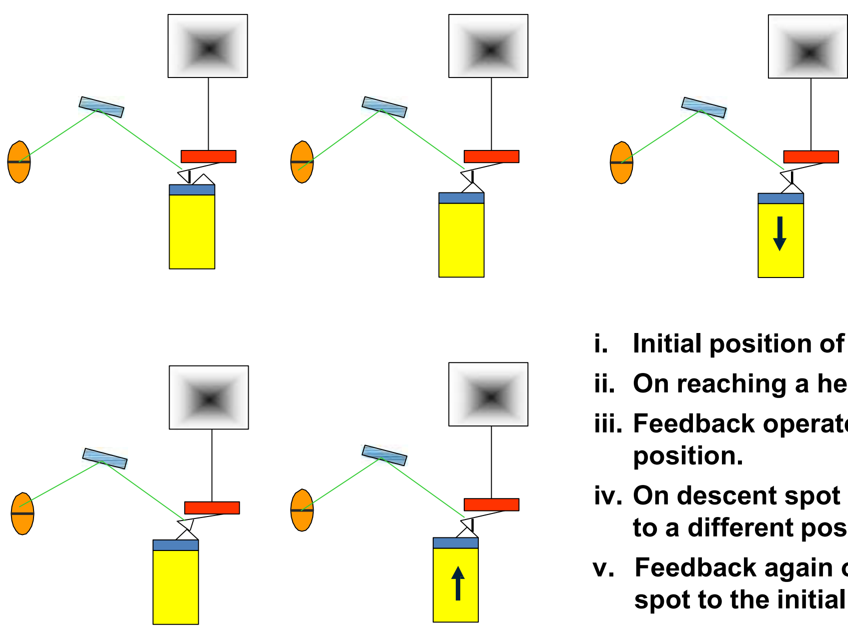

i. **Initial position of laser spot.**
ii. **On reaching a height, spot gets deflected.**
iii. **Feedback operates, brings spot to initial position.**
iv. **On descent spot deflects again, but now to a different position.**
v. **Feedback again operates to bring the spot to the initial position.**

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Nano Sensors: Lab sessions

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Prof. AJAY AGARWAL
ELECTRICAL ENGINEERING
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Class 20-21 dated 27-28th Feb 2025

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Nano Sensors

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Lecture 22 dated 4th Mar 2025

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AFM: Feedback

Constant Force mode

- **Feedback is on.**
- **Piezo responds to any changes detected & alters tip-sample gap.**
- **Fairly faithful topography.**

Constant Height mode

- **Feedback is off.**
- **Tip moves at a constant height above sample.**
- **High resolutions are achieved.**

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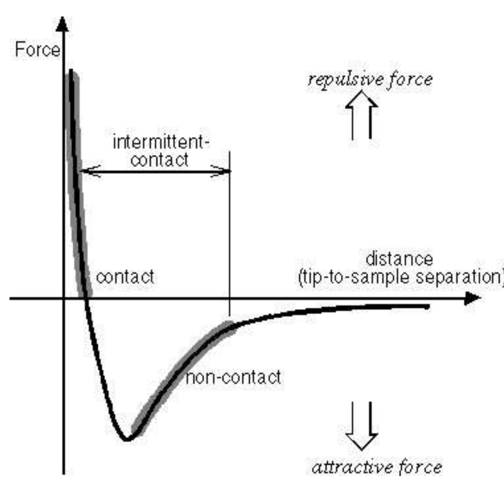
- At distances few Å apart attractive van der Waals forces act between the atoms of the tip and the sample.
- When the tip approaches closer and electron clouds repel each other the attractive van der Waals forces decrease in strength.
- When the two forces balance each other at distances ~ **chemical bond**, the force goes to zero.



A general Force-distance curve

REGIME OF CONTACT MODE AFM

- **At even closer distances repulsive van der Waals forces come into play.**

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AFM's Most Common Operating Modes

Contact Mode

- Tip-sample remain close while scanning.
- Mode is in the repulsive regime of force-plot.
- Large lateral forces exist- causing tip to drag.
- Strong repulsive forces act.

TAPPING Mode

- Cantilever oscillates at/near its resonance frequency.
- Tip taps the surface occasionally.
- Lateral forces are dramatically reduced.
- Better for softer samples.

NON-Contact Mode

- Cantilever vibrates near the surface of the sample.
- No physical contact with sample surface.
- Changes in resonant freq, vibration amplitude are detected.
- Cantilevers used are stiffer.

