09.02.2022

Atomic Force Microscope (AFM)
(Absolute) Fundamentals, (very Basic) Operation and (only Some) Applications

NT 70002

AFM

$1\ nm = 10^{-9}\ m$

very Small. → How Small?

Human Hair

50-70 μm → Length Scale

~ 1/10,000 th human Hair.

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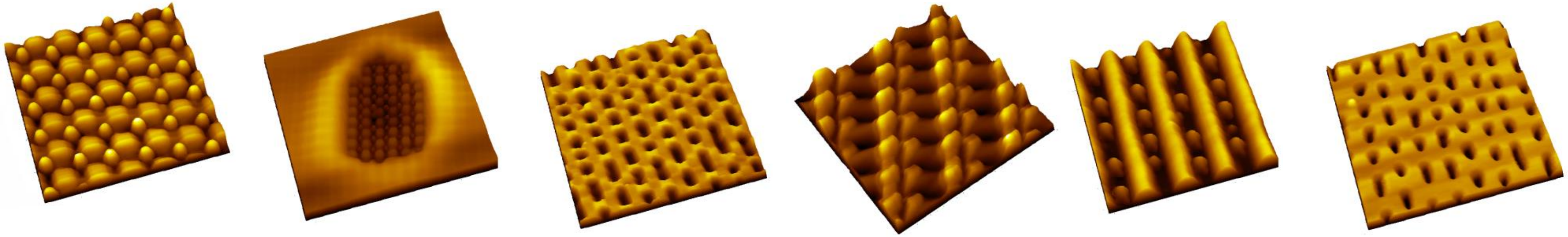


Microscopes → Optical Microscope.

5 nm

3-5 Å

1-2 Å

SEM → Scanning Electron Microscope.

TEM → Transmission Electron Microscope

Resolution ⇒

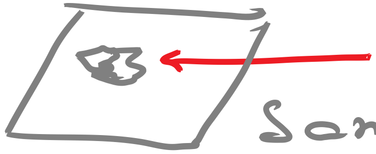
FESEM = ✓

TEM = ✓

T → Transmission.

TEM → uniqueness of TEM? →

Mostly, microscopes allow to see/observe "Surface Features"



Feature

Sample

electron Beam

Passes thru' the sample → Internal Structure.

AFM was discovered in 1986.

Gerhard Binning and Co - workers IBM Corporation

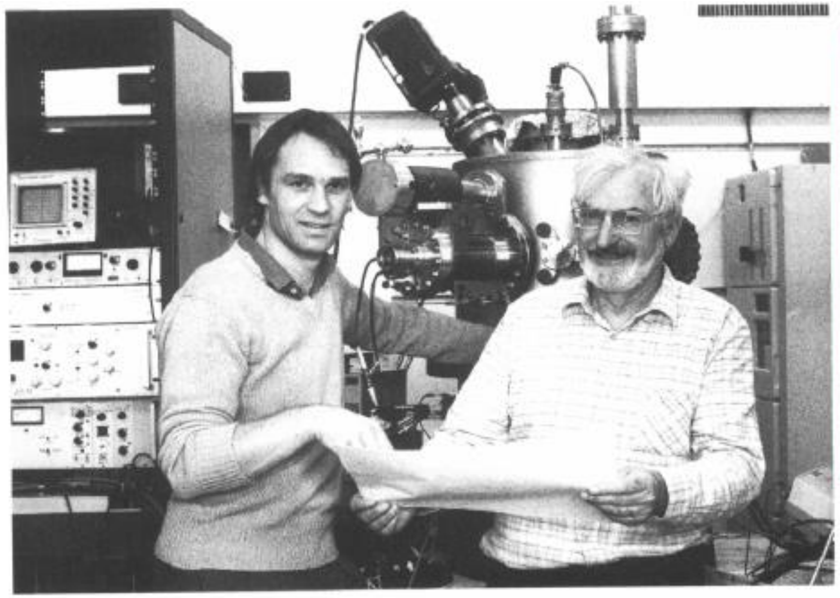
Physical Review Letters, 56,930 (1986).

(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.

Why AFM ?

STM = ? 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

⇒ What is so special about the nano scale / or Nano sized objects?

(1) Finite Size effect → Band Structure of the materials starts to change as the size becomes too small. → becomes size dependent.

Consequently

Many properties starts to change with respect to their bulk properties.

⇒ Properties that are intensive → become extensive (Size dependent)

(2) van der waal's Interaction

2 → van der waal's Interaction (or more generically intermoleculer Interaction).

London Forces

(1) Induced dipole - Induced Dipole

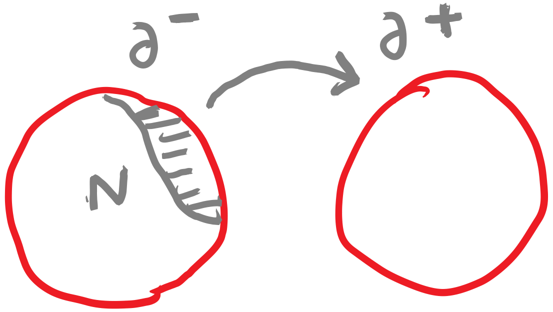
(2) Permenent dipole - Permenent Dipole

(3) P.D - I.D interactions

↳ Debye Interaction

Keesom Force

Hydrogen Bond.



Most Generic

I.D - I.D

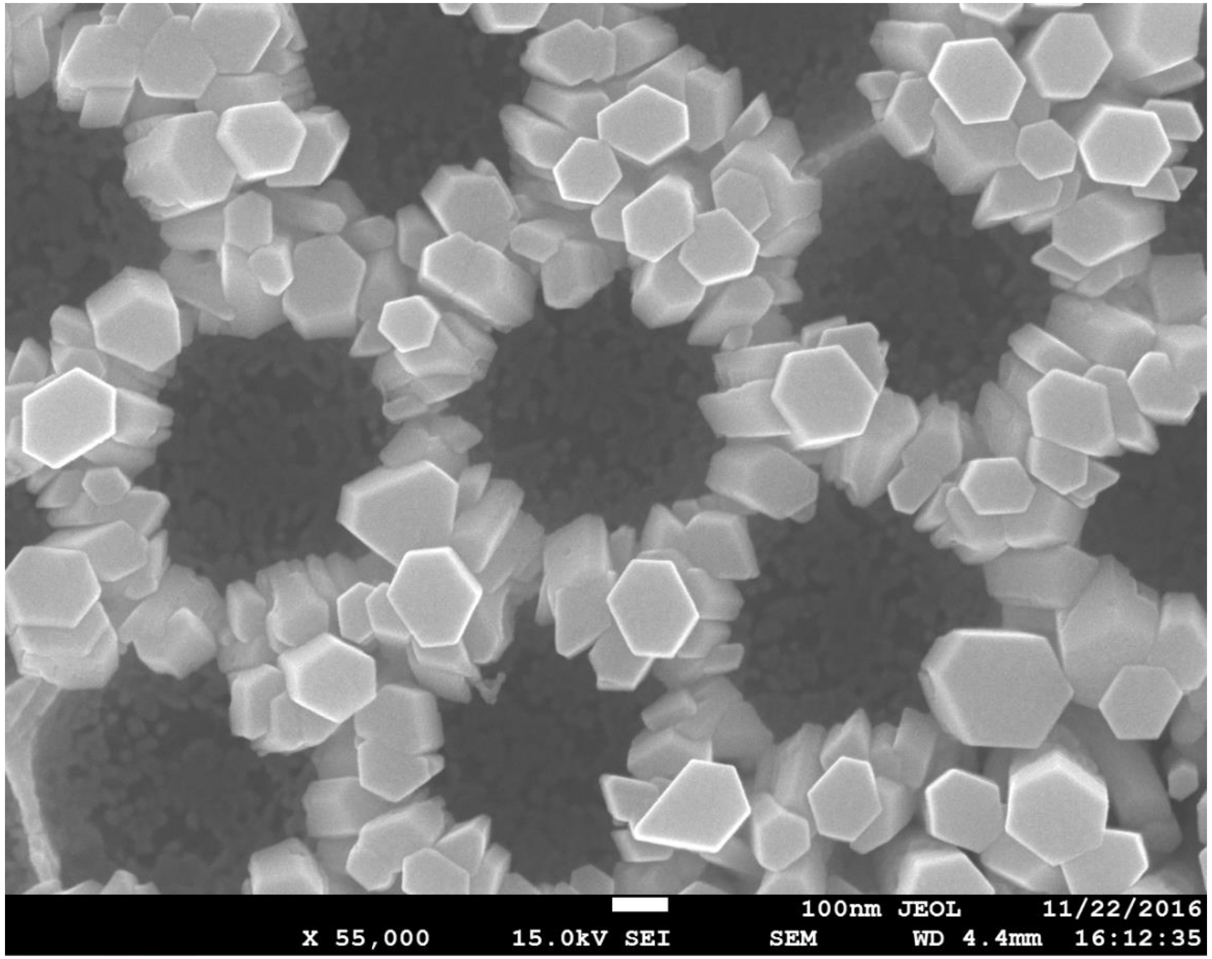
$$\phi \approx -\frac{A}{r^6}$$

mgh ↓

Second most important aspect of Nano Science / Nano Scale → It is a Length Scale that's dominated by intermoleculer Interaction.

Why AFM?

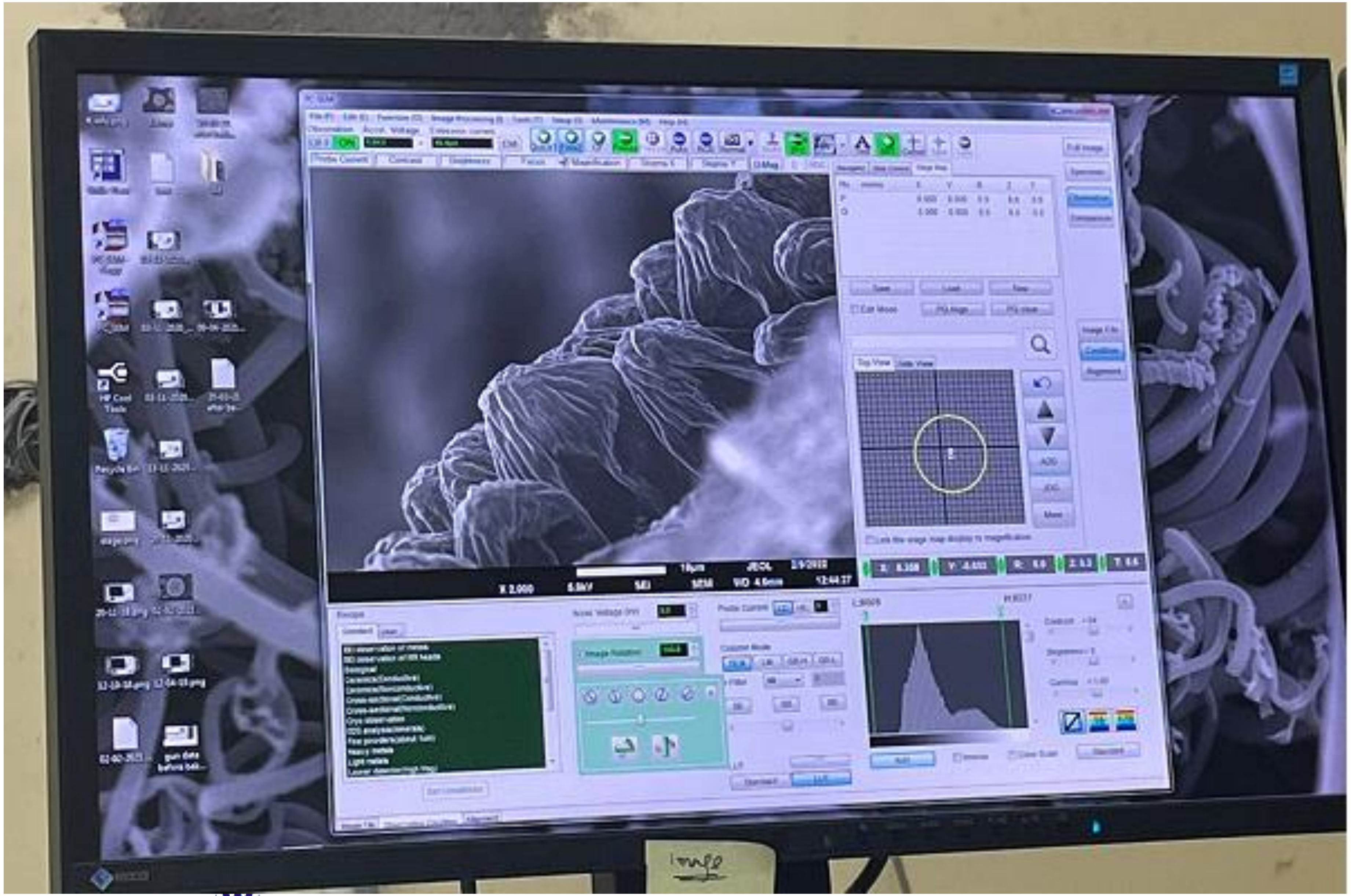
We all use a microscope to visualize small things! Here is an FESEM image.



What information is missing in this photograph?

Can you think of any limitation of this image?

Any information that is missing?



An AFM is Fundamentally totally different from ALL other Microscopes

Microscopes Generate an Enlarged IMAGE.

An AFM DOES NOT capture an Image.

The Scanning Probe Microscope (SPM) family

STM (Tunneling)

STS,STP,Scanning Electrochemical Microscope

SFM (Force)

(AFM)

Lateral Force (LFM)

Magnetic Force (MFM)

Electrostatic Force (EFM)

DPN

SPM

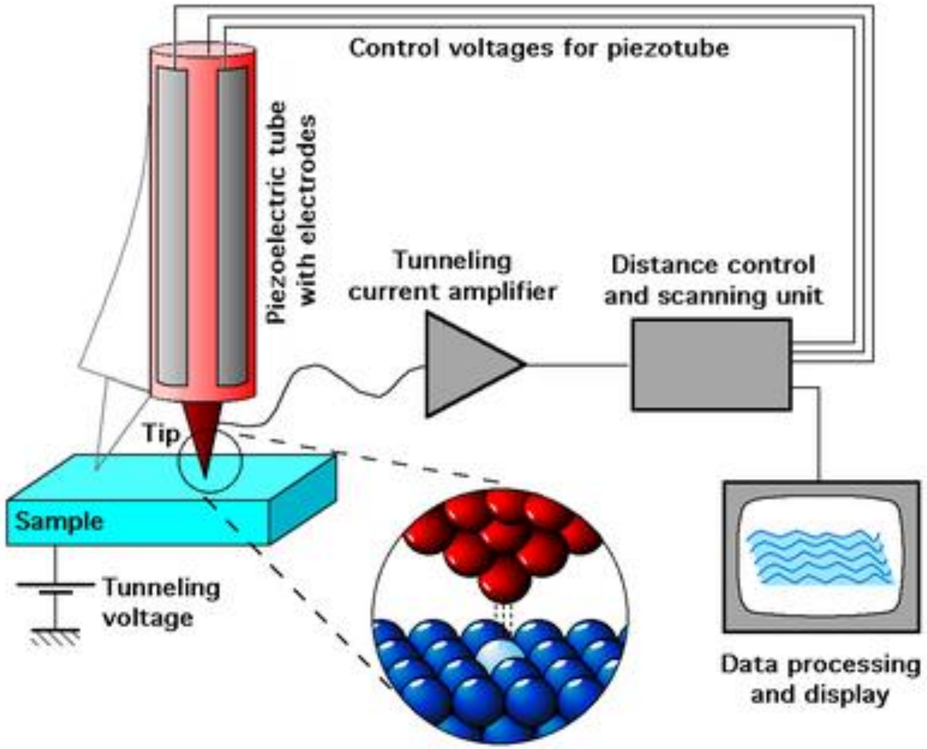
Scanning Thermal Microscope (Local Temperature)

Scanning Near Field Optical Microscope (Optical imaging)

STM (Tunneling)

The STM is based on the concept of quantum tunneling.

