Surface analysis
part I: local probe techniques

Lecture 2:

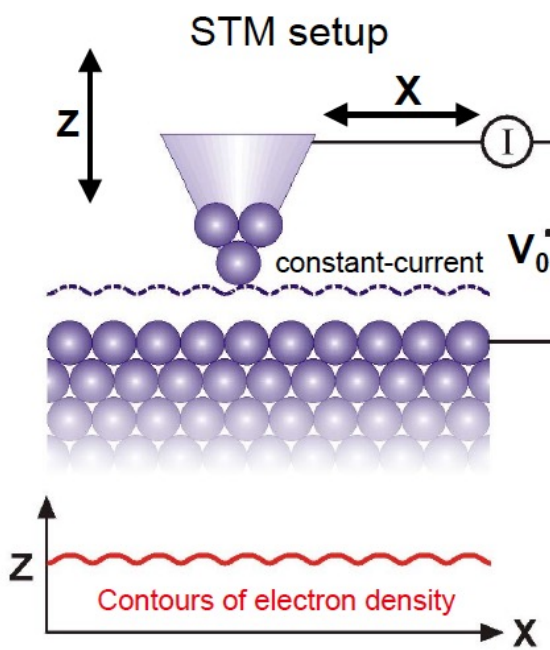
AFM – atomic force microscopy

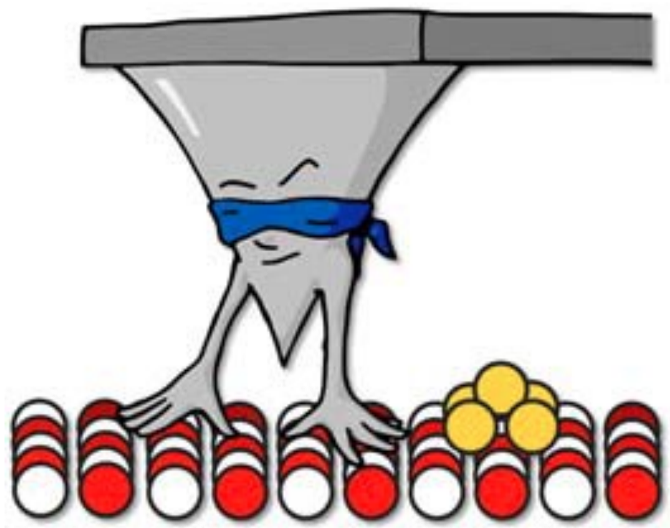
Atomic force microscopy (AFM) vs STM

Both are scanning probe techniques (local probes + scanners with feedback loops)

- STM is usable only for conductive
- STM measures electronic properties of the surface
- STM measures tunnel current

- AFM can be used for any materials
- AFM measures short-range atomic forces (or long range…)
- AFM measures mechanical deflection of a flexible cantilever (atomic forces are sufficient to induce a measurable deflection of a macroscopic cantilever)





Surface analysis, part II: local probe techniques

Lecture 2. Atomic force microscopy

• Atomic force microscopy (more general term – scanning force microscopy): introduction, concept, operation principles

• Operation modes: contact, non-contact, tapping modes…

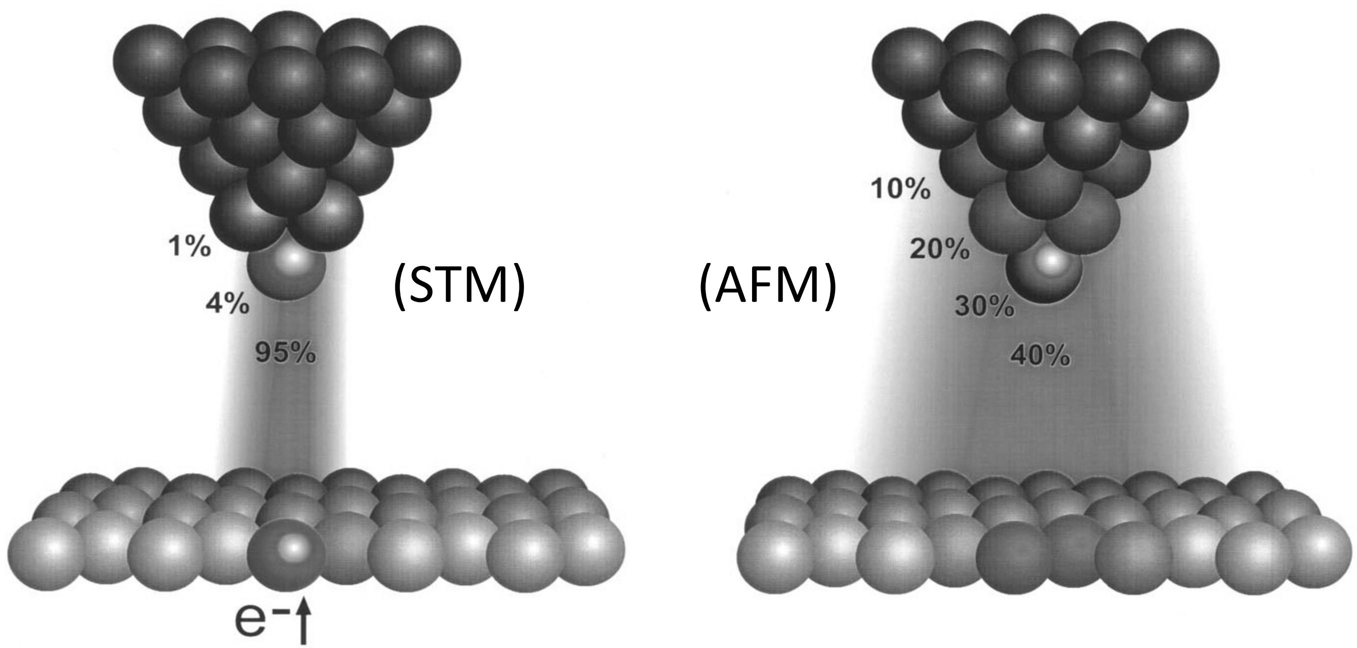
• Contact AFM

- topography analysis
- high resolution studies, vertical and lateral force microscopy
- functional techniques based on contact AFM, just some examples:

conductive AFM

Piezo-force AFM

Although the STM provides sub-angstrom resolution in all three dimensions, it is limited to conductive and semiconducting materials.



An STM gives true atomic resolution. Because the dependence of the tunneling current on the tip-to-sample separation is exponential, only the closest atom on a good STM tip interacts with the closest atom on the sample. For AFMs, the dependence of cantilever deflection on tip-to-sample separation is weaker. The result is that several atoms on the tip interact simultaneously with several atoms on the sample.

Park Scientific Instruments

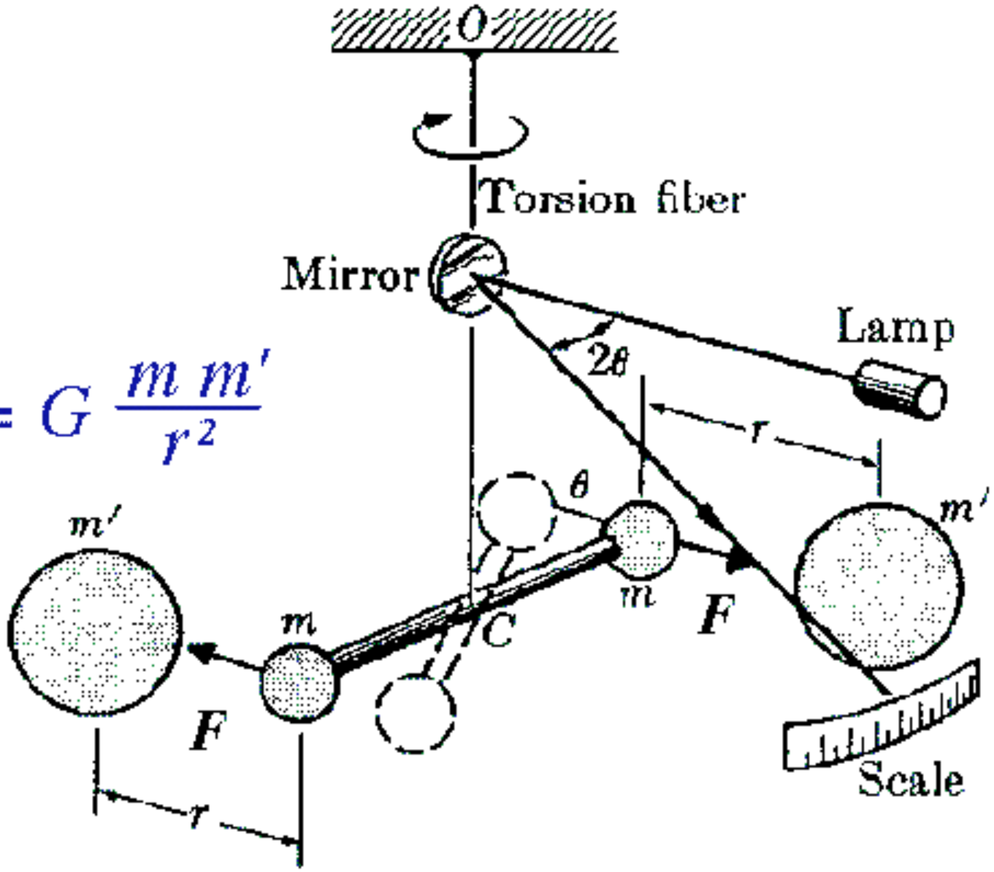
The AFM was invented in 1986 by Binning, Quate, and Gerber when they realized that the forces exerted by the tip on the sample could be used to map the topography of a sample.

Atomic force microscopy (AFM)

The key idea behind AFM technique implies that very small forces (pN to nN) are measurable by detecting mechanical movement of macroscopic objects

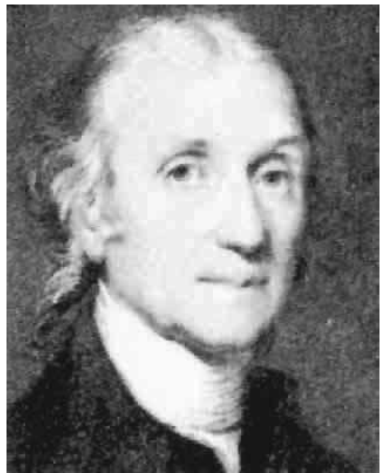
Probably the oldest scientific proof of this concept is the Cavendish Experiment

$$F = G\frac{m\,m'}{r^2}$$

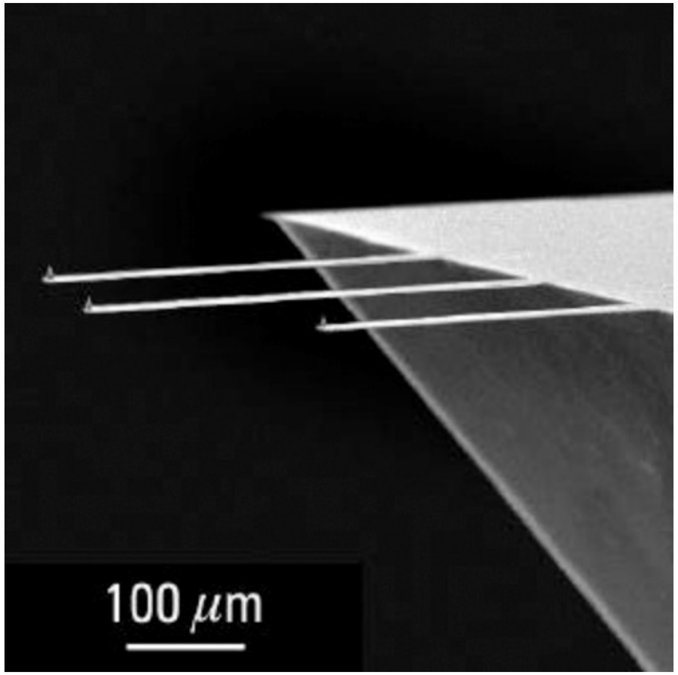


$$G = 6.673 \times 10^{-11}\ \frac{\mathrm{Nm}^2}{\mathrm{kg}^2}$$

In 1798, **Sir Henry Cavendish** determined, for the first time, the constant G



Atomic forces and AFM force sensors



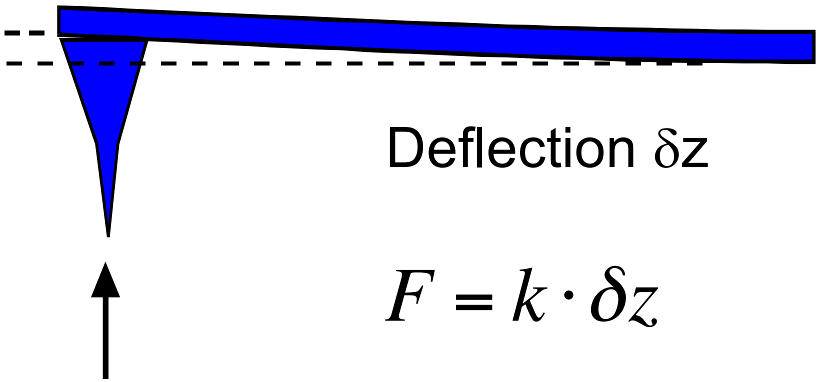
The spring constant of AFM cantilever should be that it would be deflected by the atoms it is mapping.

The vibrational frequency and mass of a typical atom are 10^{13} Hz and 10^{-25} kg giving an interatomic coupling $(C_{at} = k)$ **in solids of 10 N/m.**

Resonant frequency $w_0 = \sqrt{k/m}$

$$c_{at} = w_{at}^2 \cdot m_{at} = 10\ N/m$$

This gives a figure for a cantilever spring constant.



$$F = k \cdot \delta z$$

Typical spring constant range
0.1-10 N/m

Excursion into history: invention of AFM

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- In order to amplify atomic forces Binnig et al. came up with an idea to put the atomic probe (a tip) on a long flexible cantilever (with metal coating)
- The deflection of cantilever was detected by STM
- The resolution was 0.1nm (Z) and 3 nm (XY)
- The system maintained constant tunneling current (hence constant deflection)
- The image represents an isoforce map of the surface
$F=k\Delta z$

Atomic Force Microscope

G. Binnig[a] and C. F. Quate[b]
Edward L. Ginzton Laboratory, Stanford University, Stanford, California 94305

and

Ch. Gerber[c]
IBM San Jose Research Laboratory, San Jose, California 95193

(Received 5 December 1985)

The scanning tunneling microscope is proposed as a method to measure forces as small as 10^{-18} N. As one application for this concept, we introduce a new type of microscope capable of investigating surfaces of insulators on an atomic scale. The atomic force microscope is a combination of the principles of the scanning tunneling microscope and the stylus profilometer. It incorporates a probe that does not damage the surface. Our preliminary results *in air* demonstrate a lateral resolution of 30 Å and a vertical resolution less than 1 Å.

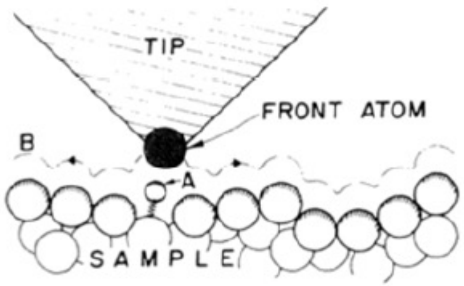


FIG. 1. Description of the principle operation of an STM as well as that of an AFM. The tip follows contour *B*, in one case to keep the tunneling current constant (STM) and in the other to maintain constant force between tip and sample (AFM, sample, and tip either insulating or conducting). The STM itself may probe forces when a periodic force on the adatom *A* varies its position in the gap and modulates the tunneling current in the STM. The force can come from an ac voltage on the tip, or from an externally applied magnetic field for adatoms with a magnetic moment.