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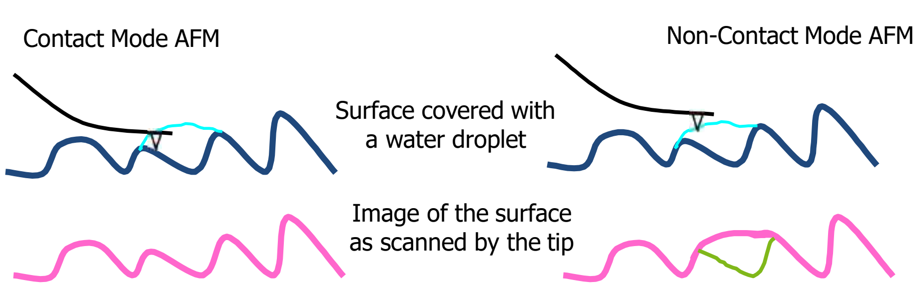
Difference between Contact and Non-contact Mode



- Tapping mode provides the best resolution.
- The lateral forces present in the contact mode are dramatically reduced in this mode giving a higher lateral resolution.

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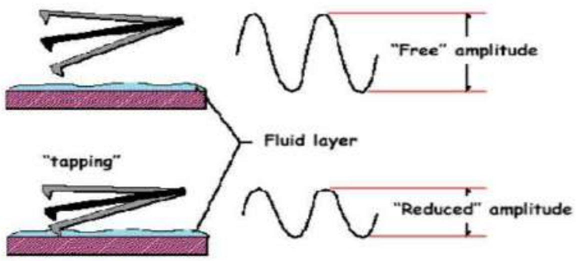
Non-contact vs. tapping mode

- Both are based on a Feedback Mechanism of constant oscillation amplitude.
- Non-contact mode: amplitude set as ~ 100% of "Free" amplitude;
- Tapping mode: amplitude set as ~ 50 -60% of "Free" amplitude.
- Tapping mode provides higher resolution with minimum sample damage.
- **Most of times, non-contact mode is operated as tapping mode.**



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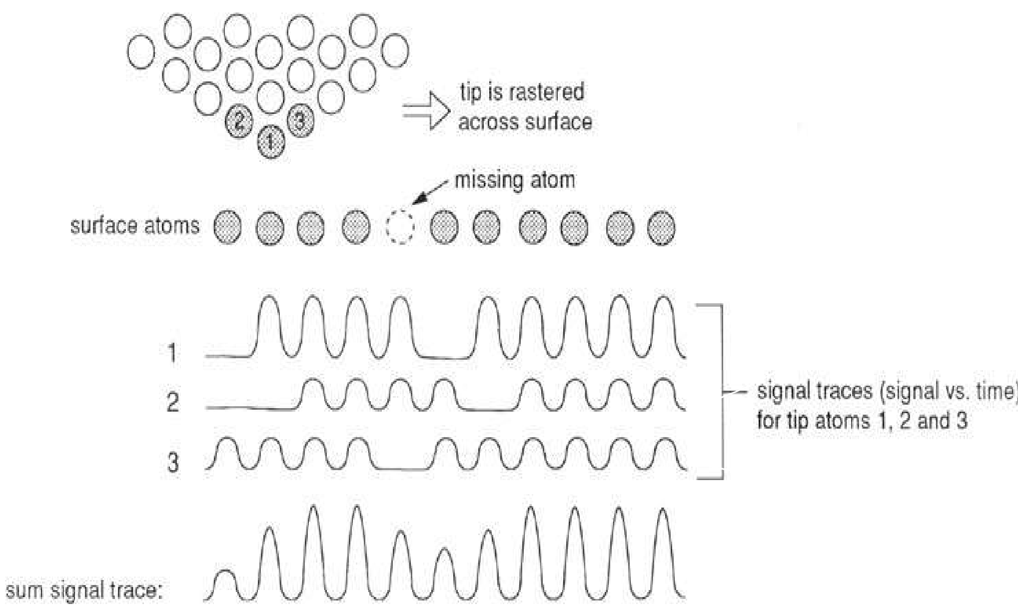
Effect of Tip-Shape on Imaging



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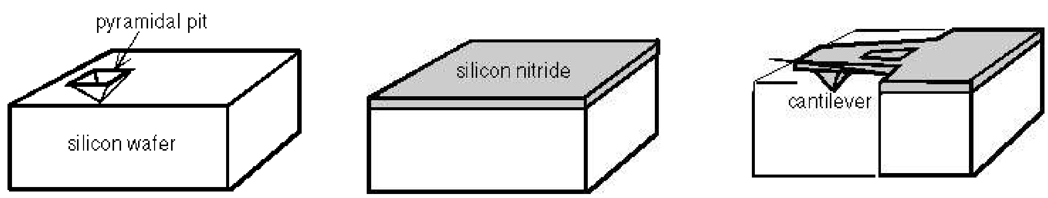
1.Silicon tips:
- Silicon conical tips are made by etching into the silicon around a silicon dioxide cap.
- They high aspect ratio but they may break more easily than the pyramidal or tetrahedral geometries.

