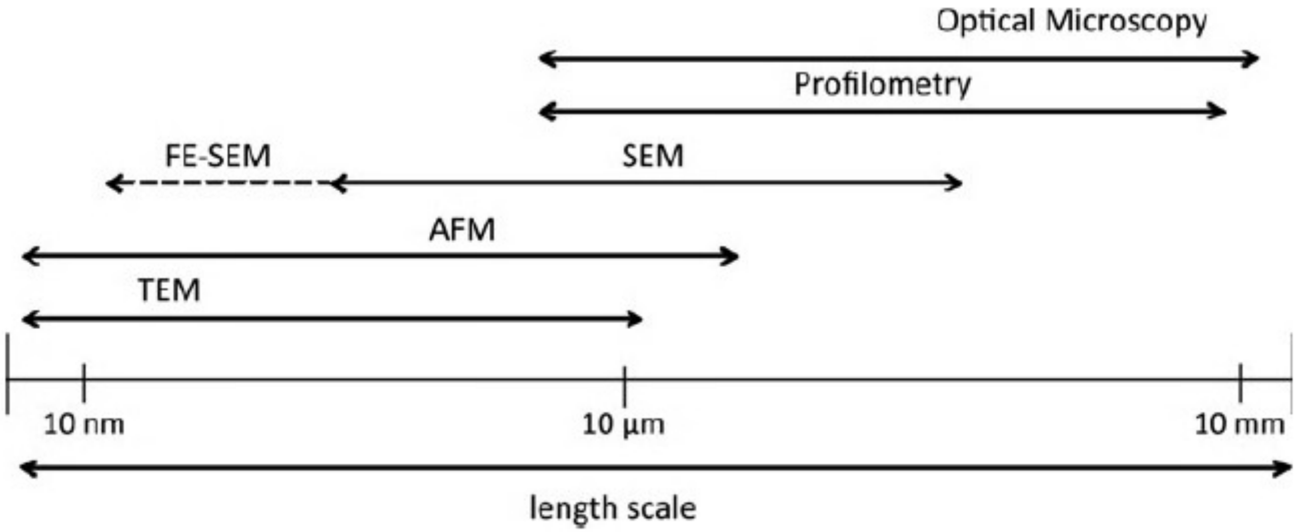
Atomic Force Microscope

Microscope at different length scales

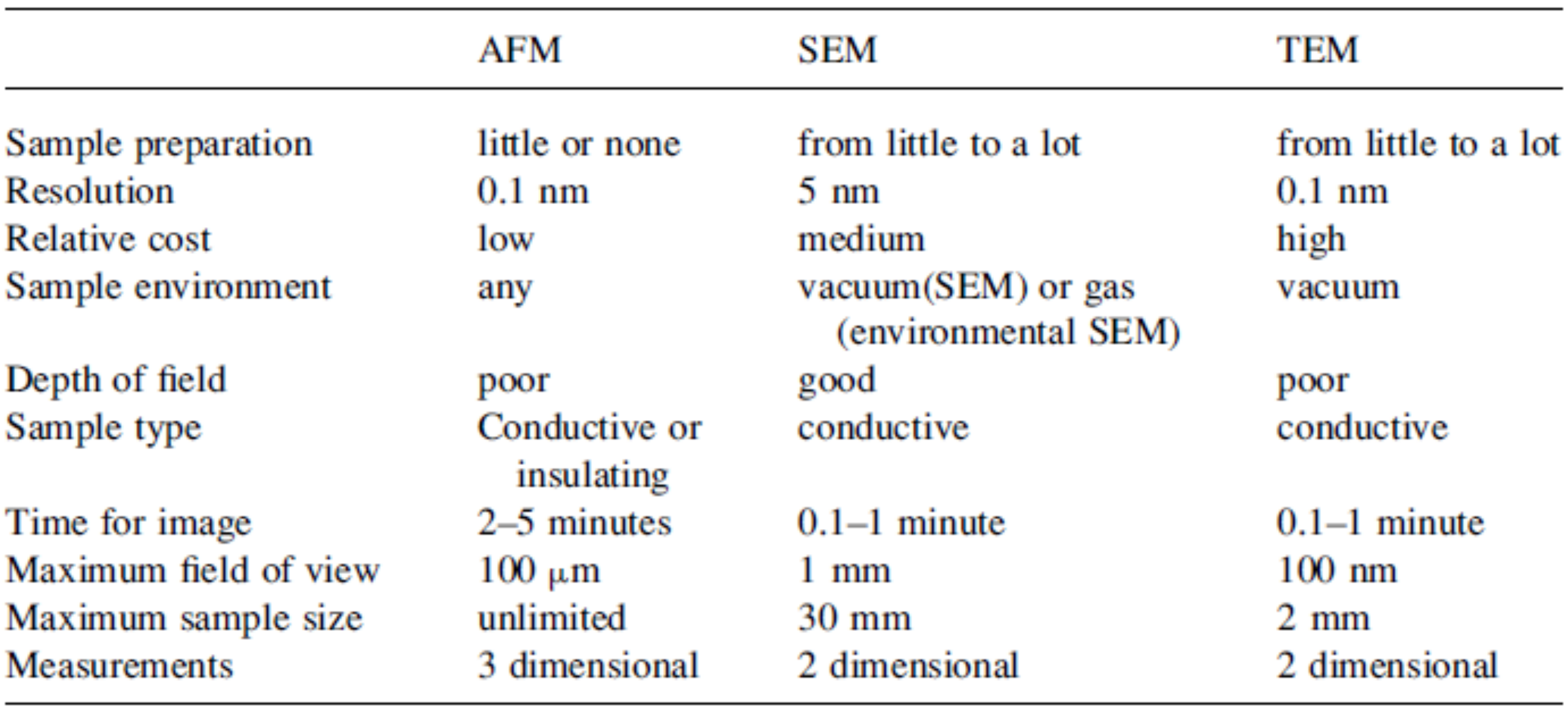


Atomic Force Microscope has among the widest range of spatial resolution.

Its unique in its ability to magnify in the 3rd dimension (Z) as well.

Downside being, any scanning area greater than $100\mu m$ is largely not practical

How AFM compares with other Techniques ?



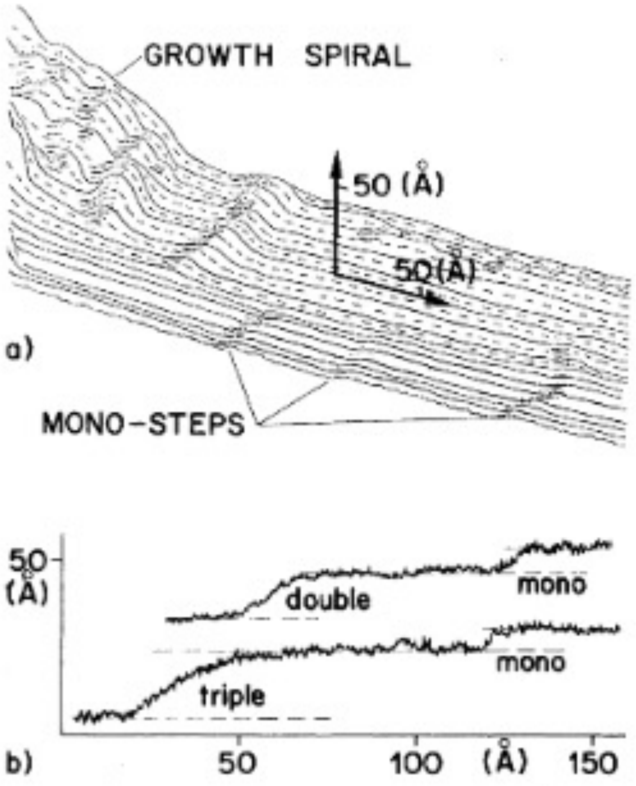
	AFM	SEM	TEM
Sample preparation	little or none	from little to a lot	from little to a lot
Resolution	0.1 nm	5 nm	0.1 nm
Relative cost	low	medium	high
Sample environment	any	vacuum(SEM) or gas (environmental SEM)	vacuum
Depth of field	poor	good	poor
Sample type	Conductive or insulating	conductive	conductive
Time for image	2–5 minutes	0.1–1 minute	0.1–1 minute
Maximum field of view	100 μm	1 mm	100 nm
Maximum sample size	unlimited	30 mm	2 mm
Measurements	3 dimensional	2 dimensional	2 dimensional

AFM with decent overlap with optical microscope spatial range is almost a logical next step

A study of Surfaces

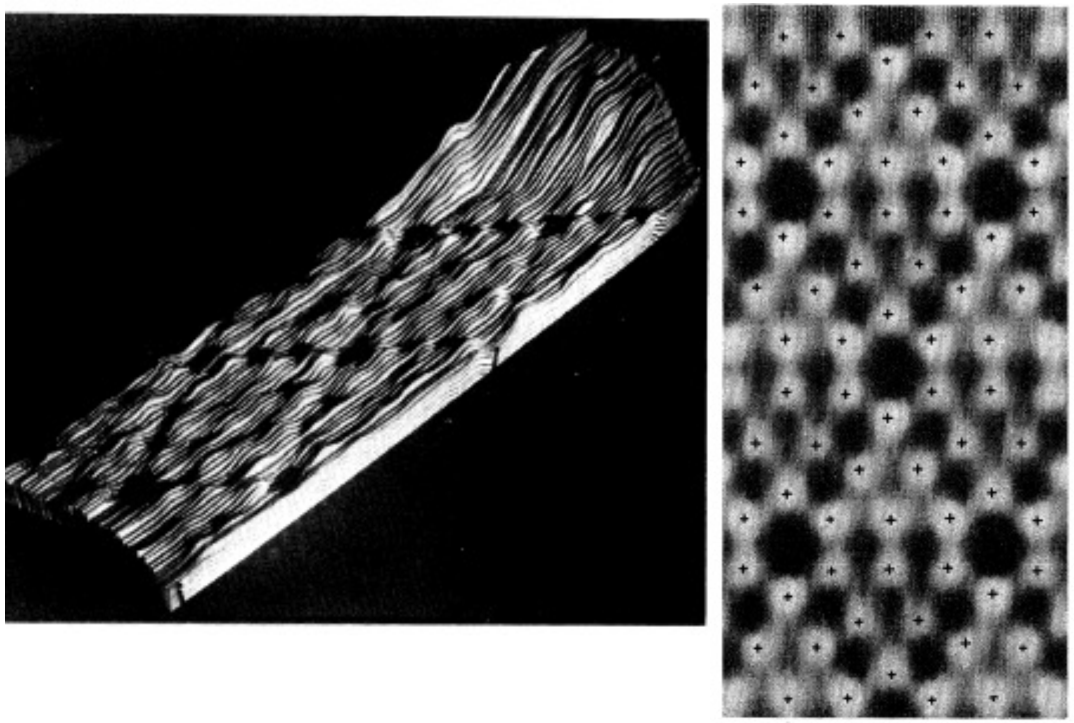
- Surfaces are areas of abnormality in relation to the bulk material
- Atoms/ ions are subjected to assymteric forces
- Surfaces are the only part of the material accessible to chemical change
- The ratio of the surface to the bulk atom density is a function of the material size and shape