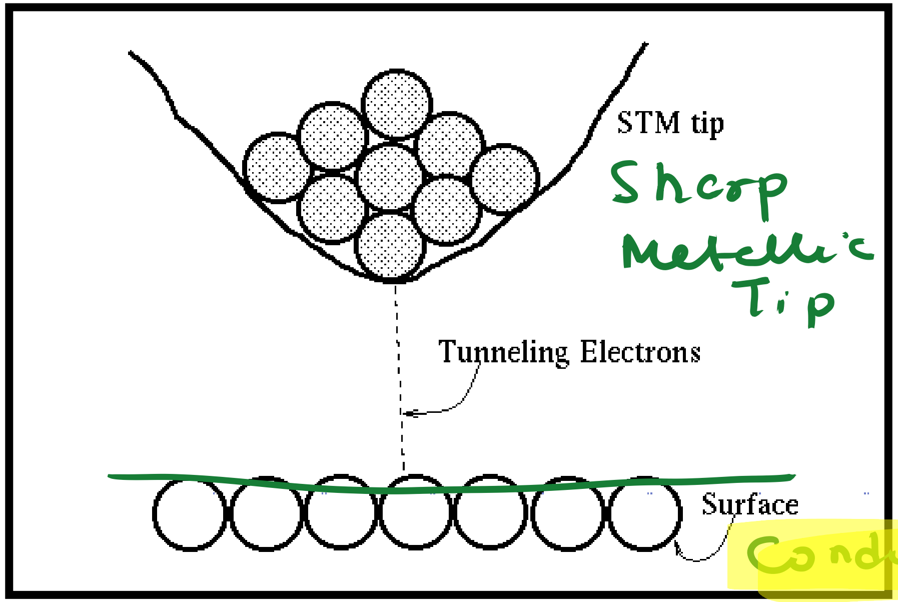
When a conducting tip is brought very near to the surface to be examined, a bias (voltage difference) applied between the two can allow electrons to tunnel through the vacuum between them.

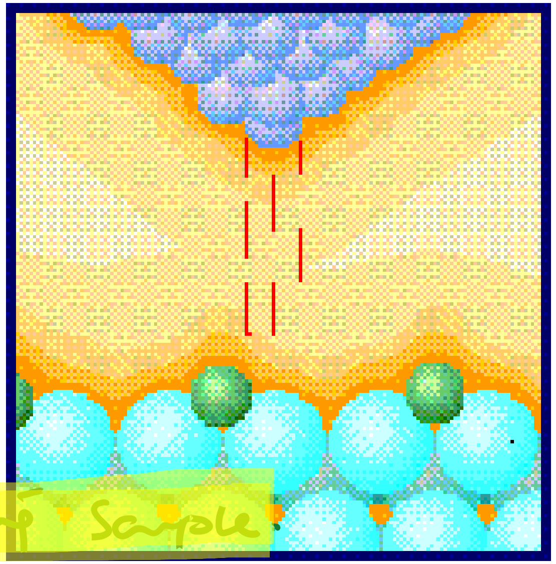


The resulting *tunneling current* is a function of tip position, applied voltage, and the local density of states (LDOS) of the sample.

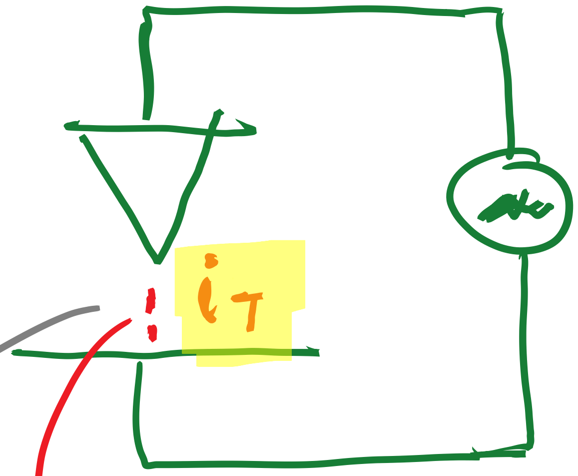
Information is acquired by monitoring the current as the tip's position scans across the surface, and is usually displayed in image form.

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





Tunneling Current

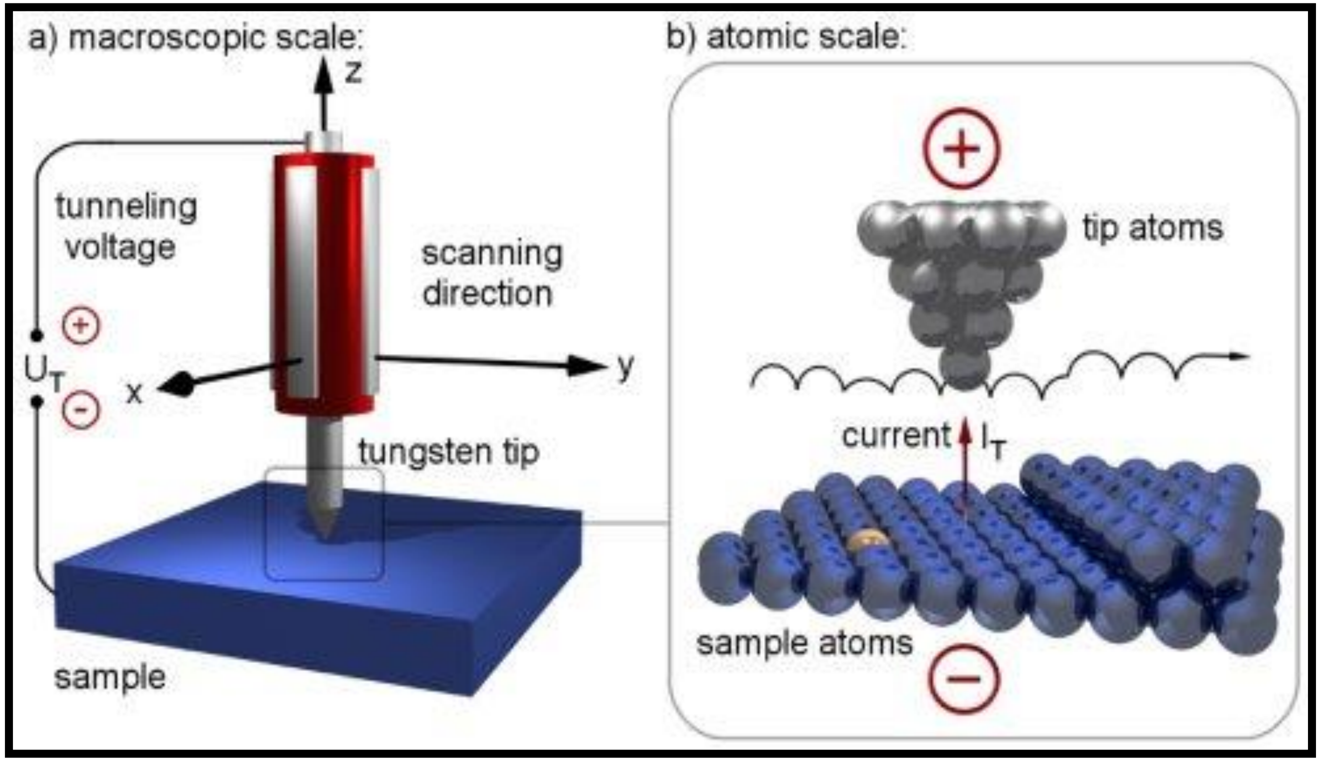


Flow of Electron

$i_T \rightarrow$ Depends on the Sepn distance.

$i_T \rightarrow 0$

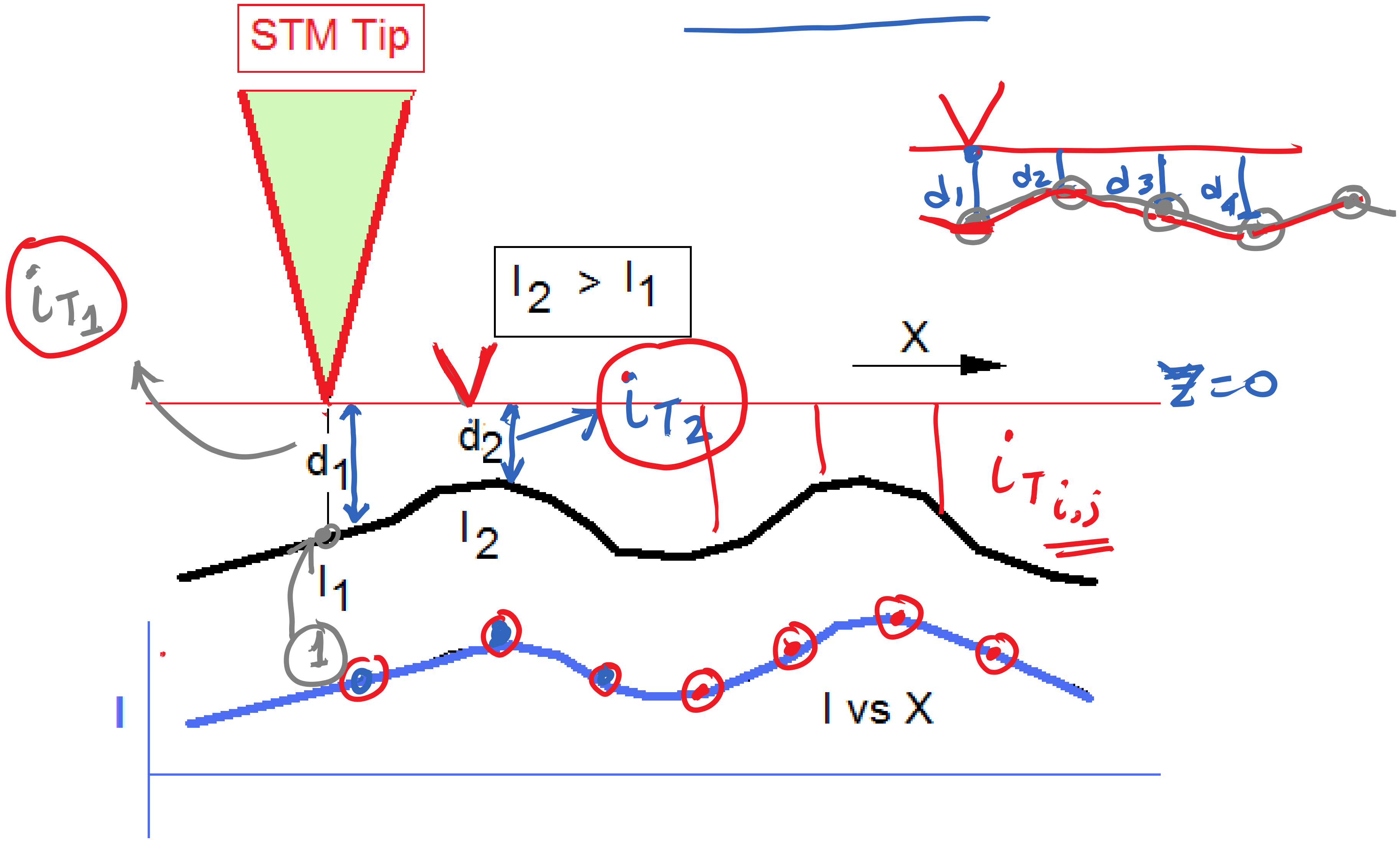
When a sharp conducting tip is sufficiently close to the surface of another conducting sample (~ 1nm), and a bias is applied, a tunneling current gets established



The tunneling current is exponentially proportional to the distance and thus via a feedback loop the tip can be maintained at a constant distance from the surface by maintaining a constant tunneling current.

Constant Height Mode STM

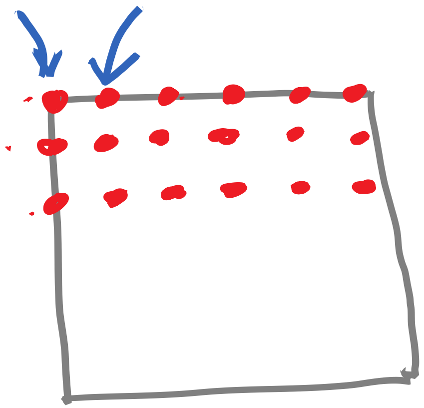
$i_{T_2} > i_{T_1}$ ← ??



eight or

tunneling current varies

lata set, the



Rastering

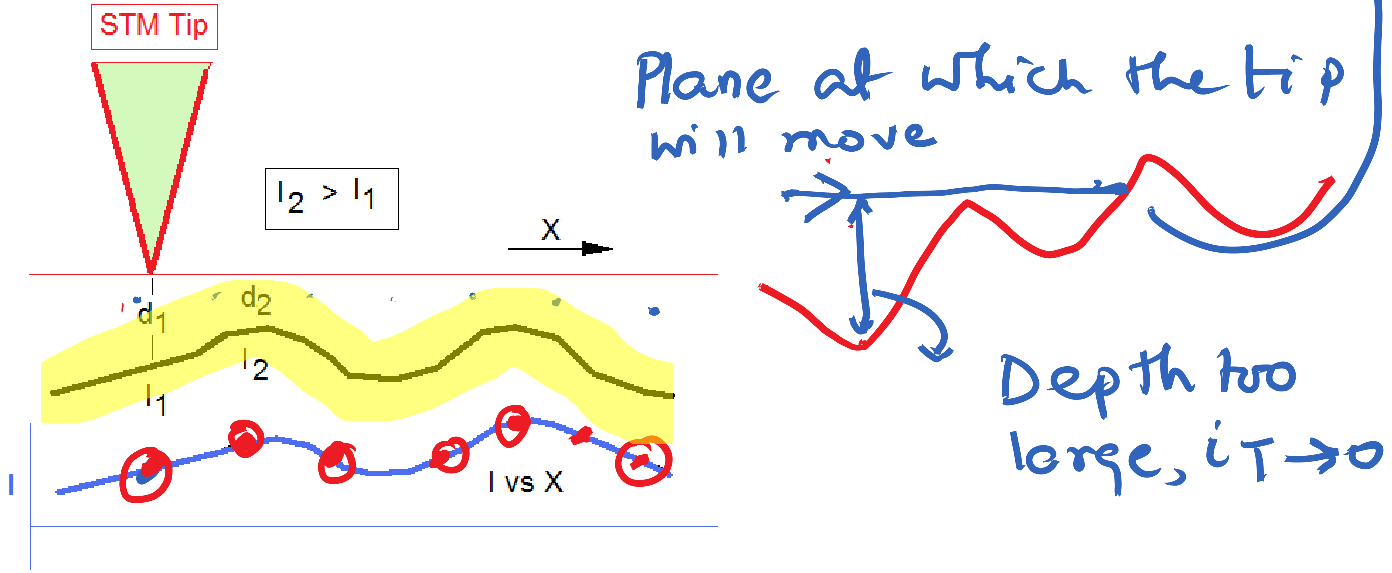
Constant Height Mode STM

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.

Tip Crash

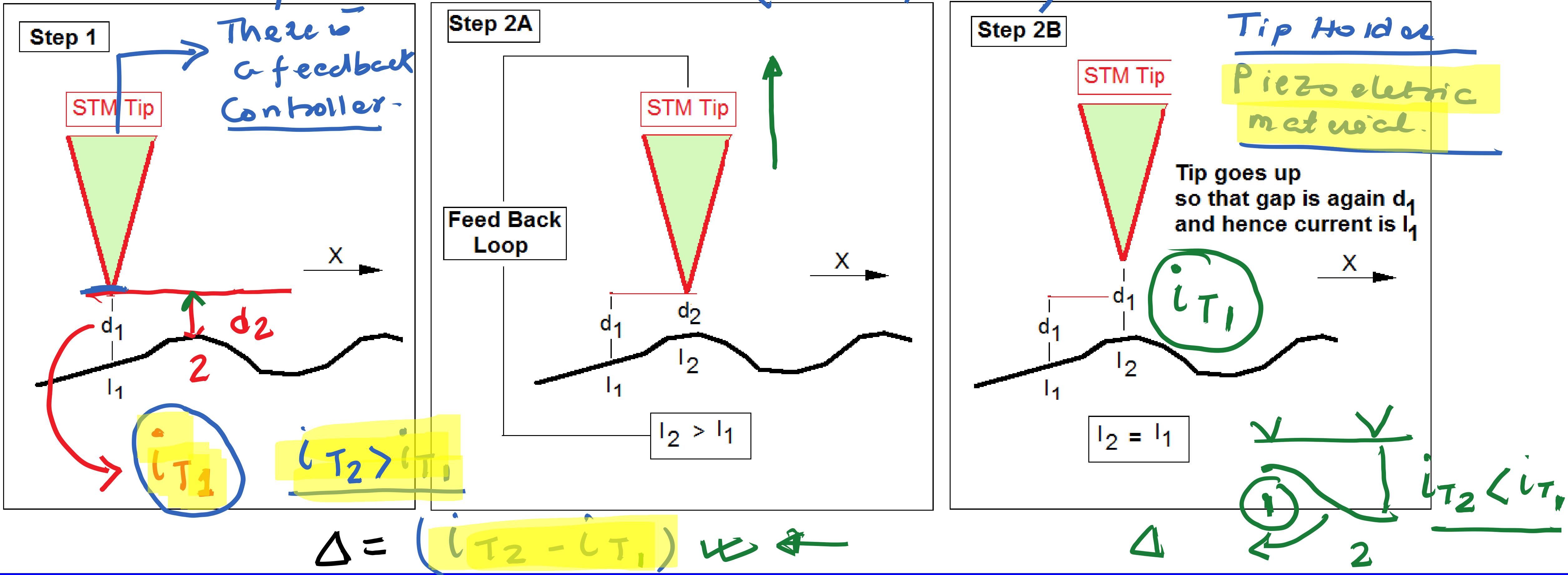


IIT Kharagpur

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.

