UNIVERSITÀ DI PISA

Introduction to Atomic Force Microscopy

Quantum Optics Lab a.a. 2020/2021

‘Probe microscopies’

1982: Scanning Tunnelling Microscope invented

Simple physical principle: a metal tip is scanned on a surface at constant tunnel current.
The current is kept current by a feedback loop controlling the voltage (V_P) applied to a piezoelectric.
This changes dynamically the position of the tip (i.e., its distance from the surface).
Monitoring V_P so provides a picture of the surface (topography $\otimes$ density of states).

Surface Studies by Scanning Tunneling Microscopy

G. Binning, H. Rohrer, Ch. Gerber, and E. Weibel
IBM Zurich Research Laboratory, 8803 Rüschlikon-ZH, Switzerland

(Received 30 April 1982)

Surface microscopy using vacuum tunneling is demonstrated for the first time. Topographic pictures of surfaces on an *atomic scale* have been obtained. Examples of resolved monoatomic steps and surface reconstructions are shown for (110) surfaces of $CaIrSn_4$ and Au.

PACS numbers: 68.20.+t, 73.40.Gk

G. Binning, H. Rohrer, Ch. Gerber, and E. Weibel, *Phys. Rev. Lett*. **49**, 57-61 (1982)

‘Probe microscopies’

1982: Scanning Tunnelling Microscope invented

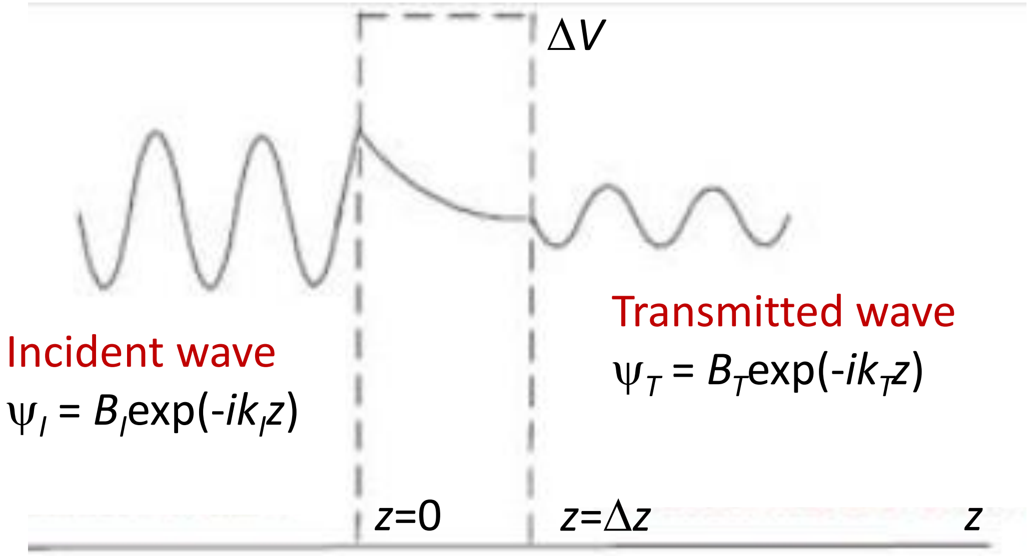
Simple physical principle: a metal tip is scanned on a surface at constant tunnel current.
The current is kept current by a feedback loop controlling the voltage (V_P) applied to a piezoelectric.
This changes dynamically the position of the tip (i.e., its distance from the surface).
Monitoring V_P so provides a picture of the surface (topography ⊗ density of states).

VERY sensitive:
Tunnel current across a barrier of height ΔV (=U-E) and width Δz:

$J_T \sim \psi^2 \sim \exp(-A\, \Delta V^{1/2}\, \Delta z)$
with
$A = 4\pi \times (2m)^{1/2} / h = 1.025$ Å^{-1} eV$^{-1/2}$
m: free-electron mass, ok for a vacuum tunnel barrier.