First atomic step scanned using tunneling microscope



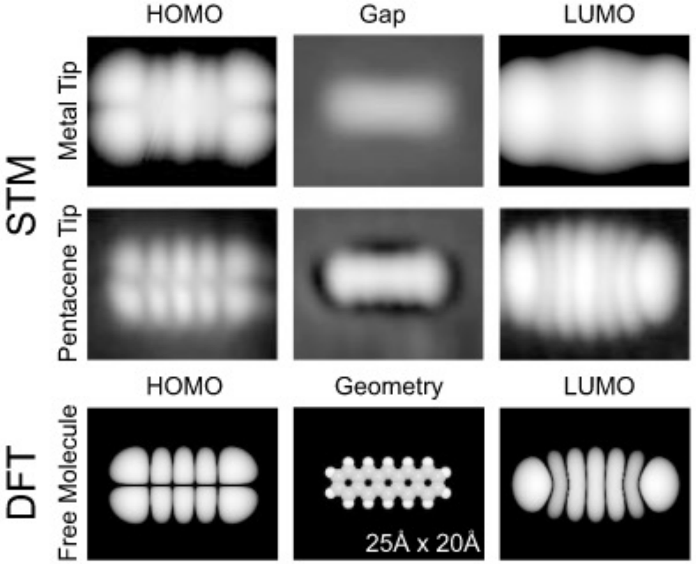
Binning et.al., 1982

Silicon 111 surface reconstruction

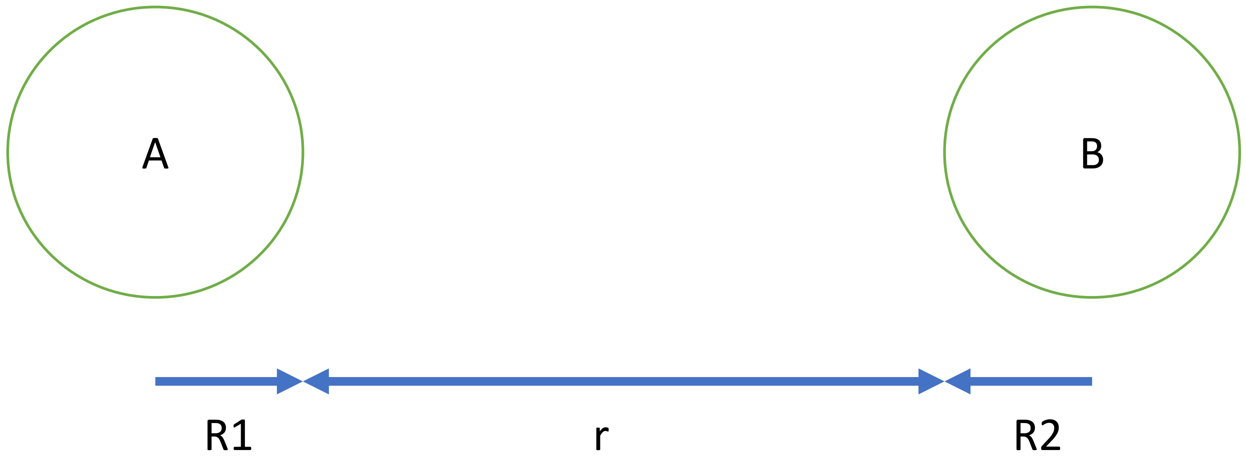


First scanning tunneling image taken by Binning et. al in 1983

Electron orbital imaging on pentacene



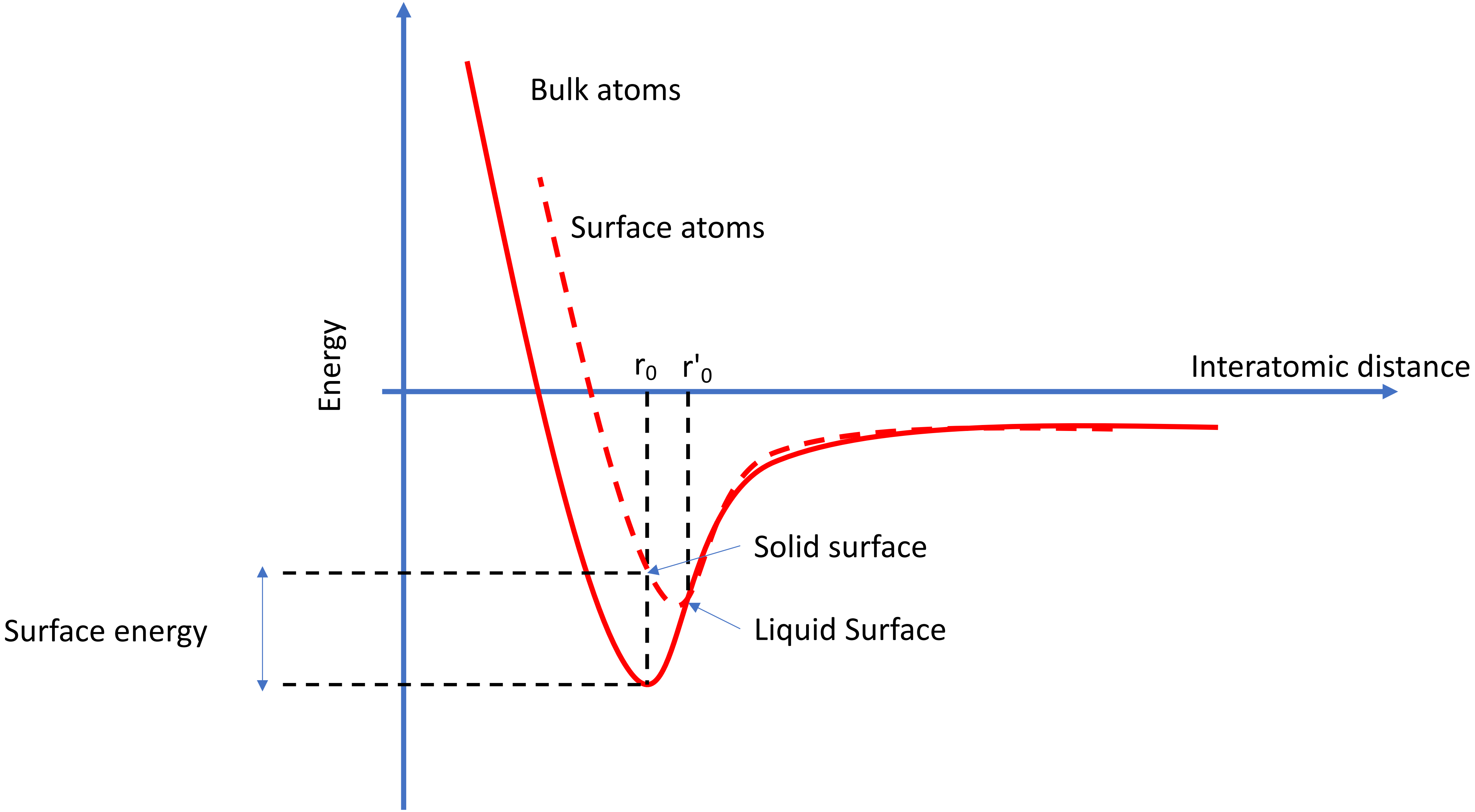
Jascha Reep, Gerhard Meyer, PRL, 2005



For the simplest case of two balls of radius R1 and R2 separated by r

The van der Waals' force $F_{VW} = -\frac{AR_1R_2}{(R_1+R_2)6r^2}$

A – Hamaker coefficient


Bulk atoms
Surface atoms
Energy
r_0
r'_0
Interatomic distance
Solid surface
Liquid Surface
Surface energy

Surface reconstruction

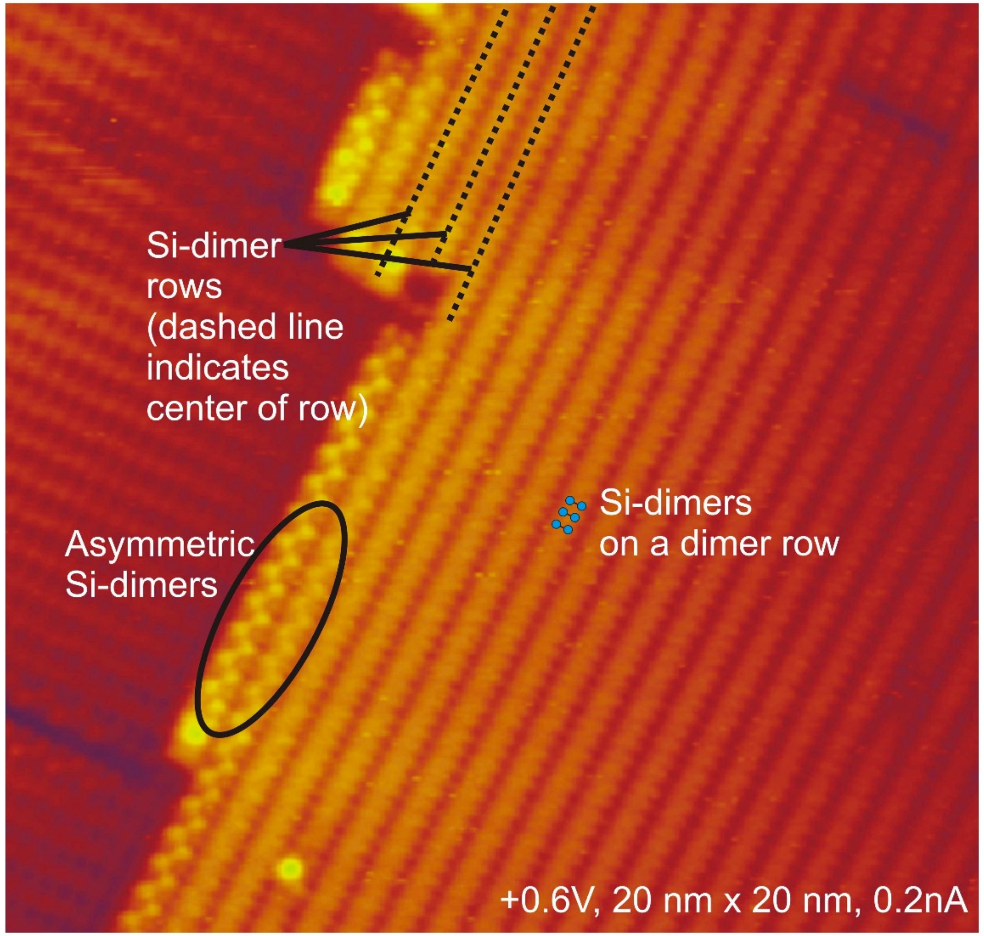
Si – 100 surface reconstructs to form dimers.

If ‘a’ is the side of the origin FCC cube,

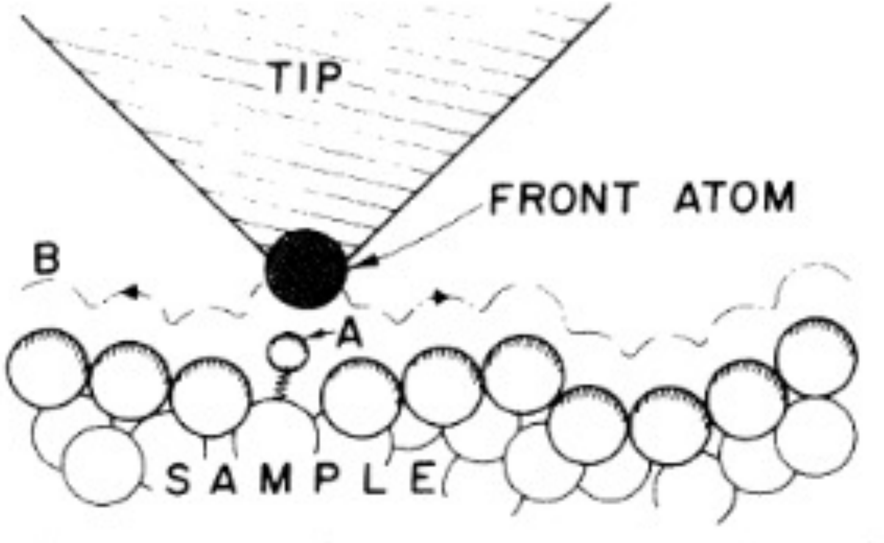
The surface reconstructs such that the periodicity is 2x1

Si – 111 surface reconstructs to give multiple surfaces
With periodicity $\sqrt{3} \times \sqrt{3}$ and $\sqrt{31} \times \sqrt{31}$

Such notation of the surface is called as Wood’s notation.



A tip : - An intentionally sharpened material to reduce the cross section of interaction



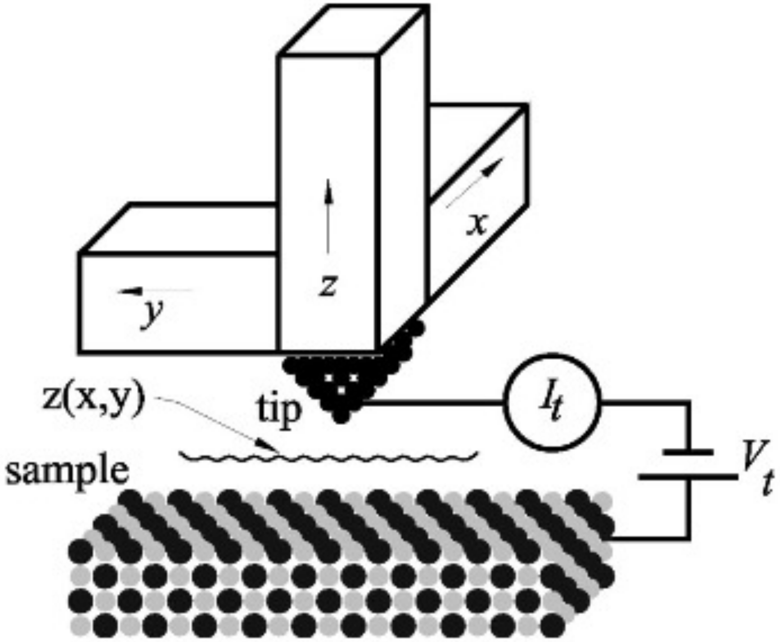
The tip follows the ‘B’ contour on the sample surface.

This is ensured by having the same tunneling current in STM

And by having the same force on the top in AFM.

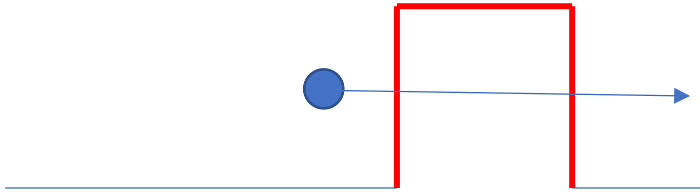
But interatomic forces $\sim 10^{-18}\ N$.

How can you make a spring so sensitive to measure that forces ?



The current in the tunneling microscope is proportional to the gap between the tip and the surface

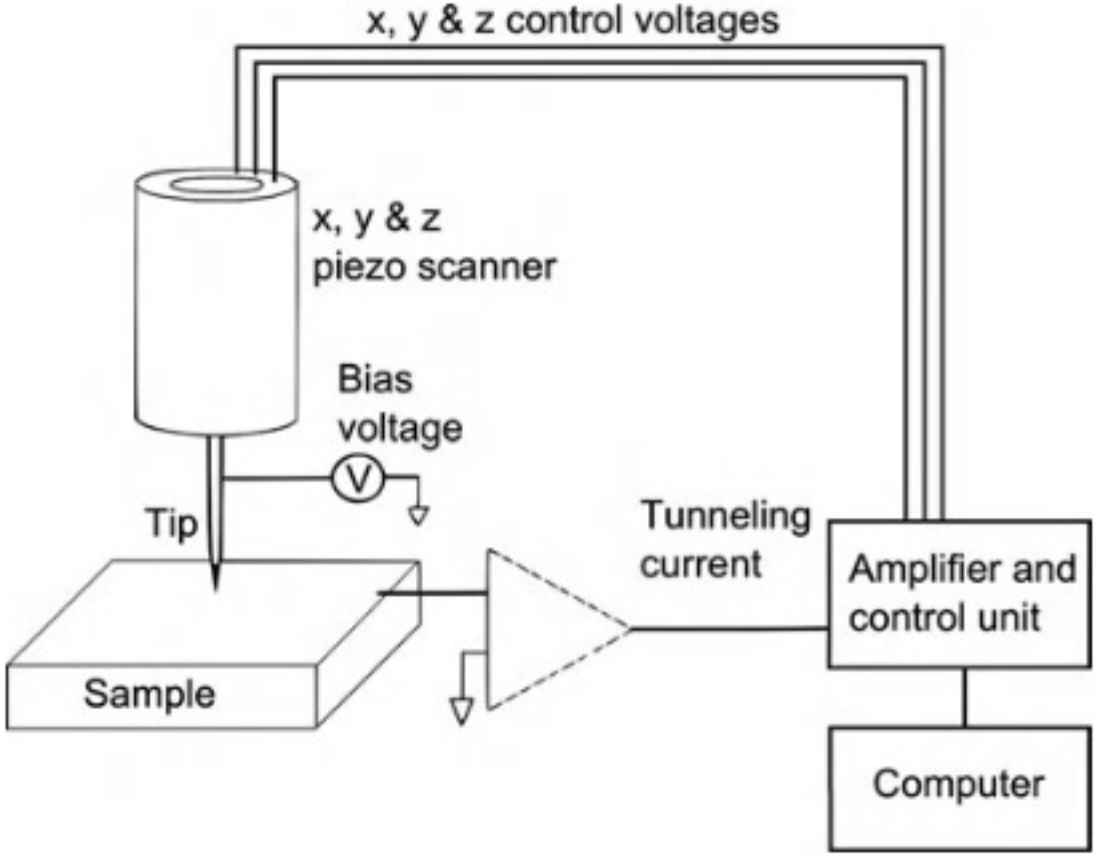
Quantum mechanical tunneling



$$I = I_0 e^{-2K_t z}$$

$$K_t = \frac{\sqrt{2m\phi}}{\hbar}$$

A Typical STM set-up



One should ask: Why is it a microscope ?