2. Silicon nitride tips
- SiN tips are fabricated by depositing a layer of silicon nitride over an etched pit in a crystalline silicon surface as shown in the figure.
- Silicon nitride is a harder material than silicon, which also makes silicon nitride tips more durable than silicon tips.



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Some Facts

- Typical forces between probing tip and sample range from **10^{-11} to 10^{-6} N.**
- The interaction between **two covalently bonded atoms** is of the order of **10^{-9}N** at separations ≈Å.
- In non-contact mode the separation between tip & sample ~ **10 to 100 nm**.
- van der Waals, electrostatic, magnetic or capillary forces are into play.
- At smaller separations ~Å the probing tip is in contact with the sample.

Here, ionic repulsion forces allow the surface topography to be traced with high resolution.

This results in a higher resolution for imaging in contact mode.

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Some Images …

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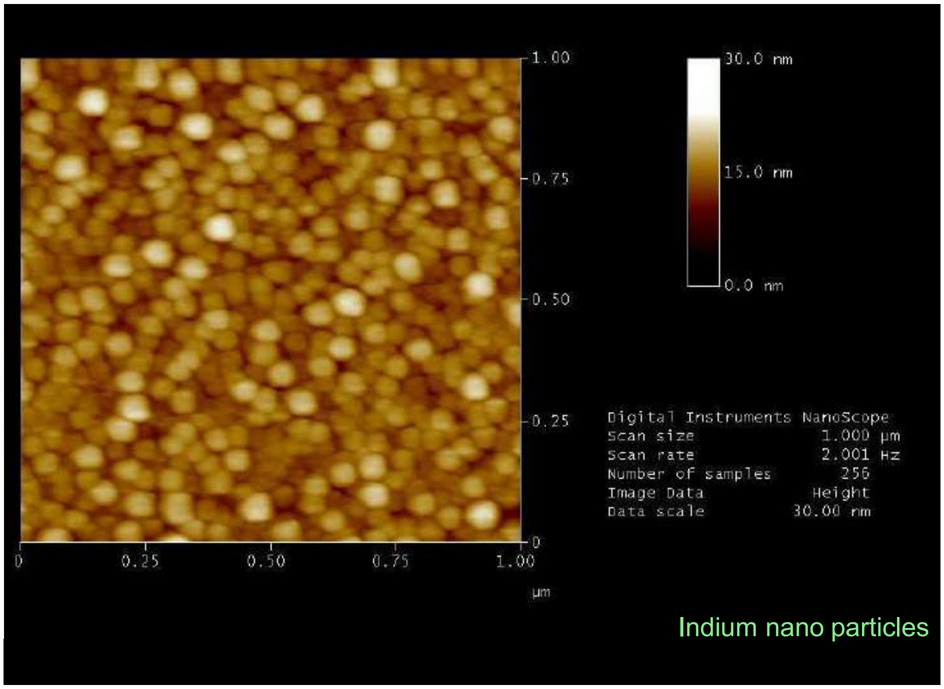