That can adjust the vertical movement of the tip. (Set point)



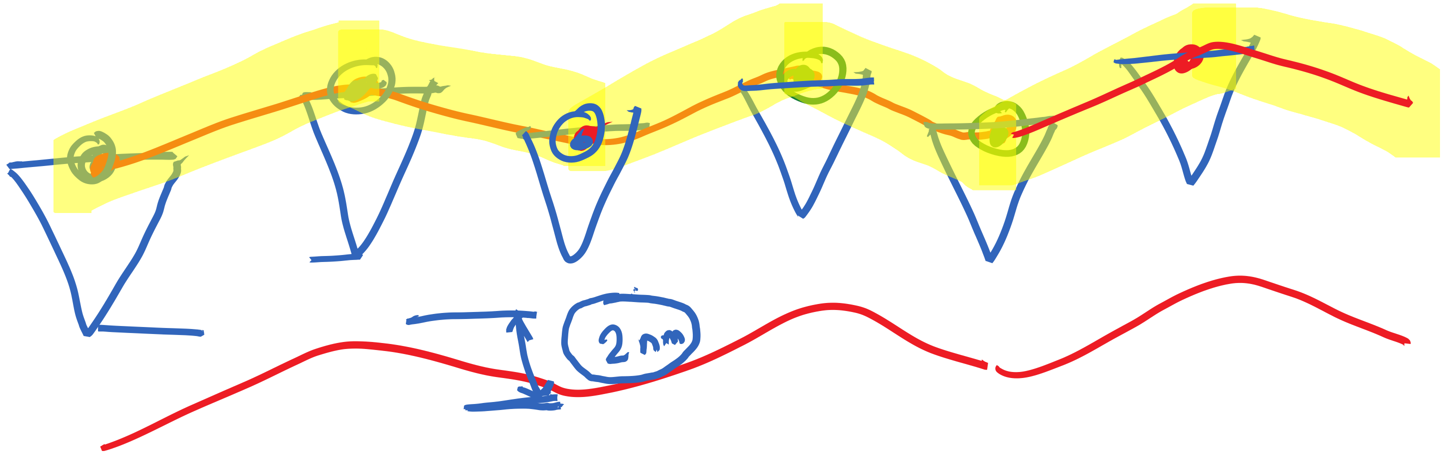
$\Delta = (i_{T_2} - i_{T_1})$

Limitation? = ?

PZT

Constant Current Mode

Piezo: If we apply mech stress, electricity gets generated.
or Apply electric field → Size/shape changes.



Mathematically rendered.

Actual Surface

Tunneling → STM Tip.

Constant Height Mode → Limitations.

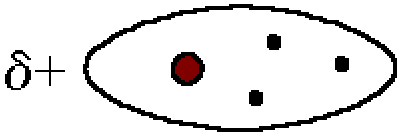
→ Constant Current Mode → Integration of a Feed back controller to the Tip.

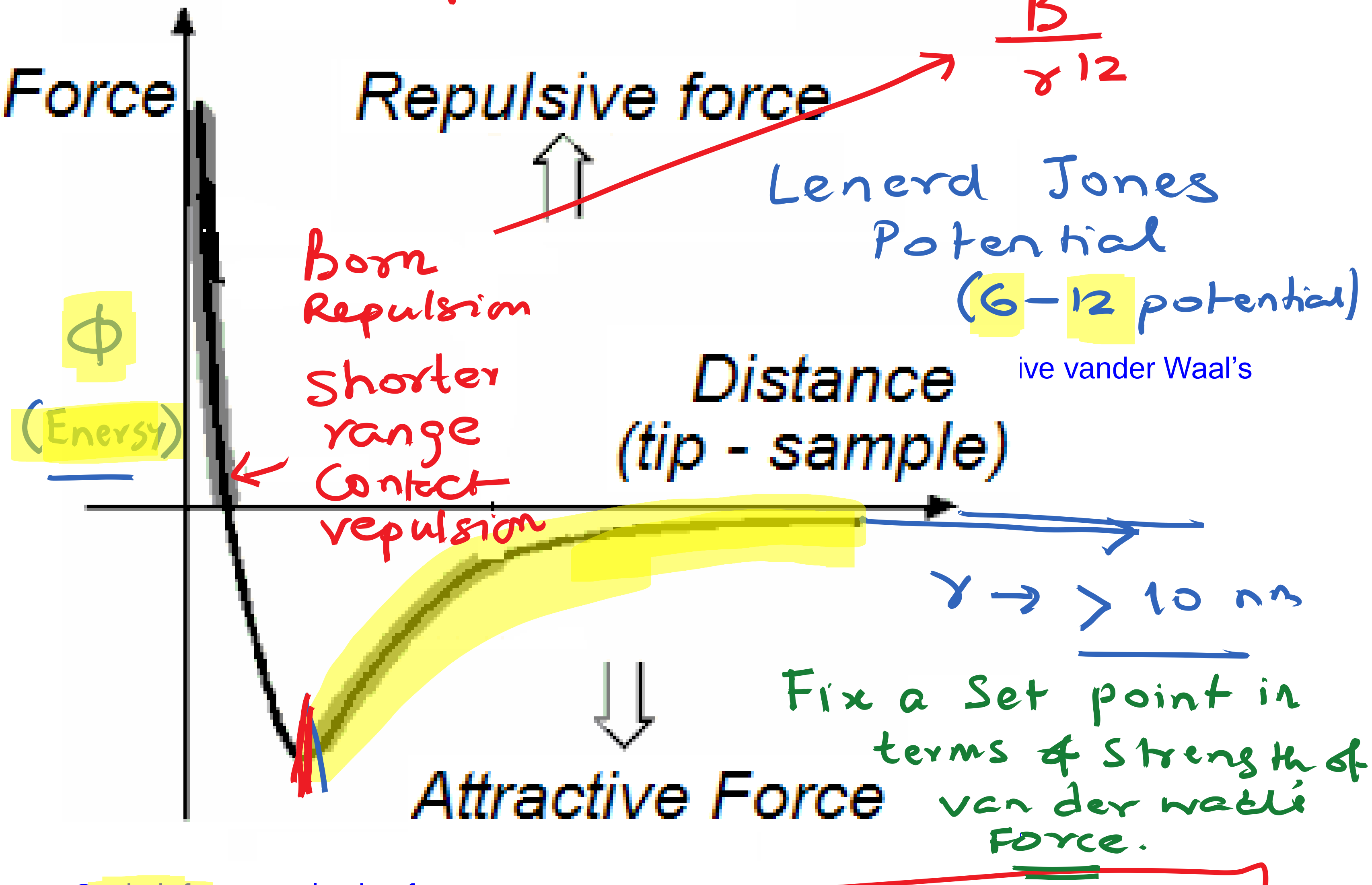
1. $(i_{T_2} - i_{T_1})$ ← Which is very small.
2. Vertical movement of the Tip

How to extend for non metallic surfaces?

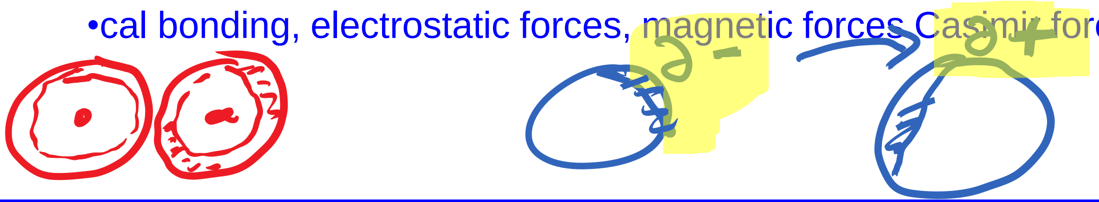
Replace Tunneling? Current

Origin of intermole





- For a charge neutral sample scanned in ai ... ive vander Waal's interaction.
- Between two molecules (or atoms), the na
- The signature of non retarded van der Wa
- Between two surfaces, the scaling or deca
- The signature of the interaction extends to
- This force is in the range of inter-atomic fo
- There can be host of other type of interacti
- cal bonding, electrostatic forces, magnetic forces Casimir forces, solvation forces etc.

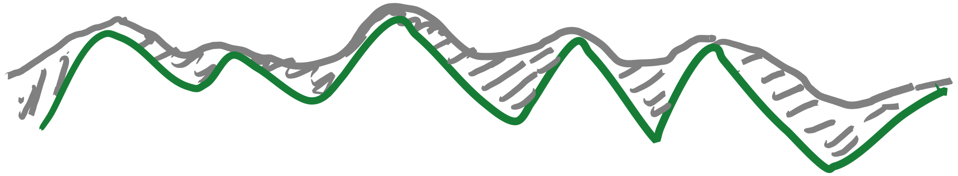


$O(r) \approx 10$ nm

$$\boxed{\phi = \frac{B}{r^{12}} - \frac{A}{r^{6}}}$$

Coating will be conformal

Practical Condition



1) Tunneling Current is going to be replaced by van der waal's forces.

Any Problem: → How to measure the variation in the strength of vdw force.

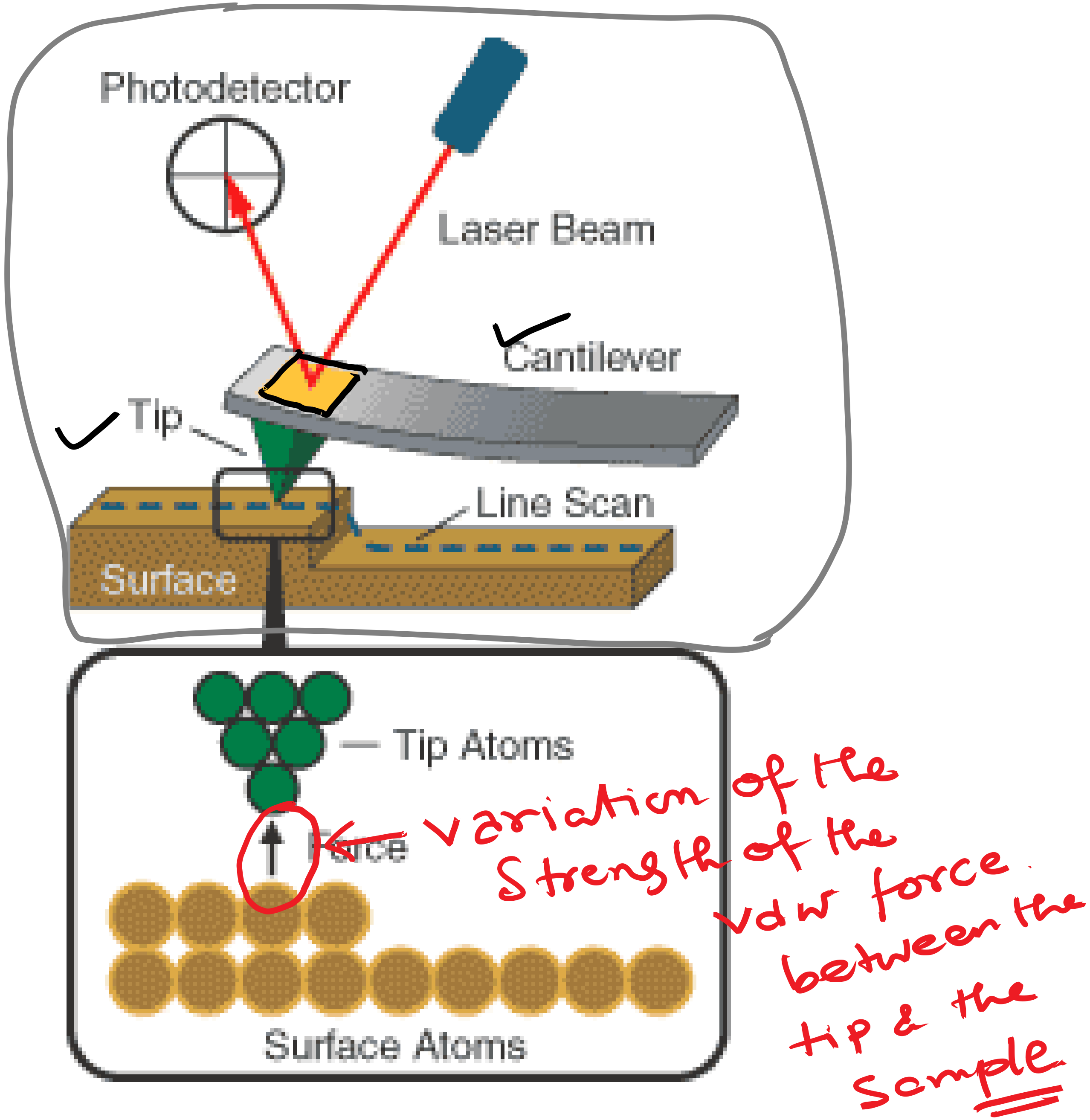
What is an AFM

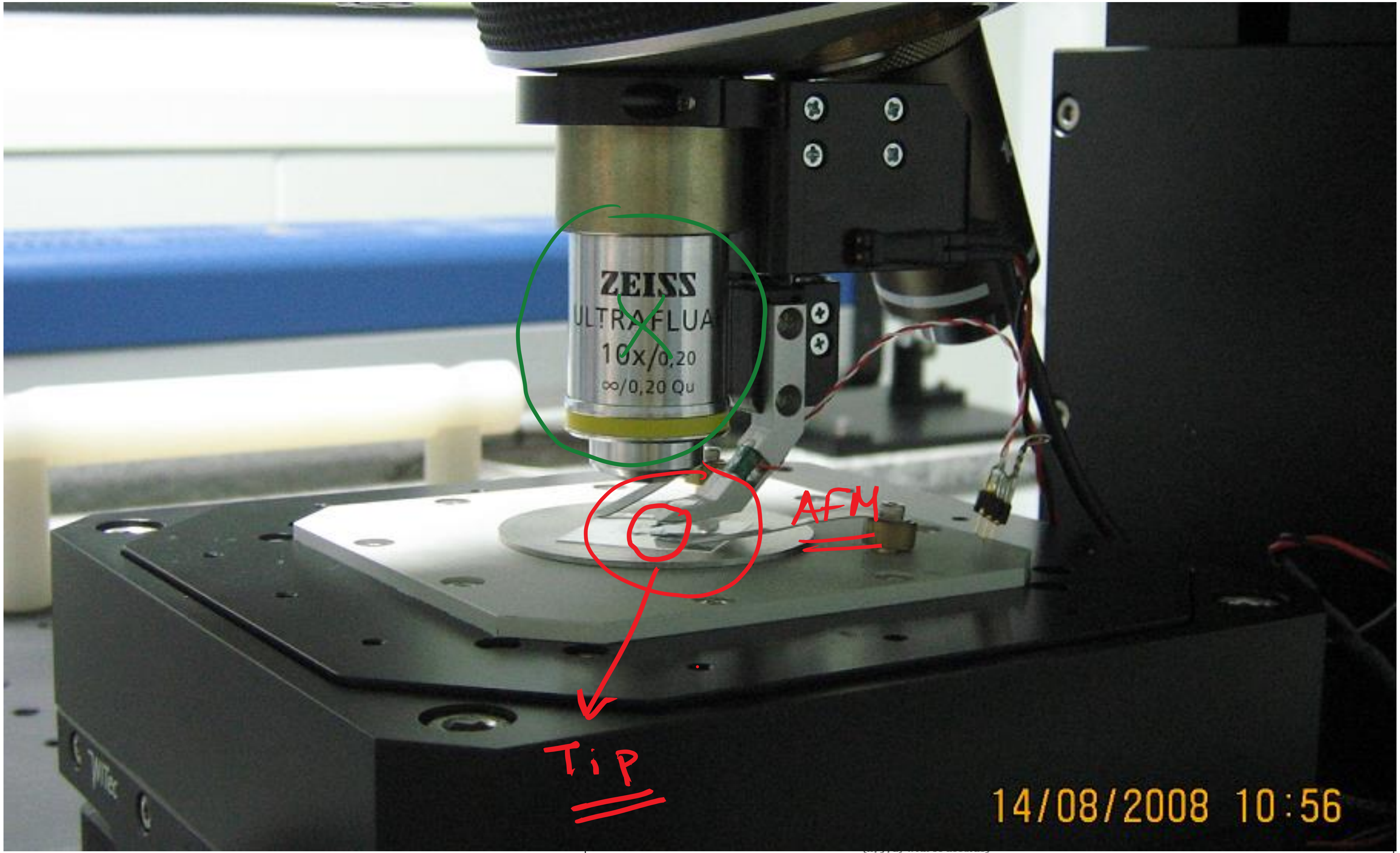
• In an Atomic Force Microscope the imaging (or more accurately, the information about the topography) of a surface is done based on the modulation of interaction forces between two atoms (or molecules).

• 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.