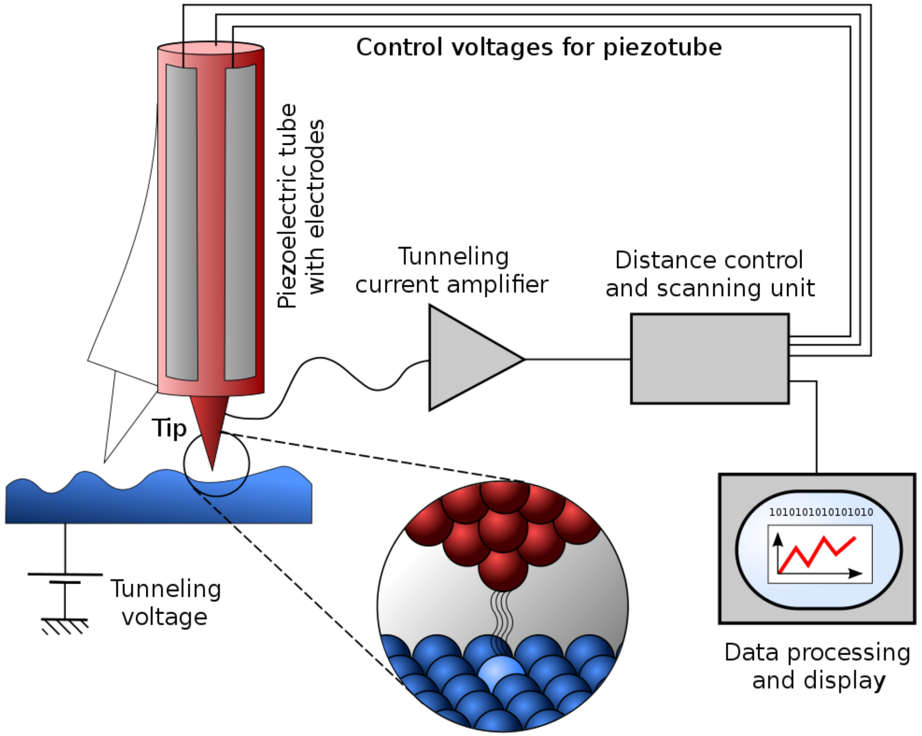
Barrier height = work function

If $\Delta V \sim$ the order of eV, a change of Δz by a single atom (~ 2-5 Å) can change J_T up to three orders of magnitude
→ overall resolution in z: << 1 Å



Exponential decay
$\psi = B\exp(-k_D z)$

$k_D^2 = (2m/\hbar^2) \times \Delta V$



Field of Nanoscience Opened

1986:
G. Binnig and H. Rohrer, inventors of the STM, rewarded with the Nobel Prize in Physics

But…

- **Highly clean surfaces needed** (contamination layers can be locally insulating and prevent tunneling);
- **Conductive samples needed** (metallics, semiconductors, or very thin dielectrics);
- **UHV conditions needed** (<10^{-7} Pa, < 10^{-9} mbar ~ 10^{-9} torr)**, since surfaces in ambient conditions dynamically change in a continuous way due to adsorption/desorption of atoms and molecules** (UHV condition somehow relaxed after some years, now measurements in air possible, + interactions with various field e.g. magnetic, optics etc.)

Early STM experiments soon showed that when the tip-sample distance was small enough that a current could flow, significant forces would act (in addition to the tunneling current) → put them to use in another technique

1986: Atomic Force Microscope invented

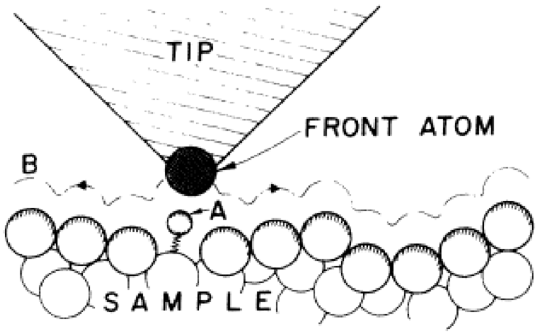
Atomic Force Microscope

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.