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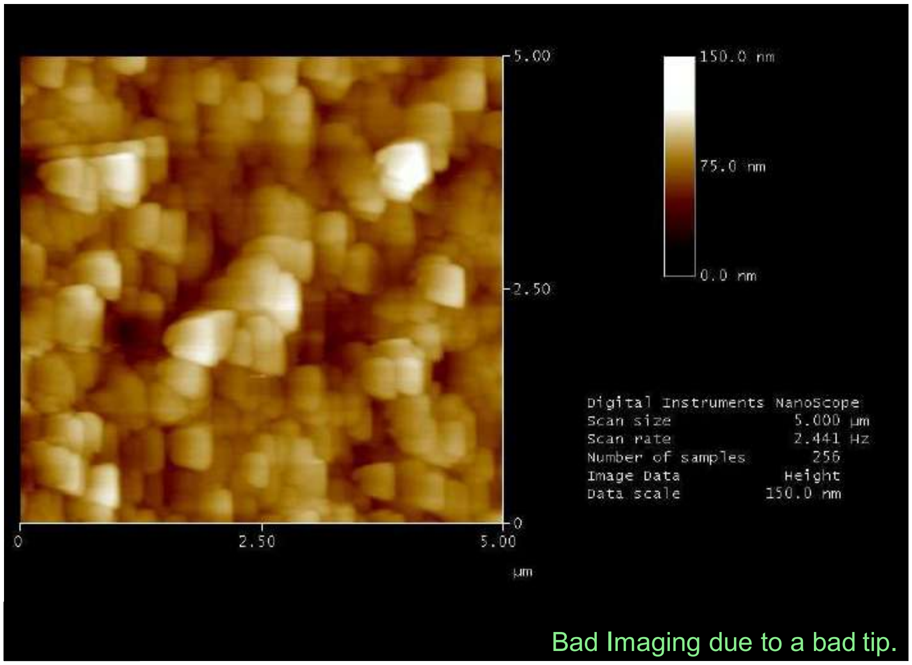
5.00
2.50
0
0
2.50
5.00
µm
150.0 nm
75.0 nm
0.0 nm
Digital Instruments NanoScope
Scan size 5.000 µm
Scan rate 2.441 Hz
Number of samples 256
Image Data Height
Data scale 150.0 nm
Bad Imaging due to a bad tip.

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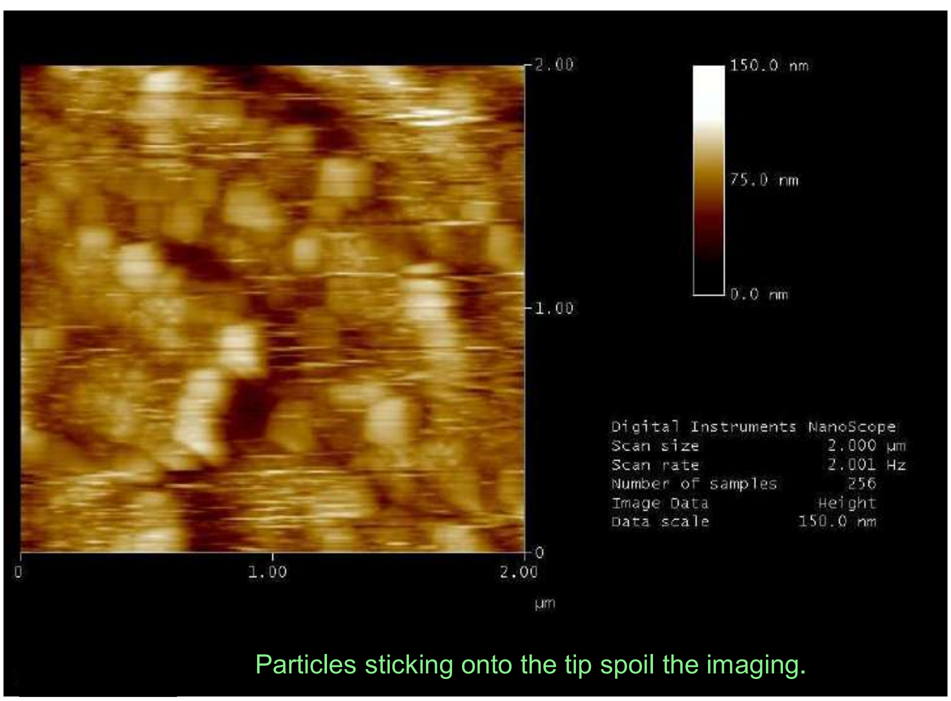
2.00
1.00
0
0
1.00
2.00
µm
150.0 nm
75.0 nm
0.0 nm
Digital Instruments NanoScope
Scan size 2.000 µm
Scan rate 2.001 Hz
Number of samples 256
Image Data Height
Data scale 150.0 nm
Particles sticking onto the tip spoil the imaging.