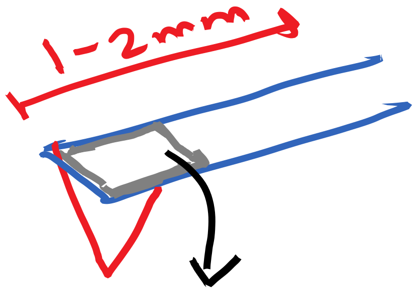

ZEISS
ULTRAFLUAR
10x/0,20
∞/0,20 Qu
AFM
Tip
14/08/2008 10:56

- **Probes or Tips** and Cantilever for mounting the tips
- Photo detector (with laser Source)
- Stepper motors for coarse movement
- Sample stage and Ruster Scanning
- Data Rendering

Alignment (Operational Aspect)

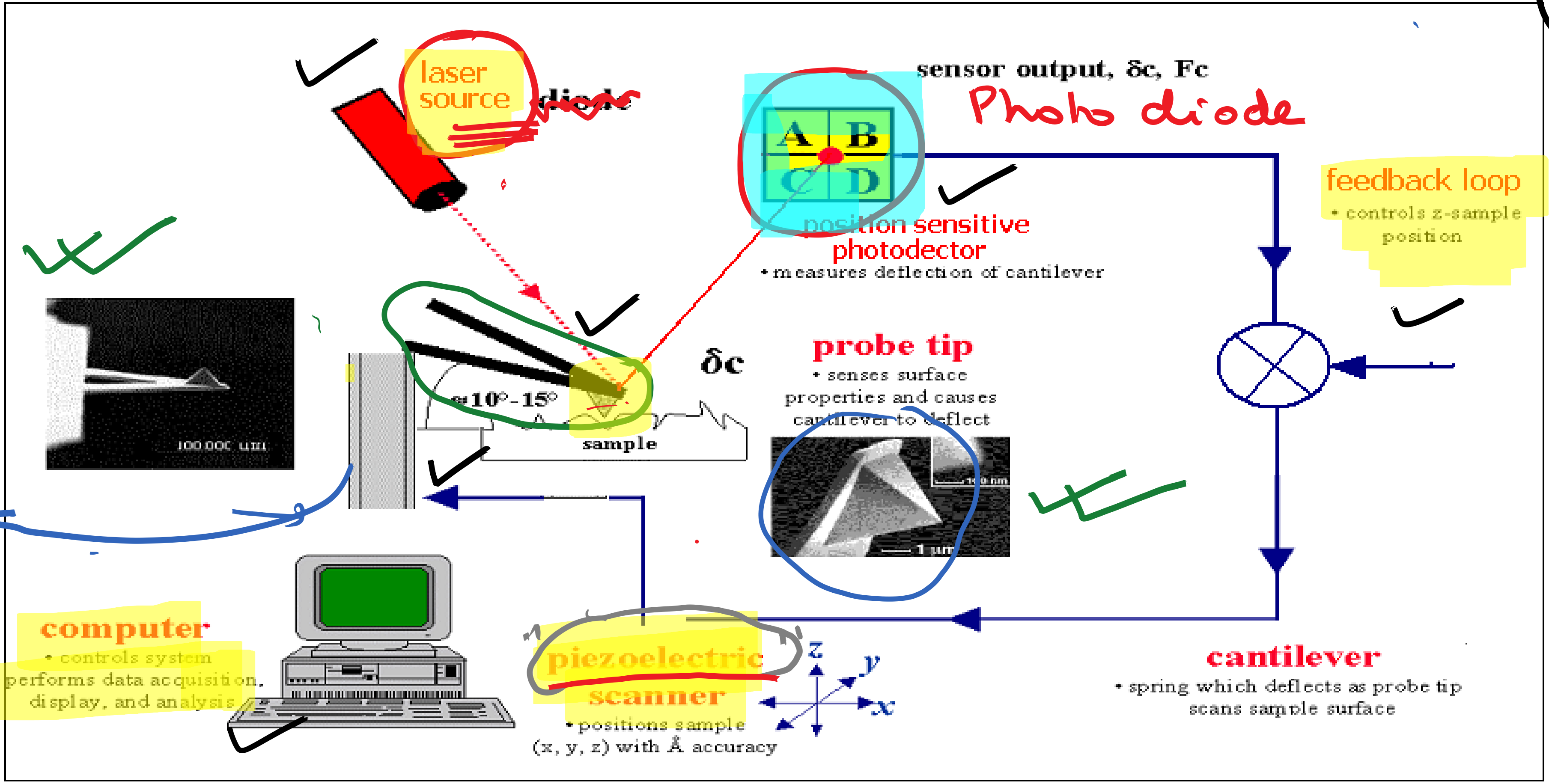
Feed back control module

Scanning Modes (Opt. Aspect)



Mirror

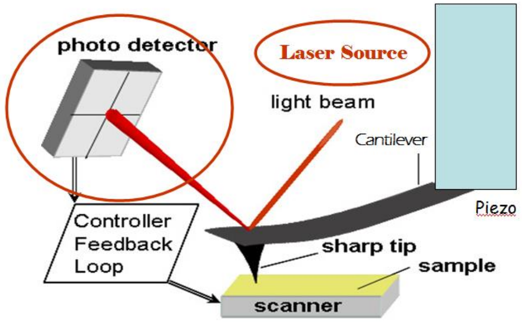
$r \simeq 10\text{-}12$ nm



You need electric signal to change it's size.

AFM Probes or Tips

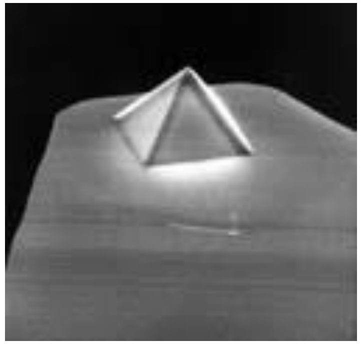
- ***Tips are some sort of a sharp probe that is used to "Scan" the surface.***
- ***This is the closest approximation of a single molecule probing the surface.***
- ***Tip diameters are typically ~ 15 – 25 nm (Hundreds of molecules)***
- ***Resolution is a major function of tip Size and Geometry***

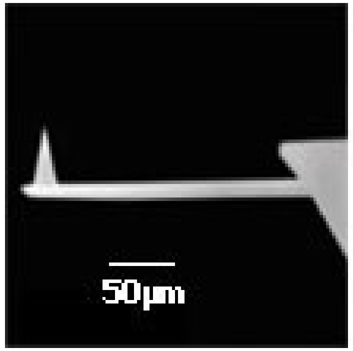


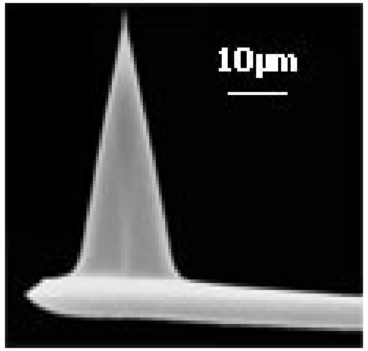
The cantilever behaves like a spring

Its stiffness is critical

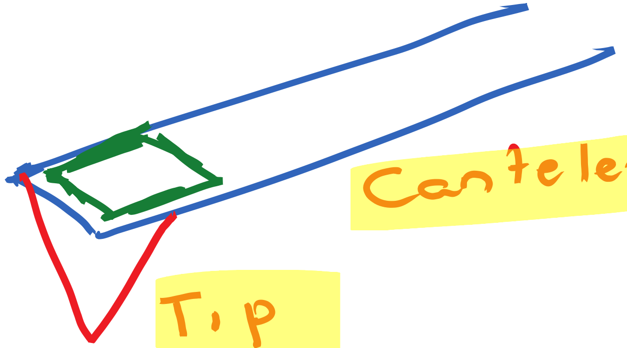
Stiffness determines the spring constant





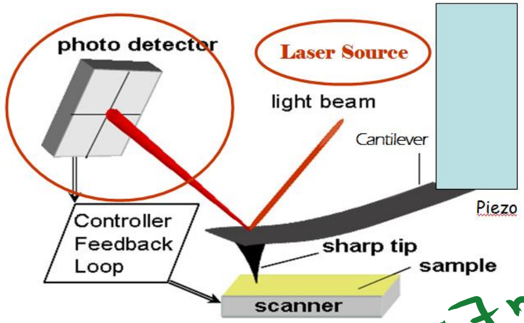


AFM Probes or Tips

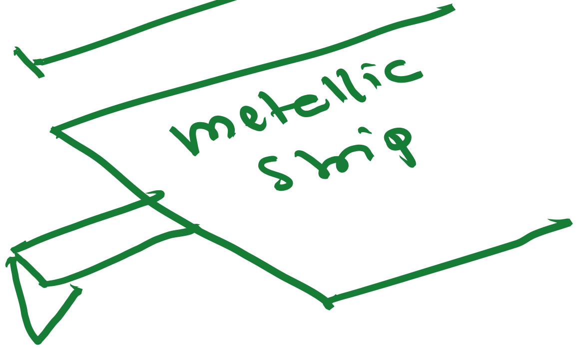


Cantelever

Tip

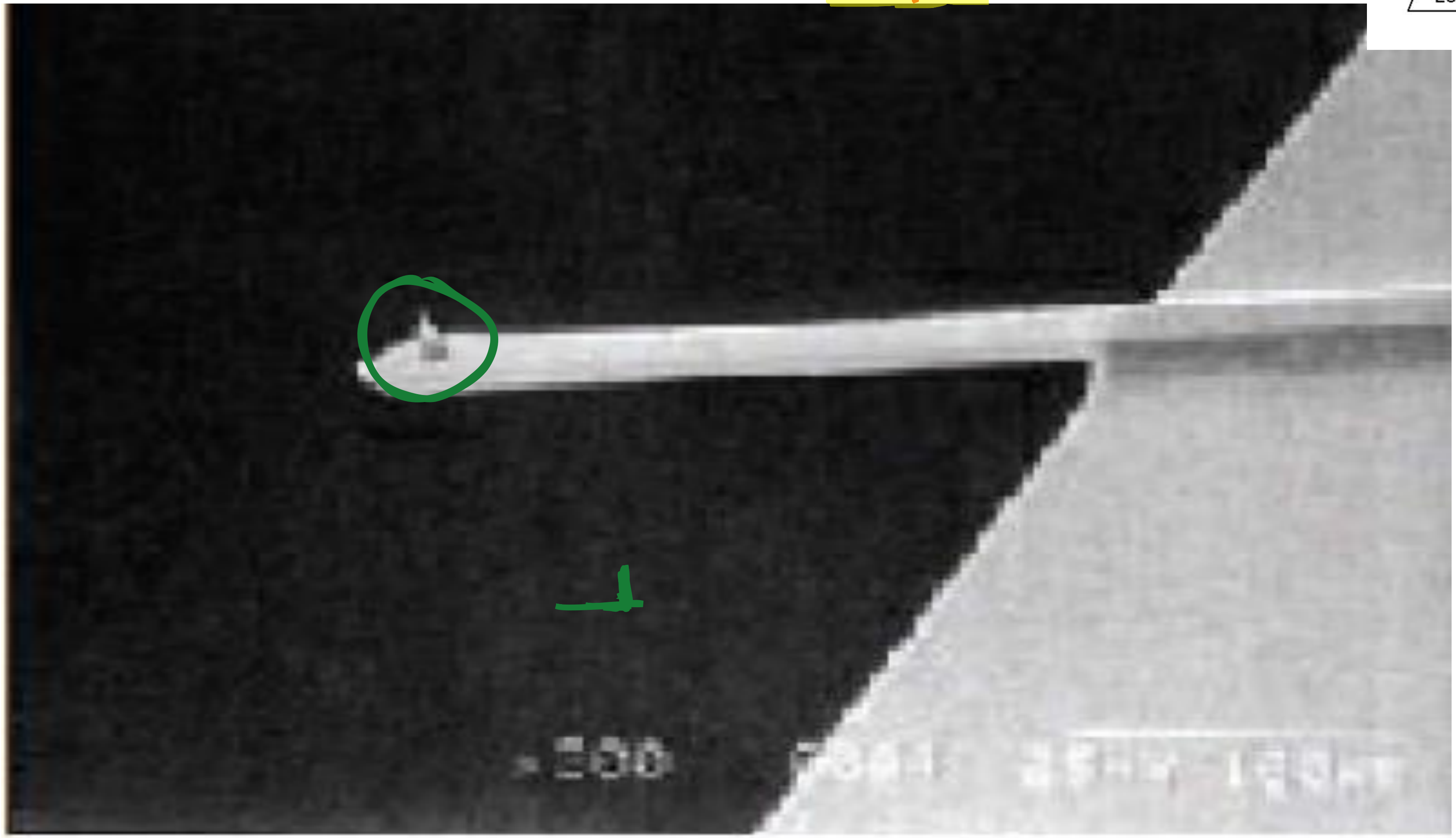


6-7m long



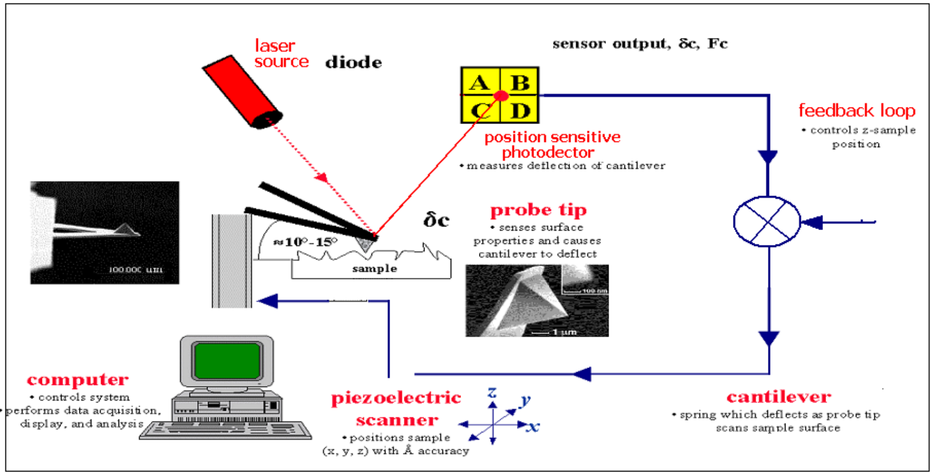
metellic strip

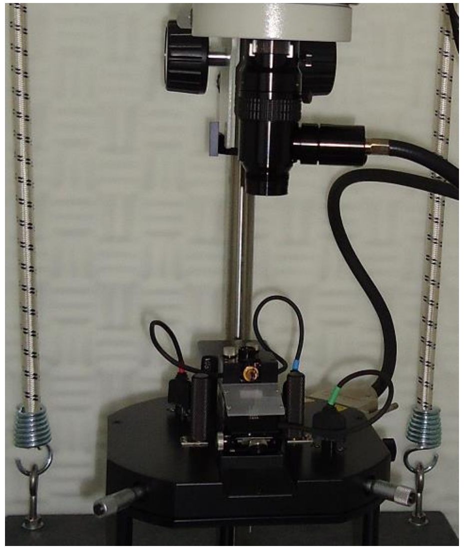
Cantilever chip

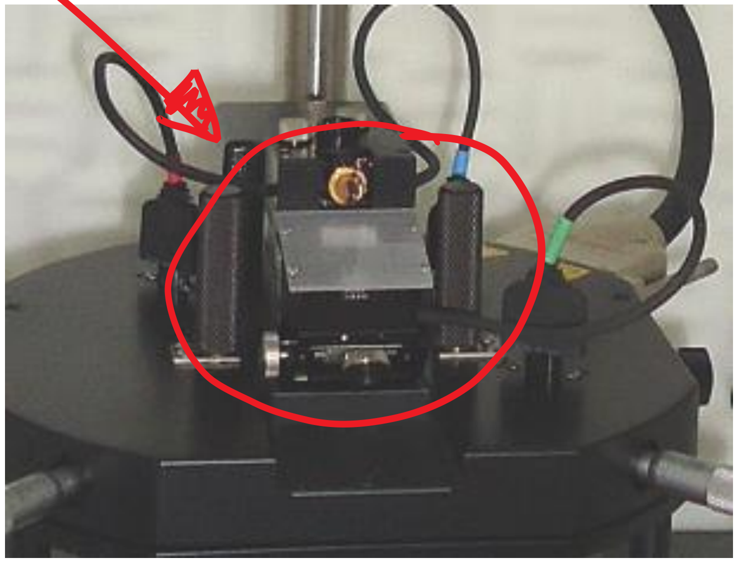


Silicon /

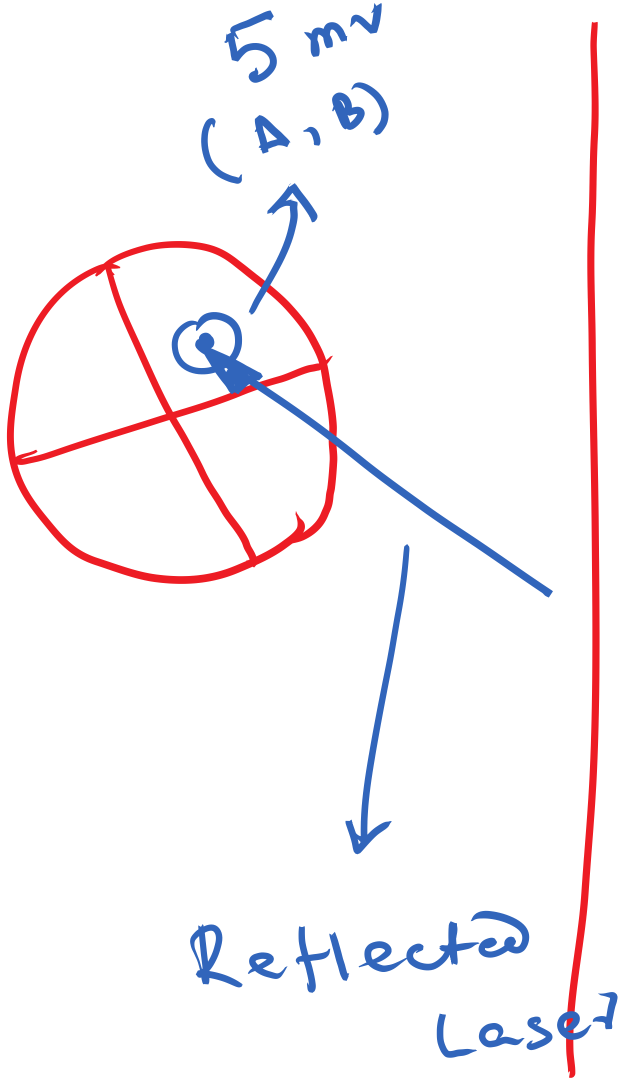
Si_3N_4


laser source
diode
sensor output, δc, Fc
A B
C D
feedback loop
• controls z-sample position
position sensitive photodetector
• measures deflection of cantilever
δc
probe tip
• senses surface properties and causes cantilever to deflect
≈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