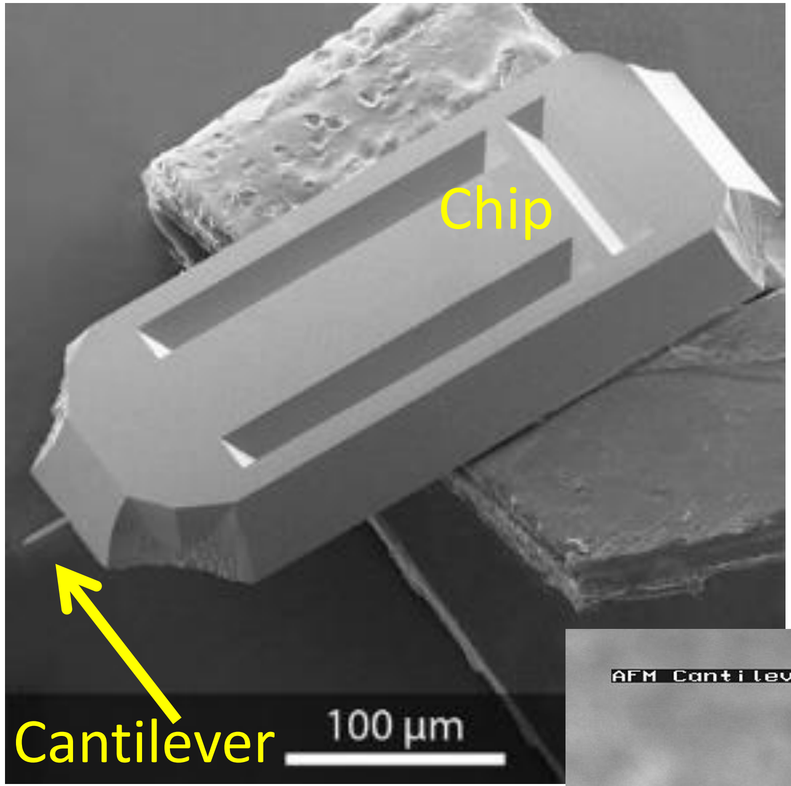
G. Binning, C. F. Quate, and Ch. Gerber, *Phys. Rev. Lett*. **56**, 930-61 (1986)