As the tip moves across the sample, the tip height is changed to maintain the same current.
This height of the tip maps the topography of the sample from the tunning current expression we saw before

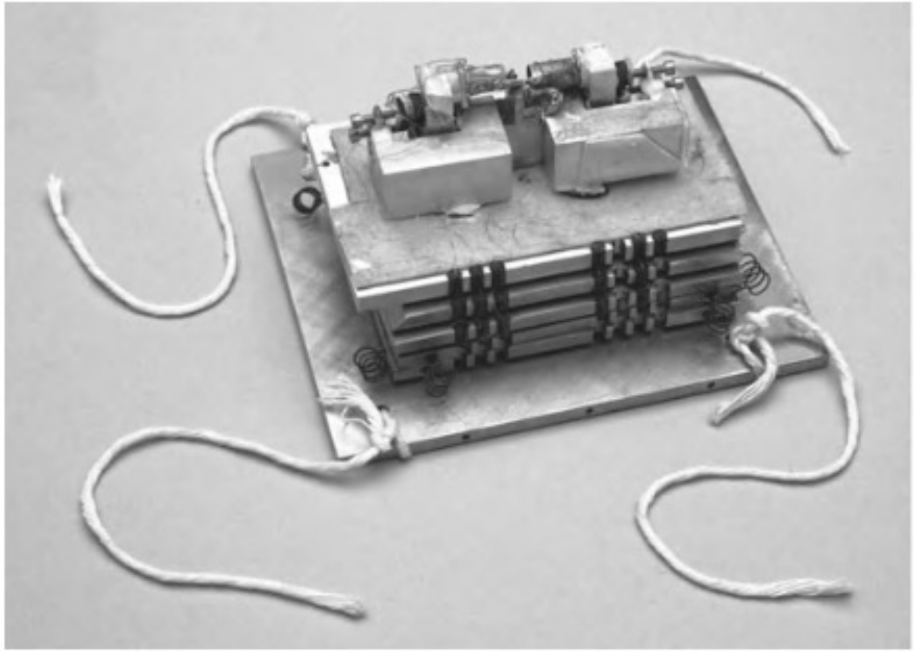
Atomic Force Microscope

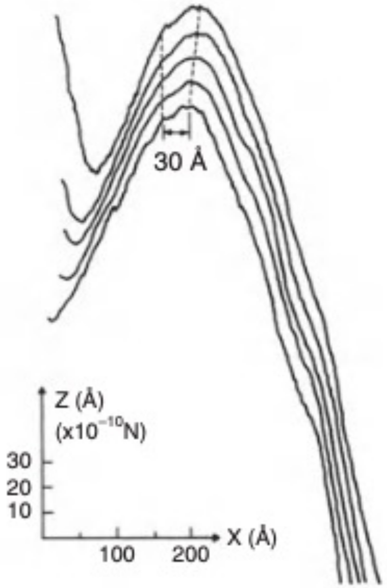
While STM could provide atomic resolution of the surface, it requires the sample to be conducting!

In addition, the sample, tip and the apparatus needs to be in vacuum and electronics cooled to reduce noise.

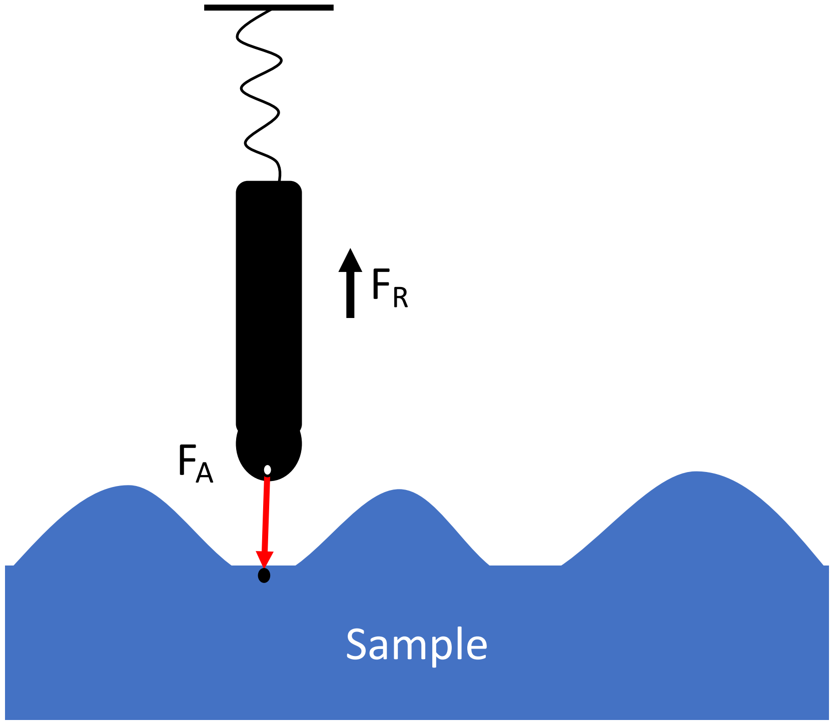
Force microscopy aims to obtain the same spatial resolution with the need for low temperatures, vacuum and the conducting samples

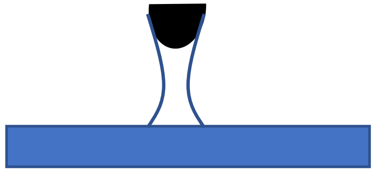
First AFM built by Binning et. al., in 1986





Forces between Tip and the Sample





Meniscus in hydrophilic substrates

Attractive force:

1. van der Waals : $(F_{vdW}\ \alpha \frac{1}{z^2})$

   Van der Waals interaction is typically from neutral particles.

   However, the underlying phenomena is Coulombic interaction between charge fluctuations in neutral particles

2. Electrostatic force: $(F_{Es}\ \alpha U^2 \frac{1}{z})$

   If there exists a potential difference U, then there is an attractive force between the tip and the sample

3. Capillary Force:

In hydrophilic substrates, moisture from room temperature can form a meniscus with the tip. This causes a capillary force driving the liquid up the tip due to surface tension effects

Repulsive Force

Pauli Exclusion / ionic repulsion:

Valence electron wavefunctions can extend few 10’s of nm.

Pauli’s idea excludes two electrons with same energy and spin in close proximity.

This leads to e repulsive potential $V_R \; \alpha \left(\frac{\sigma}{z}\right)^{12}$

where σ is the distance at which the potential is zero.

For the simplest case where in there is only van der Waal’s attractive and Pauli’s repulsion, the total potential

$$V_T = -K\left[\left(\frac{\sigma}{z}\right)^{6} - \left(\frac{\sigma}{z}\right)^{12}\right]$$

This potential is called as Lennard-Jones potential.

The force due to the potential is then $F_{LJ} = -\frac{\partial V_T}{\partial z}$

Averaged van der Waal's force on the tip

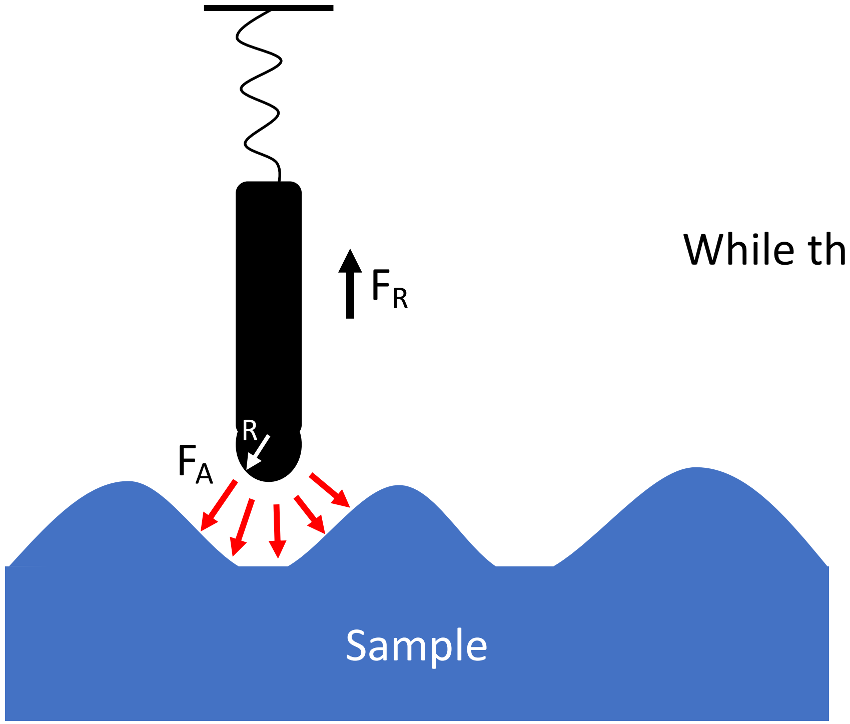
Hamaker's potential

$$V = -\frac{A_H R}{6z}$$

Thus the force

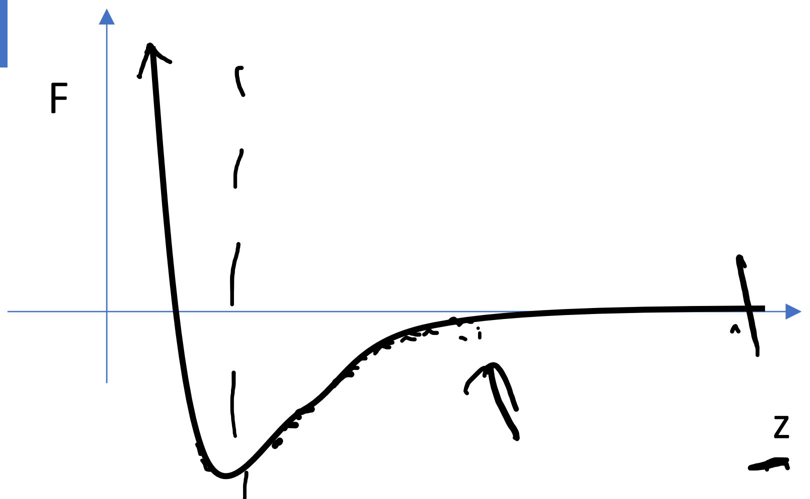
$$F_{vdW} \alpha \frac{1}{z^2}$$

Its important to note that super short-range potential z^{-6} leads to a long-range force when averaged over several interacting atoms



While the repulsive forces is sharply short-range.

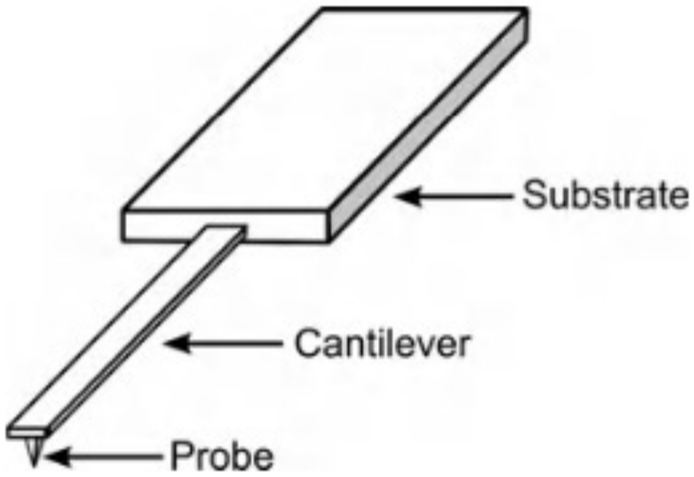
$$V_R \; \alpha \; z^{-12}$$



Design of a tip

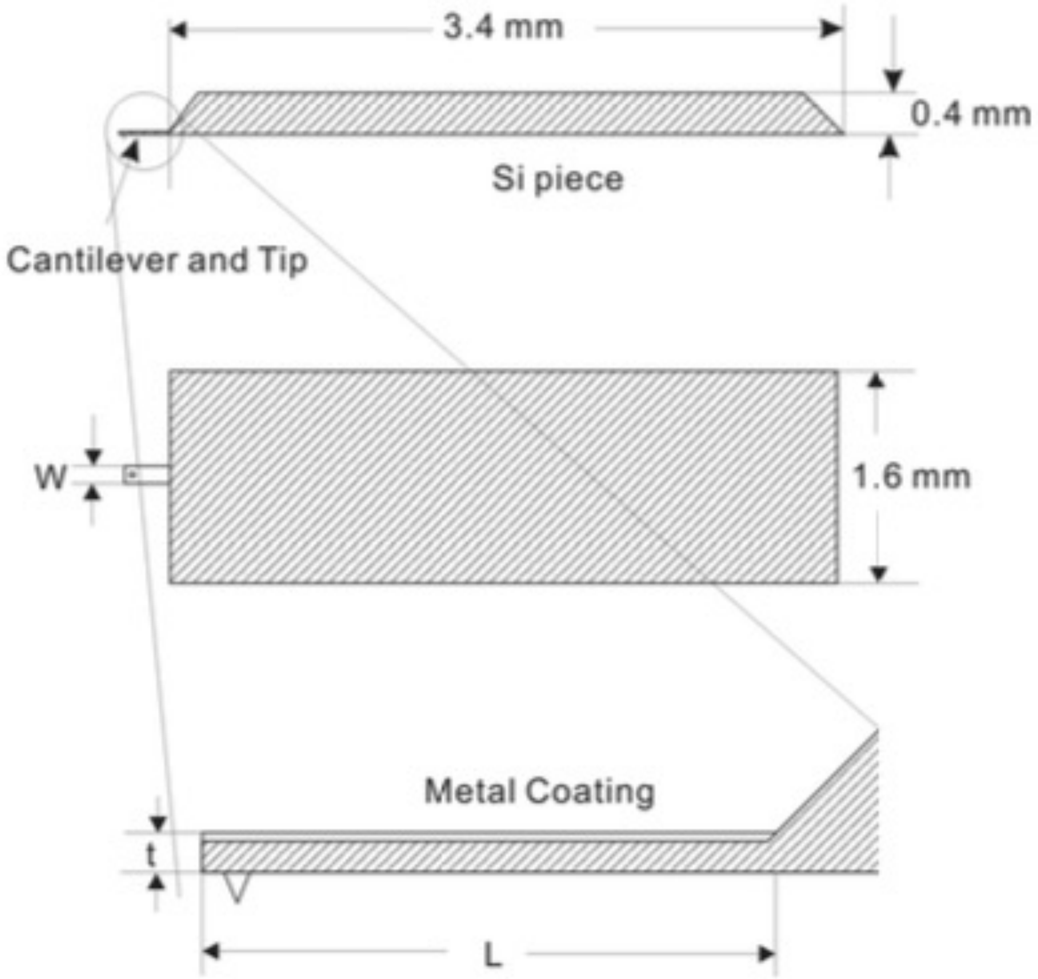
AFM tip is used to sense tip – sample interaction (Force)

Hence a mechanically pliant structure is required and is operated in elastic regime.