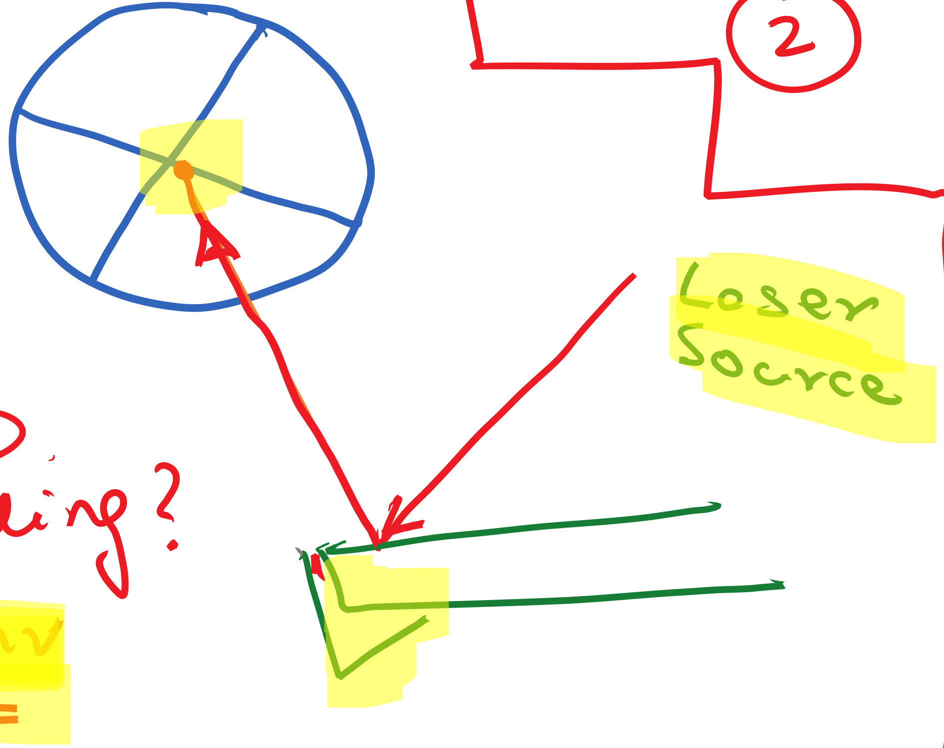


Alignment



QPD reading?

0 mv

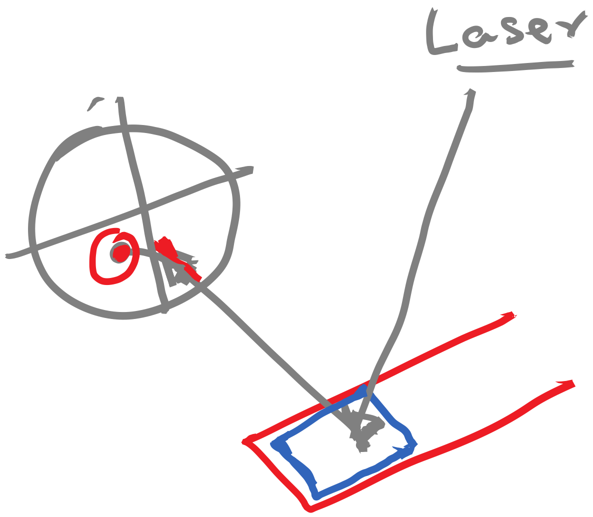


Alignment of the Cantelever

① Tip → Mounted on the Cantelever.

② Laser Source

③ 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.

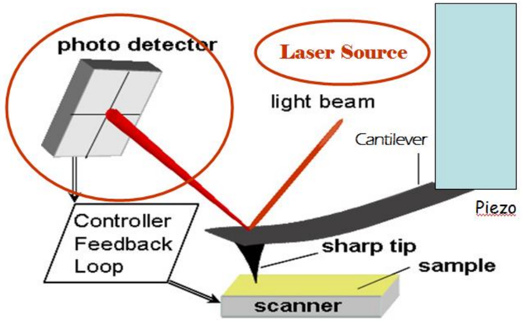
QPD (Quadrant Photo Diode)

SPD (Split Photo Diode)

PSD (Position Sensitive Photo Diode)

measure (A, B) for every point

(x, y)



C1 → Lower voltage

C2 → Higher voltage

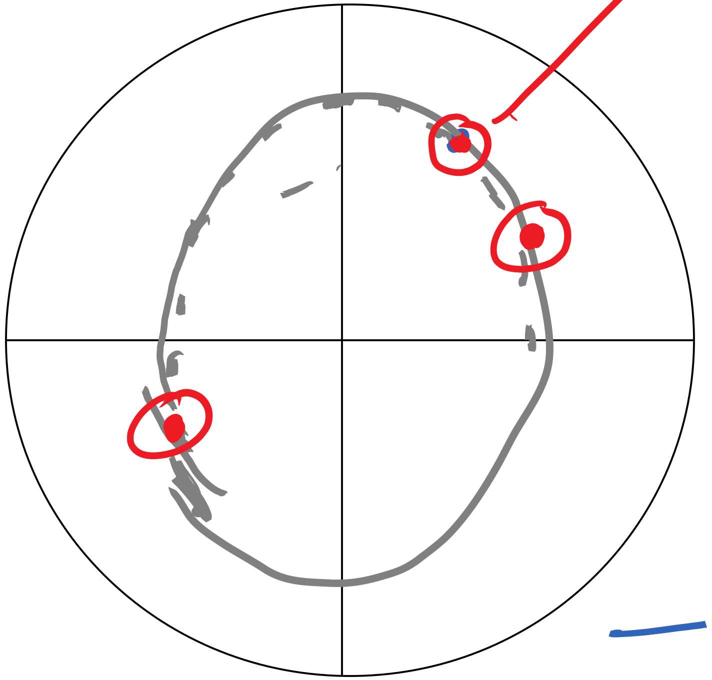


Photo Diode:

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

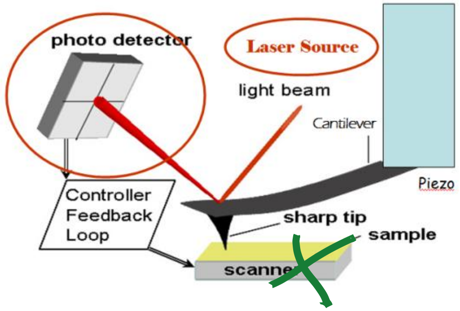
Here, the voltage generated is a function of the position at which the light falls.

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

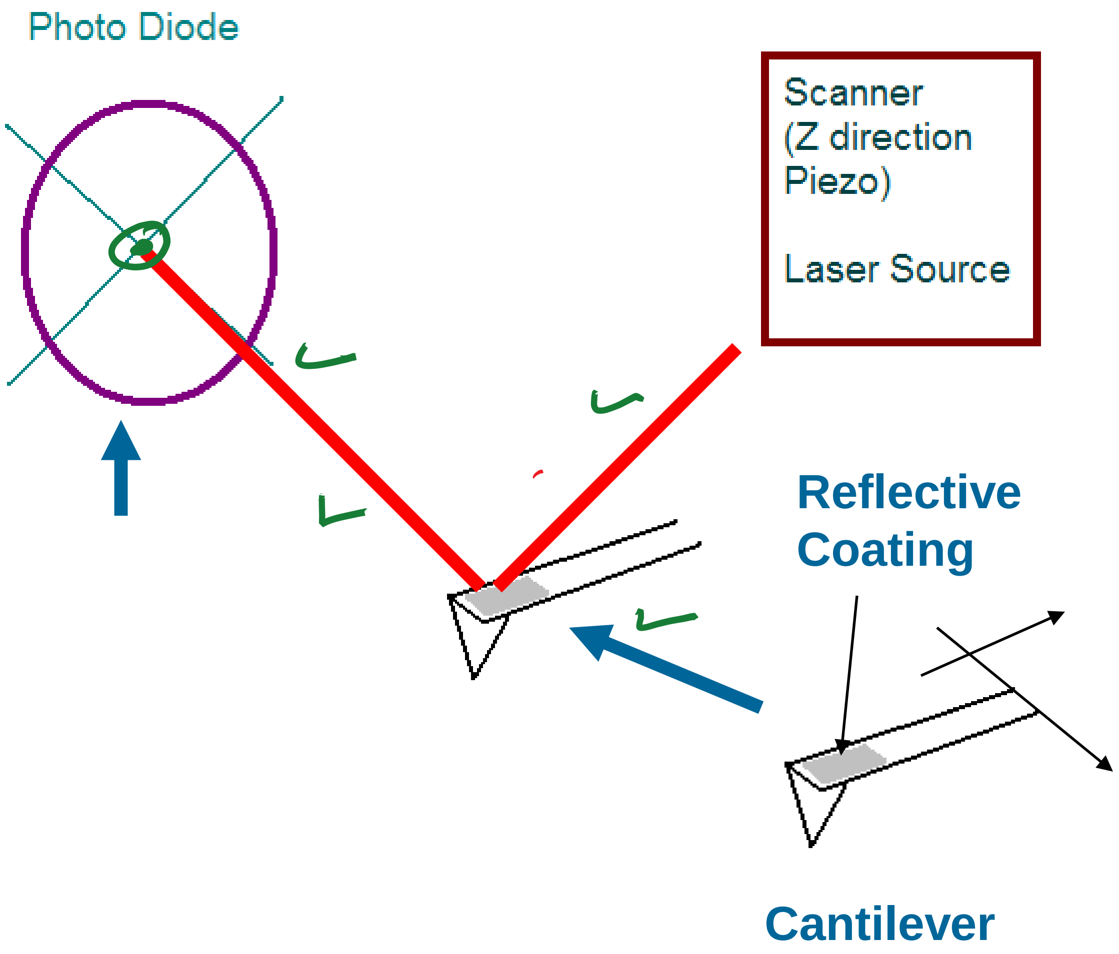
— Light falls at the center = 0 mV

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

Alignment



Reading 0 Volts!



3 Different Components

(1) Laser Source.

(2) Tip and Contelever

(3) QPD

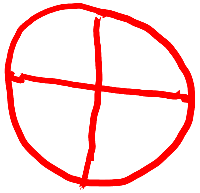
You optically Couple these 3.!!

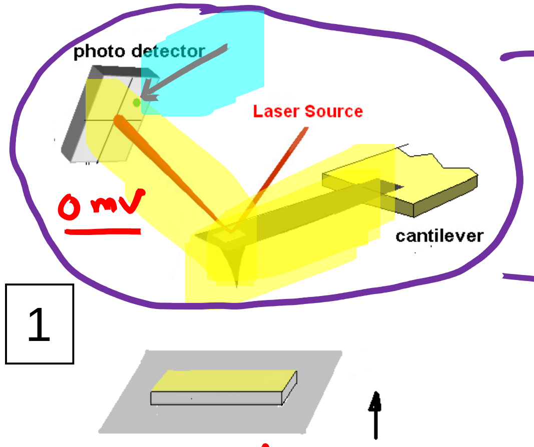
- Tip is far away (few microns to ~ mm) from sample surface.

Approach

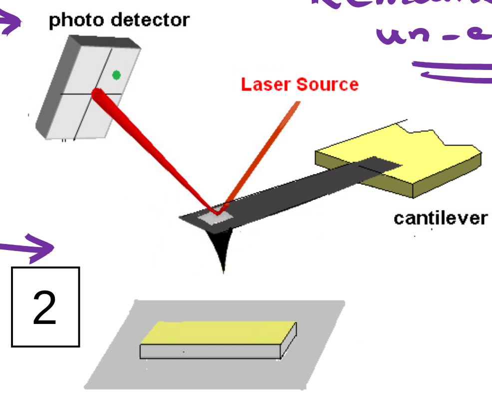
Set a Set Point

Where you want your
Stepper motor to stop



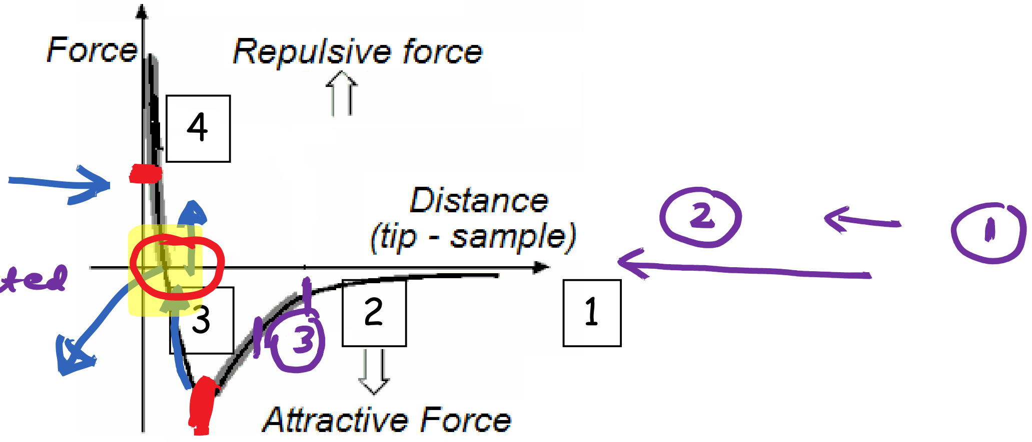


sample
(Far away from the tip)



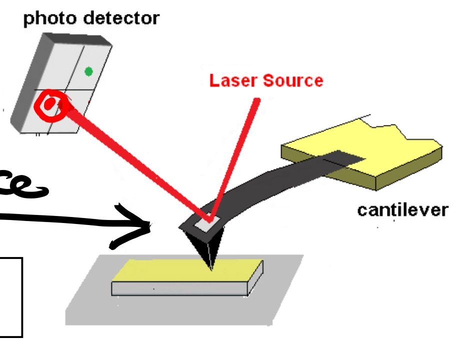
The sample moving up → still far away from the tip →

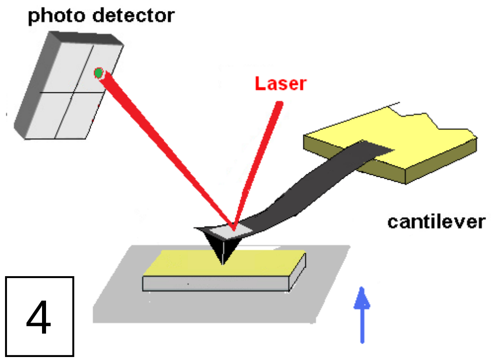
Remains un-effected

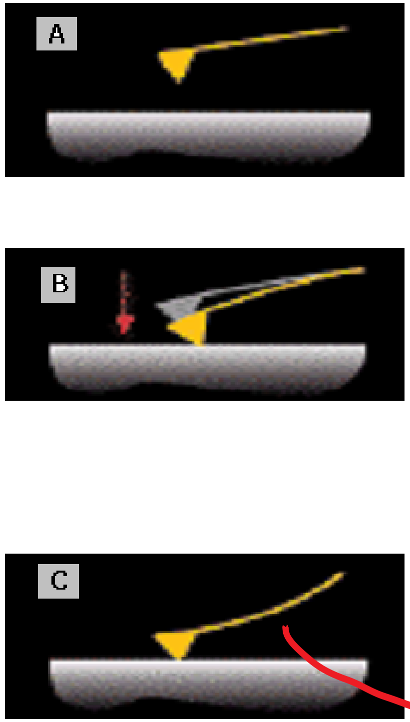


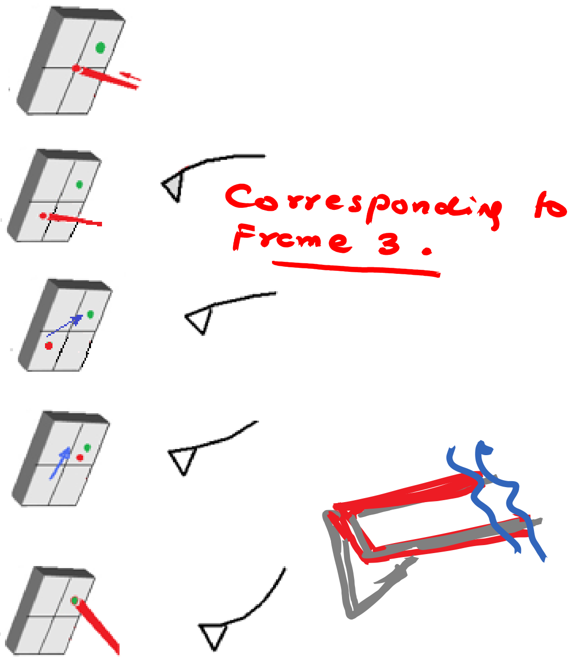
Point of Contact

The tip has started to experience attraction due to the presence of the surface!!