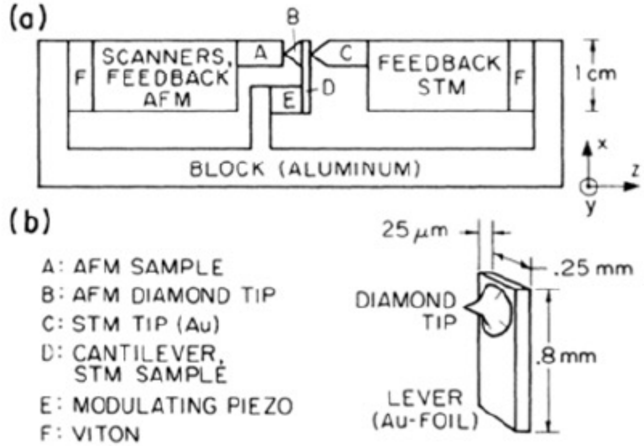
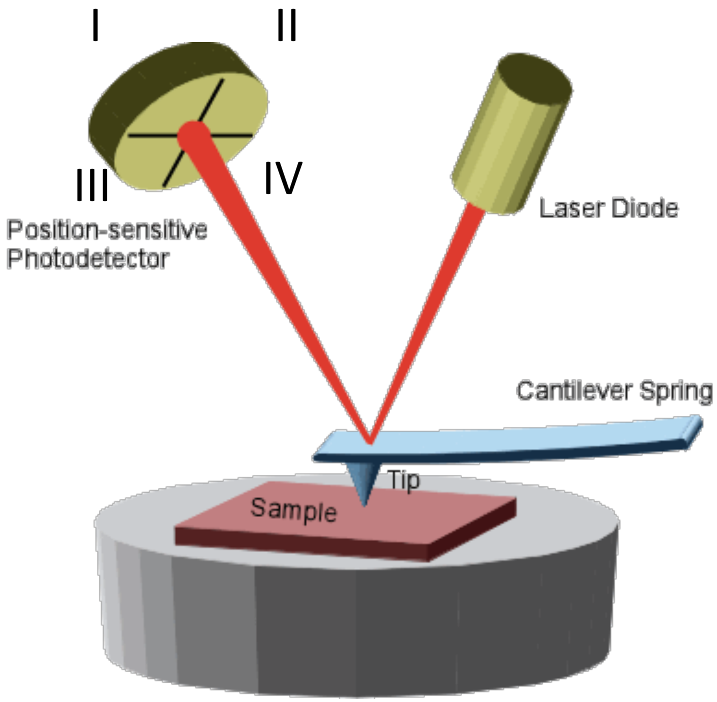


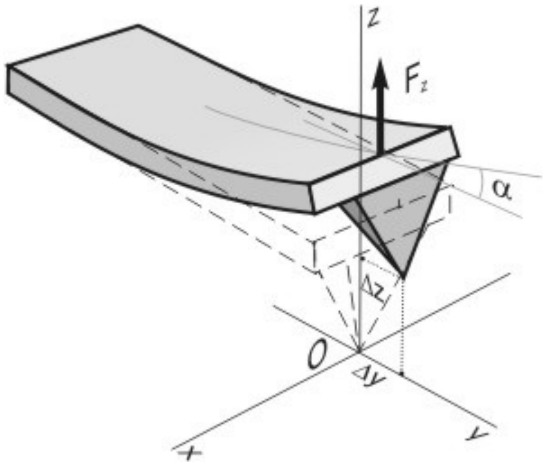
FIG. 2. Experimental setup. The lever is not to scale in (a). Its dimensions are given in (b). The STM and AFM piezoelectric drives are facing each other, sandwiching the diamond tip that is glued to the lever.

Modern AFM concept: deflection detected with laser

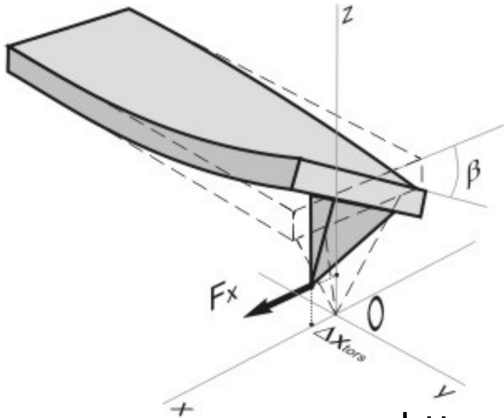
4-quadrant photodiode



Deflection:
(I + II) - (III + IV)



Torsion:
(I + III) - (II + IV)



- In modern AFM the deflection is monitored by laser reflected from the back surface of the cantilever. The reflected intensity is measured by 4-quadrant photodiode

- The movement of cantilever is amplified by placing photodiode far (cm range) from the cantilever

- Vertical (deflection) and lateral (torsion) movement of the cantilever can be measured separately (vertical and lateral force microscopy)

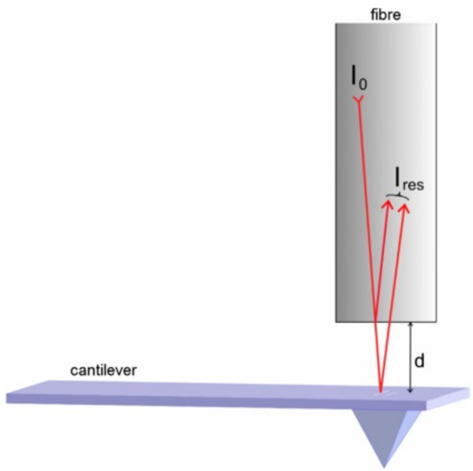
https://www.youtube.com/watch?v=jRAqhFdwt20

Detection of cantilever movement: interferometer

• In some special cases (like cryogenic AFM) when space is limited a very compact interferometer-based solution is used

• The laser light is conducted to the cantilever via the optical fiber, the cantilever is positioned close (some tens of microns) to the optical fiber edge

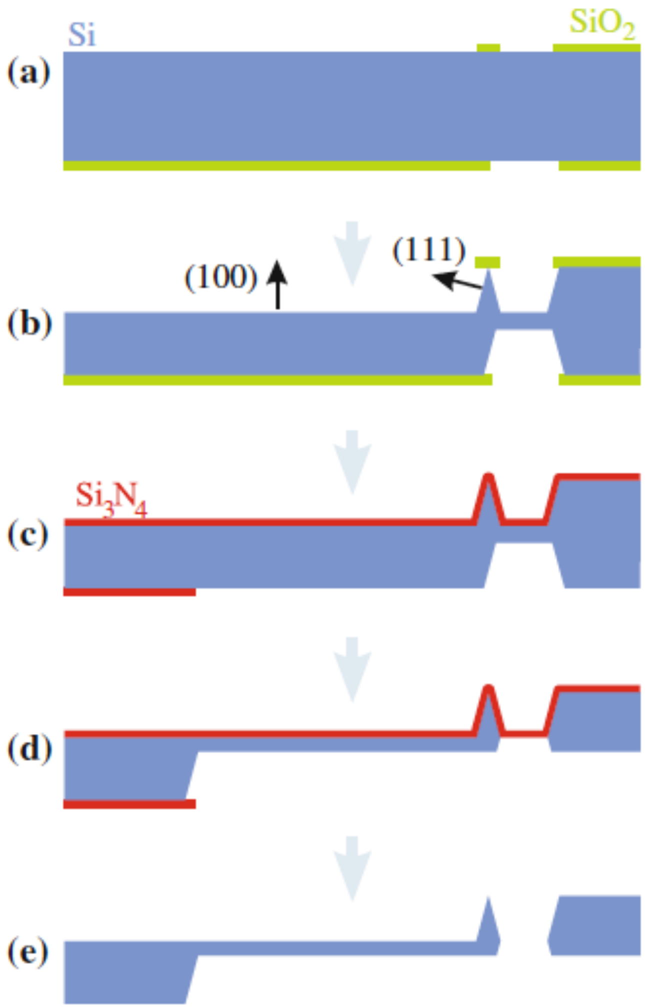
• Due to the interference between the beams reflected from the fiber edge and cantilever surface the measured light intensity is a periodic function of tip height – from Max to Min – $\lambda/4$

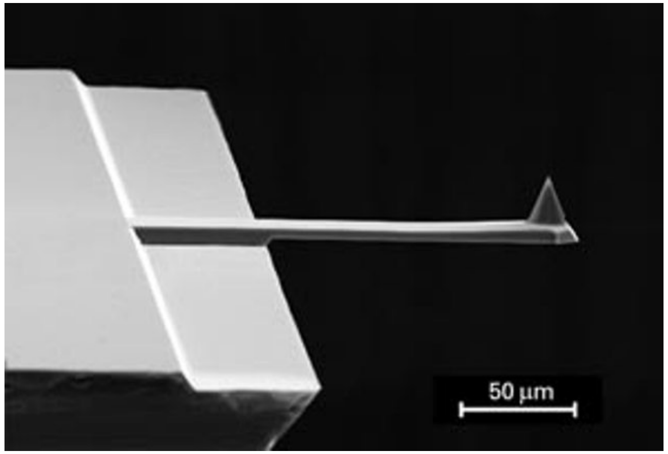


• cantilever deflection is detected by measuring the interference signal change with *pm* resolution

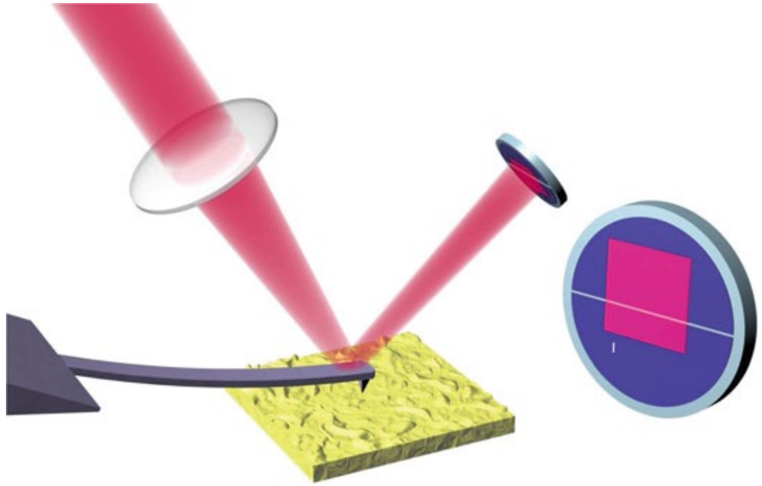
- Advantages :
  - deflection can be easily quantified
  - very compact arrangement suitable for UHV, cryo-systems
- Disadvantages :
  - Complicated and sometimes difficult to align
  - Lateral force detection is problematic (almost impossible)

Microfabrication of a Si cantilever using alternating lithographic patterning and wet chemical etching



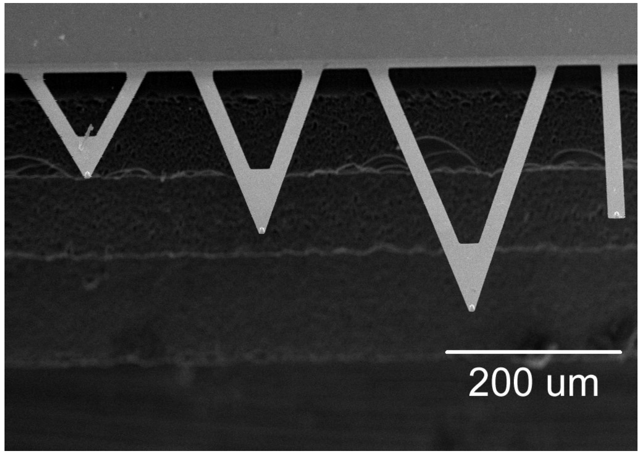


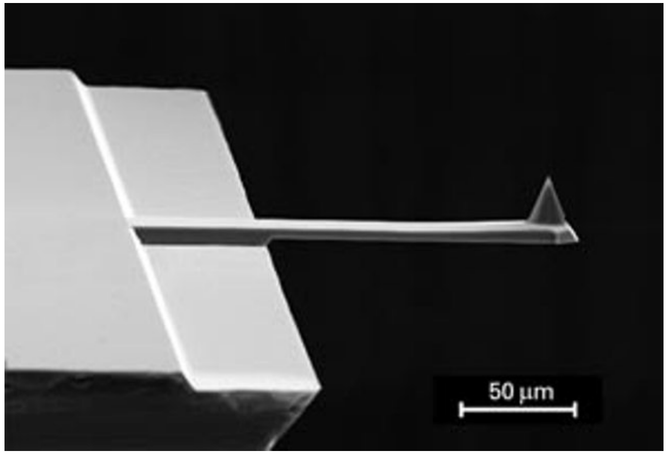
Resonant frequency $w_0 = \sqrt{k/m}$

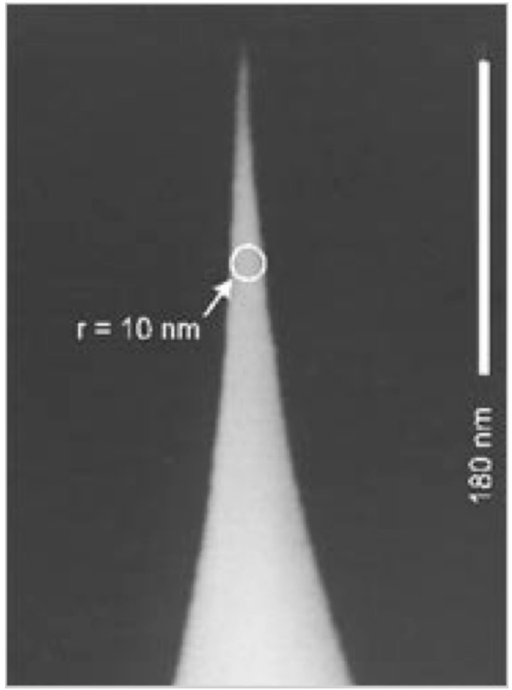


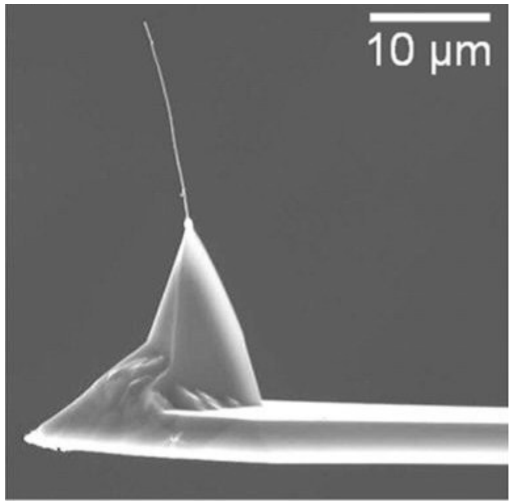
Reflecting back side is important

Cantilevers









• Unlike STM tips AFM cantilevers require micro fabrication facilities, the tip is typically prepared using lithography process

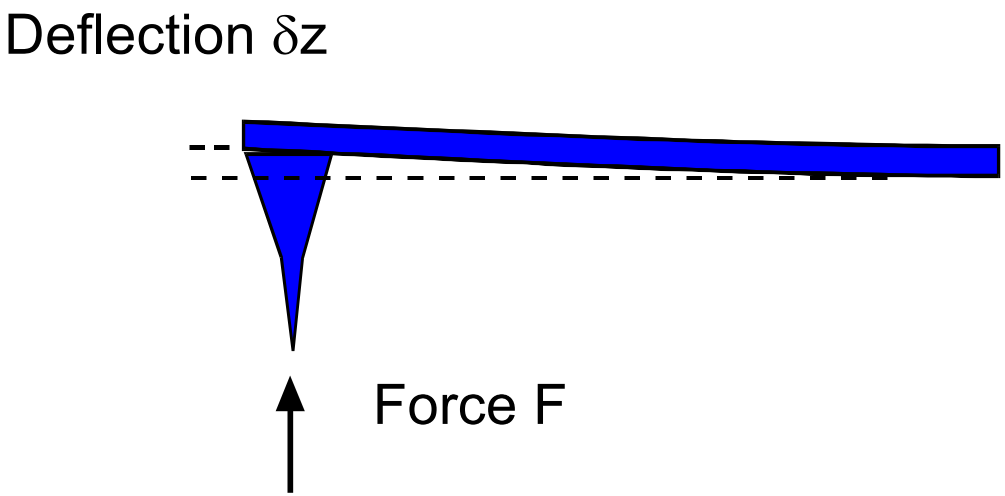
• The material typically used is Si or SiN, the tip curvature radius can be R < 10 nm

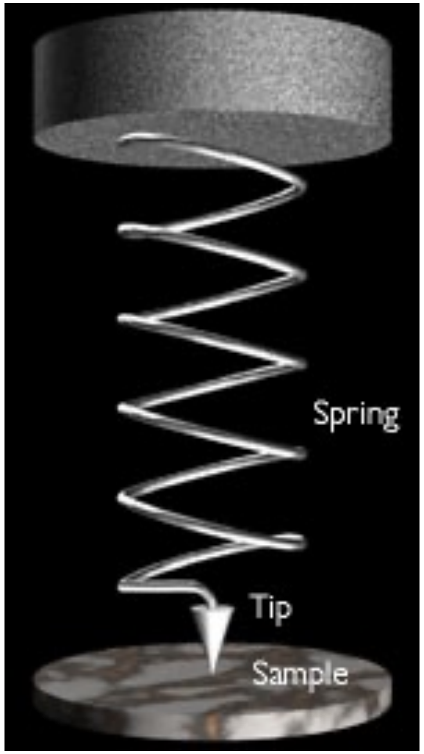
• The back side can have reflective coating for stronger reflected signal

• There is a broad range of solution for tip functionalization for special measurements:

- conductive coating (Pt, Au, Ti),
- magnetic coating (Co),
- hard coating (diamond)
- nanotubes for special applications
- chemical functionalization (tips terminated with molecules of special type for probing chemical forces and other interactions)

Cantilevers for AFM force sensors



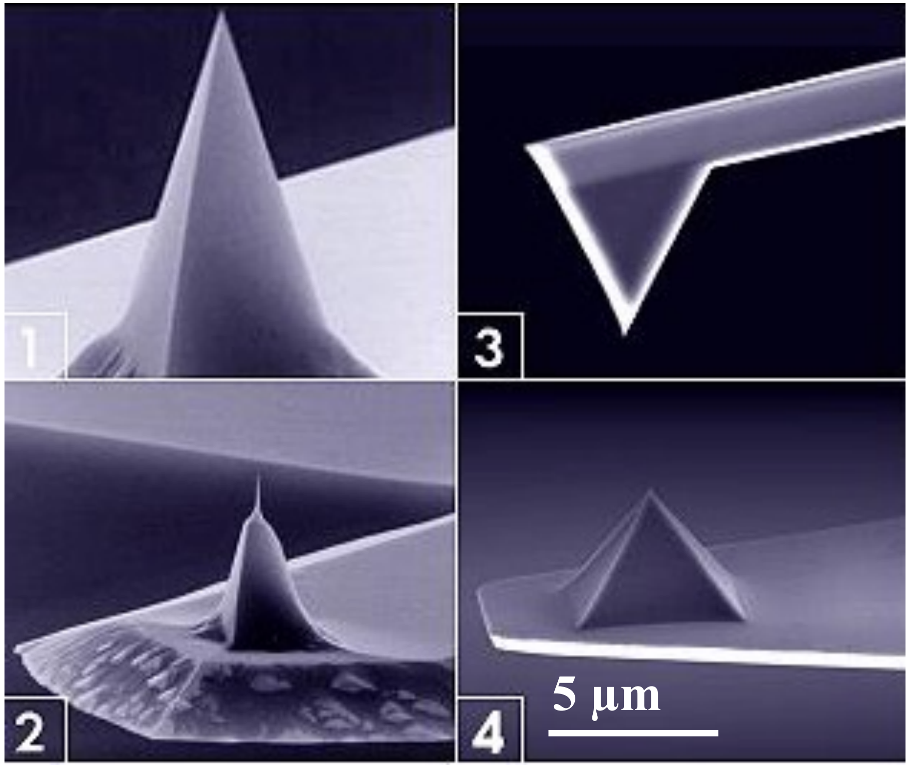


The force sensor is a straightforward application of Hooke's Law: $$F = k \cdot \delta z$$