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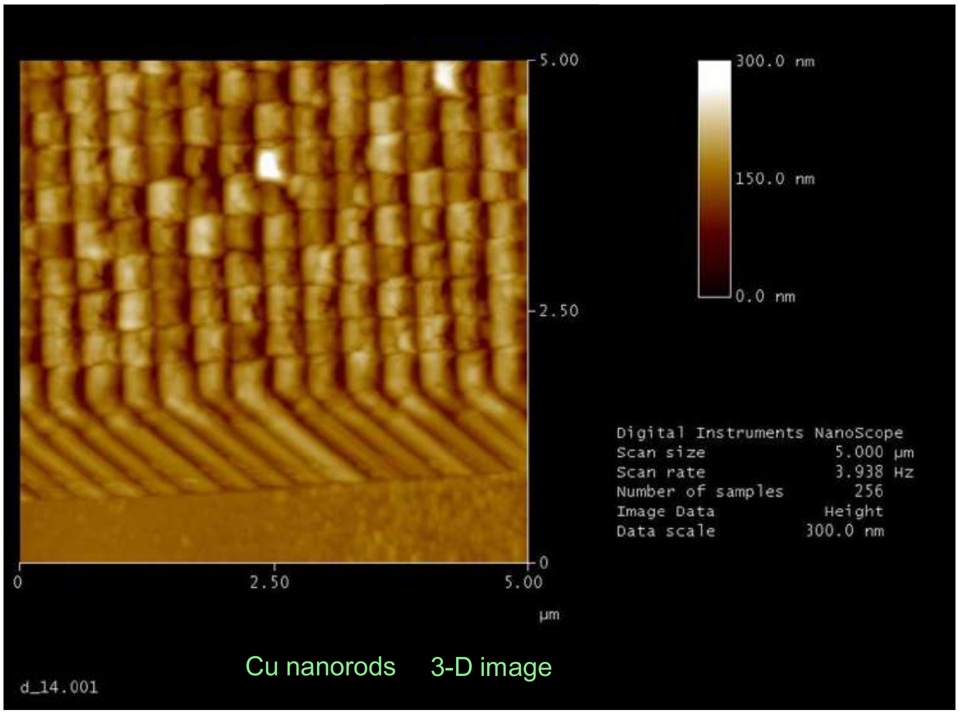
5.00
2.50
0
0
2.50
5.00
µm
300.0 nm
150.0 nm
0.0 nm
Digital Instruments NanoScope
Scan size 5.000 µm
Scan rate 3.938 Hz
Number of samples 256
Image Data Height
Data scale 300.0 nm
Cu nanorods
3-D image
d_14.001

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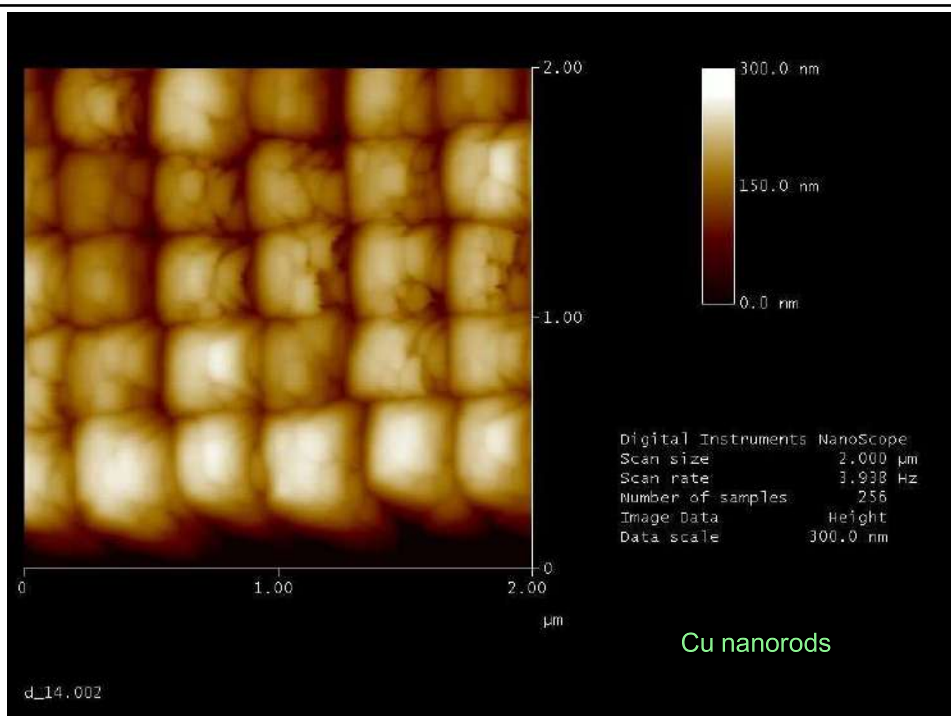
2.00
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µm
300.0 nm
150.0 nm
0.0 nm
Digital Instruments NanoScope
Scan size 2.000 µm
Scan rate 3.938 Hz
Number of samples 256
Image Data Height
Data scale 300.0 nm
Cu nanorods
d_14.002