Spring constant $k = Ewt^3/4L^3$

Design of commercial AFM tips



Examples of Tips realized in experiments

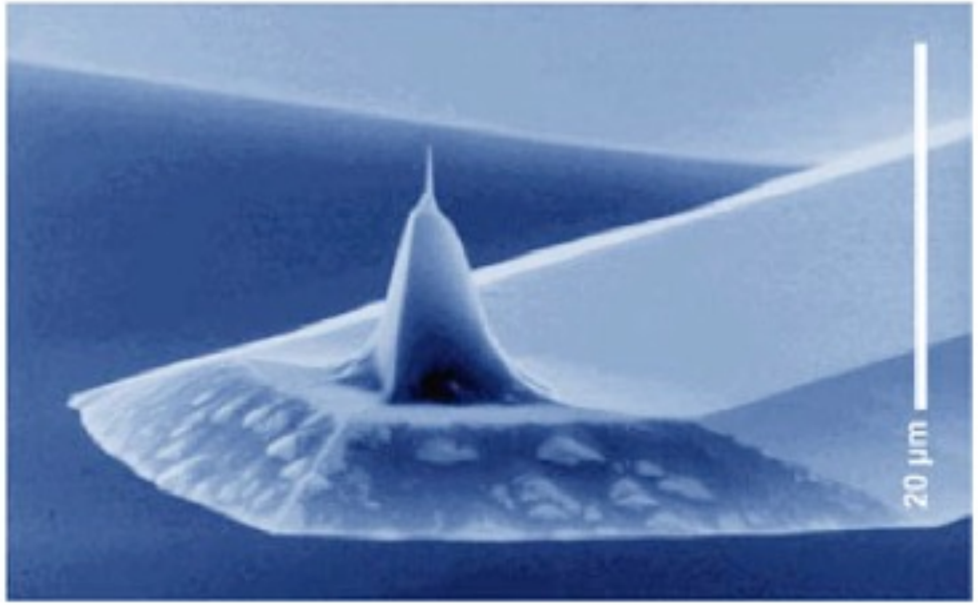


FIG. 8. (Color in online edition) Scanning electron micrograph of a micromachined silicon cantilever with an integrated tip pointing in the [001] crystal direction (Wolter *et al.*, 1991). This is a Pointprobe sensor made by Nanosensors GmbH und Co. KG, Norderfriedrichskoog, Germany D-25870. Photo courtesy of Nanosensors GmbH & Co. KG.

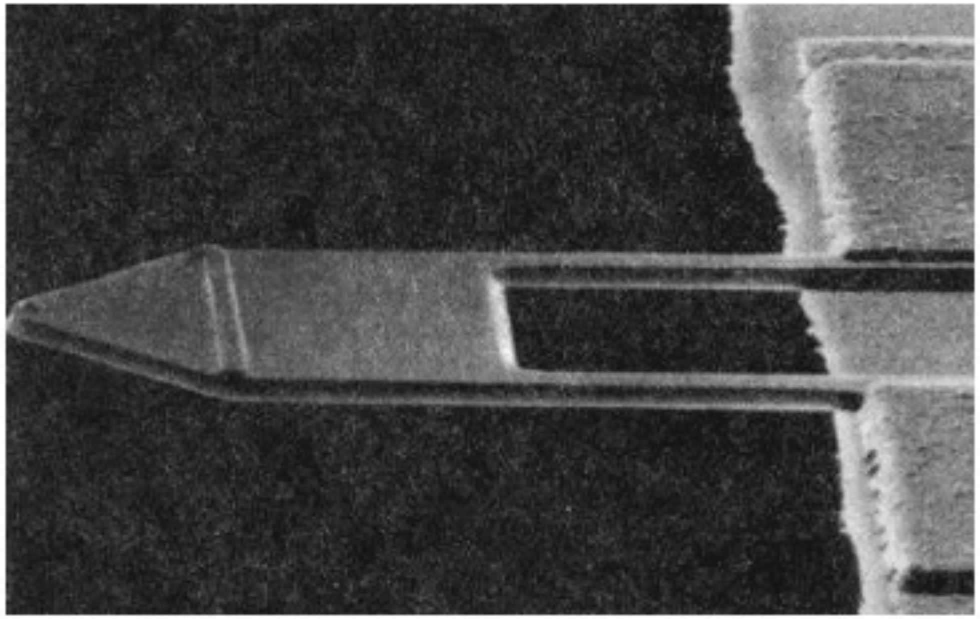


FIG. 10. Scanning electron micrograph of a piezoresistive cantilever built from silicon. Length, 250 μm; full width, 80 μm; thickness, 2 μm. From Tortonese *et al.*, 1993.

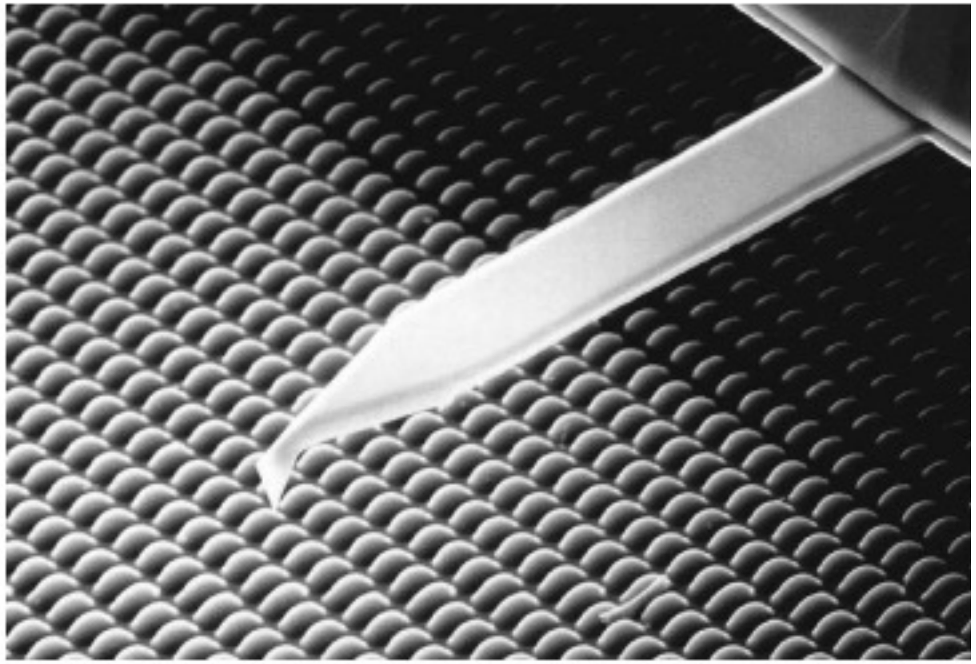


FIG. 9. Scanning electron micrograph of a micromachine con cantilever with an integrated tip pointing in the [001] crystal direction. In this type, the tip is etching free so that the sample area adjacent to the tip is visible in an optical microscope. Length, 120 μm; width, 30 μm; thickness, 2.8 μm; $k = 15$ N/m; $f_0 = 300$ kHz. Photo courtesy of Olympus Optical Co. Ltd, Hachioji, Tokyo 192-8507, Japan.

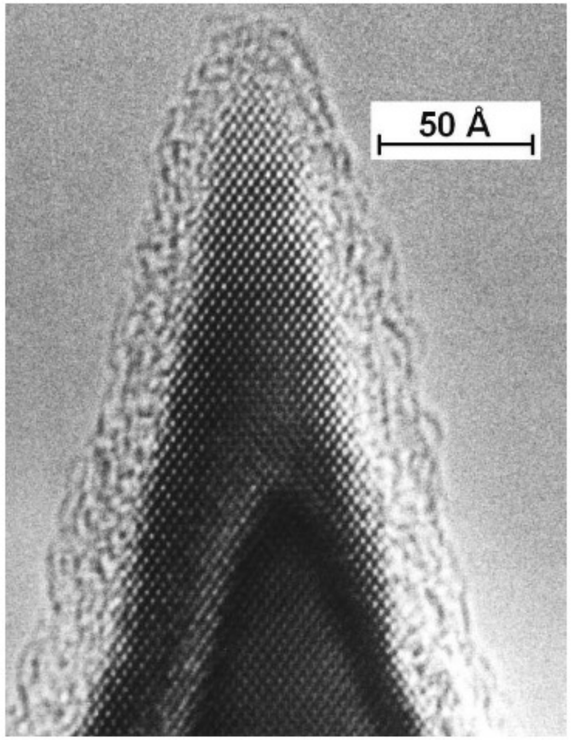


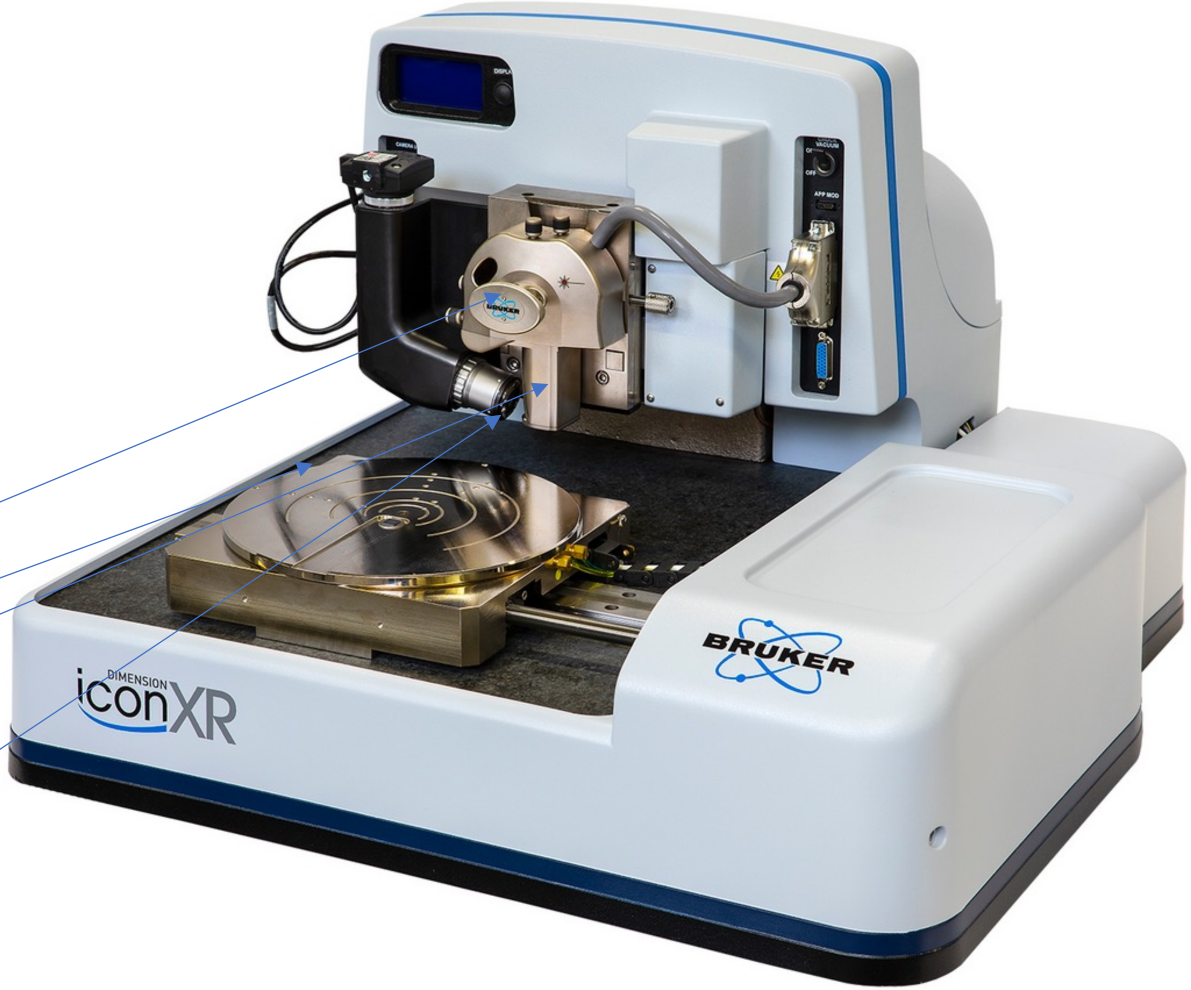
FIG. 13. Transmission electron micrograph of an extremely sharp silicon tip. The native oxide has been etched away with hydrofluoric acid before imaging. The 15–20-Å-thick coating of the tip is mostly due to hydrocarbons which have been polymerized by the electron beam. Interestingly, the crystal structure appears to remain bulklike up to the apex of the tip. From Marcus *et al.*, 1990.

FIG. 15. Scanning electron micrograph of a cleaved single-crystal silicon tip attached to the free prong of a qPlus sensor. The rectangular section is the end of the free prong with a width of 130 μm and a thickness of 214 μm. The tip is pointed in the [111] direction and bounded by $(\bar{1}\bar{1}\bar{1})$, $(1\bar{1}\bar{1})$, and $(\bar{1}1\bar{1})$ planes according to the method of Giessibl *et al.* (2001b). Figure courtesy of Christian Schiller taken from Schiller, 2003.

AFM Instrumentation

Basic components:

1. Microscopic Stage
2. Control Electronics
3. Computer



Microscopic Stage:

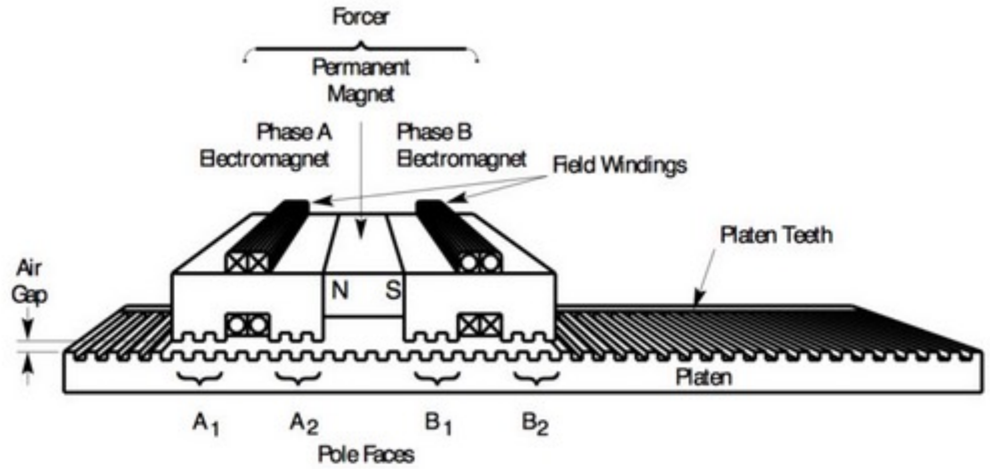
1. Scanner head
2. Sample holder
3. Force Sensor
4. Integrated optical microscope

Linear Translations

However, for an AFM, there is a need for atomical resolution!