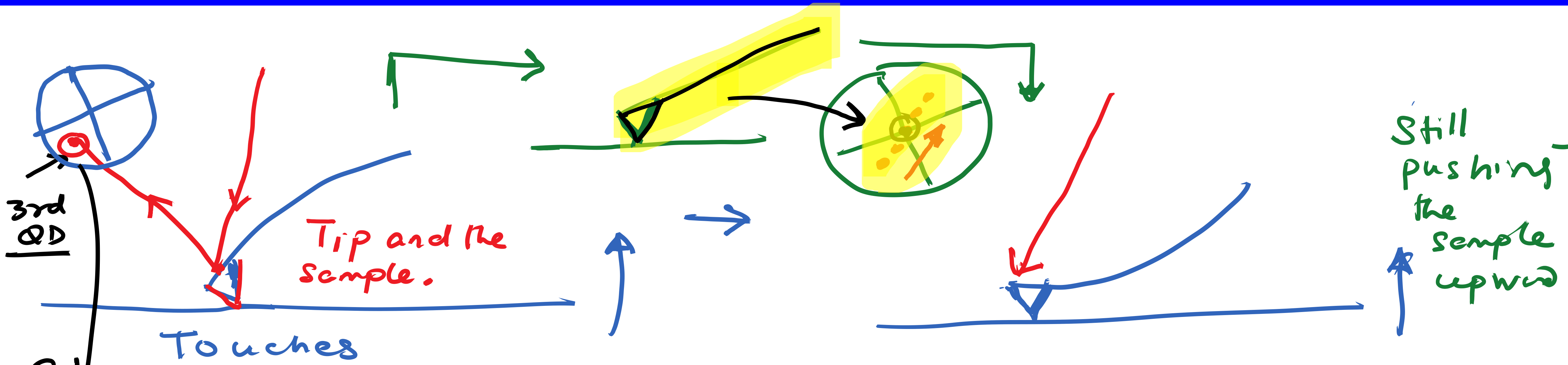


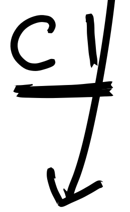


Corresponding to Frame 3.

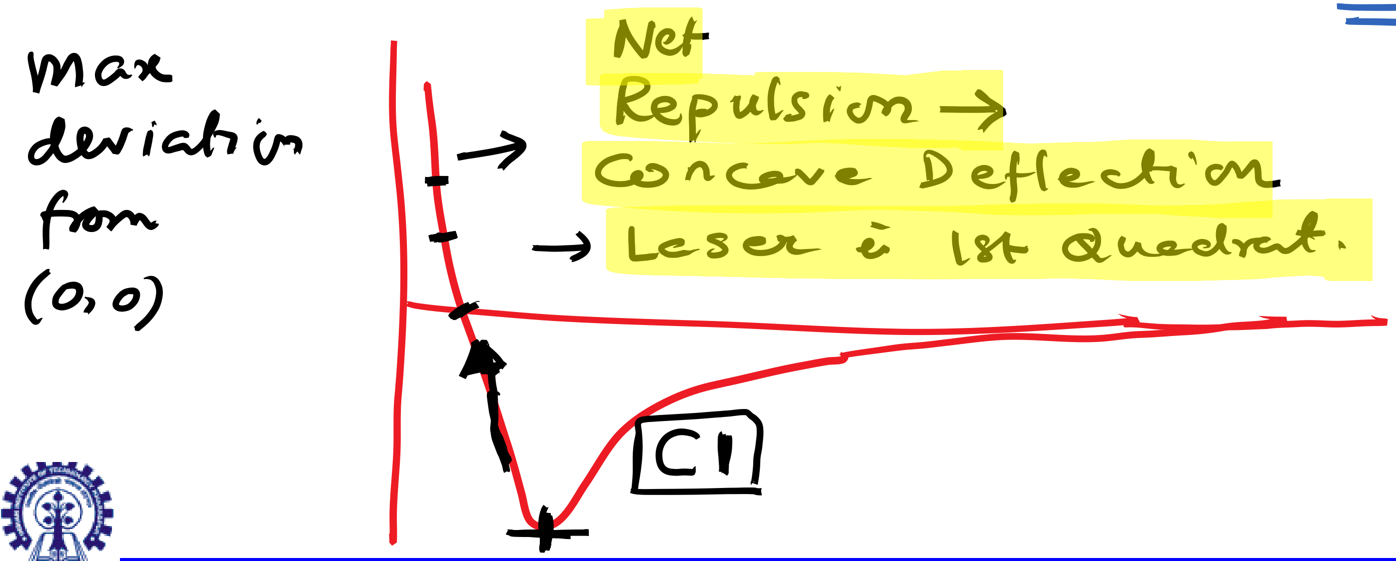
Once approach is complete the Piezo elements get activated

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





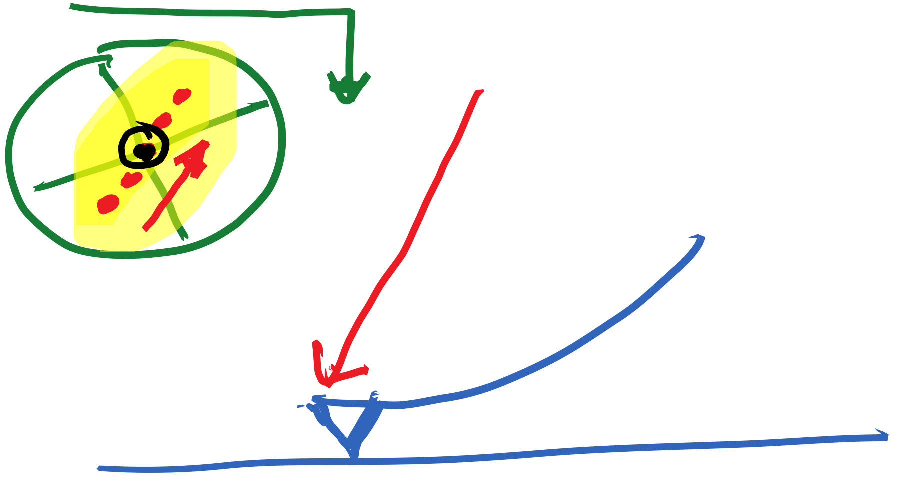
Nature of the deflection changes from Convex to Concave!



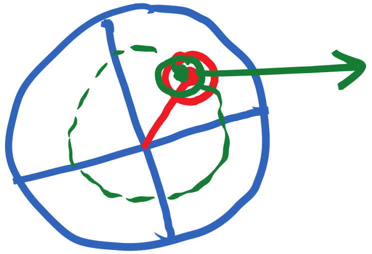
Tip-Sample Interaction:
There are three simultaneous
Signatures:-

(1) Deflection of the Cantilever
(2) Position of the reflected Laser
(3) On the Force Curve

When does the upward movement of the Sample Stop?



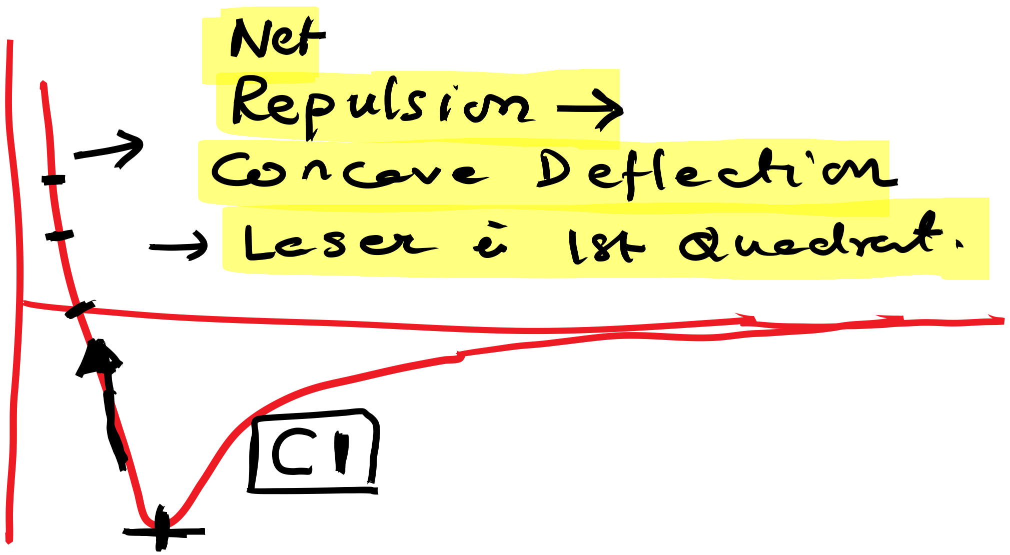
Still pushing the sample upward



Set point (3 mV)

↳ 3 mV or 5 mV → Operator-prerogative

softer sample → Lower set point



Tip-Sample Interaction:

There are three simultaneous signatures:-

(1) Deflection of the Cantilever
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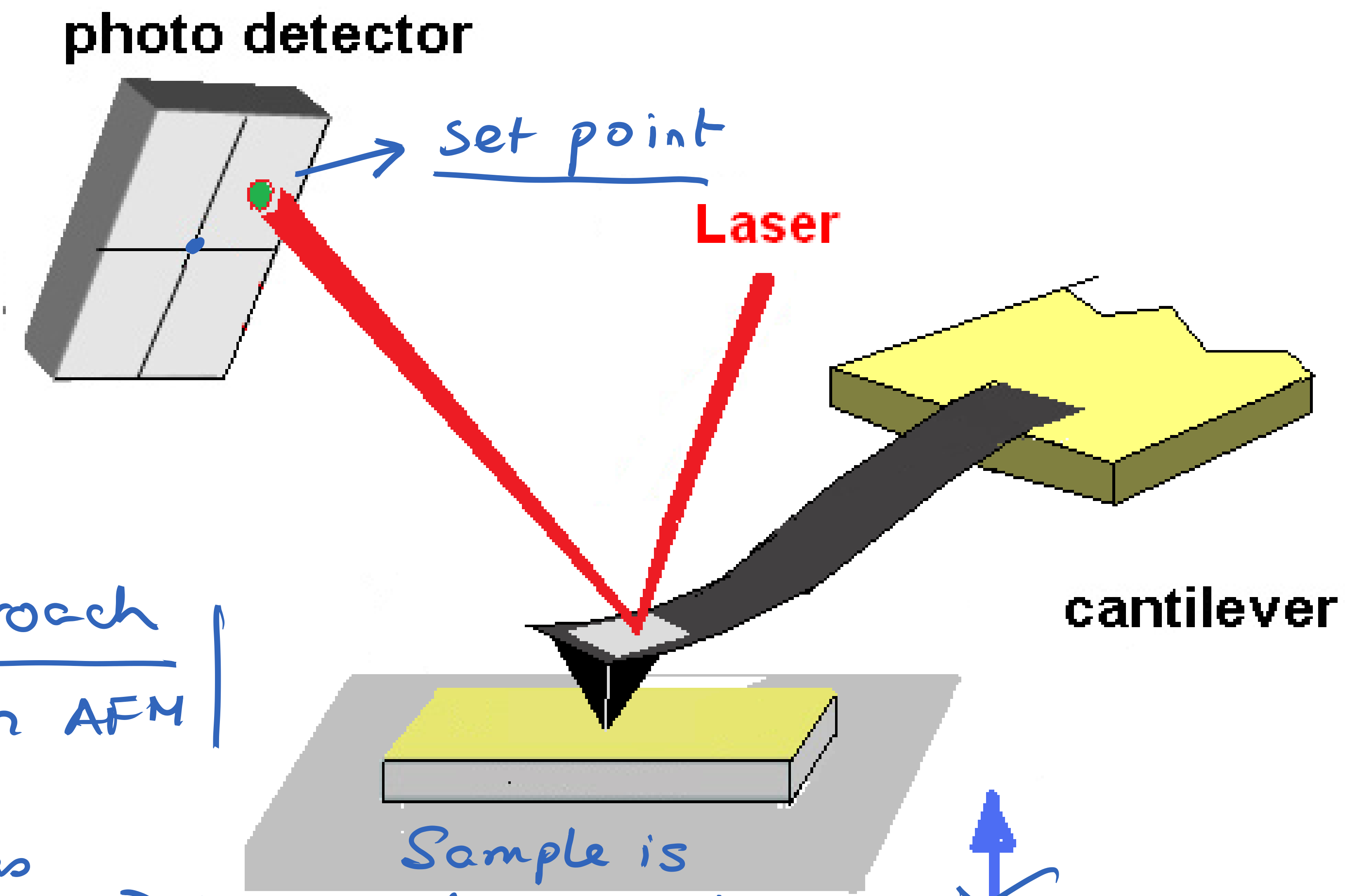


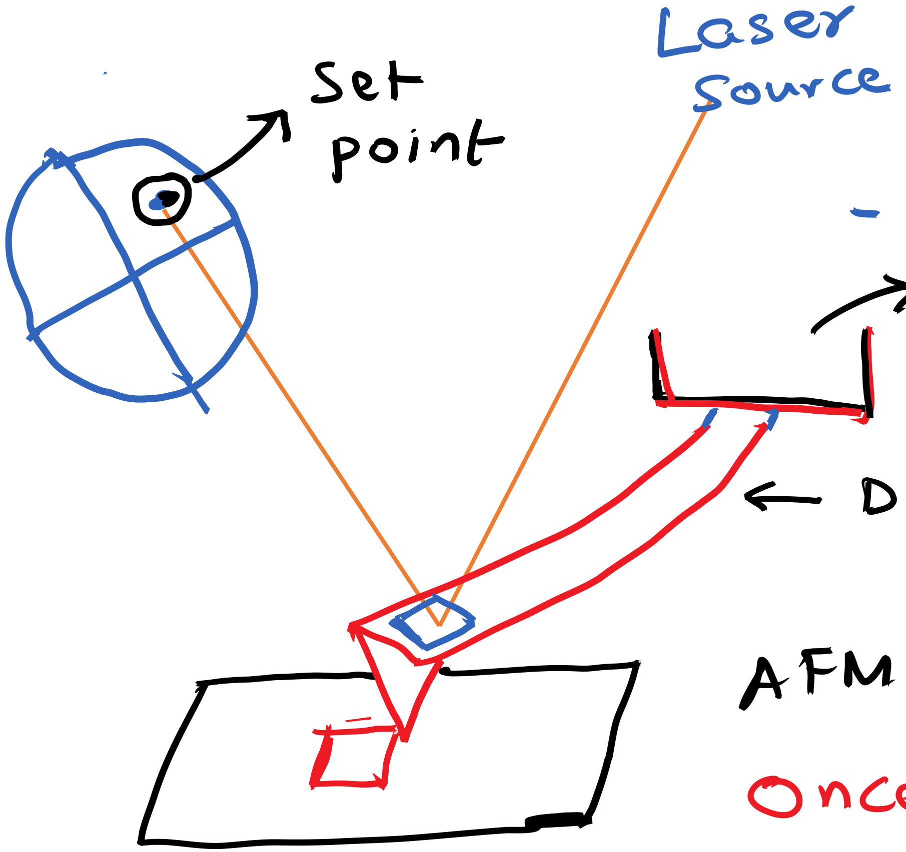
Approach of an AFM

Tip has approached the Sample

Sample is also coupled.

movement has stopped

16th Feb 2022



Piezo Electric Scanner.
(Controls the movement in X, Y and Z directions)

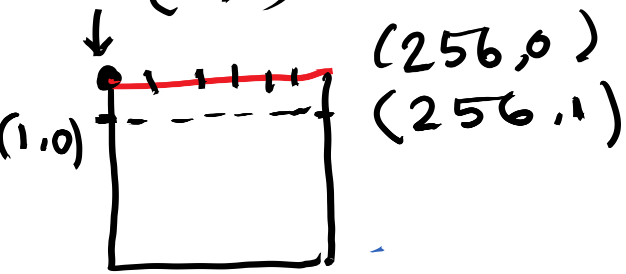
← Deflection

AFM has approached

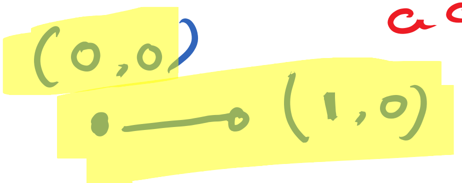
Once the Tip has approached, the Piezo Now gets activated.