A typical value for k is 10 N/m (can be within a range of 0.1 -50 N/m)

Loading force range: 0.1 to 10 nN

Cantilevers for AFM force sensors



Resonant frequency $w_0 = \sqrt{k/m}$

Resonant frequency range: 30-200 kHz

Cantilever made of Al foil (4x1x0.01 mm^3) would have a spring constant of 10 N/m. However, its resonant frequency will be only 1 kHz (too low)

For a high resonant frequency cantilevers are microfabricated from Si, Si_3N_4

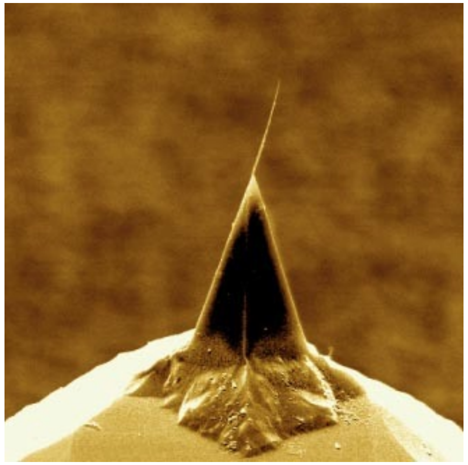
Typical cantilever dimensions:

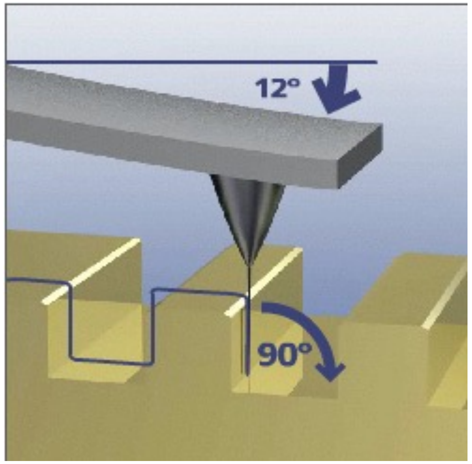
- Width 20-50 µm
- Length: 50-400 µm
- Thickness: 1 µm

Where to get probes? Each supplier has online shop e.g. https://www.brukerafmprobes.com

Carbon Nanotube Tips for extreme topography imaging

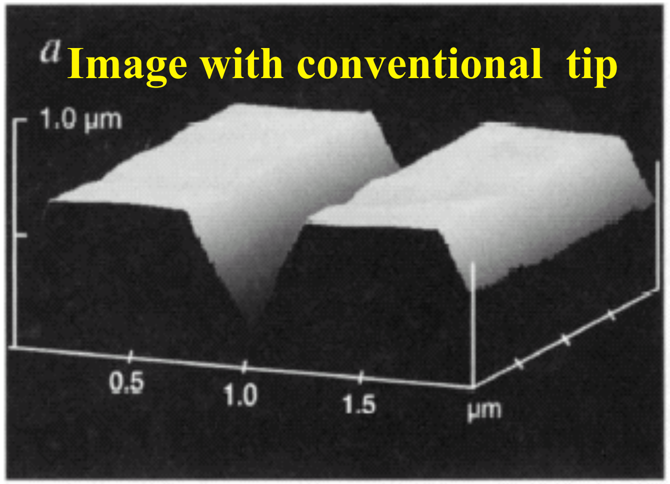
AFM tip with attached CNT (10 nm in diameter)

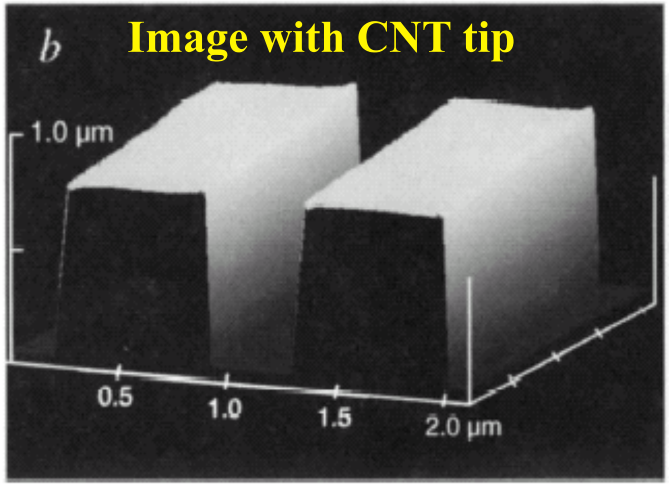




- **Provide high resolution**
- **Show little evidence of wear**
- **Promising for critical dimension metrology of semiconductors, and nanobiological investigations**

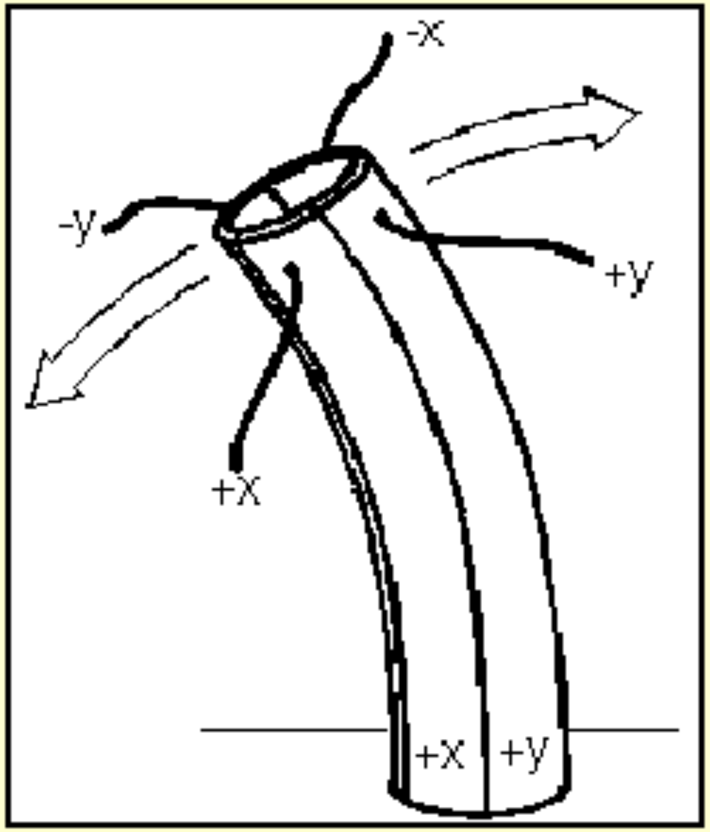
Tip geometry effect on imaging



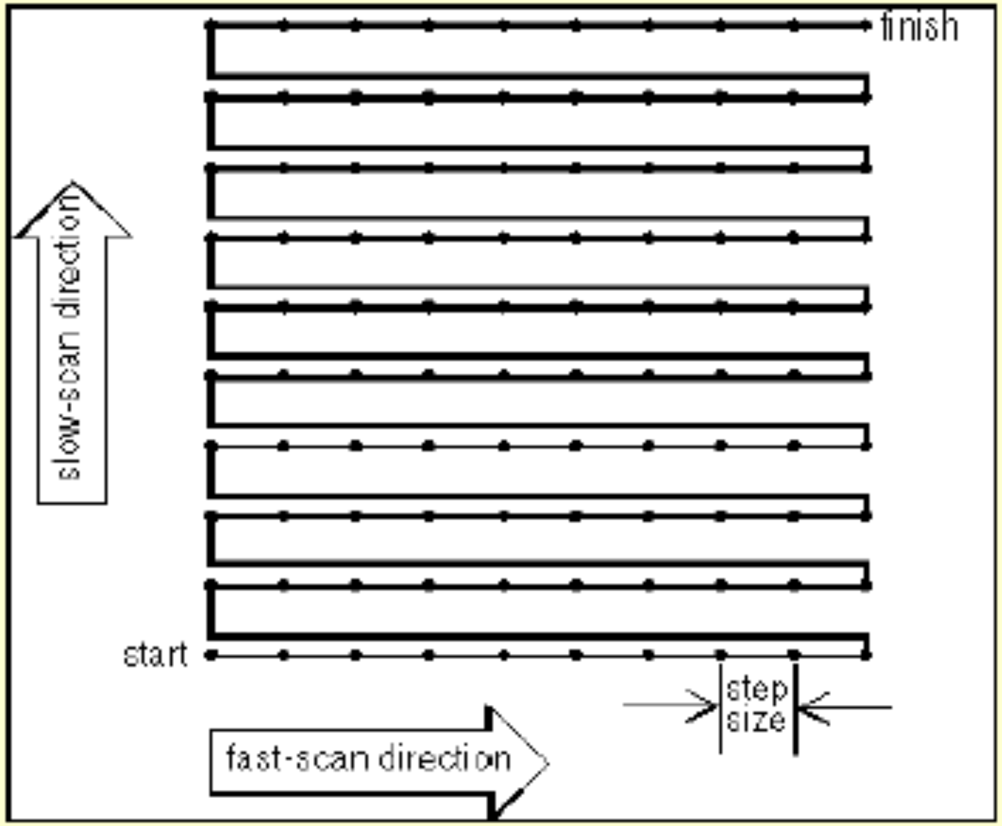


Lateral resolution of 2 nm can be achieved (~10 nm for conventional Si tip)

AFM imaging: raster scan using piezoelectric tube



Piezoelectric tube
(for details see the lecture on STM)



Raster scan

Like in STM AFM image is collected by raster scan using piezoelectric tube. There is a fast scan direction (line scan) and slow direction (step between lines). Choice of scanning direction matters!

The resolution is formally defined by scanning area and density of points (area can be vary from tens of microns to some angstroms, the number of points is typically some 200x200 to 1000x1000 dots). In reality the resolution obviously depends on the tip radius, sample surface and roughness, stability of the environment and other factors

AFM imaging : feedback loop

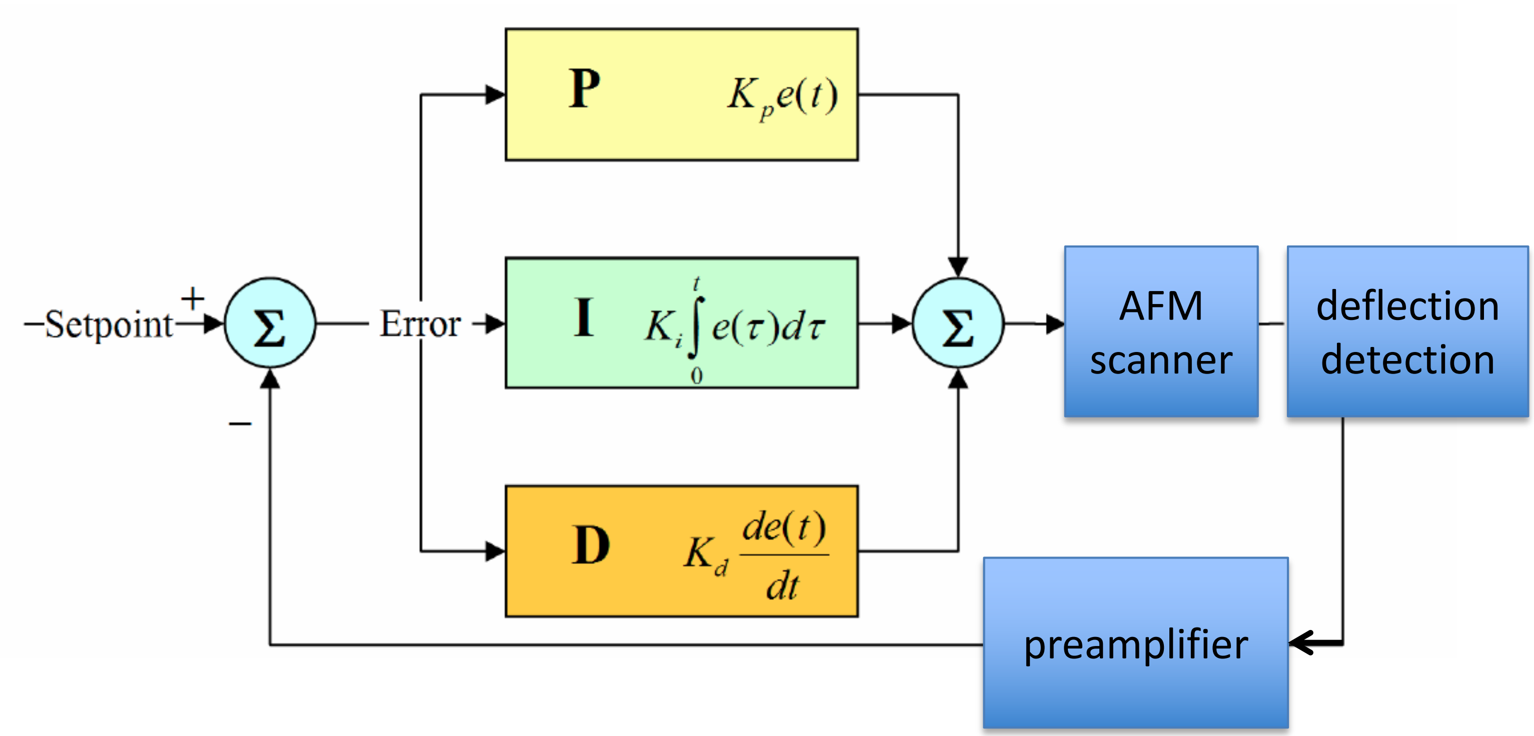
• AFM uses feedback loop similar to STM.
• In case of STM in constant current mode there is a regulator that vertically adjusts the tip position in order to keep the tunneling current at a preset value (setpoint).

• In case of contact AFM the setpoint is a deflection value (or voltage value measured from photodetector)

The feedback loop regulation uses PID (proportional-integral-differential) amplifier. The system constantly measures deflection and compares it with the reference value (setpoint). The difference is error signal that is used as input for for PID controller.

This error signal is properly amplified by PID unit and finally transmitted back to the piezoscanner in order to adjust its position in a way to approach desired setpoint.

As usually proper adjustment of the gain coefficients is essential for getting correct results



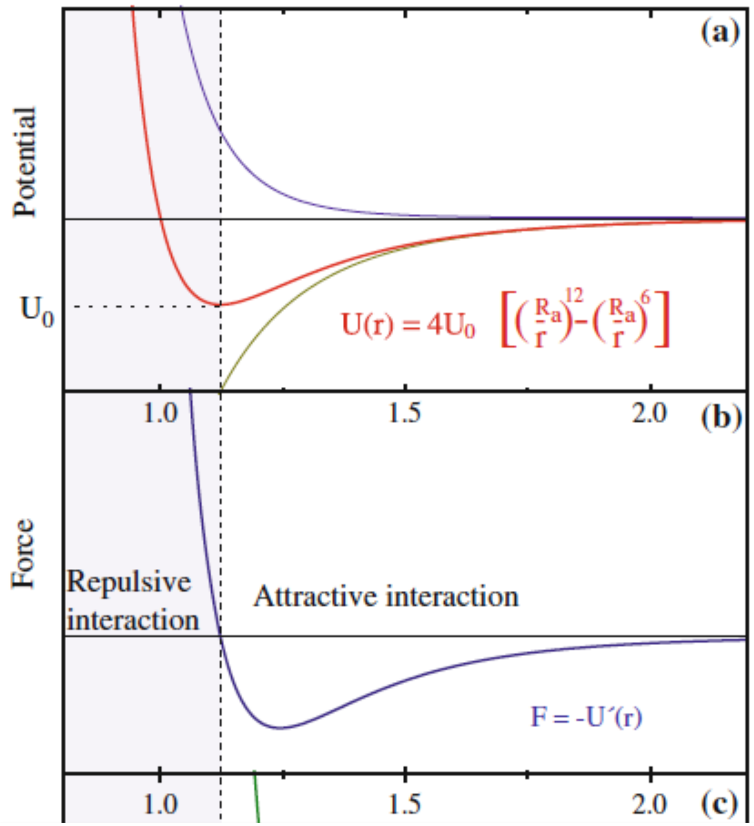
AFM imaging : relevant forces

• Unlike STM AFM can use a great variety of tip-sample interactions.

• ***Short-range forces*** arise from overlap of electron wave functions and from repulsion of the ion cores. Therefore their range is comparable with extension of wavefunctions (angstroms). Short range forces can be either attractive or repulsive. The attractive forces occur when the overlap of electronic wave functions reduce the total energy (like molecular binding). On the other hand the Pauli exclusion principle can lead to repulsive forces due to strong orbital overlap. The ionic repulsion acts over small distances where the screening of the ion cores by electrons falls away.