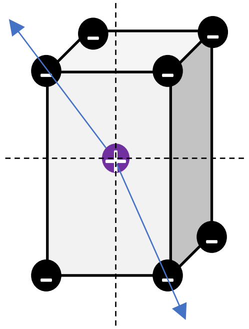
Linear translations in Å resolution is required.

From inches - ~ $1\mu m$



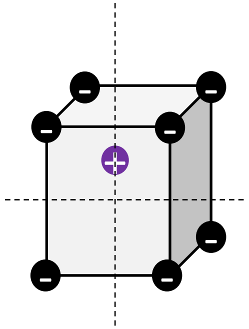
Piezoelectricity:

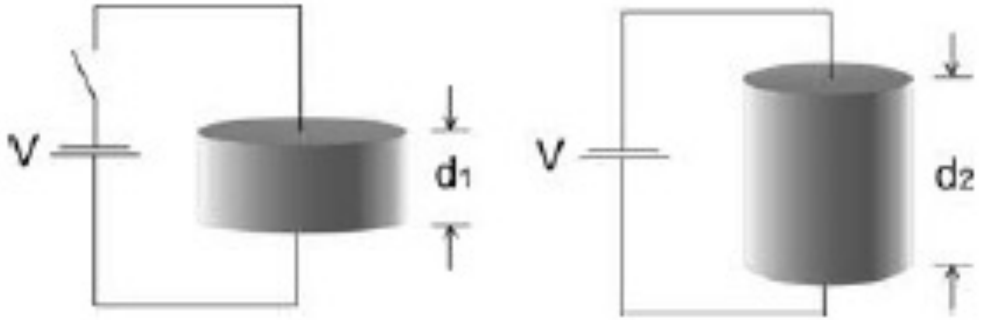
In non-centrosymmetric crystals, strain causes generation of electric dipoles



Equal and opposite dipoles

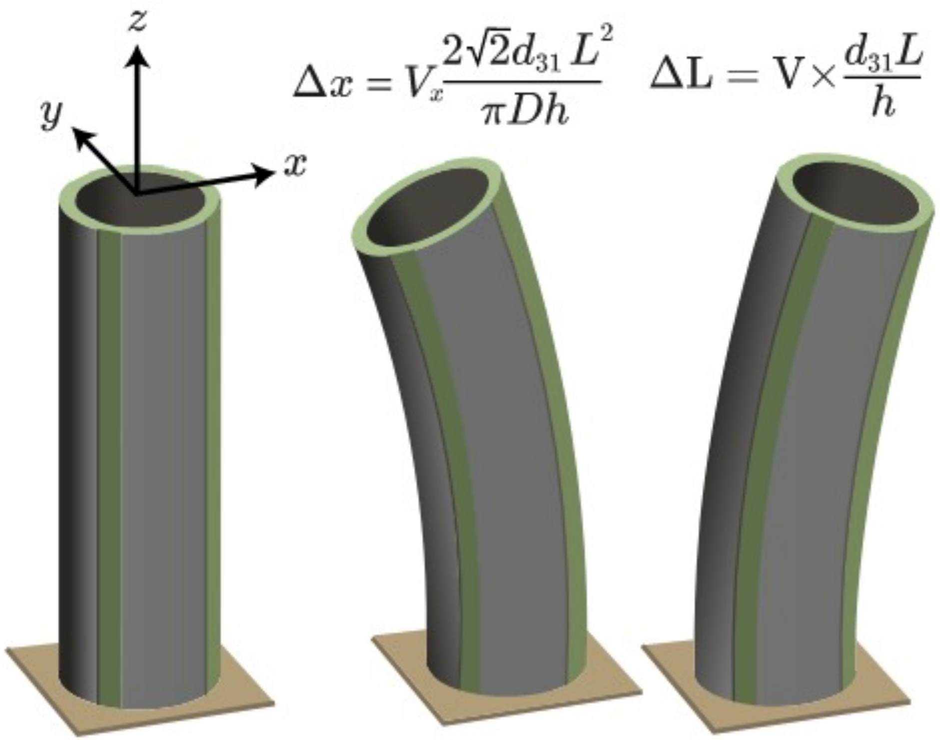
Uncompensated dipoles

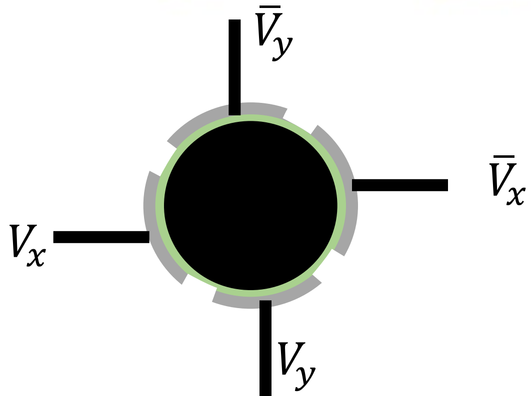




You either apply strain and get dipoles;
Or force a potential and observe mechanical strain

3-D motion using Piezoelectric transducers





When the base of the tube is fixed,
V_x voltage along x axis, V_y voltage along Y

$$\Delta x = V_x \frac{2\sqrt{2}d_{31}L^2}{\pi D h}$$
$$\Delta y = V_y \frac{2\sqrt{2}d_{31}L^2}{\pi D h}$$

d_{31} is the piezo strain, L – length of tube, D – outside diameter, h – tube thickness.

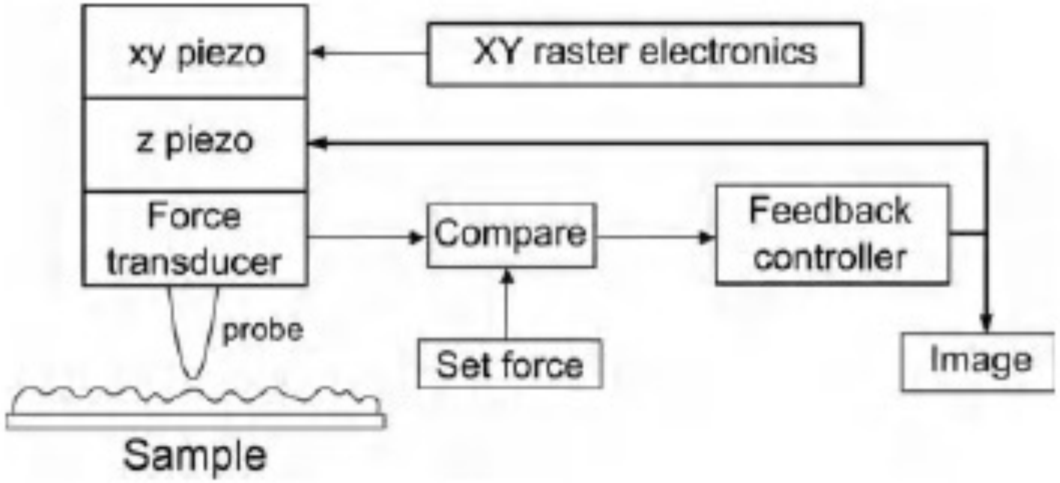
If a potential V is applied to all four terminals,

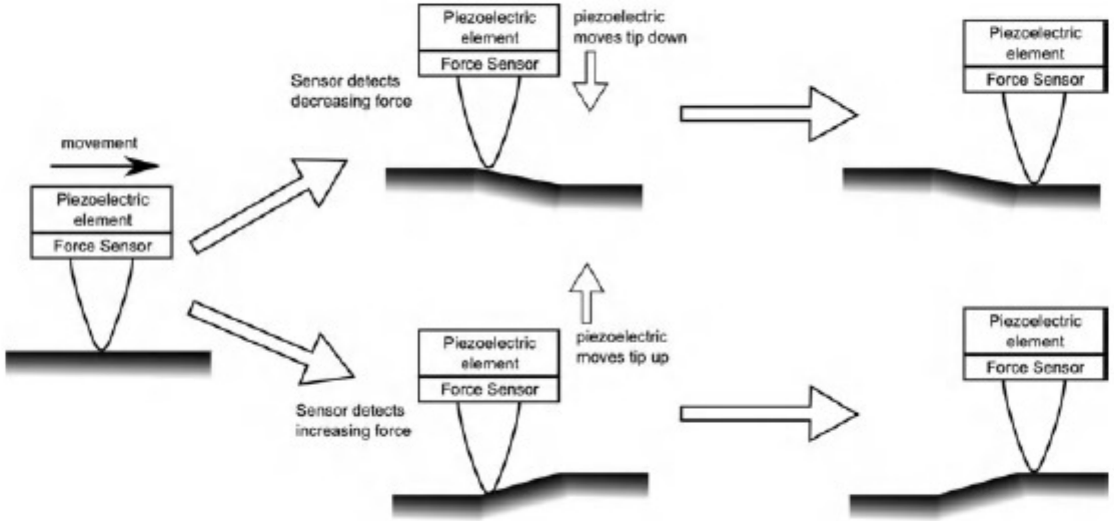
$$\Delta L = V \times \frac{d_{31}L}{h}$$

Since, +Vx and –Vx are applied to diametrically opposite sides, one side contracts and other side expands. The tube ‘bows’ in a fashion to accommodate the differential strain

Typical expansion coefficient: $\sim 0.1\ nm/V$
Thus, atomic scale movements are possible using Piezoelectric transducers

Typical Block diagram of AFM head



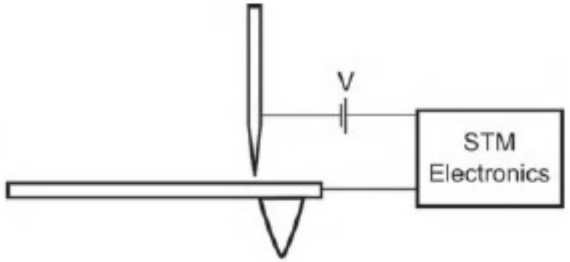


The XY Piezo in addition to linear transducers are used to raster the sample surface. At each point on the surface, the force transducer translates the force sample interaction

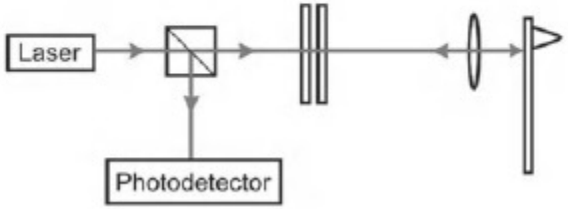
A feedback loop is used to ensure that the force between the tip and the probe remains the same. ‘z’ transduction need to bring the force to its set value is noted as the sample height

Force Sensors

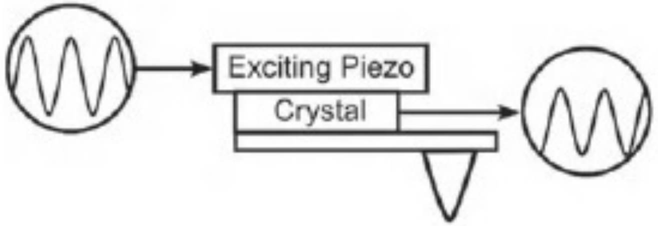
A force central to AFM design. Some common designs



A STM based design

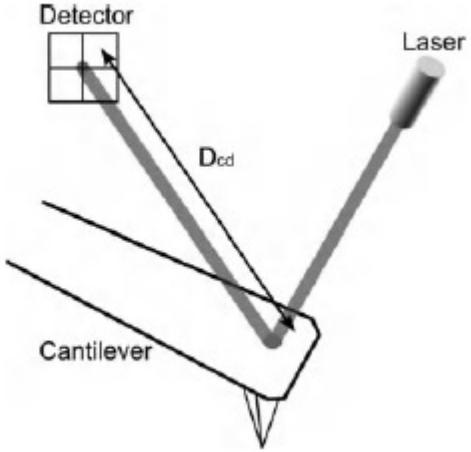


Traditional interferometer

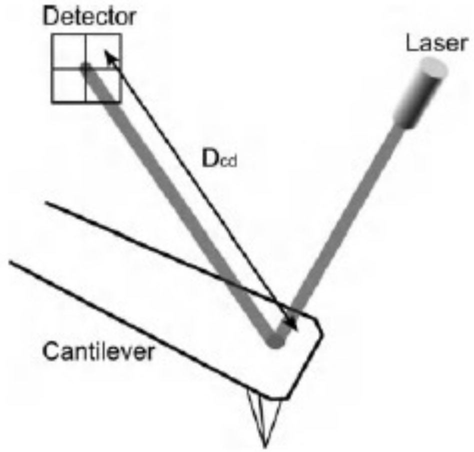


Dynamic measurement (Phase and amplitude)

Standard Piezoelectric sensor



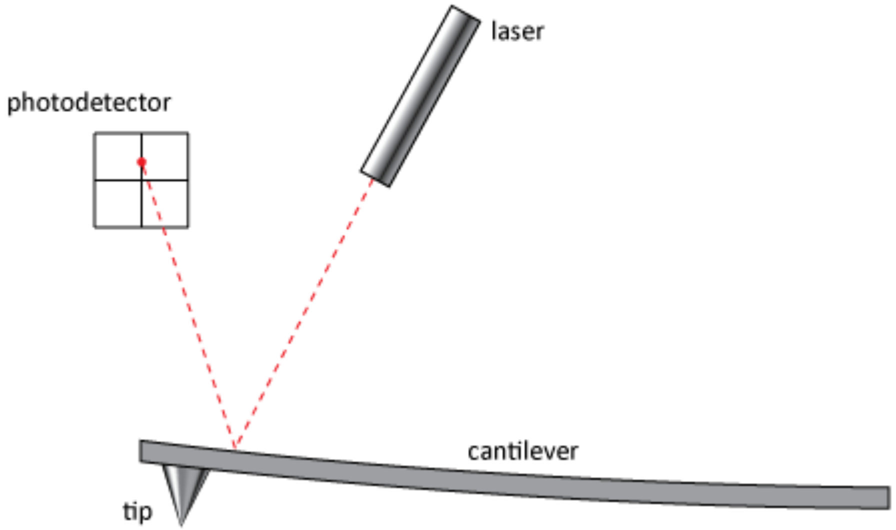
Most-commonly used technique

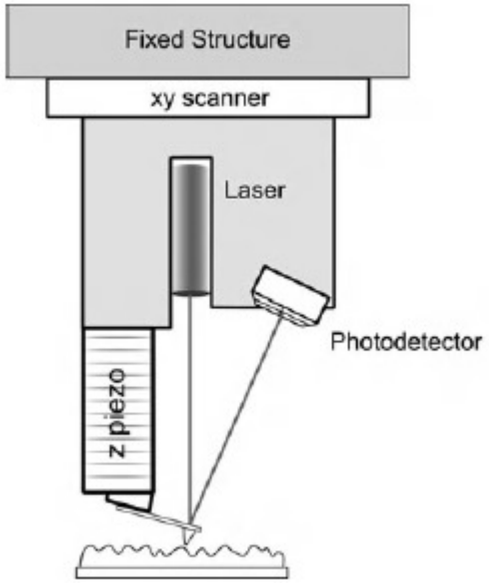


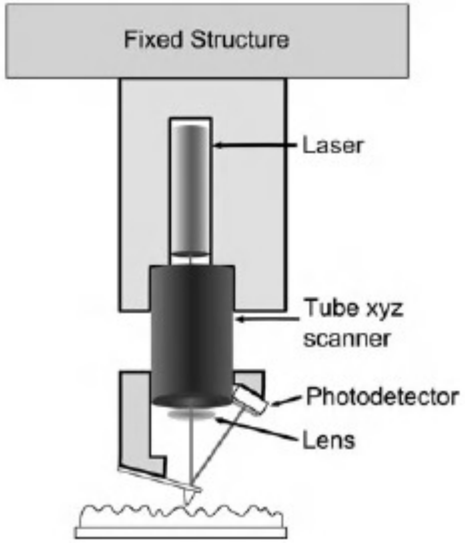
Cantilevers are typically 30 - 600μm long

20 – 60 μm wide and 0.2 – 1 μm thick.