The vertical movement of the sample towards the tip was by a stepper motor.

Point of Approach (0,0)

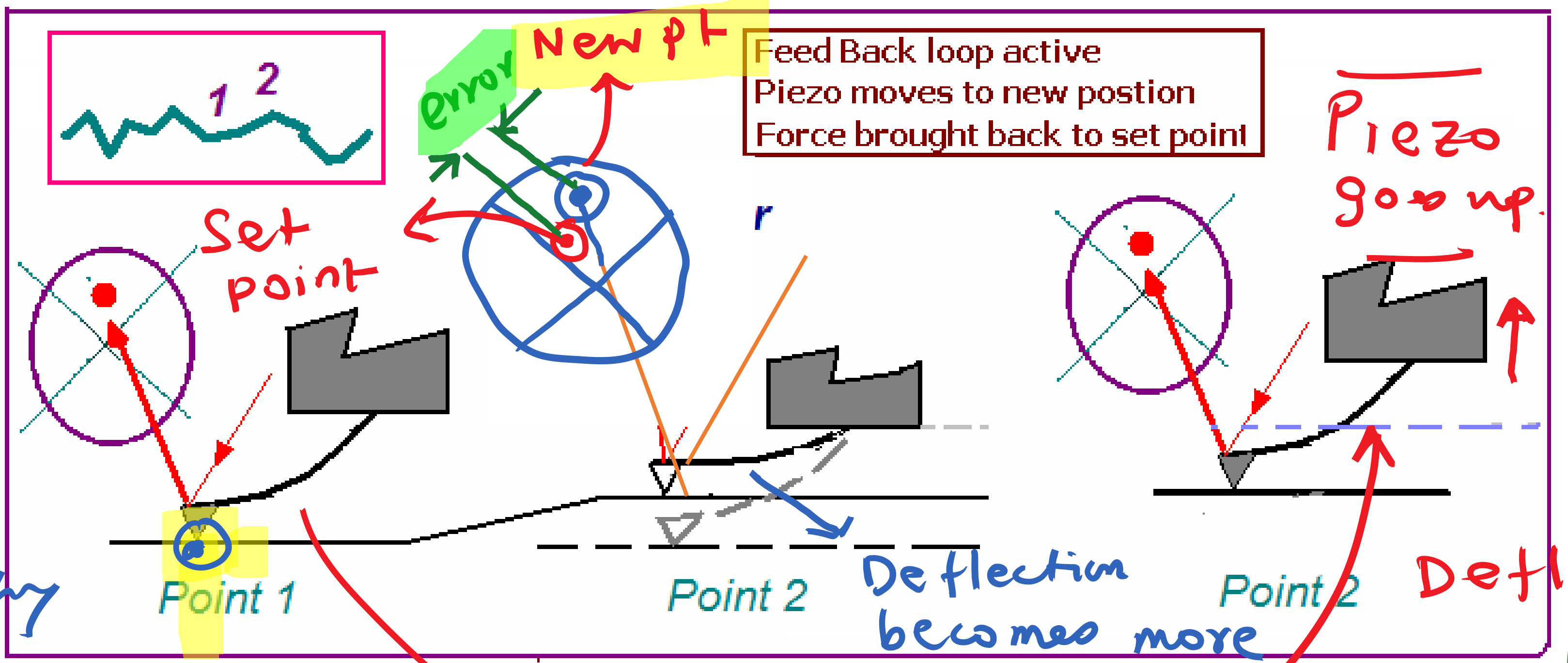


Rastering.



Beam Bounce Technique

=> Tracking the Topography



When sample has an elevation (less separation distance)

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

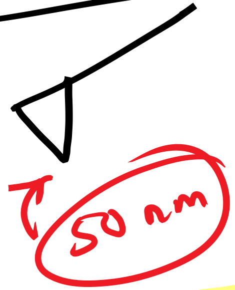
- Sample has a depression (separation distance more)

✓ Less force exerted on the cantilever. Piezo comes down.

- Feedback loop works on the error in the Force signal

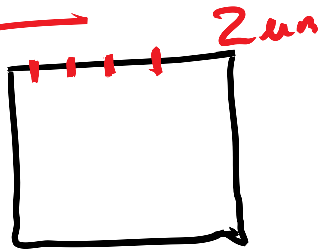
  - The deflection is converted into change in voltage and the controller, register in the feedback loop controls the force or distance and records the topography.

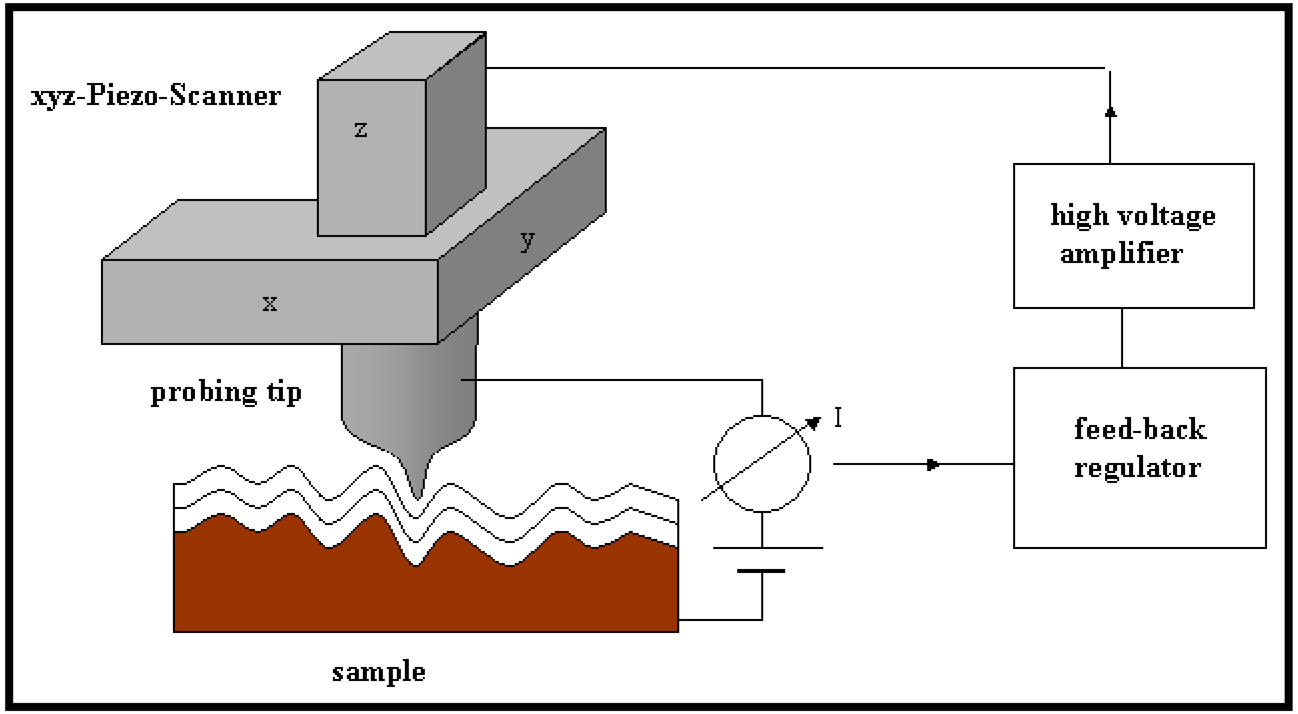
Typical R Tips ~ 10 nm



Lateral resolution.

CONTACT MODE AFM

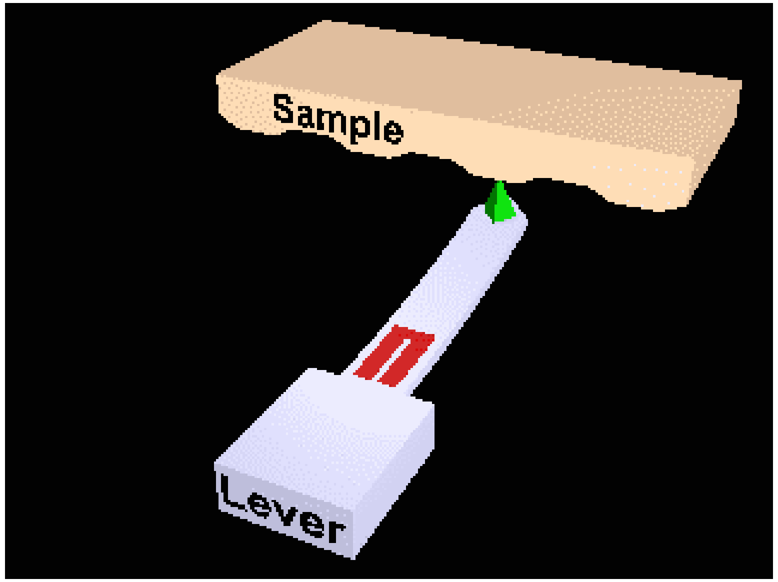


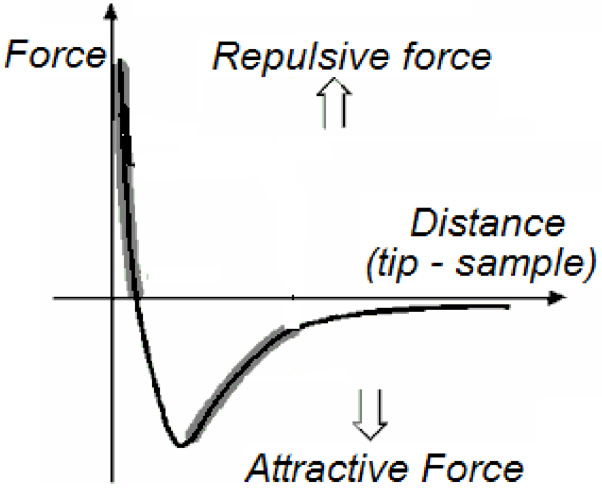


The ability to precisely position the probe of an AFM is made possible by an XYZ Piezo-Scanner which coupled to a feedback regulator keeps track of the tunneling current and precisely positions the tip accordingly.

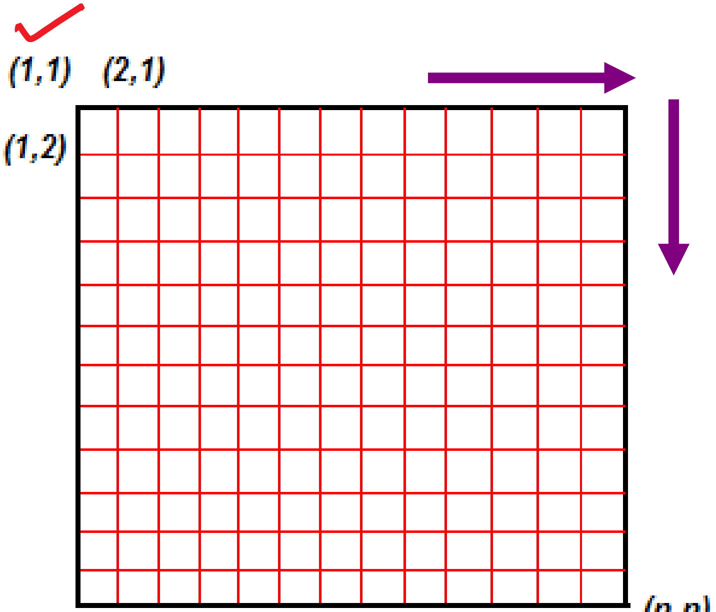
Contact Mode

- 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



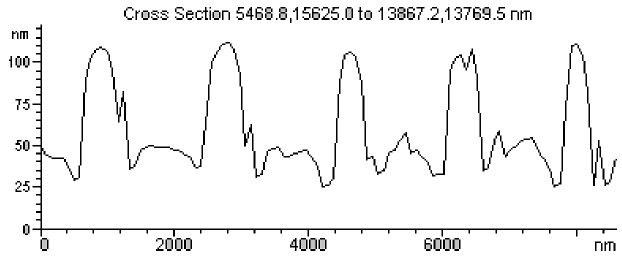
Nested do Loop

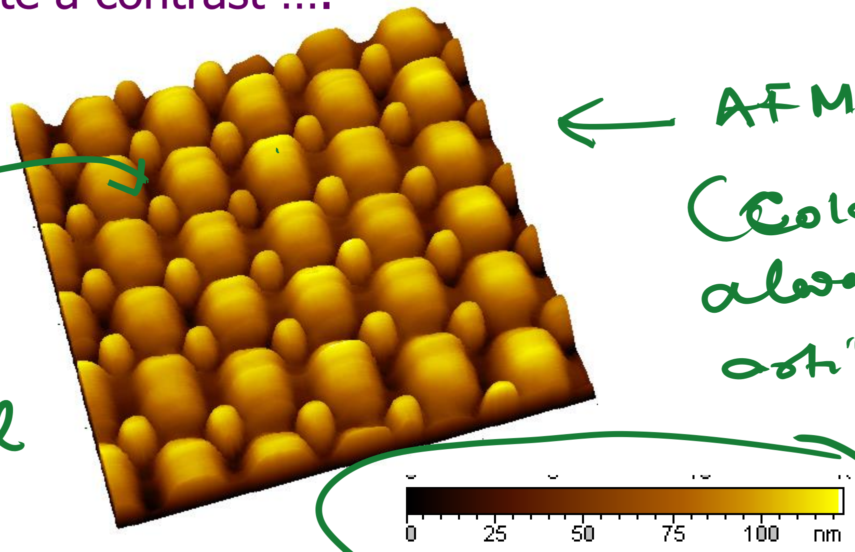
x	y	z
1	1	0.213
2	1	- 0.947
3	1	- 0.776
4	1	0.312
….	….	….
1	2	- 0.398
…	….	…..
n	n	0.512

min, max

Normalise between 0 and 1.

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





AFM image (Color is always artificial)

AFM image is incomplete without the vertical scale bar.

In AFM images, colors are always artificial

Soft Surfaces

Tapping modes: frequency

Intermittent Contact mode / or Tapping mode

Resonant oscillation frequency:

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

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

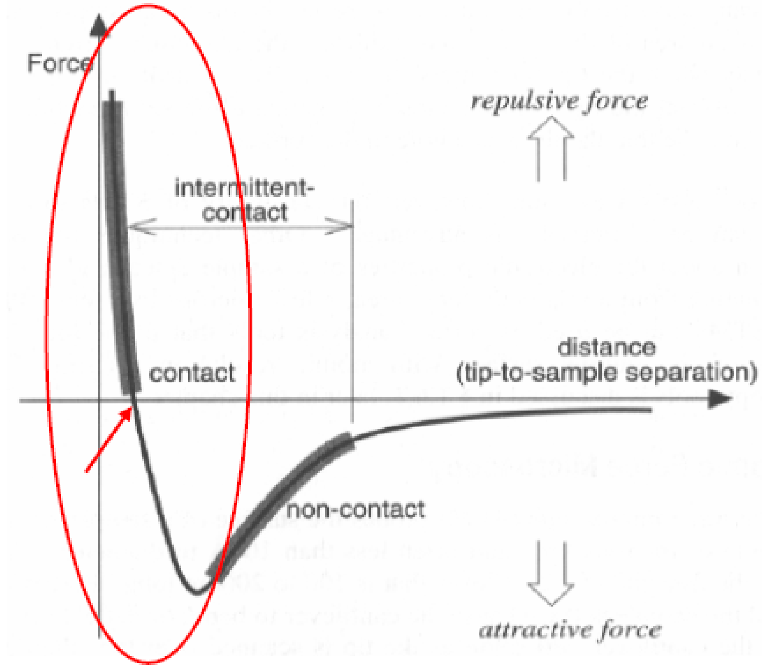
$$\boxed{k_{eff} = k - \Delta F}$$

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

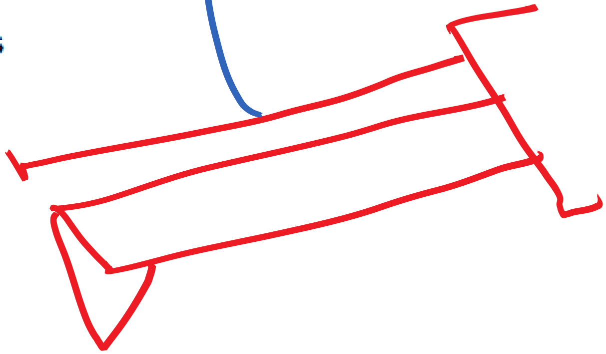
movement

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

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



Additional Drive to oscillate
Behaves like a spring.