• Lennard – Jones potential (tip-sample)

$$U_{\mathrm{LJ}}(r) = 4U_0\left[\left(\frac{R_a}{r}\right)^{12} - \left(\frac{R_a}{r}\right)^{6}\right]$$



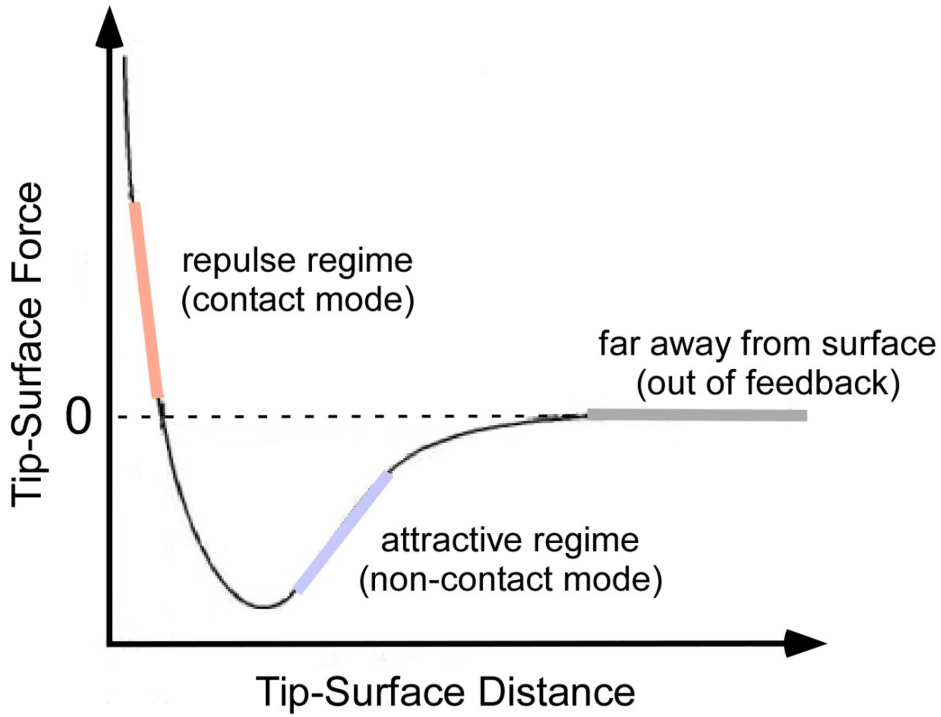
Normalized distance

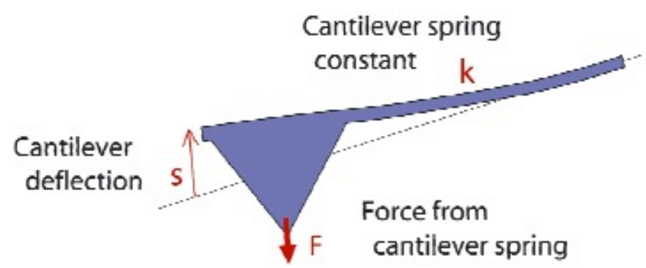
AFM imaging : relevant forces

• Unlike STM AFM can use a great variety of tip-sample interactions.

• ***Short-range forces*** arise from overlap of electron wave functions and from repulsion of the ion cores. Therefore their range is comparable with extension of wavefunctions (angstroms). Short range forces can be either attractive or repulsive. The attractive forces occur when the overlap of electronic wave functions reduce the total energy (like molecular binding). On the other hand the Pauli exclusion principle can lead to repulsive forces due to strong orbital overlap. The ionic repulsion acts over small distances where the screening of the ion cores by electrons falls away.

• In case of ***contact AFM*** (static measurement of deflection of the cantilever) the position of the tip is given by equilibrium of attractive and repulsive forces + external force due to cantilever spring constant. The deflection of the cantilever is used as a feedback signal (to generate an error signal)





Cantilevers can be thought of as springs where the Hooke's law applies:
F=-k*x, where x is displacement.
For practically used cantilevers ***k*** is within the range 0.01 – 40 N/m

AFM imaging : relevant forces

- ***Van der Waals forces*** are electrostatic forces between dynamic dipoles. They always present and attract even chemically inert noble gas atoms. This interaction is induced by temporary fluctuating dipoles that form even in non-polar molecules due to fluctuations.

A simple model (sphere tip apex, infinite surface) can be used for evaluation purposes:

$\boldsymbol{F_{VdW}=HR/6D^2}$, where $\boldsymbol{D}$ is the tip-sample distance,
$\boldsymbol{R}$ is tip radius and $\boldsymbol{H}$ is Hamaker constant ~ 10^{-19}J.

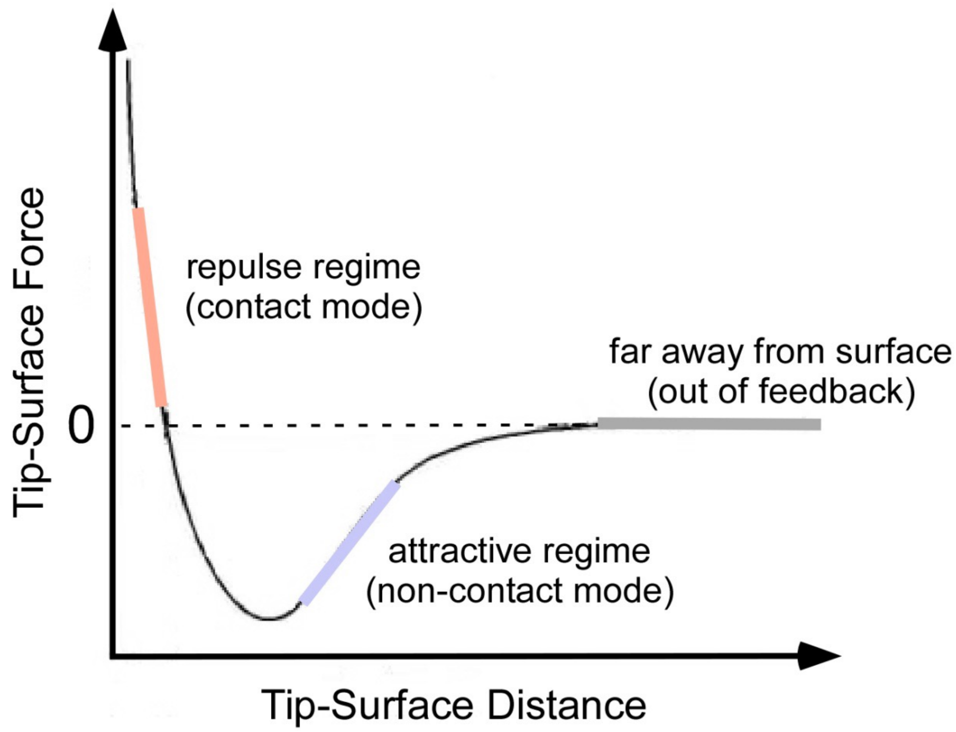
For R=30nm and D=0.5 nm in vacuum $\boldsymbol{F_{VdW} \sim 2nN}$

The distance range where Van der Waals forces dominate is usable for ***Non-contact AFM***

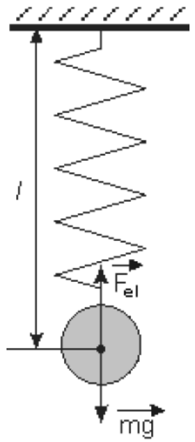
- ***Electrostatics forces*** (long-range, attractive or repulsive) will be discussed later

- ***Capillary forces*** attractive forces that influence tip behavior when measuring in air or liquid (will be discussed later)

- ***Magnetic forces (long-range)*** influence tip with magnetic coating when magnetic sample is Measured – will be discussed later

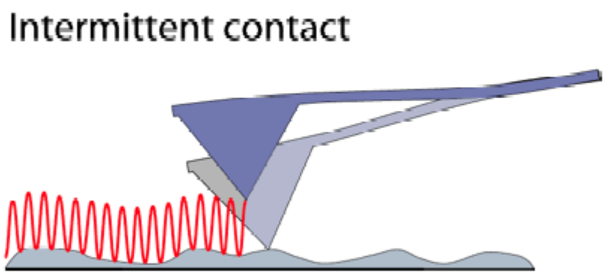


Dynamic imaging modes

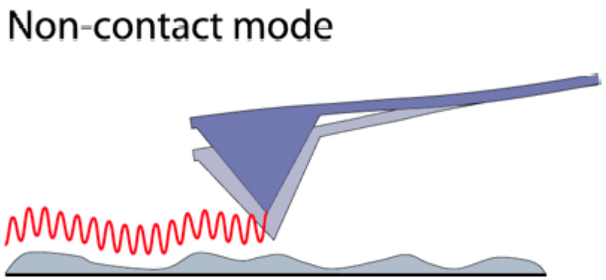


• Dynamic modes rely on oscillation of cantilever close to the surface

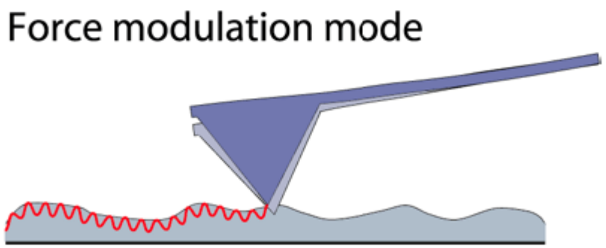
• cantilever can be considered as a spring characterized by a resonance frequency like a classic spring pendulum oscillating at f_0
$f_0 = 1/2\pi * (k/m)^{1/2}$, where ***k*** is spring constant and ***m*** is effective mass



• ***Non-contact mode***: cantilever oscillates close to the surface without touching it. Because of the tip-sample interaction (attractive van der Waals forces) the resonance frequency and amplitude changes. Unlike contact mode where the static deflection is monitored, here the monitored parameters are amplitude or frequency of vibration.
This mode is used for atomic resolution in ultra-high vacuum

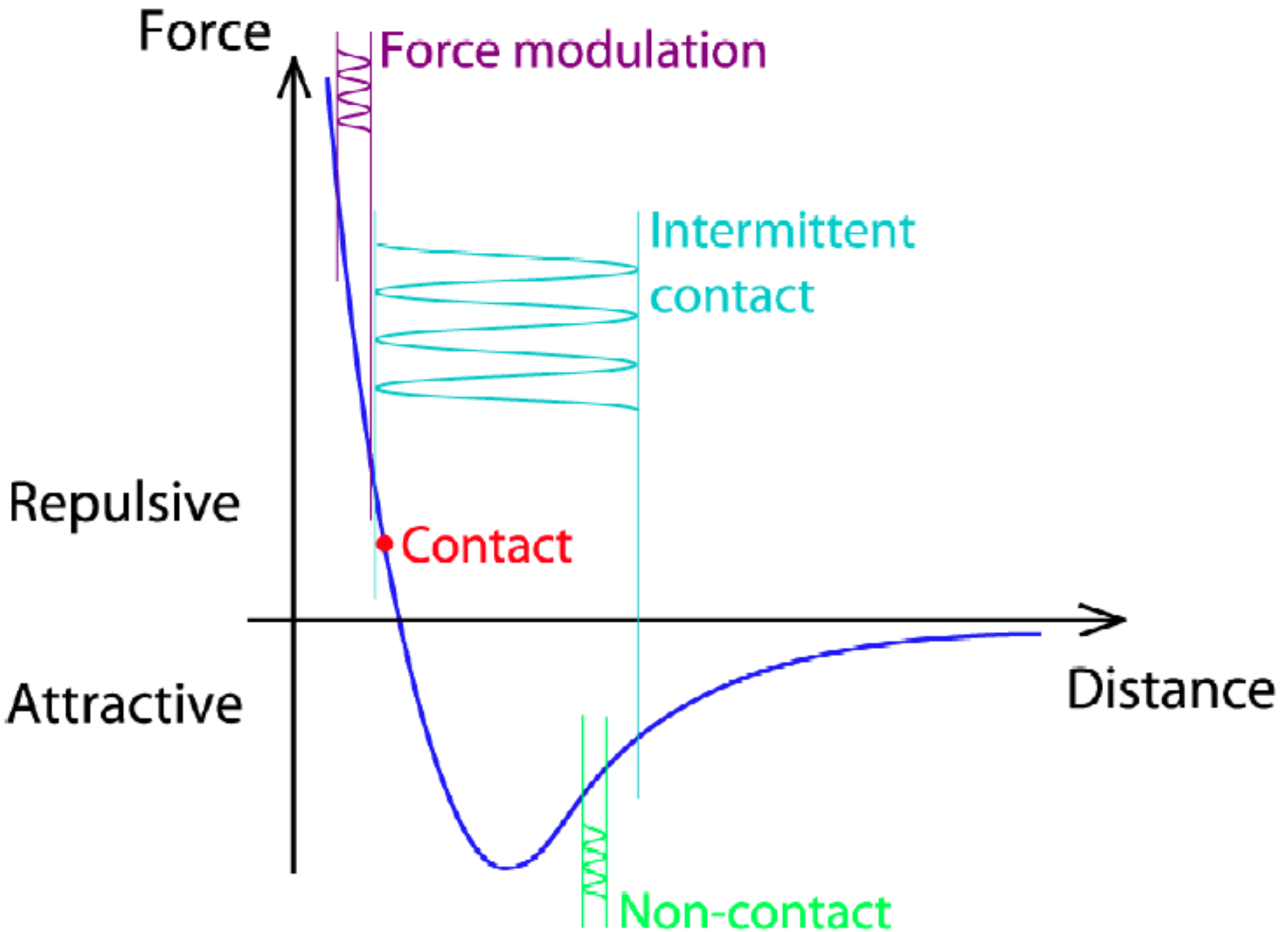


• ***Intermittent contact mode (tapping mode)***: cantilever oscillates and tip makes repulsive contact with the surface at the lowest point. This technique representing a combination of contact and non-contact mode is often used for imaging in air
Feedback loop: oscillation amplitude



• ***Force modulation mode***: tip oscillates but does no leave the surface. This technique maps the elastic properties of the sample

Dynamic imaging modes and forces vs. distance



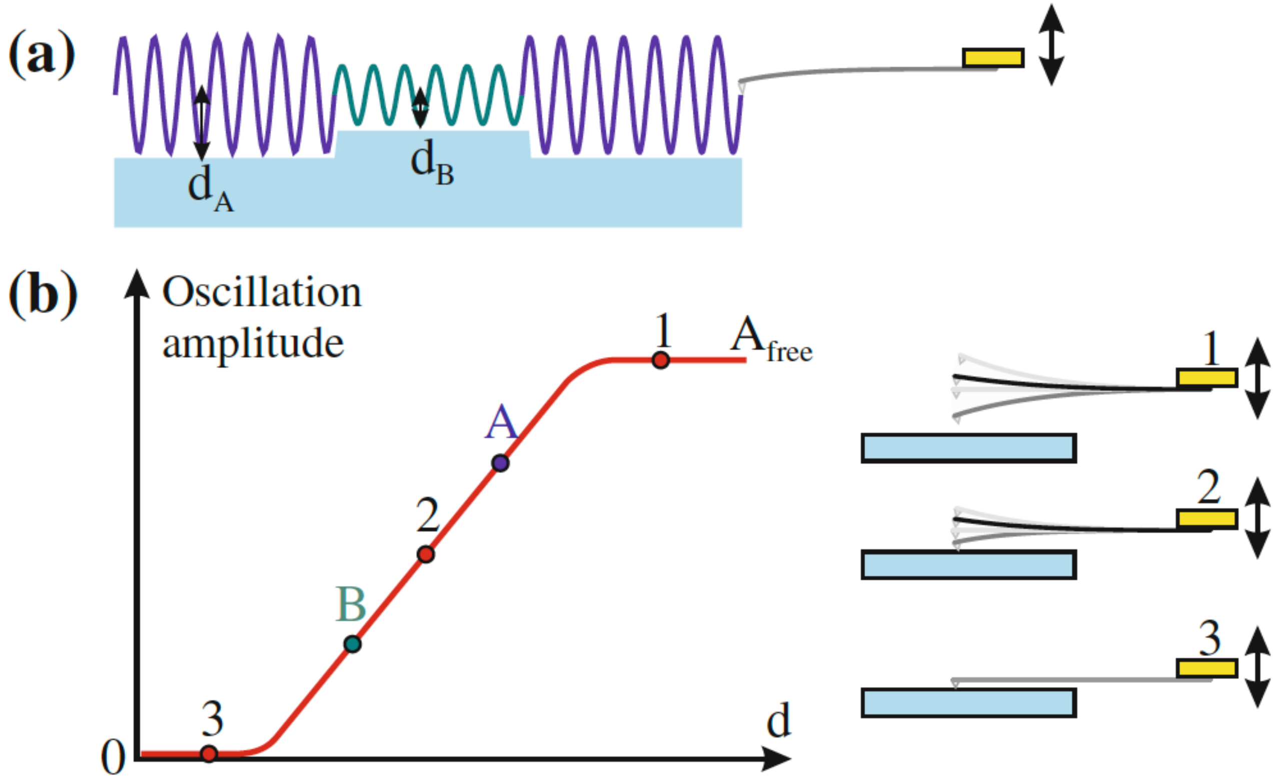
- ***contact mode***: stationary tip remains at close distance in the range where repulsive forces dominate.

- ***force modulation mode***: oscillating tip remains at close distance in the range where repulsive forces dominate.