Position sensitive photo detection





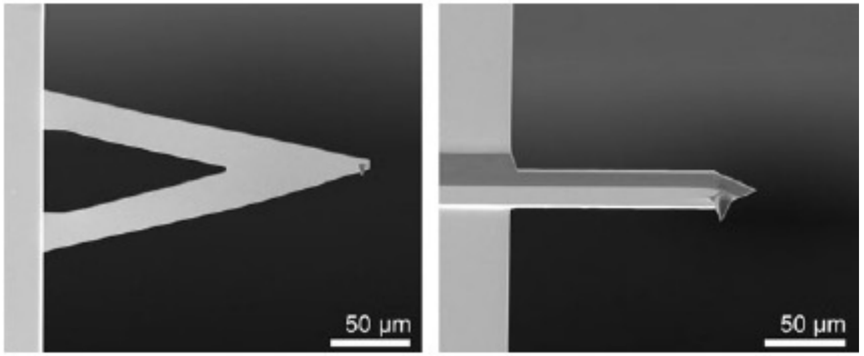


Either the laser and detector assembly is moved by X-Y stage.

Or the cantilever and the detector is moved. Lens focuses the light.

Typically the lens focuses the light at an angle to the cantilever to allow for imaging the sample and the cantilever together

Operational Modes of AFM



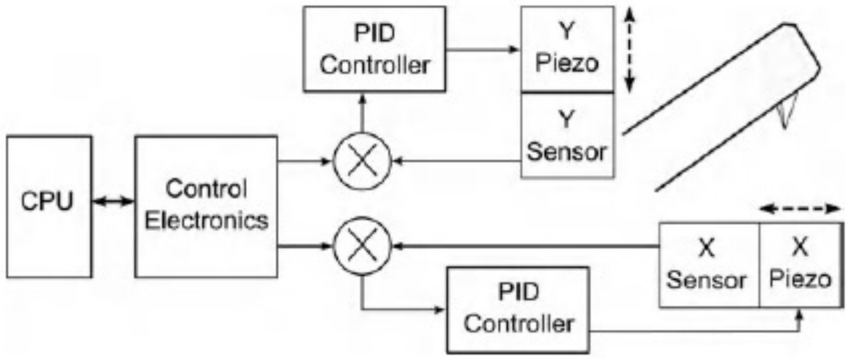
Contact

Non-contact mode

Typically a Triangular shaped less stiff cantilever is used (K < 1 N/m)
Made of Si_3N_4 like material
The tip is brought to contact in a static mode
Bending of the cantilever is measured

Typically a rectangular stiff material is used.
Like Si
The cantilever is made to oscillate close to the surface
The sample-tip interaction forces the oscillation amplitude and phase to change

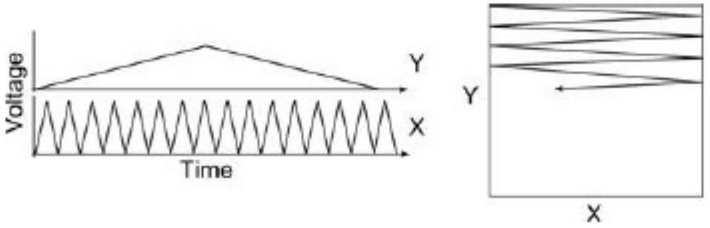
Generating an AFM Image



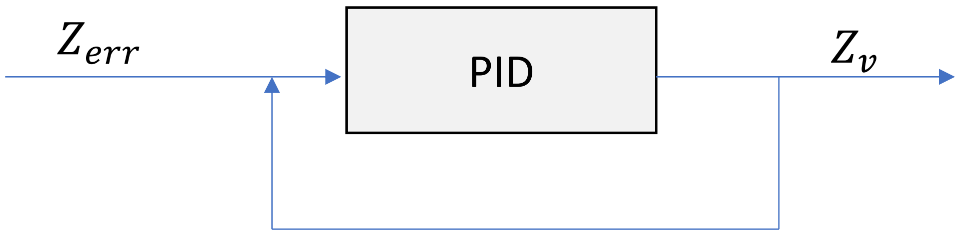
Steps:

1. Generate scanning signals for X-Y stage movement
2. Take input signal from force sensor and feed to z-piezo
3. Get output control to X-Y-Z motor
4. Generate signal for oscillating probe. Measure phase and amplitude
5. Collect signals and display on computer

Generating the X-Y raster



The Z potential is set using a feedback setup



$$Z = p * V_{err} + i * \int Z_{err} dt + D \times \frac{dZ_{err}}{dt}$$

Static Interaction of Tip with Sample

A cantilever with the sharp tip is moved close to the surface.

At each point, there is sample height determined force $F(z)$

The probe tip is assumed to be much stiffer than the cantilever and the sample

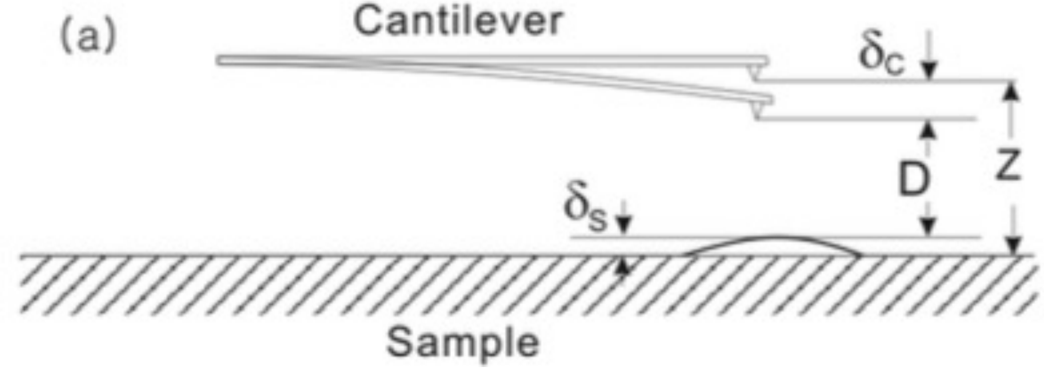
Typical inter-atomic force constants in solids – 10 – 100 N/m

In biological samples - ~ 0.1 N/m.

Hence the cantilever stiffness is typically set at 0.01 – 5 N/m

The cantilever deflection $\delta_c = -F_c(z)/k$ – Hooke's law

Where F_c is the elastic force on the cantilever.



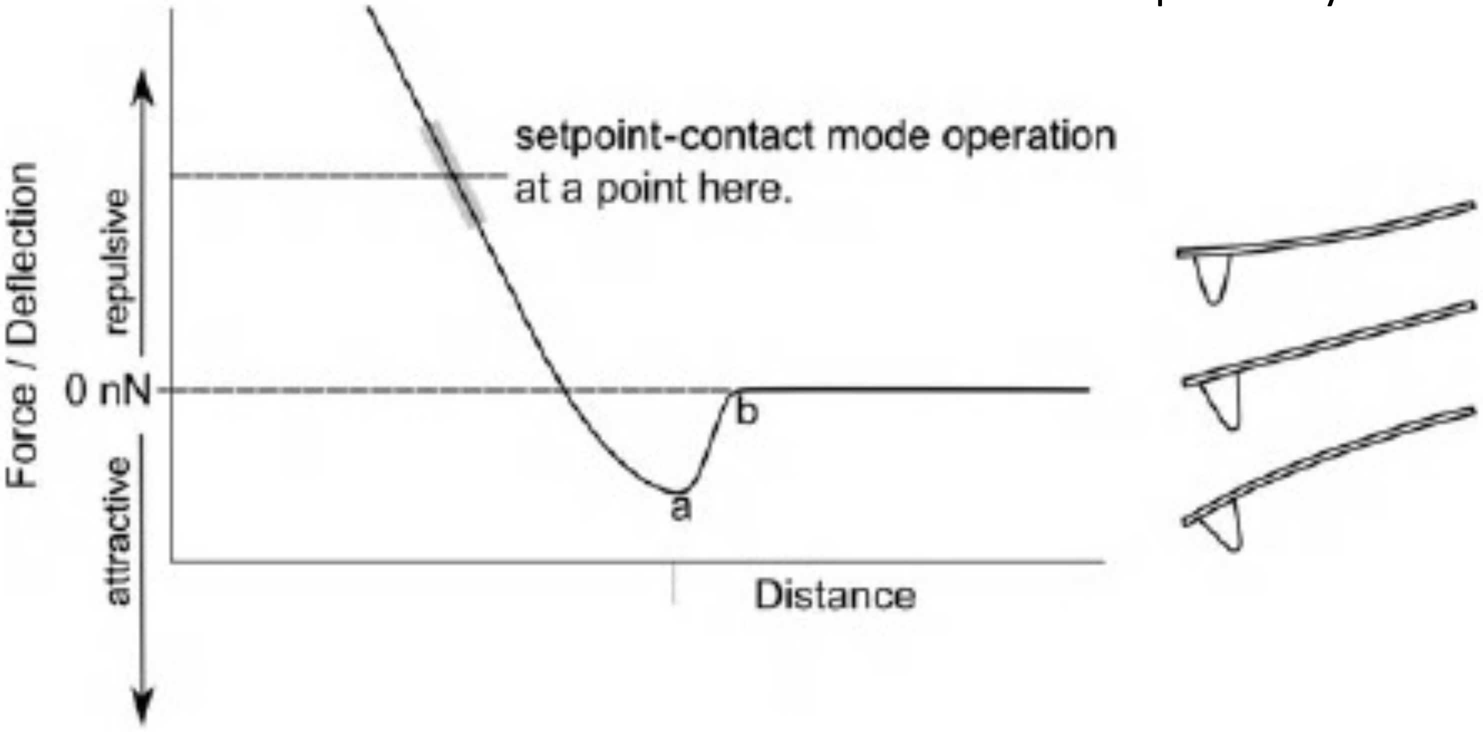
Say, the cantilever is near a hill with D separation. There will be tip bending and a restoring force from the cantilever.
At equilibrium the cantilever restoring force is same as the sample-tip interaction force. So, the net force on the cantilever is 0. However, the cantilever at 0 force is at a different height (Z)

Force-distance curve

In contact mode, the tip Z is moved such that the force remains constant. This point is typically in the repulsive region as shown.

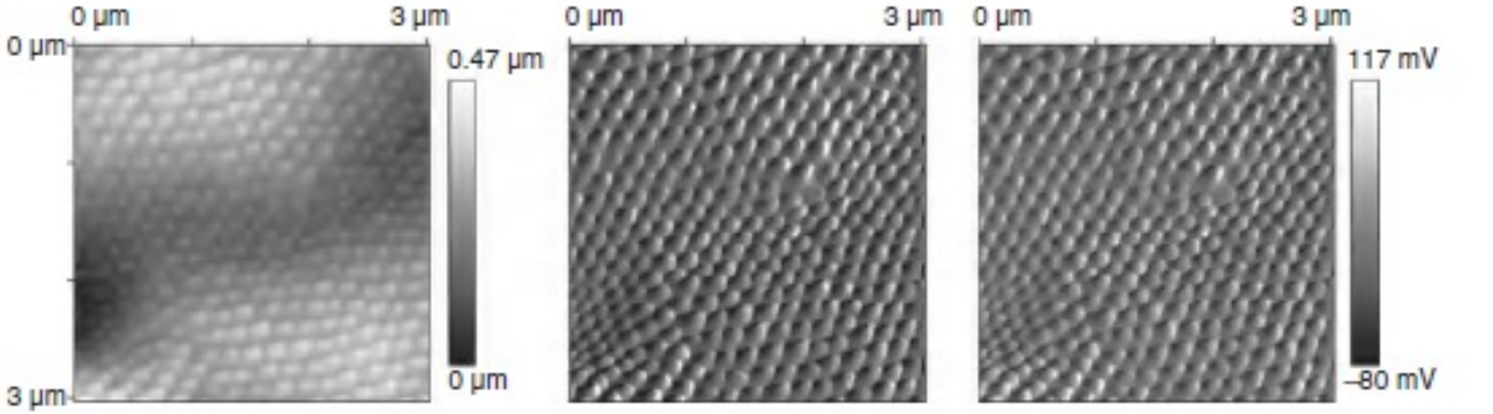
The tip is always touching.