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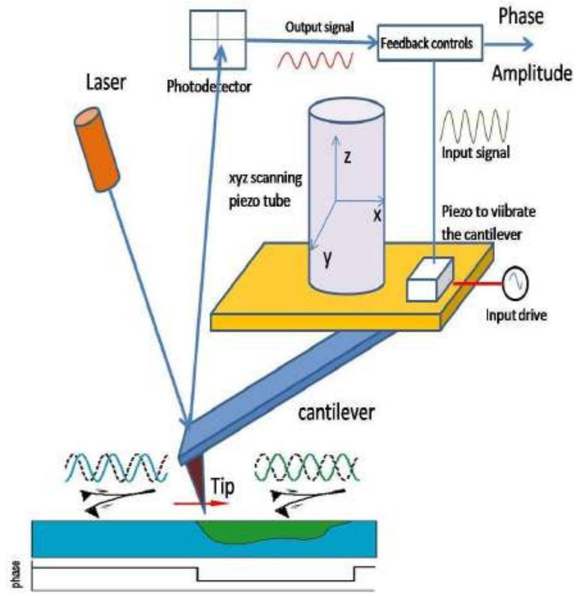
Phase Imaging

- Phase image: The phase lag of the cantilever oscillation, relative to drive signal
  - ➢ Simultaneously imaged with topography
- Phase lag is dependent on factors such as: viscoelastic properties of the surface, composition, adhesion and friction
- Helpful in identifying multi-phase morphology of polymer blends



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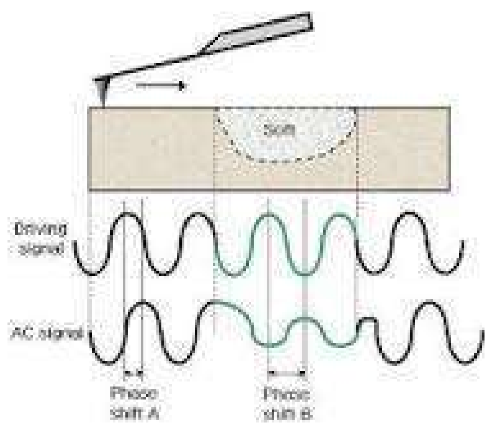
Phase Imaging

- Phase Imaging refers to the monitoring of the phase lag between the signal that drives the cantilever oscillation and its output signal as shown in Figure.
- Changes in the phase lag reflect changes in the mechanical properties of the sample surface.



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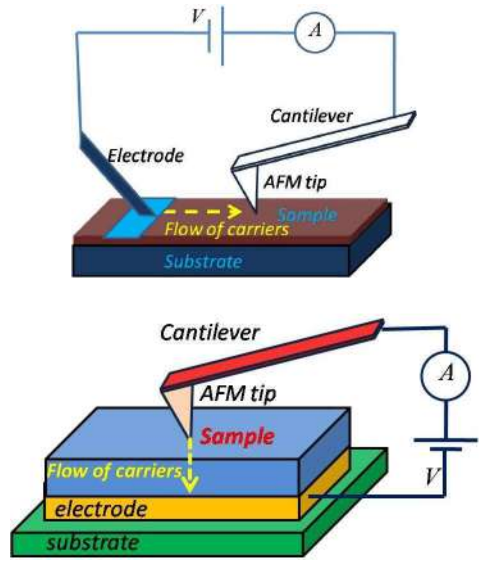
Current sensing - AFM

- CS-AFM measures the local electrical conductivity of a material at nanoscale
- Current flow direction in the sample can be controlled: either lateral or transverse direction through appropriate electrode configuration
- Local Electron/ hole mobility can be estimated from local I-V plots
- Local photocurrent can be measured by conducting I-V spectroscopy under light illumination