- ***Intermittent contact mode (tapping mode)***: the tip moves between the attractive and repulsive parts of the curve

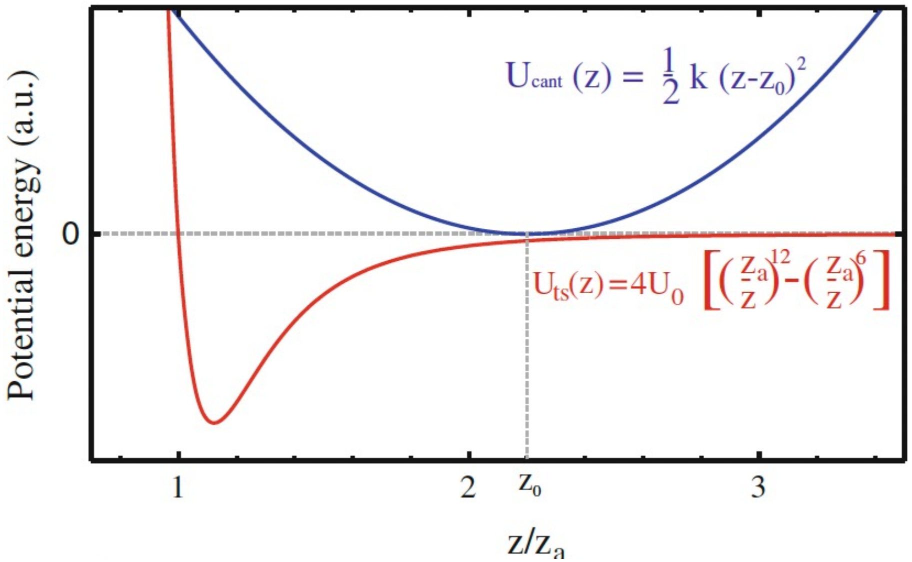
- ***Non-contact mode***: the tip oscillates within the attractive part of the curve without entering in contact with the sample (typically used in vacuum)

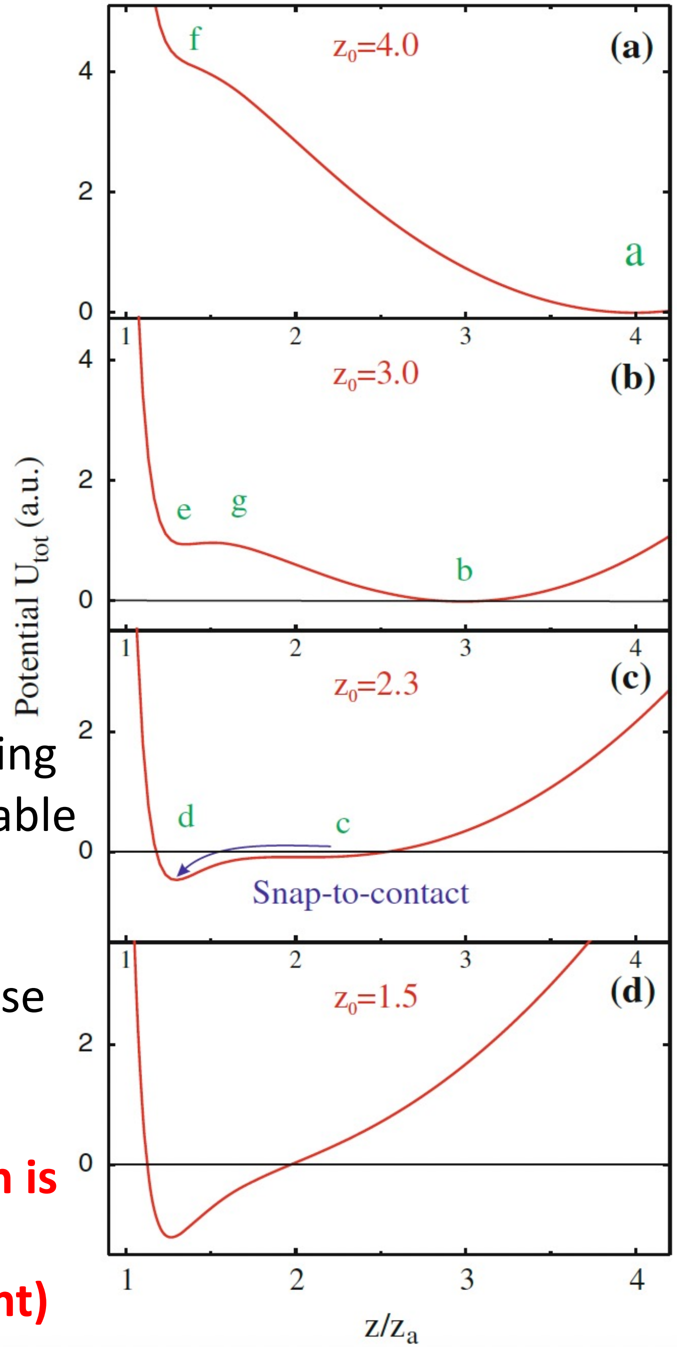
Intermittent contact mode (tapping mode): amplitude vs distance



Here the oscillation traces for two different average tip-sample distances, when operation is performed in constant height mode, i.e. without feedback.
It was found experimentally and also from simulations that the oscillation amplitude as reduced approximately linearly when decreasing the average tip-sample distance d, once the oscillation path reaches the repulsive regime
In tapping mode detection, a certain amplitude A is chosen as the amplitude setpoint for the z-feedback

“Snap-to-contact” : behavior of cantilever near the surface





-If the cantilever tip is far from the surface (corresponding to large values of z0), the spring potential provides a stable potential minimum at z ≈ z0

- If the cantilever comes closer to the surface, the potential minimum close to z0 vanishes, then the cantilever tip will find a new stable minimum close to the sample surface

This abrupt jump of the cantilever equilibrium position is called snap-to-contact

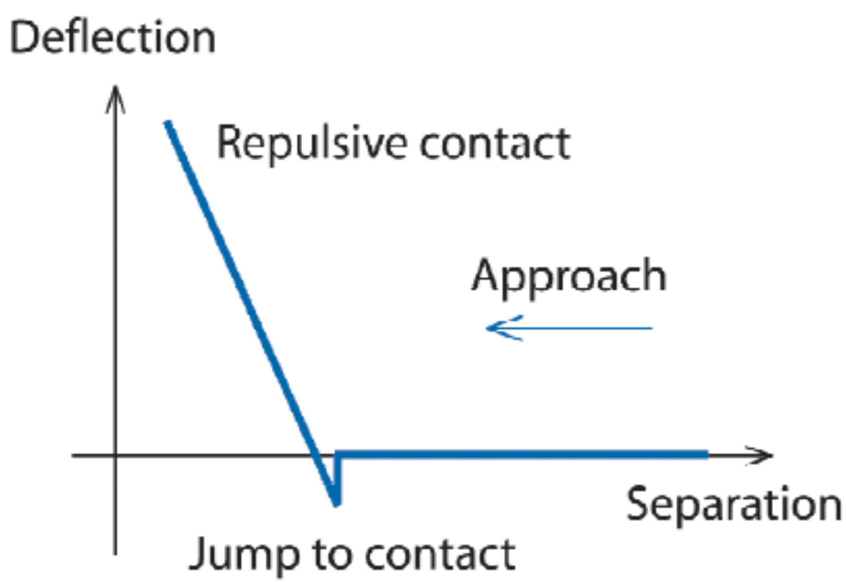
How to avoid? Large Z_0 distance, high K (spring constant)

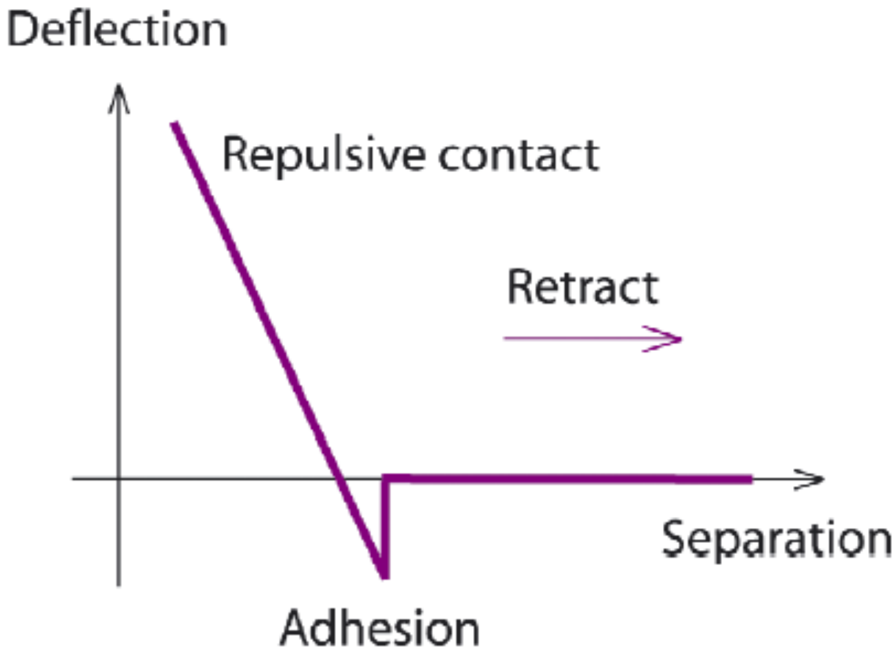
“Snap-to-contact” : contact vs tapping mode

- *In the contact mode of AFM, the measurements are performed with the tip snapped into contact, i.e. in a regime in which the repulsive tip-sample interaction prevents any further approach toward the surface*

- *In dynamic AFM measurements (with an oscillating cantilever) snap-to-contact would stop the oscillation due to the very narrow potential minimum close to the surface – this should be avoided*

Interaction between AFM probe and surface vs. distance: force-distance curve



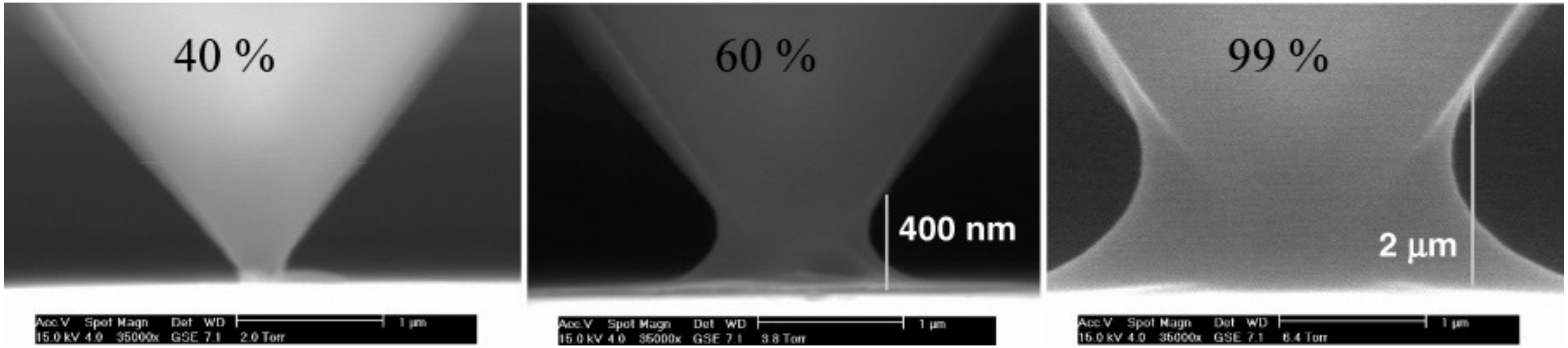


• In this example a cantilever tip approaches to a hard incompressible surface. When the tip is too far the attraction forces are weak and cantilever remains in undisturbed position. At some point attractive forces (e.g. Van der Waals forces) overcome the cantilever spring constant and the tip ***jumps*** into contact with surface (deflection downwards).

Once the tip is in contact it remains on the surface while the cantilever continues to approach. The deflection increases with the distance decreasing.

• As the cantilever is retracted from the surface, often the tip remains in contact with the surface due to some adhesion and the cantilever is deflected downwards. At some point the force from the cantilever is sufficient to overcome adhesion, and the tip brakes free

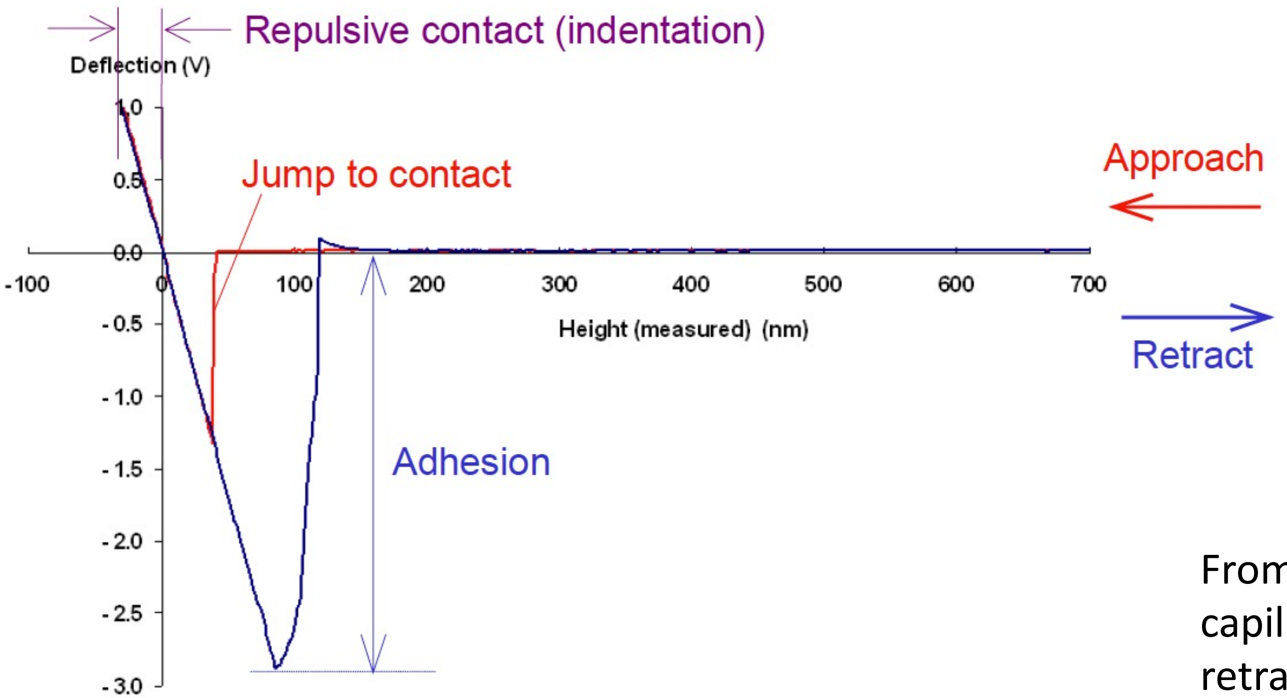
Contact between AFM probe and surface in air: role of humidity

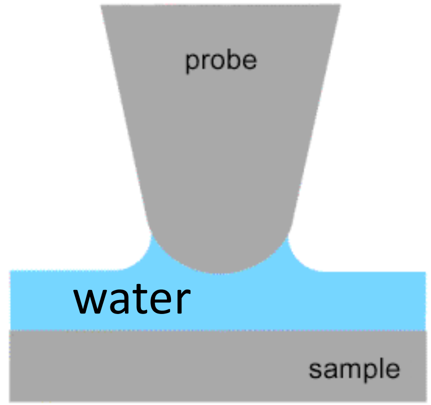


Hydrophilic surfaces are covered with a thin water layer in ambient conditions
These layers join when the tip and and sample are close together, forming a capillary neck between them (and hence induce a strong adhesion)
SEM imaging show that the size of the water meniscus can be very big and may have a complicated hysteretic dependence on the relative humidity

Weeks et al., Langmuir 2005, 21, 8096-8098

AFM in air: water meniscus and hysteretic behavior of force-distance curve





Typical interaction for an uncoated hydrophilic cantilever in air approaching a hard hydrophilic surface (like glass). Hydrophilic surfaces are covered with a thin water layer in ambient conditions These layers join when the tip and and sample are close together, forming a capillary neck between them (and hence a strong adhesion)

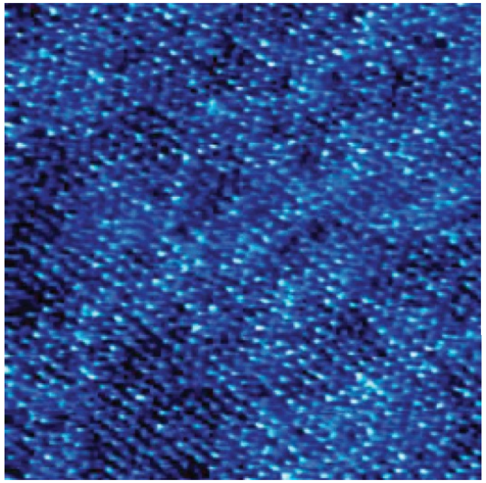
From Laplace equation the maximal capillary attraction force for retracted tip can be approximated as

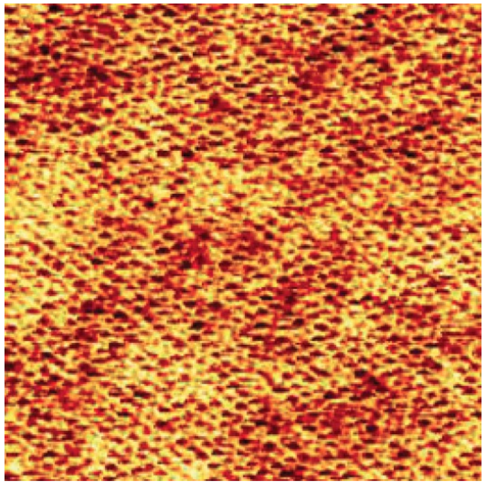
$\boldsymbol{F_{max}=4\pi R\sigma \cos\theta}$