At point b, an instability happens and the comes into contact



Force on the cantilever and its deflection are just scaled version of the same thing (related by Hooke's law)

Typical Contact Mode Image

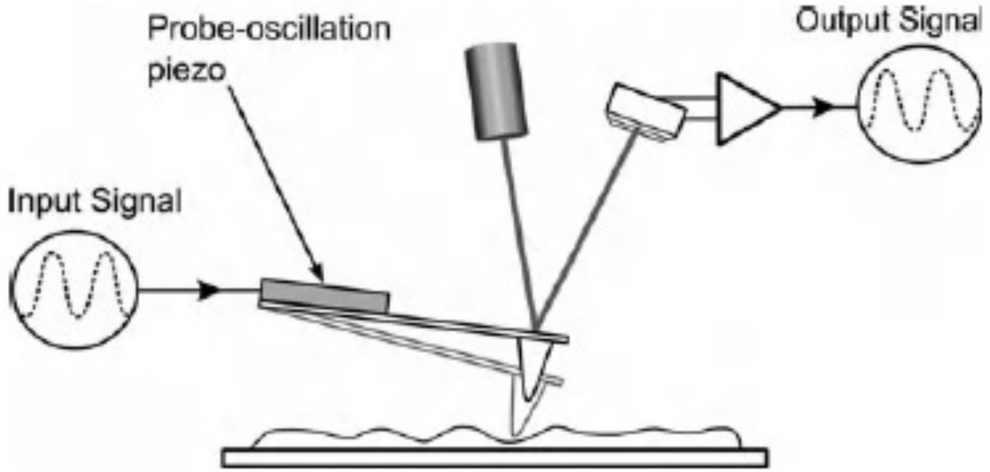


Two modes of AFM operation in contact:

1. Constant Force mode: This is the most common mode of operation. The Z-height is changed using a feedback loop to get to the same force as seen in the previous slide. In most measurements, this is called as the height image.
2. Constant height mode: This is the deflection in the cantilever before the feedback kicks in. This is the raw deflection in the cantilever as a X-Y stage is moved. This is typically displayed as deflection image.

Many times, this image is used. However, if the feedback settings (PID) is correct, the height image and the deflection image should match.

Dynamics Mode

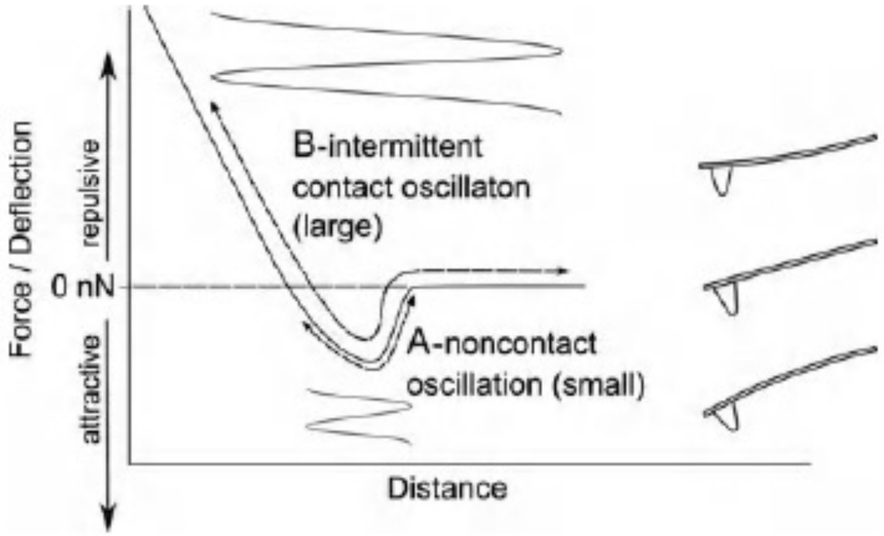


An additional piezo patch is placed on the cantilever.

The potential of vibration is set close to mechanical resonance.

The tip oscillation is collected by optical means.

The sample interaction changes the amplitude, frequency and phase of the oscillation

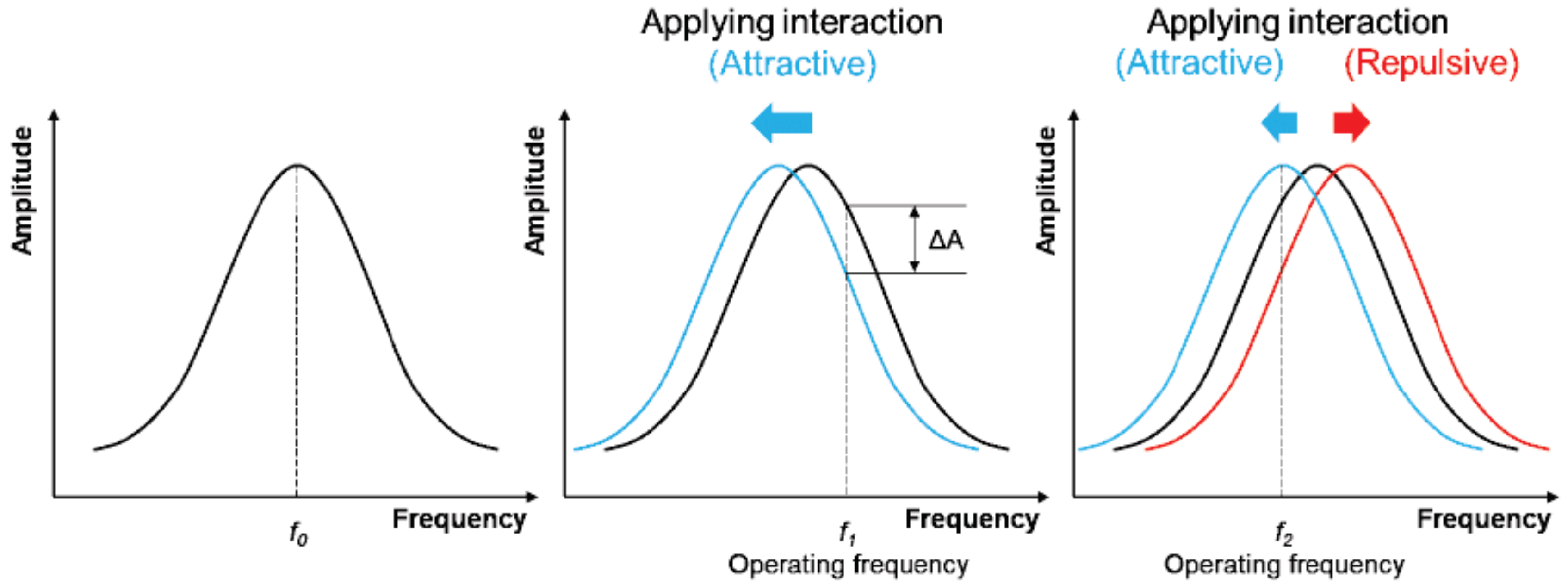


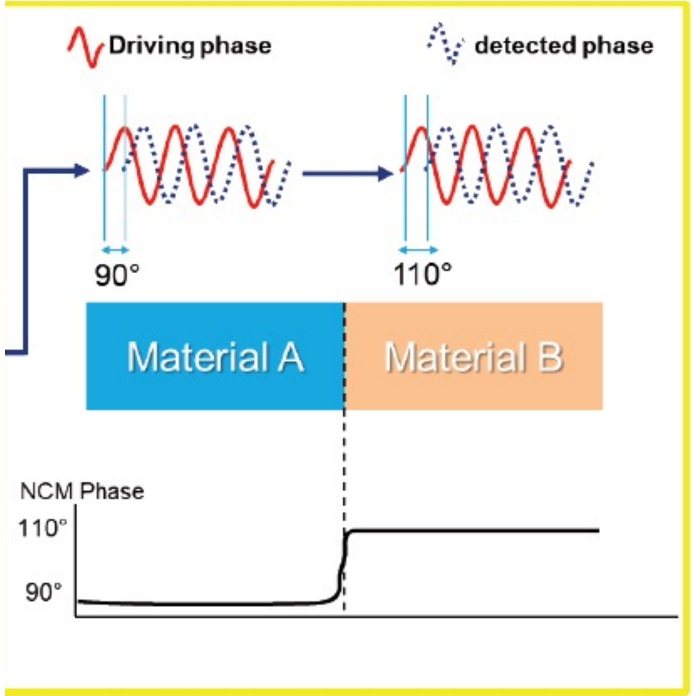
The amplitude of oscillation, determines the interaction method.

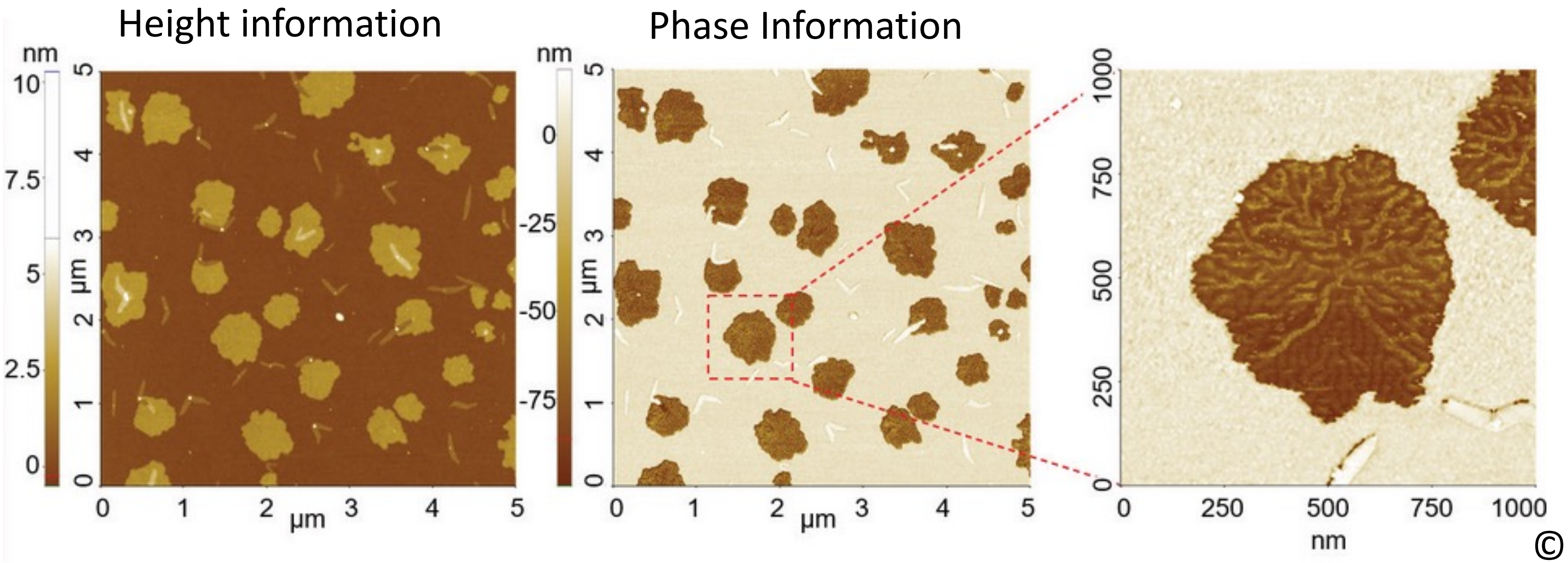
Small oscillations are mostly non contact

Large oscillators near the surface gives intermittent contact.

Resonant frequency $f_0 = \sqrt{\frac{k}{m}}$


Amplitude
Frequency
f_0
Applying interaction
(Attractive)
Amplitude
ΔA
f_1
Frequency
Operating frequency
Applying interaction
(Attractive)
(Repulsive)
Amplitude
f_2
Frequency
Operating frequency

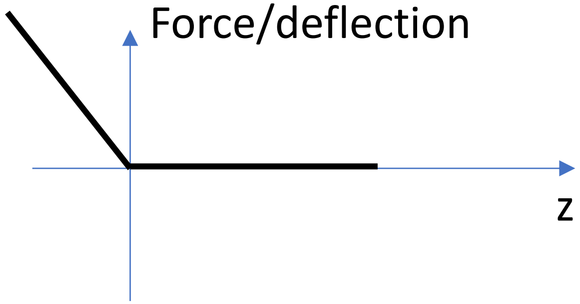

Driving phase
detected phase
90°
110°
Material A
Material B
NCM Phase
110°
90°


Height information
Phase Information
nm
10
7.5
5
2.5
0
5
4
3
2
1
0
µm
0
1
2
3
4
5
µm
nm
0
-25
-50
-75
5
4
3
2
1
0
µm
0
1
2
3
4
5
µm
1000
750
500
250
0
0
250
500
750
1000
nm

Types and modes of Tip based microscopy
Contact modes Non-contact and intermittent contact
Chemical Force microscopy
Force spectroscopy Nanoindentation Magnetic force microscopy Kelvin-Probe microscopy Nano-Lithography
Scanning Near Field optical microscopy (SNOM) Aperture SNOM, non-aperture SNOM, transmission SNOM, Collection SNOM
STM Based methods Scanning Tunnelling Spectroscopy Scanning Tunnelling Electron microscopy (STOM) Ballistic electron emission spectroscopy

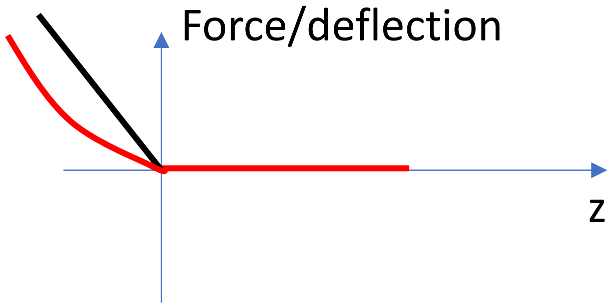
Force Spectroscopy:

Assume an ideal case: Infinitely hard sample and no surface forces

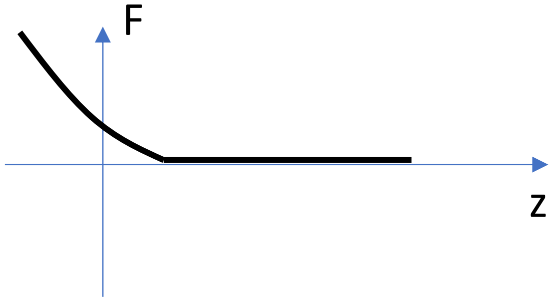


No force till contact!
After contact, there is linear deflection

Soft material no surface forces

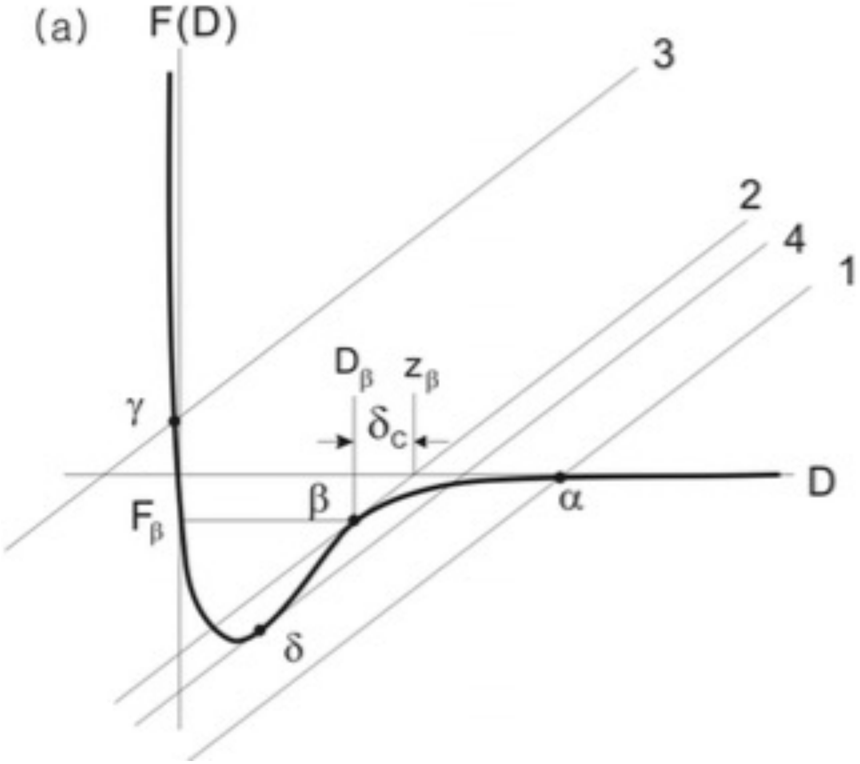


Infinitely hard with repulsive forces



As the tip comes a bit close, the repulsive force takes charge and bends the cantilever up.
This happens even before contact