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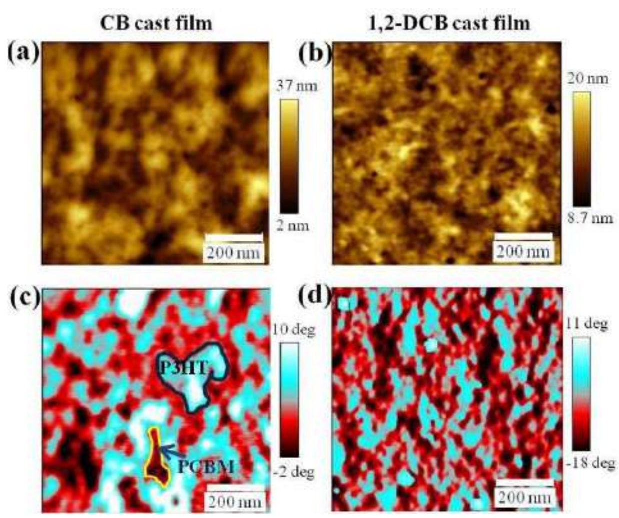
Topography and phase imaging



Topography images of poly(3-hexylthiophene): phenyl-C_{61}-butyric acid methyl ester (P3HT:PCBM) blended films spin cast from CB & 1,2-DCB solvents, respectively. (c and d) shows the corresponding phase images

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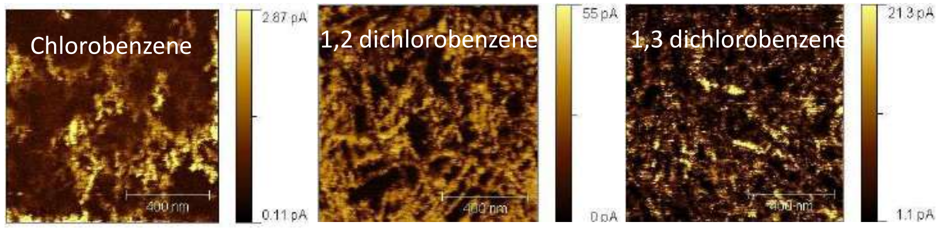
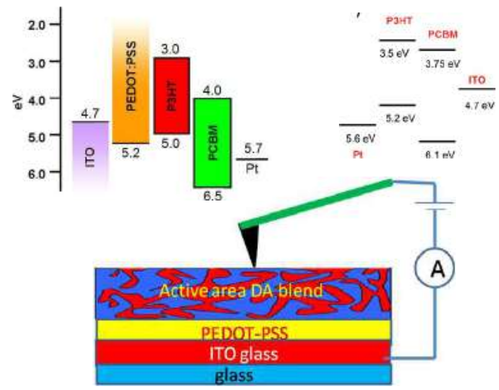
Current Sensing-AFM



- Brighter regions (higher hole current) correspond to P3HT
- Darker regions (lower hole current) correspond to PCBM
- Nanoscale charge transport pathways are clearly defined in 1,2 DCB film, which also has higher electrical conductivity as compared to CB & 1,3 DCB films



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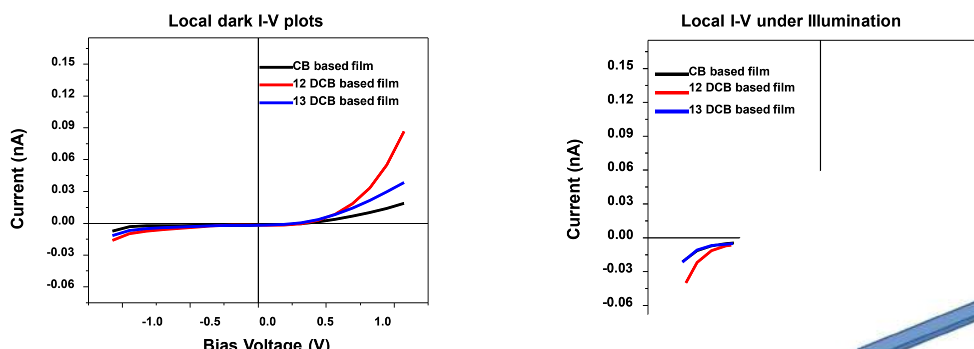
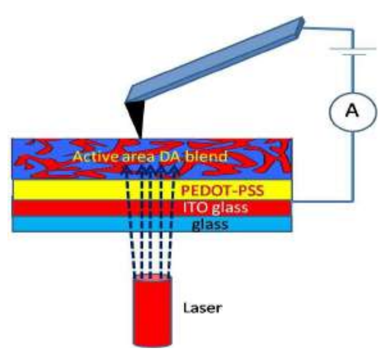
Local I-V measurements by CS-AFM



- Local I-V spectroscopy plots under dark and illuminated (680 nm, ~10^{18} photons/cm^2-sec) conditions show significant ‘photo-response’, despite weak absorption in RED region
- Local Current in all the films increased with illumination
- Dark current and illuminated photo-current was maximum in 1,2 DCB based film