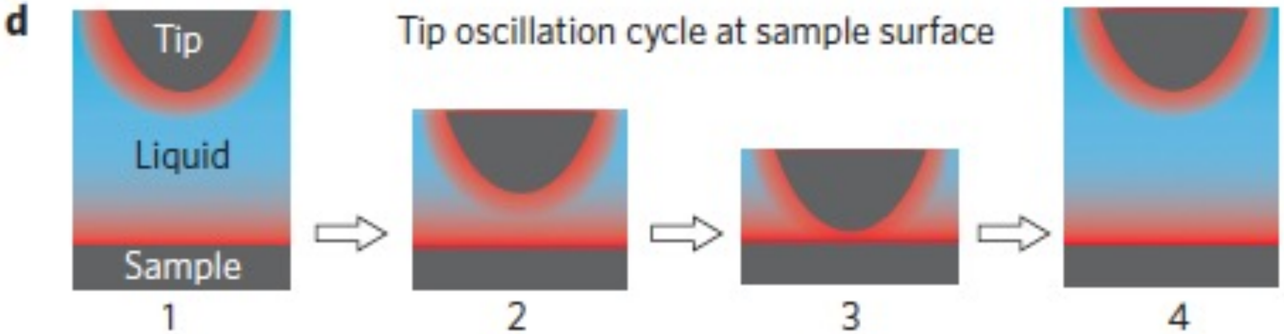
where R is tip radius, σ is surface tension, θ is contact angle (for estimation purposes $\boldsymbol{\cos\theta \approx 1}$)

Considering $\sigma=0.073$ **N/m** for water at 20°C and R=10 nm we get capillary force ~ $10^{-8} - 10^{-9}$ N (comparable or greater than Van der Waals forces)

AFM in liquid (water): high resolution topography and adhesion energy mapping





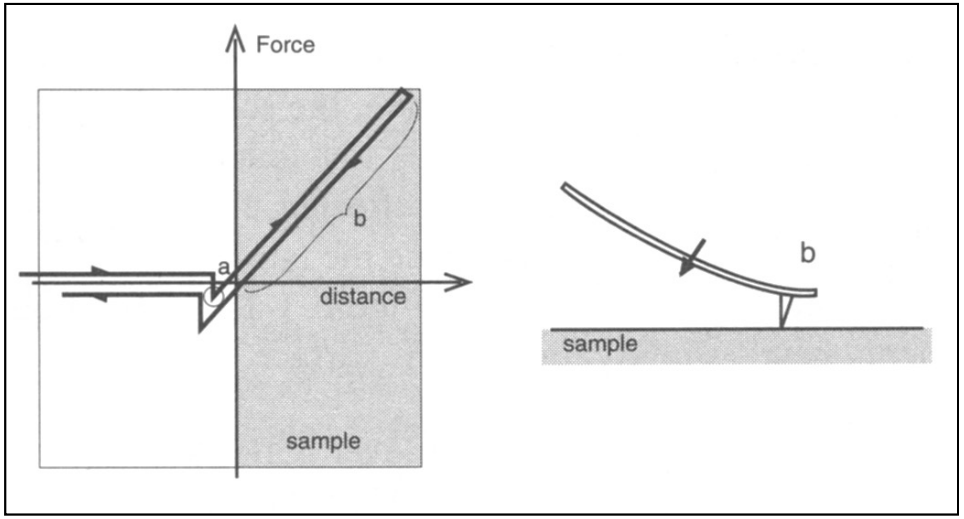


If the sample and probe tip are immersed in a liquid (here water was used) the capillary forces disappear.
left side: atomic resolution (20x20nm) images of mica (aluminosilicate) measured using non-contact AFM in constant-amplitude mode. Working amplitudes were kept in the 0.5–2 nm range, peak to peak.
Lower image: phase mapping.

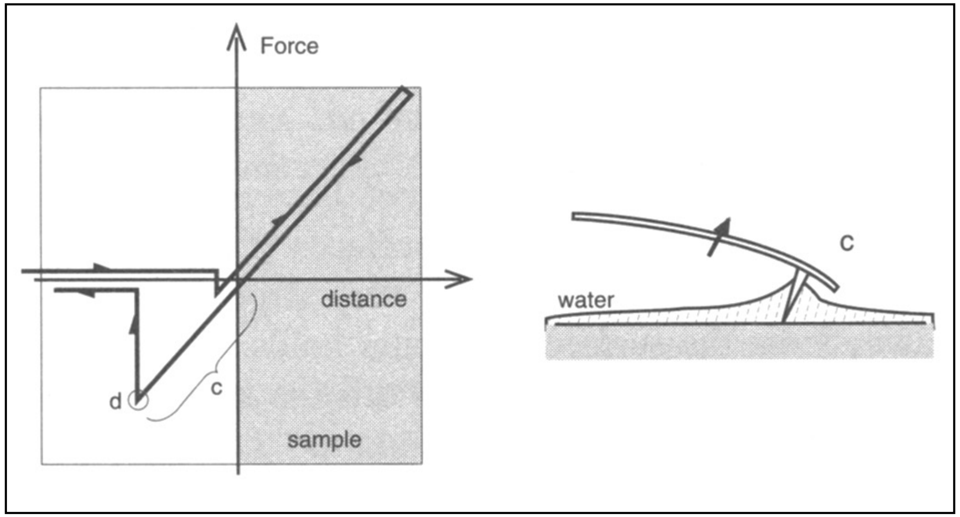
Right side: The image formation mechanism associated with short-range re-arrangement of the liquid molecules near the interface. As the tip oscillates the two interfacial liquid layers coalesce into a single layer and then separate again. The non-conservative work applied to perform this operation is directly related to the adhesion energy of both interfaces

K. Voitchovsky et al., Nature Nanotechnology, 5, 401, (2010)

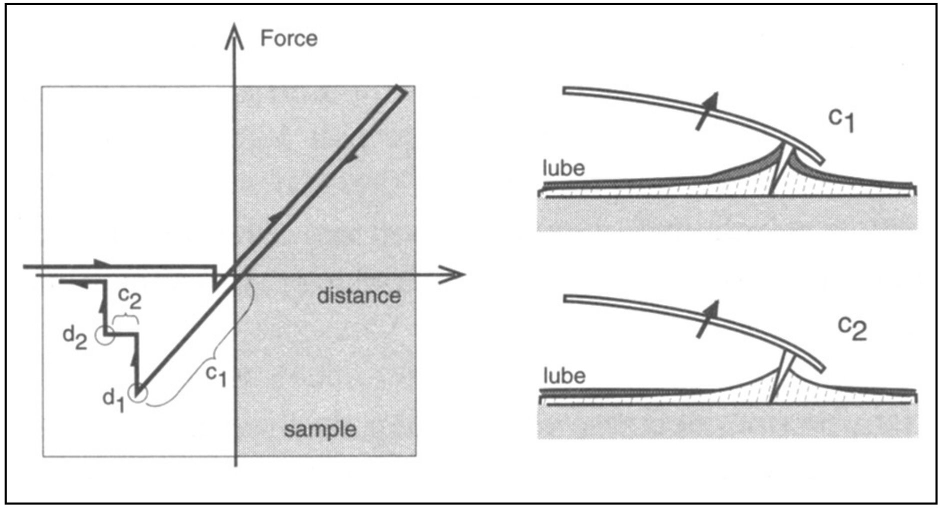
Force-distance curves



Vacuum



Humid Air

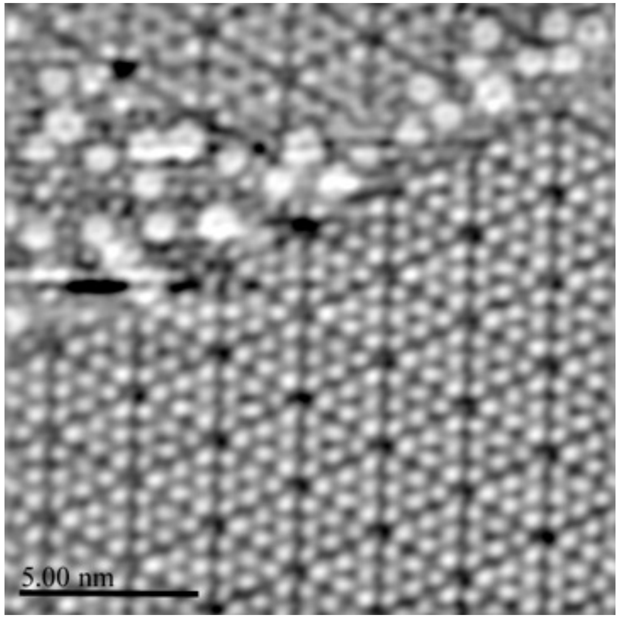


Humid air with contamination layer

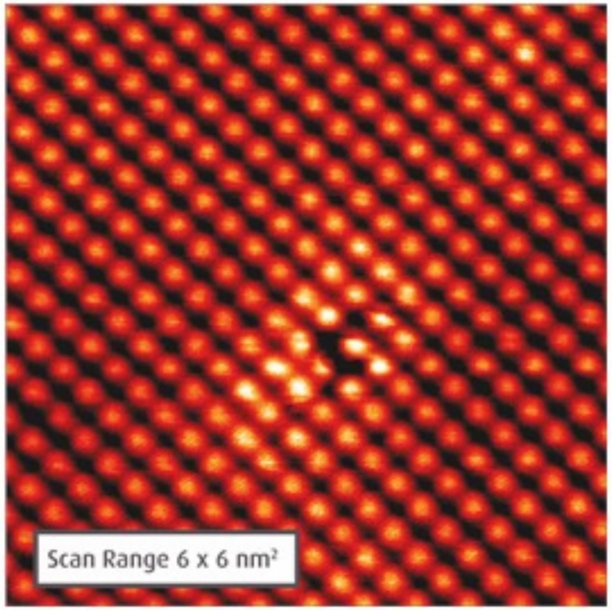
High-resolution AFM?

- **High resolution AFM is generally more challenging than STM**
- **Unlike in STM, there is a linear force-distance dependence in AFM, therefore "accidental" atomically sharp tips are not as successful nor common.**
- **The tip is contacting a larger area than a single atom. Presence of a contamination film, such as water, could spread out the point contact forces without inhibiting atomic resolution.**
- **At present the theoretical basis of atomic scale resolution is still under development.**
- **There is no universal approach to achieving atomic resolution on arbitrary materials.**
- **However is some cases atomic resolution is possible (in most cases by NC-AFM)**

Atomic resolution in NC-AFM

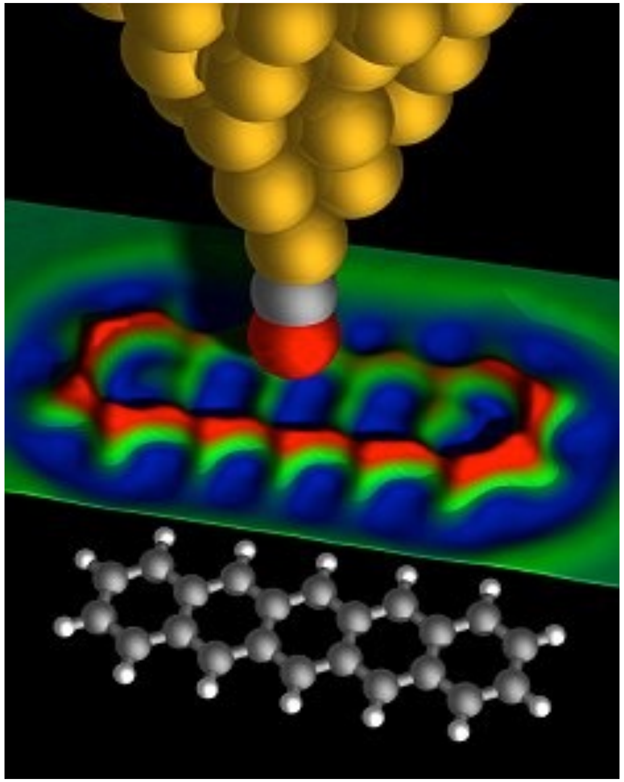


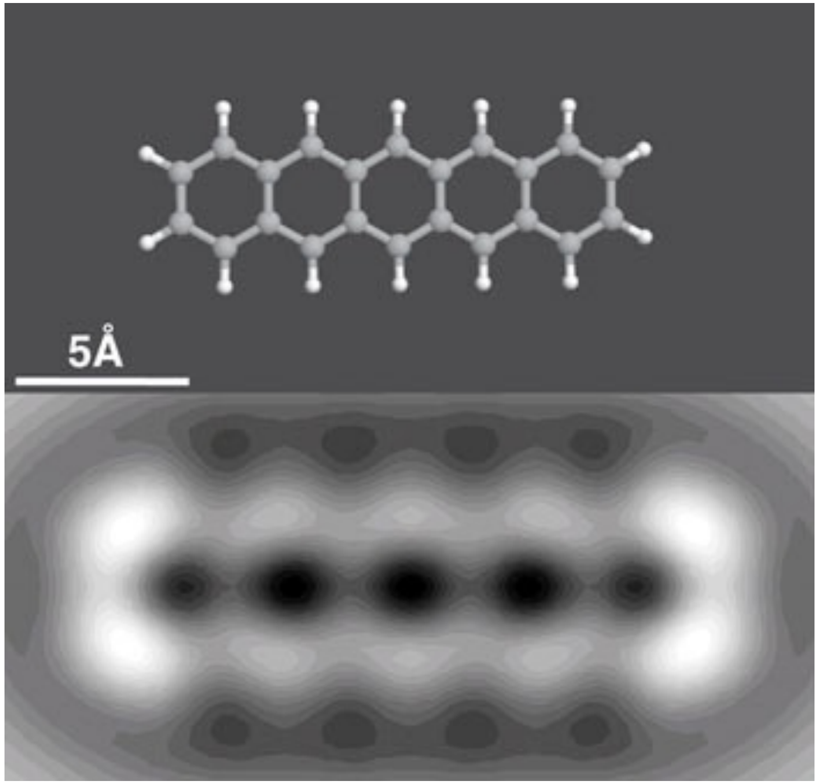
NC-AFM image of <111> Si 7x7 surface reconstruction. Image courtesy of JEOL.



UHV AFM Image of NaCl (100) on mica. Image courtesy Omicron

Atomic Imaging in NC-AFM





Imaging of individual atoms in the pentacene molecule consisting of five benzene rings fused in a line. Image was taken at 5 K in UHV with CO-molecule modified the tip.

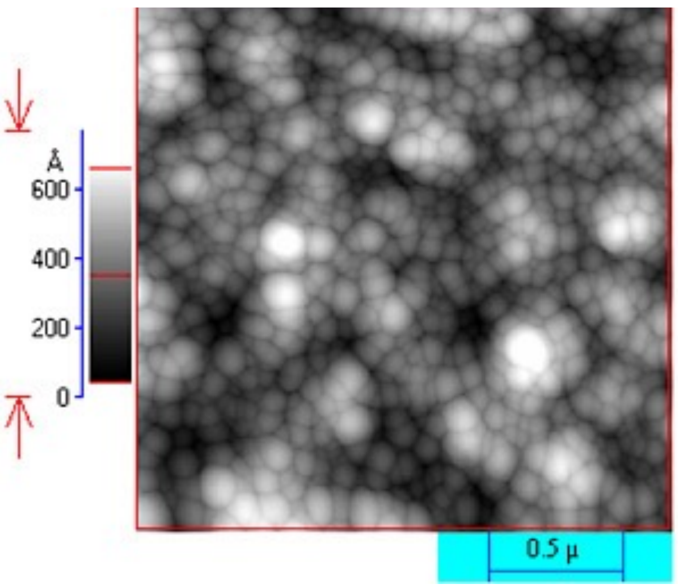
The key step is functionalizing the microscope's tip apex with suitable, atomically well-defined terminations, such as CO molecules.

Chemical forces (short-range interactions): a high-stiffness cantilever is required in order to operate at distances about 1Å. The use of constant-height operation was critical because it allowed stable imaging in the region where Δf is a nonmonotonic function of z.

Gross et al, Science 325, 1110 (2009)

CONTACT AFM: topography measurements

Example 1: 2x2 micron scan of a polycrystalline (ceramics) film



Rp-v = 771 Å

= 115 Å

Surface Area = 4.193 μ^2

$$R_{rms} = \sqrt{\frac{\sum_{n=1}^{N}\left(z_n - \bar{z}\right)^2}{N-1}}$$

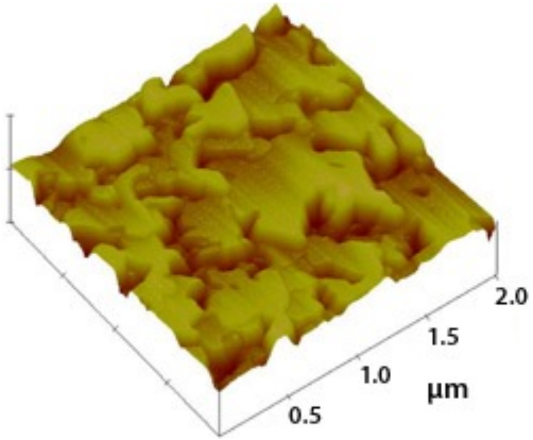
Topography measurements on micron – to – submicron scale images is one of very standard applications of contact AFM for a broad range of scientific and industrial applications
In general case these measurements are quick and easy to implement, can be performed with a great variety of cantilevers at different conditions.
The scan represents the 3D map with height represented here in grayscale.
The map can be readily processed in order to extract roughness and other essential parameters

Sometimes contact AFM is used to precisely measure profile in microfabrication processes (etching, layer deposition, lithography and other planar processes)

Example borrowed from the site of NREL (National Renewable Energy Lab.)