Force-Displacement Curve

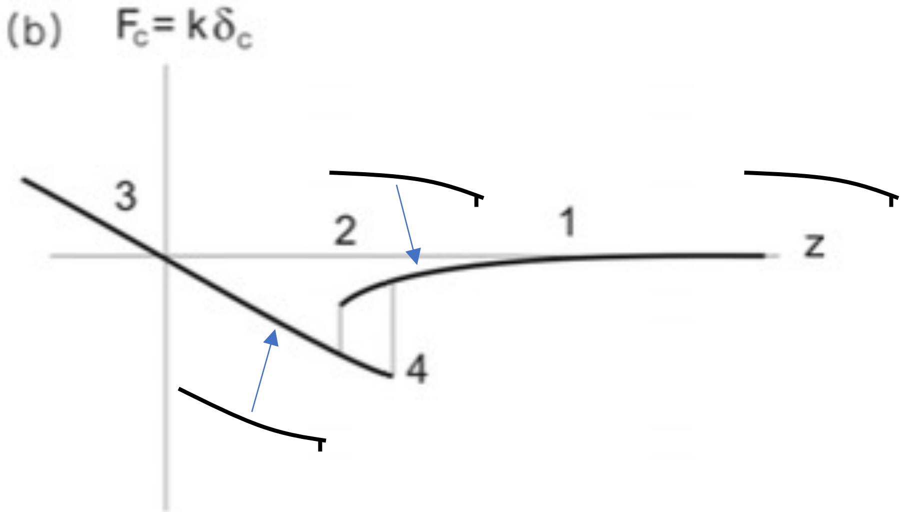


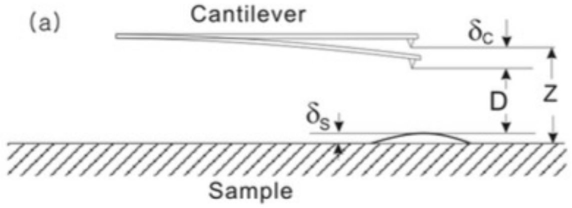
Till point 1, cantilever bends towards the sample
Right after 2, there is jump to contact. The cantilever bends away due to contact. There is onset of repulsive interaction.

Further reduction in z, increases repulsive interaction quickly. Slightly before 3, repulsive force overtakes attractive force. You have displacement opposite direction to force!

Force-Displacement Curve

Force on the cantilever vs the final distance from surface





Many a times, when the tip is retracted,
There is hysteresis.

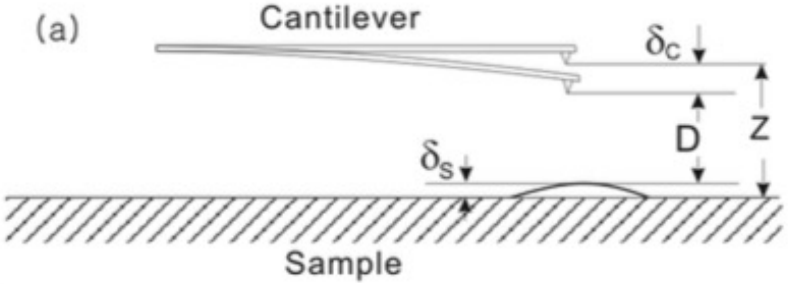
This is due to adhesion, and capillary forces.

The tip takes larger z than the jump to contact

Cantilever Force-Distance Curve

Lines 1- 4 are Hooke's law lines

Corresponding to $F_c = -k(D - z)$

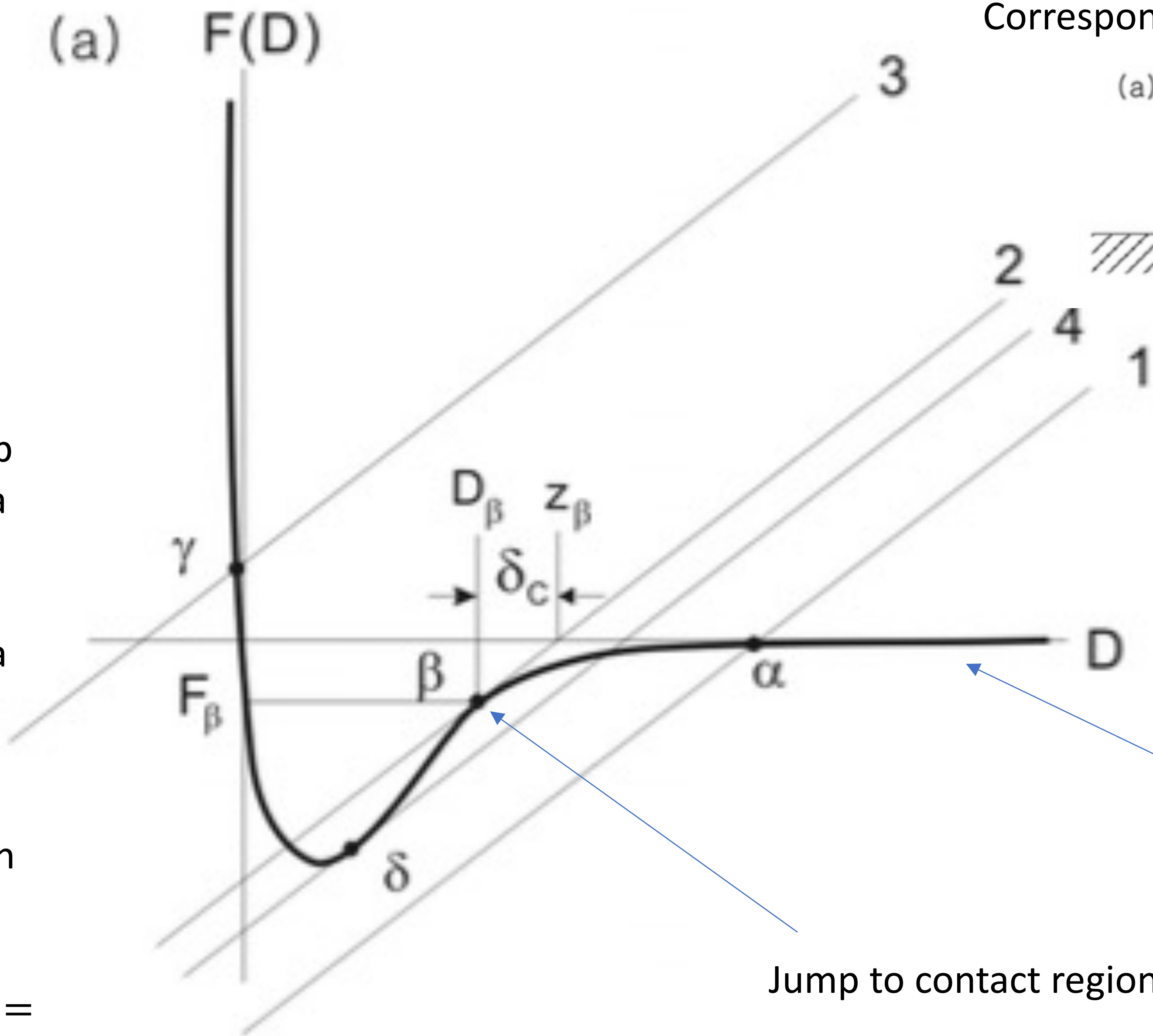


If you have moved the tip such that $D = \beta$, giving a interaction force F_β,

The cantilever will have a spring restoring force.

The cantilever bends up, moving back to a position corresponding to z_β.

The deflection now is $\delta_c = D_\beta - z_\beta$

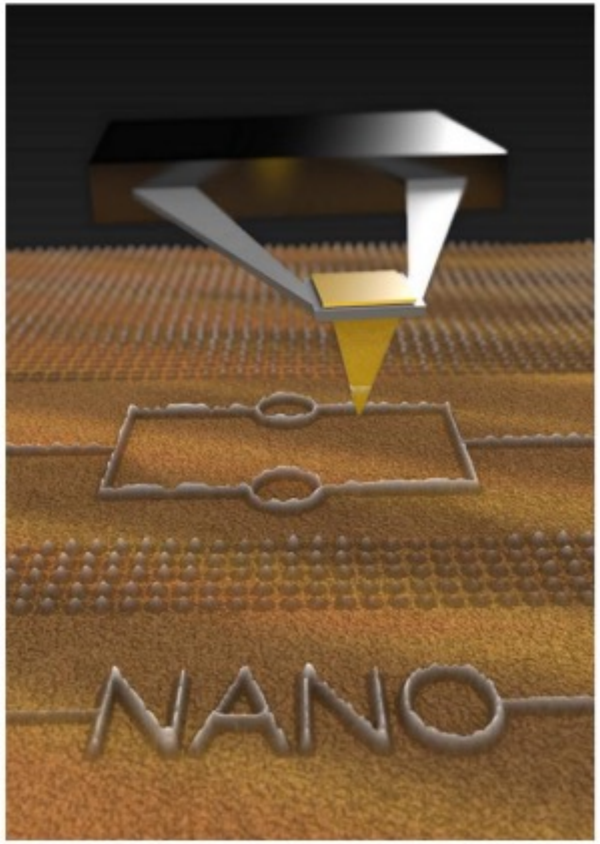


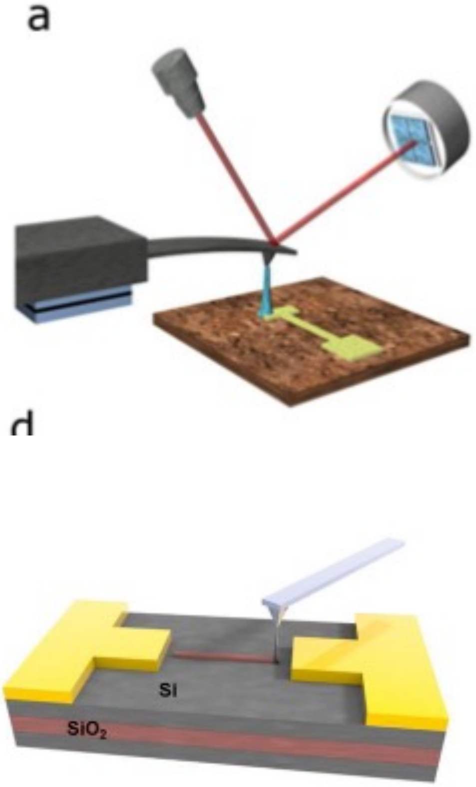
No force, No displacement
$F_c = \delta_c = 0$

Jump to contact region

Rep. Prog. Phys. **71** (2008) 016101

AFM Nanolithography





Lithography is a method of selectively removing material.

Nanoscale lithography is used in many places such as electronic devices, chemical synthesis and such

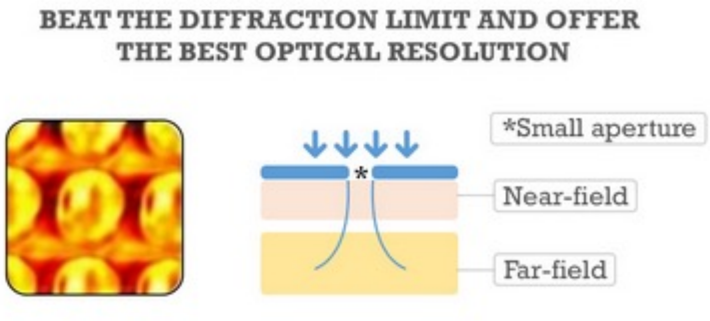
A probe can be used which can supply electric field modifying the properties of certain polymers in nanoscale.

The modified polymers are dissolved in suitable solvents.

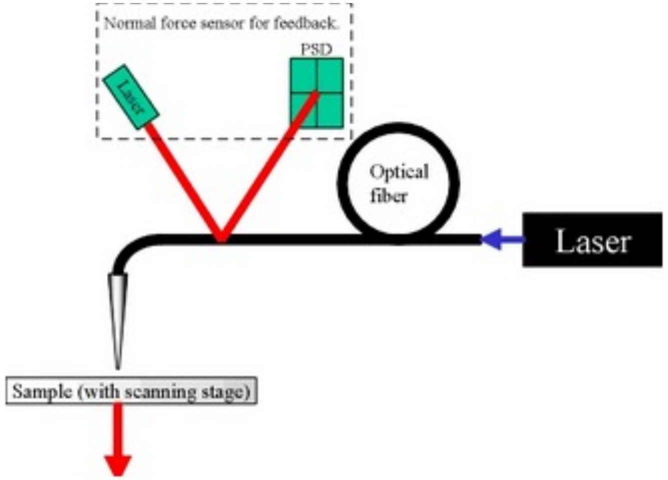
R. Garcia, et al., Advanced scanning probe lithography. Nature Nanotechnology 9, 577–587 (2014); doi:10.1038/nnano.2014.157

Near Field Microscope

Abbe's limit: You can only resolve two features if $d > \frac{\lambda}{2\sin\alpha}$ where d is the distance between points, α half angle of collection


BEAT THE DIFFRACTION LIMIT AND OFFER THE BEST OPTICAL RESOLUTION

So, use a AFM tip to go near field!
Go near the particle and collect the light at smaller wavelengths



A nanoparticle when irradiated with light forms a dipole.

The dipole oscillates with very small wavelength near the nanoparticle (near-field),
Which then diverges (diffracts) to larger wavelengths as you go far (far-field)

A. Lewis et. Al., development of a 500A spatial resolution optical microscope, Ultramicroscopy,1984

End of Atomic Force Microscopic Techniques