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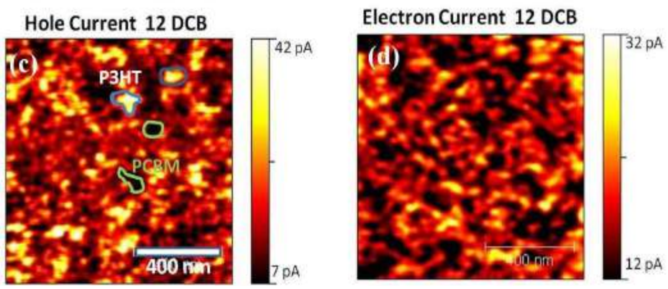
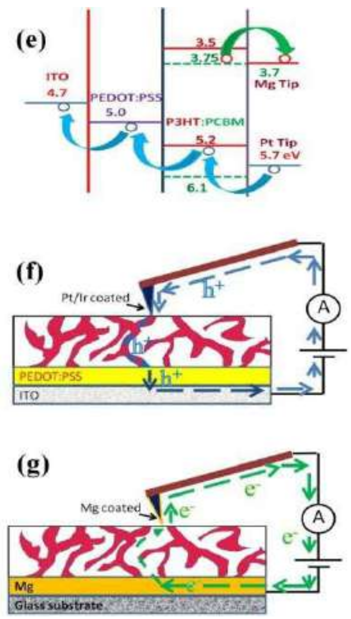
CS-AFM and selective carrier dynamics





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Hole mobility calculations from local I-V plots

- Space charge limited current density (SCLC)

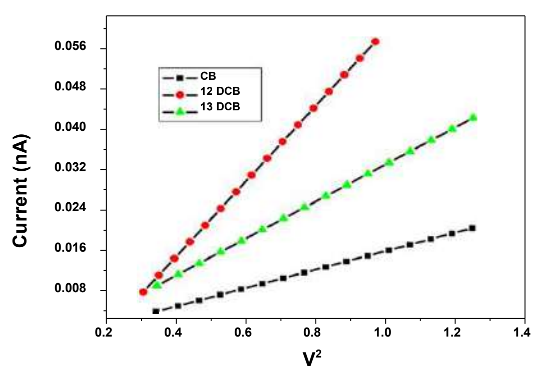
$$J = \frac{9}{8}\varepsilon\varepsilon_0\mu\frac{V^2}{L^3}$$

(Mott, N. F. Gurney, R. W. Electronic Processes in Ionic Crystals, Oxford University Press, London, 1948)

J= current density
Tip radius = 20 nm
ε = dielectric constant
μ = hole mobility
V = bias
L = thickness of film

- Hole mobility (dark) obtained by fitting the above equation
  - CB 4.64 x 10^{-3} cm^2/V-s
  - 1 3 DCB 6.89 x 10^{-3} cm^2/V-s
  - 1 2 DCB 1.40 x 10^{-2} cm^2/V-s

- Highest hole mobility in 1 2 DCB film



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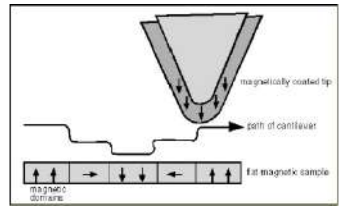
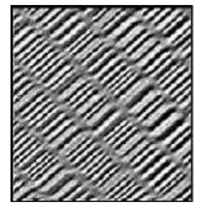
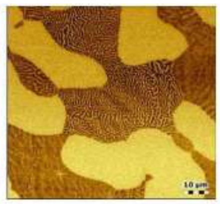
Magnetic Force Microscopy (MFM)

- Because the magnetic forces interact at greater distances than van der Waals forces, so electrical or magnetic force information can be separated from surface topography simply by increasing the tip-to-sample distance i.e. by lifting up the tip.
- Dual scanning: the tip **first acquires** surface topography in the tapping mode, then the tip is lifted up, and retraces the surface profile maintaining constant tip-surface separation.

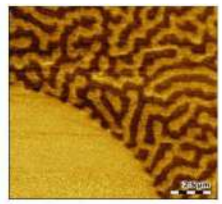




Topography of a polished stainless steel sample. Scan size: 80 µm. Height range: 50 nm.



Magnetic force microscopy (MFM) of the same 80 µm × 80 µm area. Phase range: 10°.



Magnetic force microscopy (MFM) of a smaller area. Scan size: 13 µm. Height range: 50 nm.

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Questions / Discussions

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