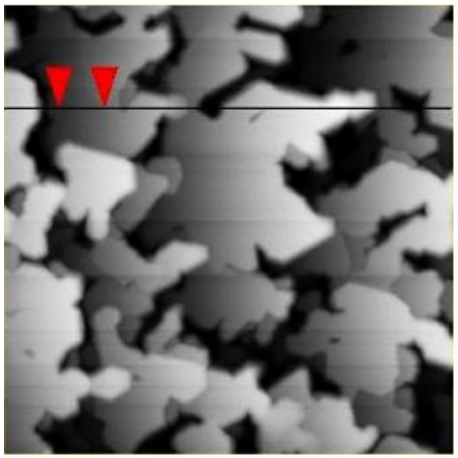
CONTACT AFM: topography measurements

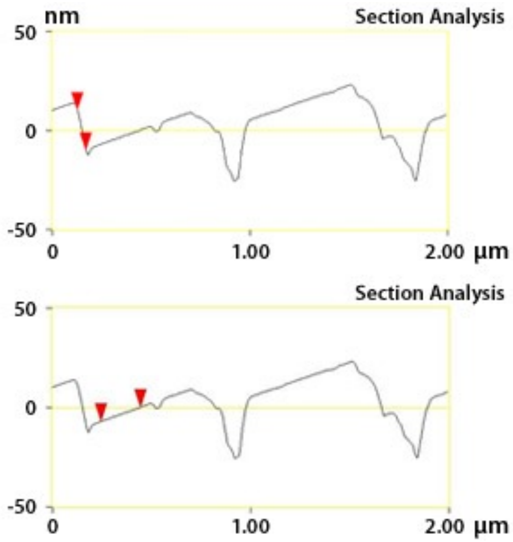
Example 2: 2x2 micron scan of epitaxial GaP film on Si



In AFM software packages 3D representations are readily available

AFM image of a GaP/Si sample. This is a real 3-dimensional representation of a GaP/Si sample surface, which can be rotated to reveal features not observed in a given orientation.





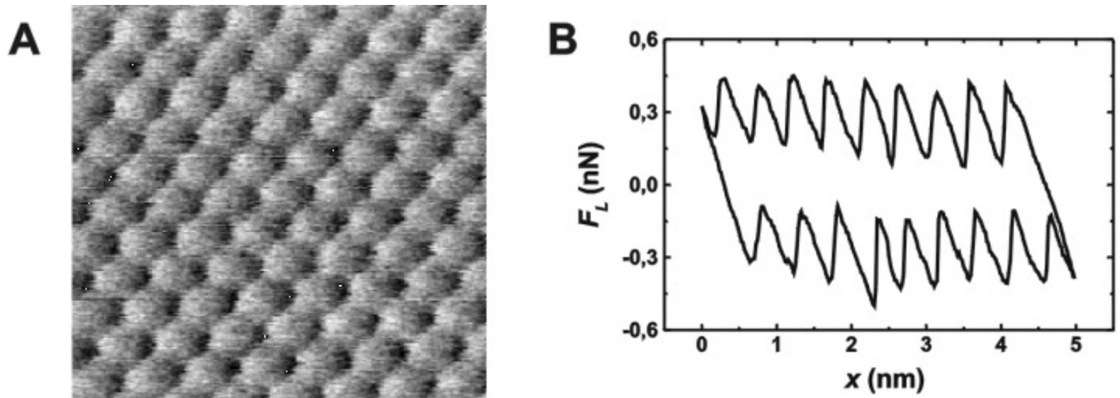
For detailed profile analysis line scans are often used.
Because of the digital character of AFM data, line scans can be generated from any part of the image, and it is also possible to measure the angle between features.

Example borrowed from the site of NREL (National Renewable Energy Lab.)

<u>CONTACT AFM</u>: topography measurements

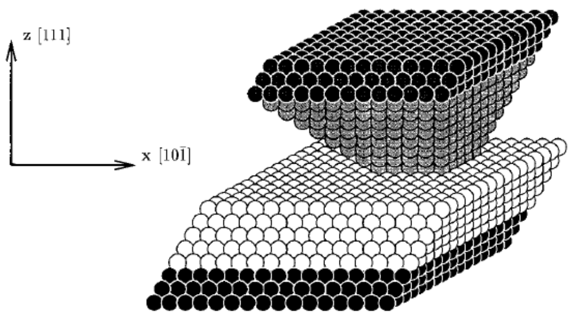
Example 3: Lateral force: detecting atomic-scale features

Contact AFM is rarely used for true atomic-scale imaging (non-contact AFM is more suitable for this purpose). However in some cases it can deliver useful information on the atomic scale. In the example shown here NaCl (100) surface is studied with contact AFM in vacuum. Lateral force 5x5 nm image reveals the periodicity of the surface lattice. This behavior of lateral force is called **<u>atomic-scale stick-slip (sawtooth behavior)</u>**. The lateral force increases while the contact is locked onto one atomic position until it is strong enough to initiate a slip to the next atomic position.



*From L. Howald et al., Phys. Rev. B **51** (1995) 5484*

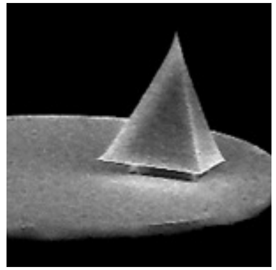
Similar friction maps have been measured on other materials including graphite and metals (Cu). By quantifying the friction force one can extract the information about the contact area.
This area is generally greater than a single atom (image on the right) so this is not a "true atomic resolution" map



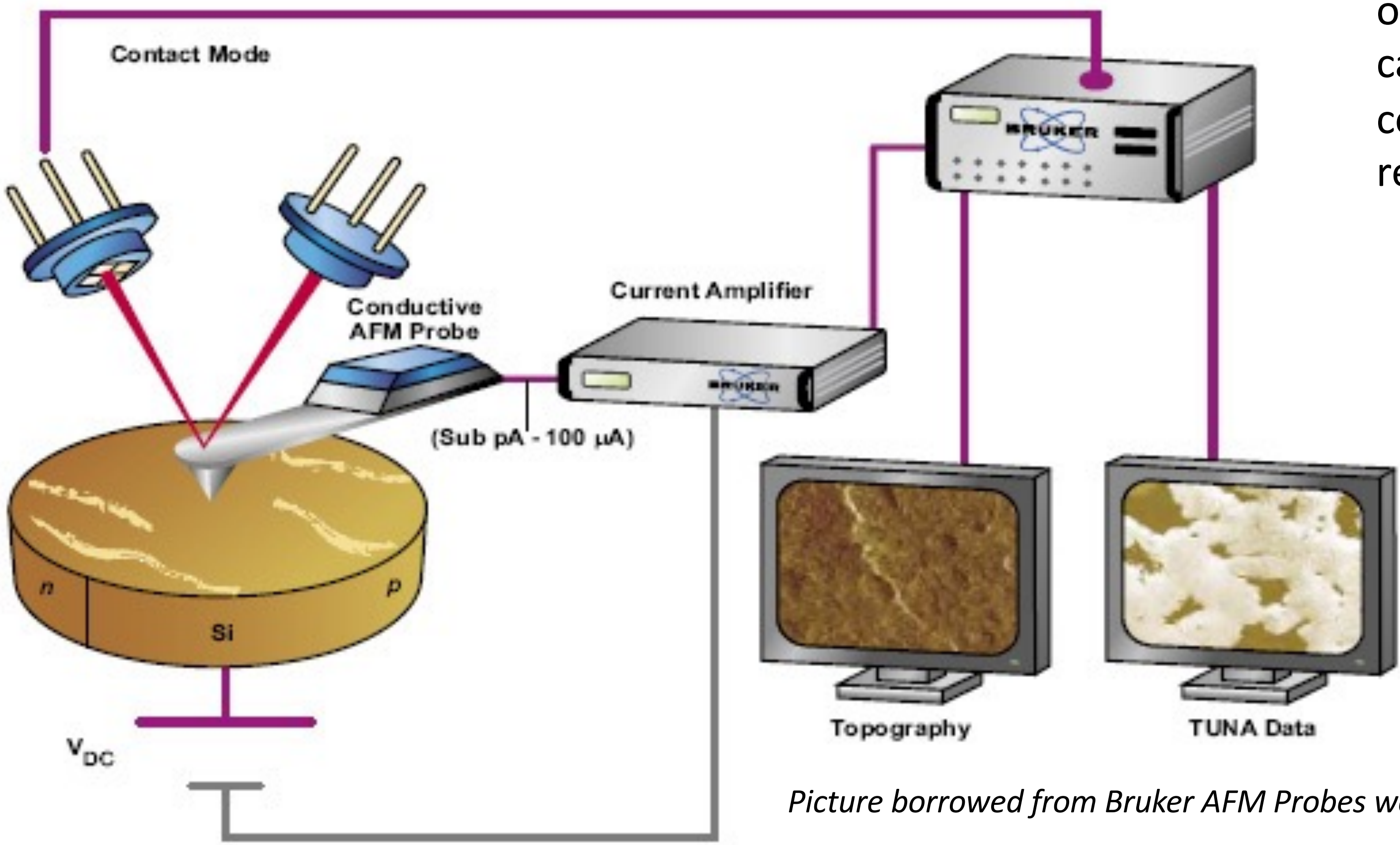
*From M. Sorensen et al., Phys. Rev. B **53** (1996) 2101*

<u>CONTACT AFM</u> – functional modes: conductive AFM

•In this mode the electrical current mapping or local I-V, or I-t characteristics are collected together with topography information
• The feedback loop relies on deflection signal (like conventional contact AFM) <u>– this is a fundamental difference from STM!</u>
• Unlike STM conductive AFM is usable for detecting very small < 1pA and irregular conduction spots in dielectric



Conductive cantilever or conventional cantilever with conductive coating is required



Picture borrowed from Bruker AFM Probes website

• Concurrent collection of topography and conduction maps allows for direct comparison between topography features (cracks, grain boundaries, steps) and zones of different conduction

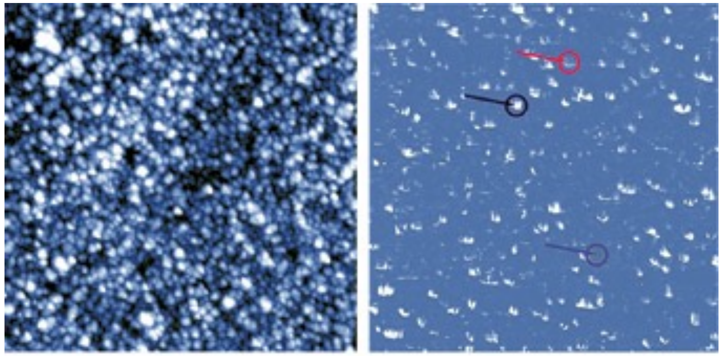
<u>CONTACT AFM</u> – functional modes: conductive AFM, example

Topography (top left) and current (top right) 2x2 μm image of a Europium-doped ZnO sample at a bias of 1.5 volts.
Lower image represents local I-V curves measured with AFM probe recorded at three specific positions indicated in current images. The curves are consistent with the current contrast map.

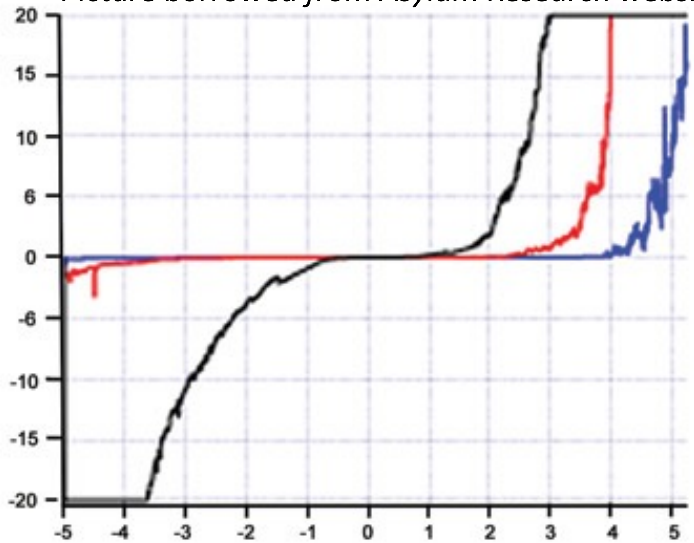
• When interpreting conductive AFM data the electronic properties of the probe have to be considered. The properties of tip-sample contact often influence (or even determine) the measured conductive response. One of the reasons is a Schottky-type diode formed at the contact due to different workfunctions of the materials.

• In order to optimize the current response the coating material has to be carefully selected depending on measured samples.

• The tip metallization is often unstable and prone to wearing. It can be destroyed by strong currents if some heavily conductive spot is encountered

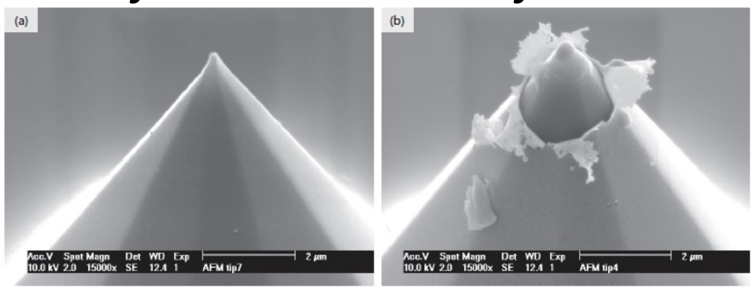


Picture borrowed from Asylum Research website



Problem with metallization:

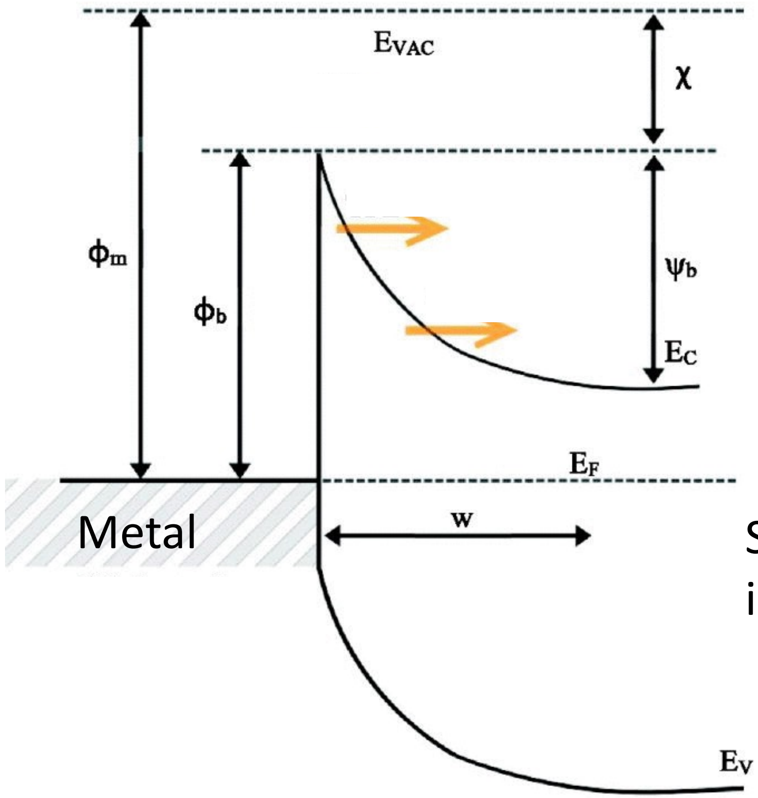
Before ***after***



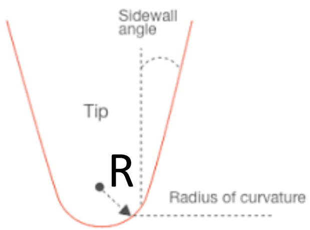
<u>CONTACT AFM</u> – probe-sample interface can be a diode

- in many cases c-AFM conduction is determined by the probe-sample interface

- this interface is a potential barrier, which may be
  - Ohmic contact (low barrier compared to kT)
  - Schottky barrier (thermoionic injection)
  - Tunneling barrier (high electric filed)