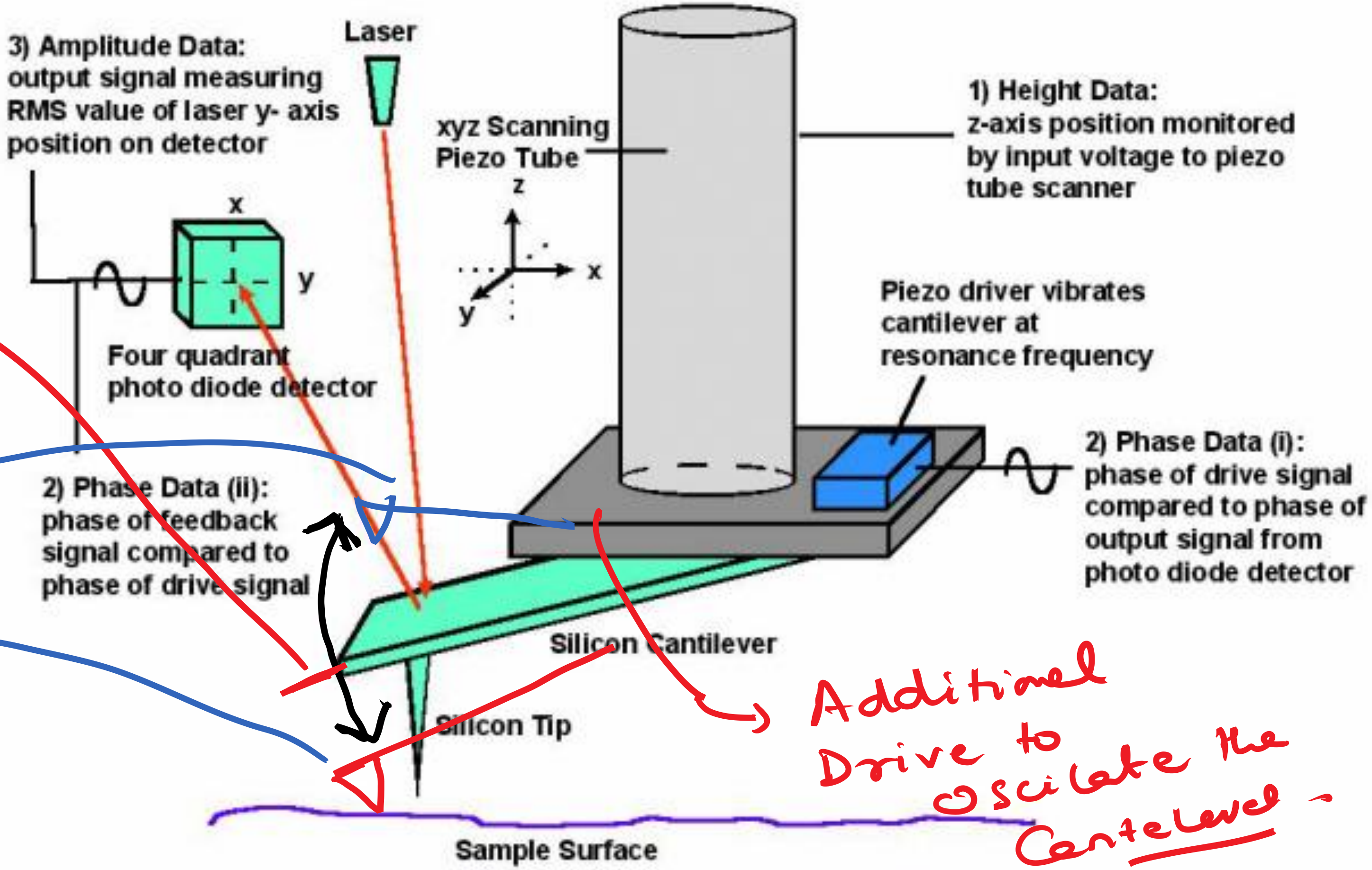
Tip is mounted on a Cantelever.

After you have aligned → Cantilever Starts to oscillate.

Tapping mode

In air

Three Types of Data Collected in Tapping Mode



Physical Oscilation of the centelever leads to oscilation of the Laser. spot on the QPD.

Tapping Mode or Intermittent Contact Mode

Oscillating tip now approaches the Surface

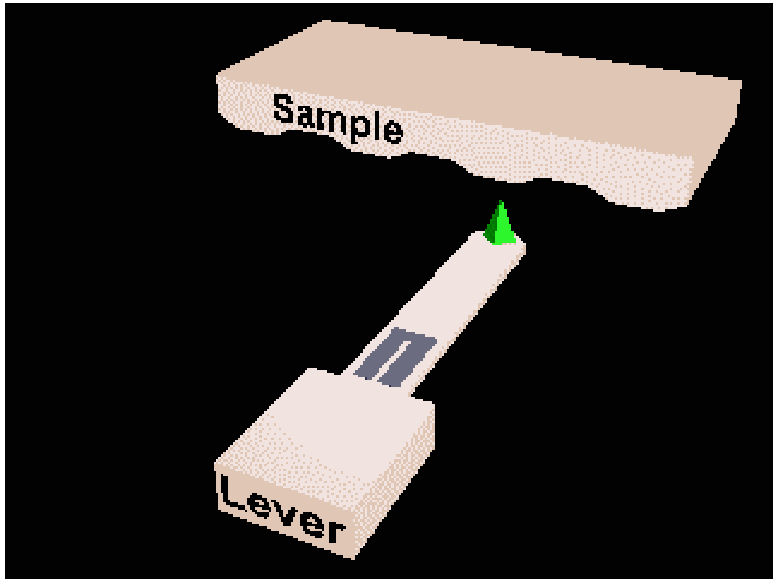
- The set point is in terms of the Amplitude of the cantilever.
- The scanner moves along the surface, at a fixed amplitude
- Scanning is done in the attractive interaction regime.
- Along with the surface profile (topography) the Amplitude of the cantilever will change
- Feed back loop activated due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

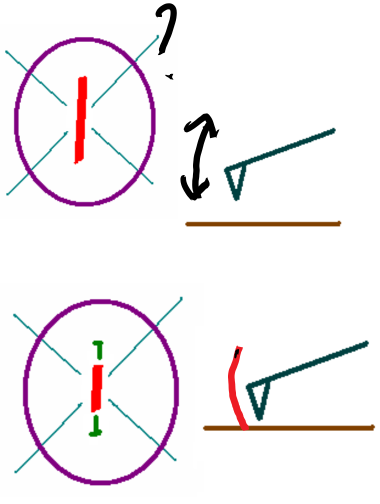
Set Point

90% of Free oscillation

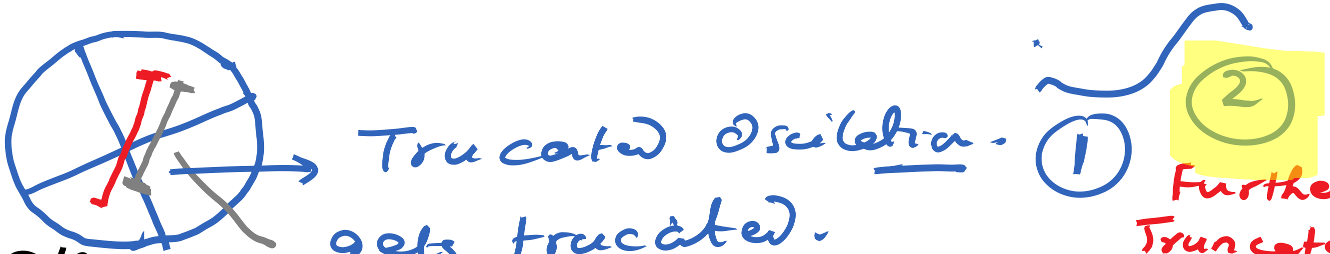
Hits the surface

As the oscillating tip comes closer to the sample - it hits the surface.





Consequence on the QPD



Truncated Oscillation.

gets truncated.

1

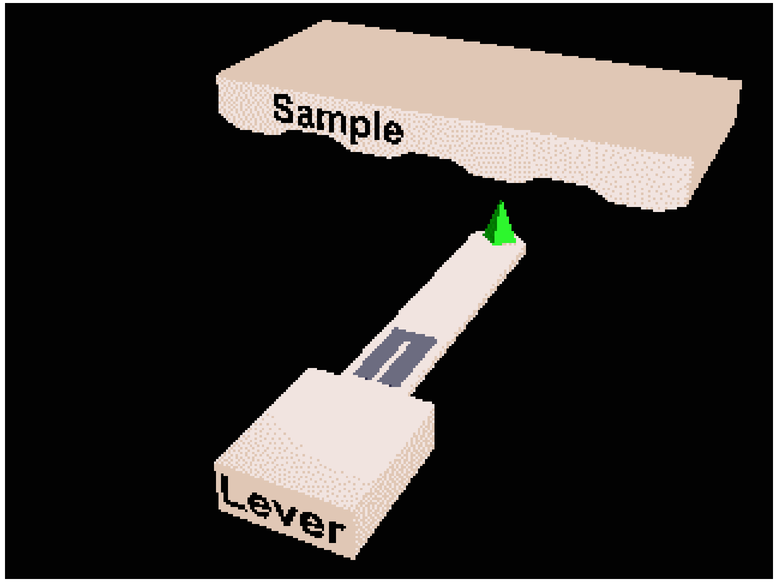
2

Further Truncated

As physical oscillation of the tip gets truncated, the movement of the Laser spot also

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.

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.

A Good Cantilever .. .. ..

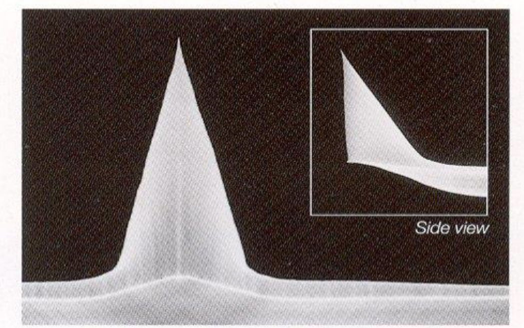
One of the most important factors influencing the resolution which may be achieved with an AFM is the sharpness of the scanning tip.

- 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.

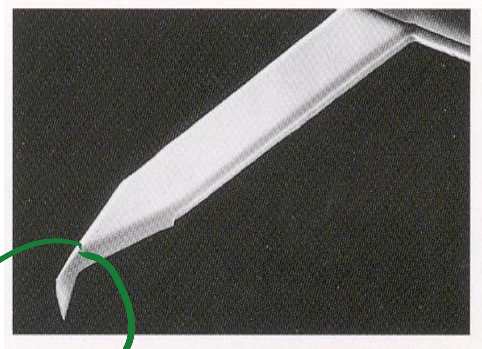
    a) Contact mode: low force constant

    b) Non contact mode: high force constant.

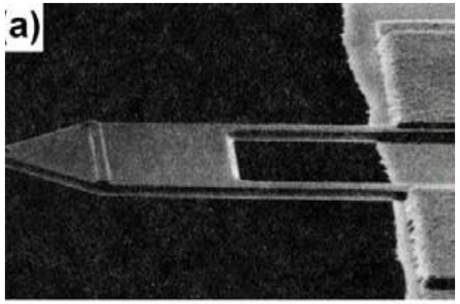
AFM Probes: Some Typical Examples



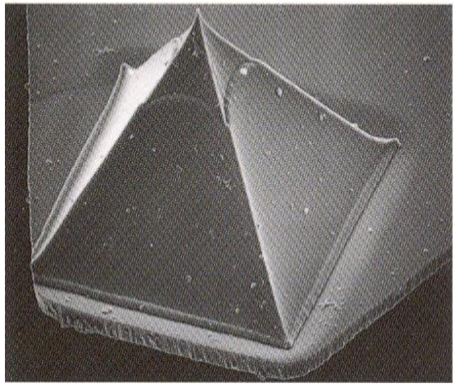
Tip Shape: Tetrahedral
Height: 10mm
Radius: <10nm
Material: Si



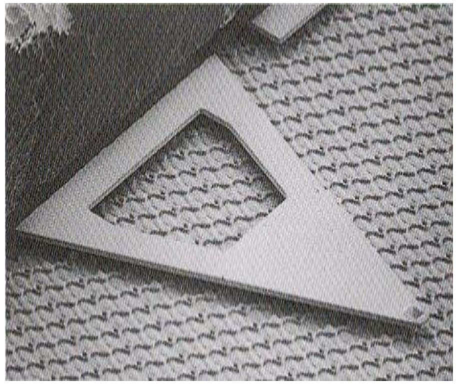
Cantilever Shape: Rectangular
Length:120mm
Thickness: 2.8mm
k : 20N/m; f = 300kHz; Material: Si



Piezo resistive cantilever (amplitude can be adjusted)



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



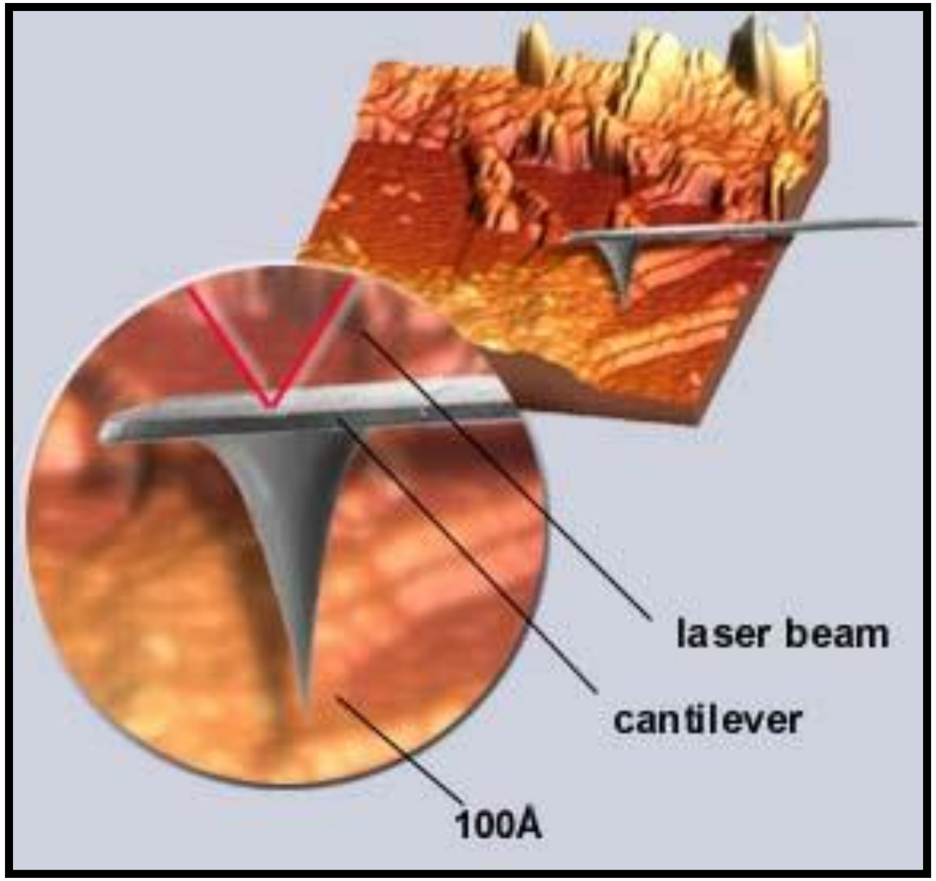
Cantilever Shape:Triangular
Length:200mm
Thickness: 0.8mm
k : 0.18N/m, f = 27 kHz; Material: SiN

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)



As with the STM the probe tip of an AFM must be very small but because there is no need to establish a tunneling current one can use a variety of materials, not just those with a low workfunction.

AFM: Beyond Topographic Imaging

① True z resolution

② It does not capture any image.

"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)