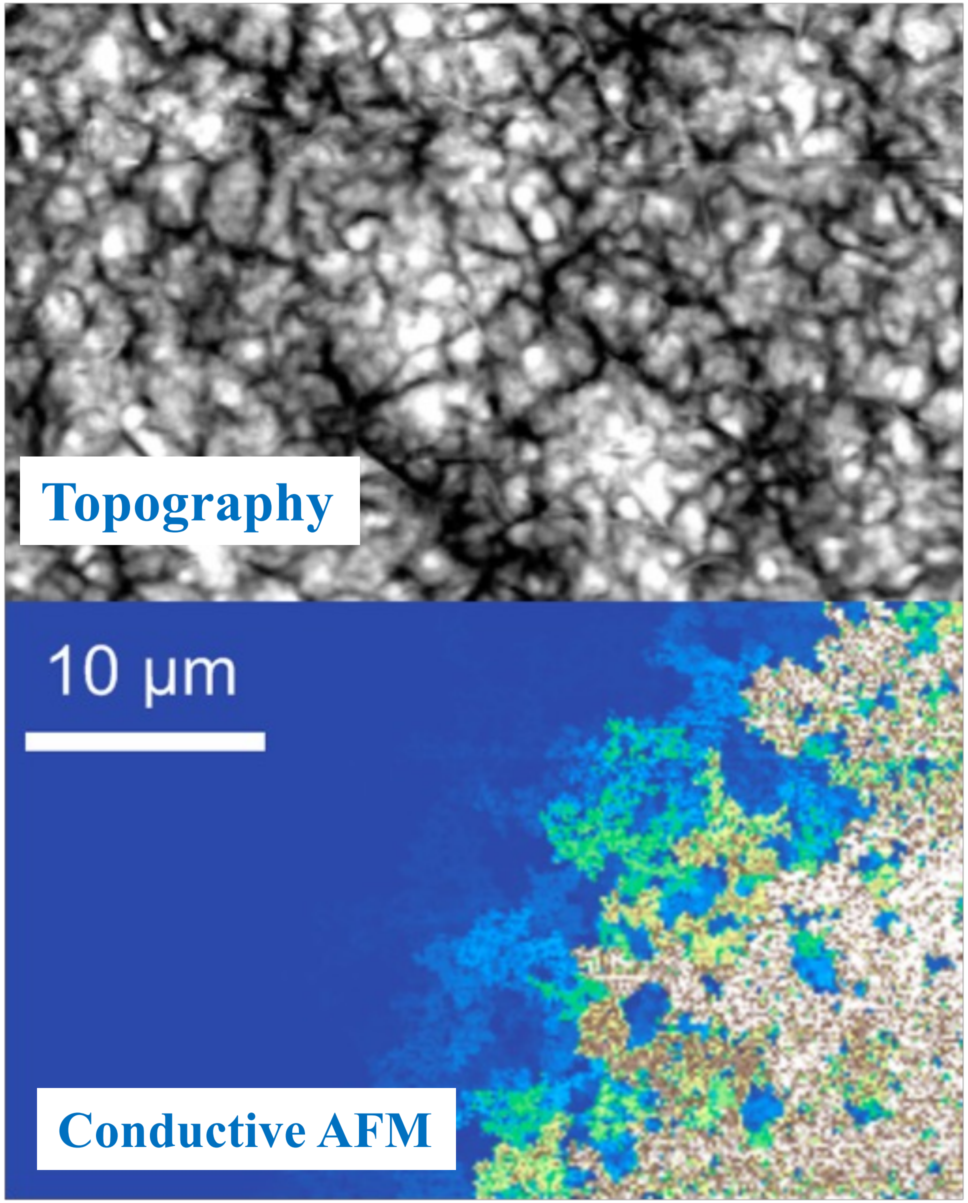
Semiconductor or insulator



- The electric field is concentrated near the probe

- In close proximity to the interface a useful approximation is V/R, where V is applied voltage, R is tip radius

Conductive AFM : example

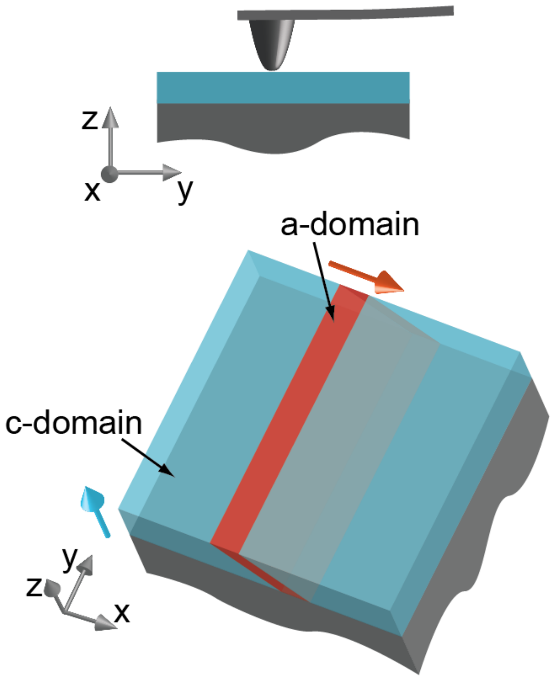


A ferroelectric copolymer film is deposited on top of the silver film on glass. From the topography, the film does not seem to have much difference. When seen by Conductive AFM, the film shows significant difference in conductivity.

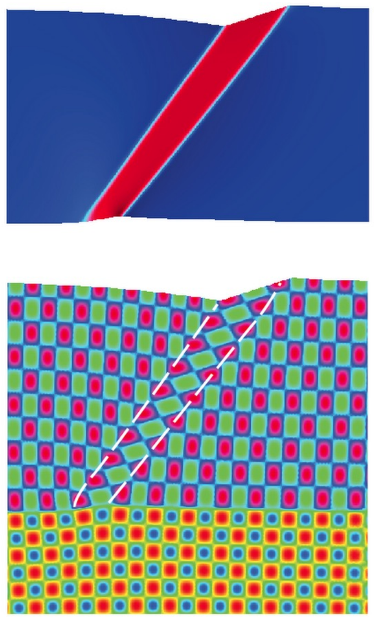
Image credit: Park Systems

Conductive AFM, example: PZT with 90° ferroelastic domains: conductive domain boundaries

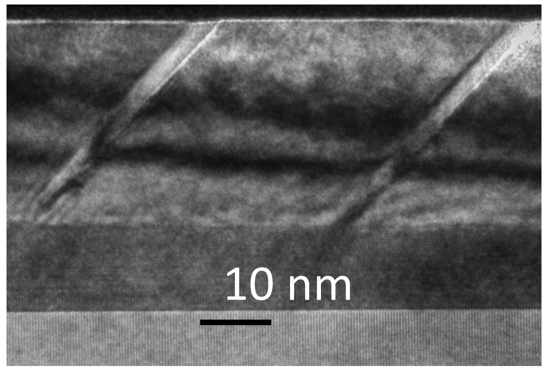
90° domains of polarization are formed in thin (50nm) epitaxial PZT films in order to reduce the mechanical energy (reduce lattice mismatch). They can be seen in topography (sub-nm vertical resolution).
Theoretical analysis suggests that the domain boundaries can show metallic conduction (conductive channels in dielectric media, which can be reconfigured!)



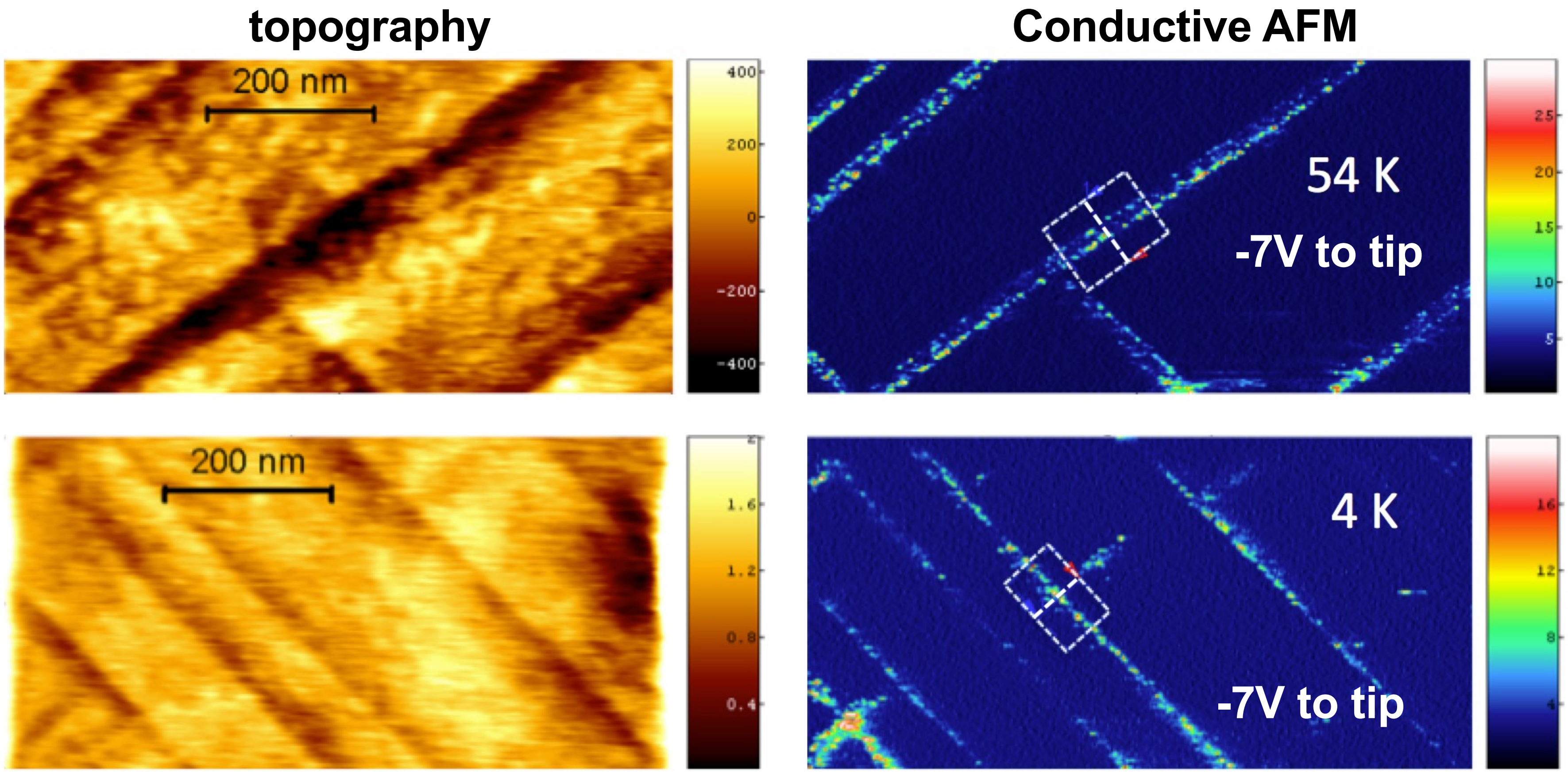
Simulation

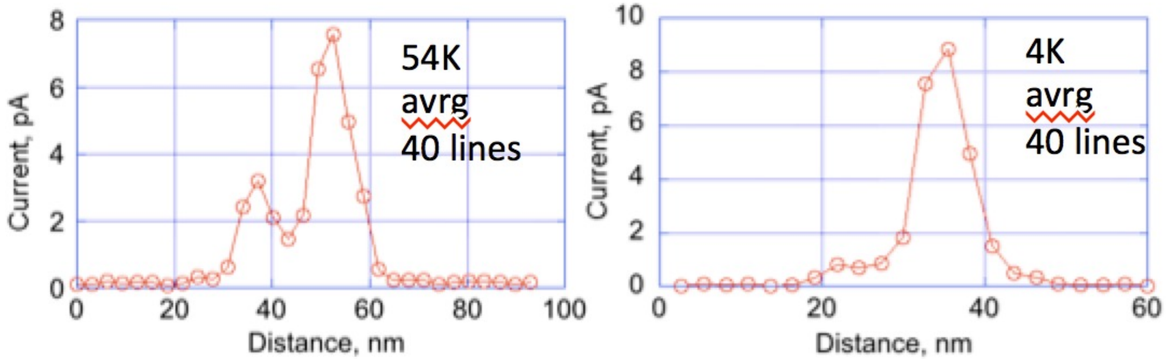


TEM image



Conductive AFM, example: PZT with 90° domains: conductive domain boundaries



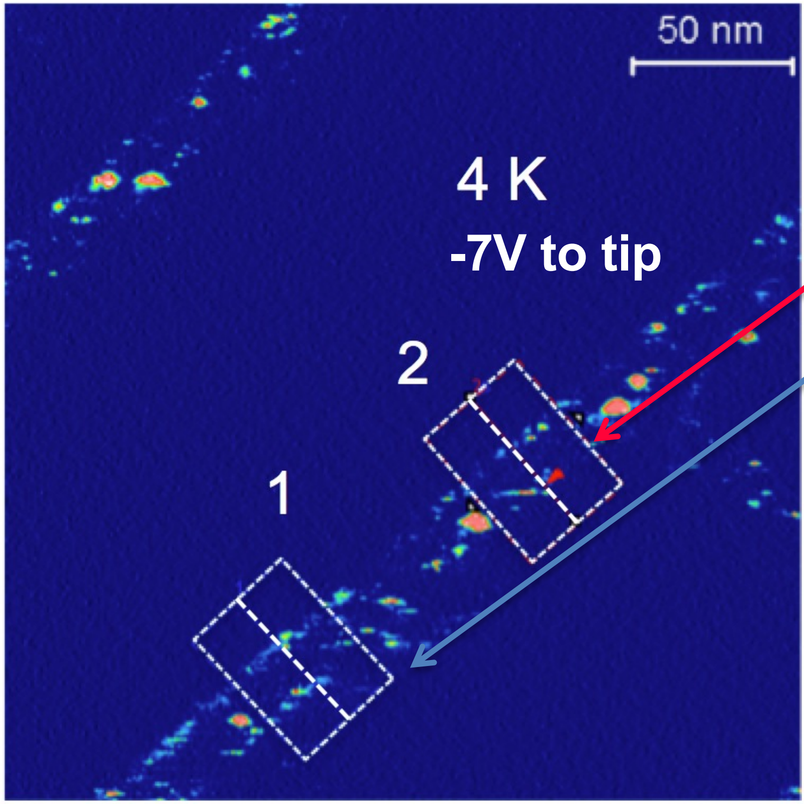


4K vs 54K: (same probe)

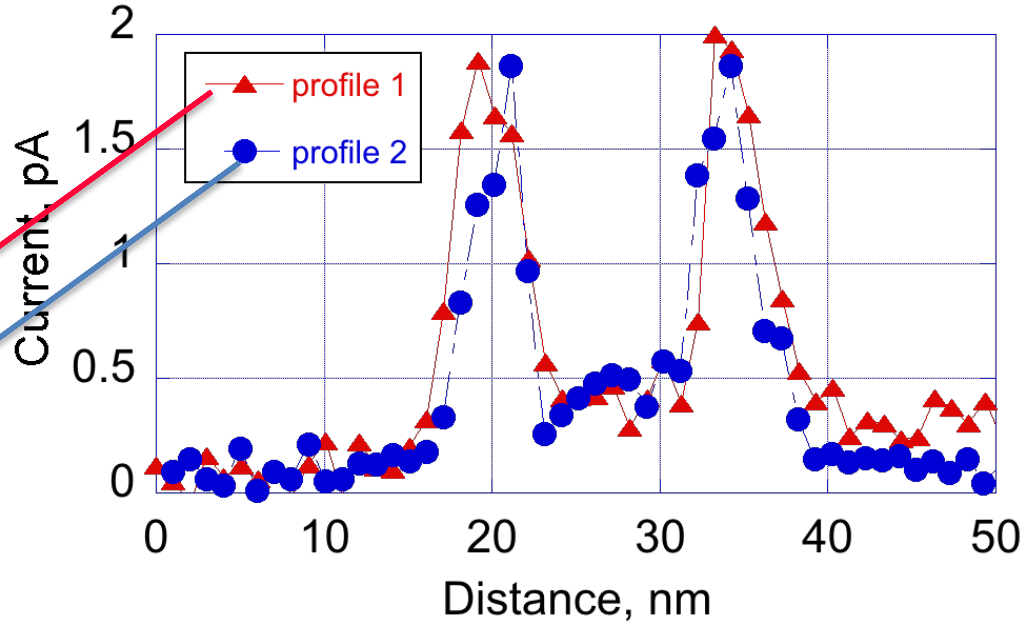
Current values are close!

Enhanced resolution c-AFM: resolving domain boundaries

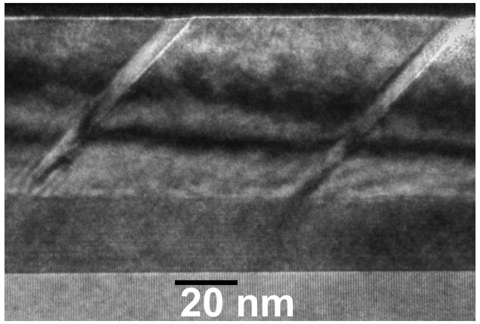
c-AFM: 250x250 nm^2 map



Conduction profile (averaged, 30 lines)



TEM cross-section



- Improved resolution: pristine diamond probe at low force (1-3nN)
- 90° -domains: clear double traces
- conduction profile width (at 50%): 4±1nm!
- TEM cross-section 12±1 nm – the width is comparable
- We see individual conductive domain walls!

I. Stolichnov et al., Nano Letters (2015)

Conductive AFM (c-AFM) + topography: a powerful characterization tool

- Conductive AFM: a local probe for precise nm-scale conduction analysis
- C-AFM in combination with topography offers a powerful characterization tool for probing electrical properties of nanostructures
- Care has to be taken for tip protection (mechanical, electrical), the metallization can be deteriorated during the scan
- Special probes are available for heavy duty experiments (doped diamond tips for long durability, metal tips for high current)
- Choice of right probe is essential!
- Note the difference between c-AFM and STM
- Environmental control can be important (electrochemistry!)

Conductive AFM (c-AFM) + topography: environmental control

A simple approach (without going for UHV):

- Use a scanner with environmental control (varying temperature + gas flux, typically N_2)
- Dry your sample by high T (if possible) – remove absorbed water from the surface
- Cool it down maintaining the gas flux
- run the measurement without any effect from environmental oxygen and moisture