1. Tunnelling tip (STM) replaced by a force-sensing **cantilever (AFM)**.

2. Feedback loop: Constant tunnelling current (STM) replaced by **constant force (AFM)**

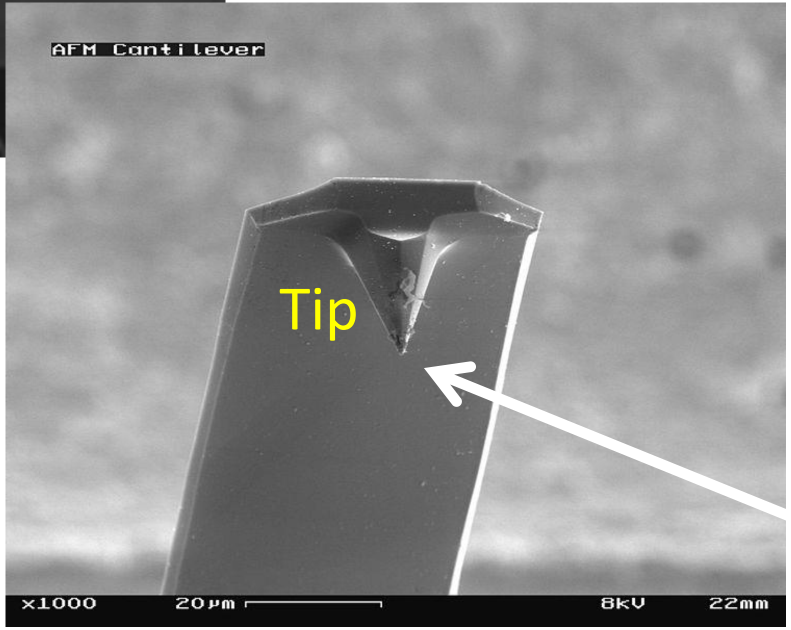
Cantilever 1/2

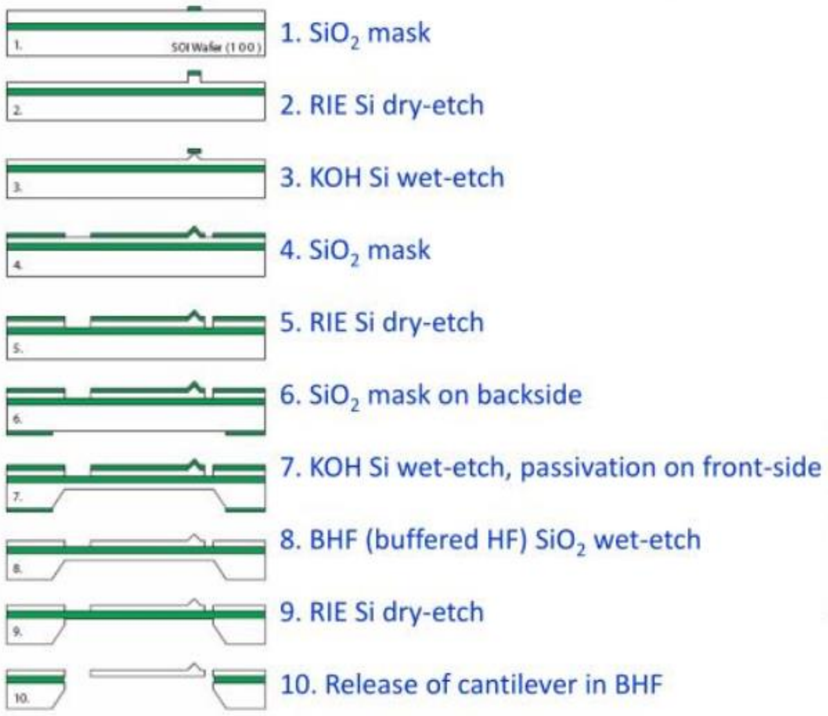
UNIVERSITÀ DI PISA



High Resolution, Tapping (Dynamic) Mode AFM Probe with AFM Tip at the Very End of the AFM Cantilever, OPUS by MikroMasch
www.nanoandmore.com

f_R = 300 kHz
k = 26 N/m





More info: T. Wakayama et cl., *Sensors and Actuators A* **126**, 159-164 (2006).

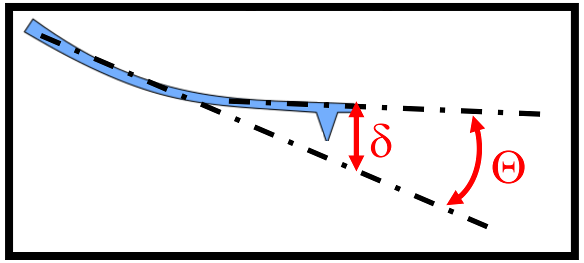
Ideally only one atom interacting with the target surface: the resolution clearly depends on the tip curvature radius

Materials: Al foil + diamond (1986), SiO_2, Si_3N_4, today mostly done by Si micromachining (MEMS technology, i.e. anisotropic etching along crystalline directions)

Cantilever 2/2

Dynamometer: tool to measure forces, by transducing forces into **calibrated** displacements

Similarly, a cantilever beam can experience a displacement (deflection) proportional to the sensed force



Many different cantilevers are commercially available:
They are different for:
-Dimensions and shape, typical values: 0.1-0.5 mm:
-Elastic constant (materials and design, typical values: 0.05-50 N/m);
-Tip coating (conductive, super-hard, etc.) and curvature radius.

Cantilever choice depends, for instance, on:

- Operation mode (contact/non contact)
- Quantities to be probed (e.g., if an electric field is needed, a conductive tip has to be used)
- Hardness and wear of the sample to be probed
- Possible material manipulation (e.g., nanoindentation requires super-hard tips)

Forces involved and characteristic range:

Tip-surface forces 1/3

UNIVERSITÀ DI PISA

Long range:
Electrostatic force in air: 100 nm
Double layers formed in liquids (also electrostatic): 100 nm

Short-range:
Van der Waals (dipole-dipole): 10 nm
Hydrogen-bonding forces: 0.2 nm

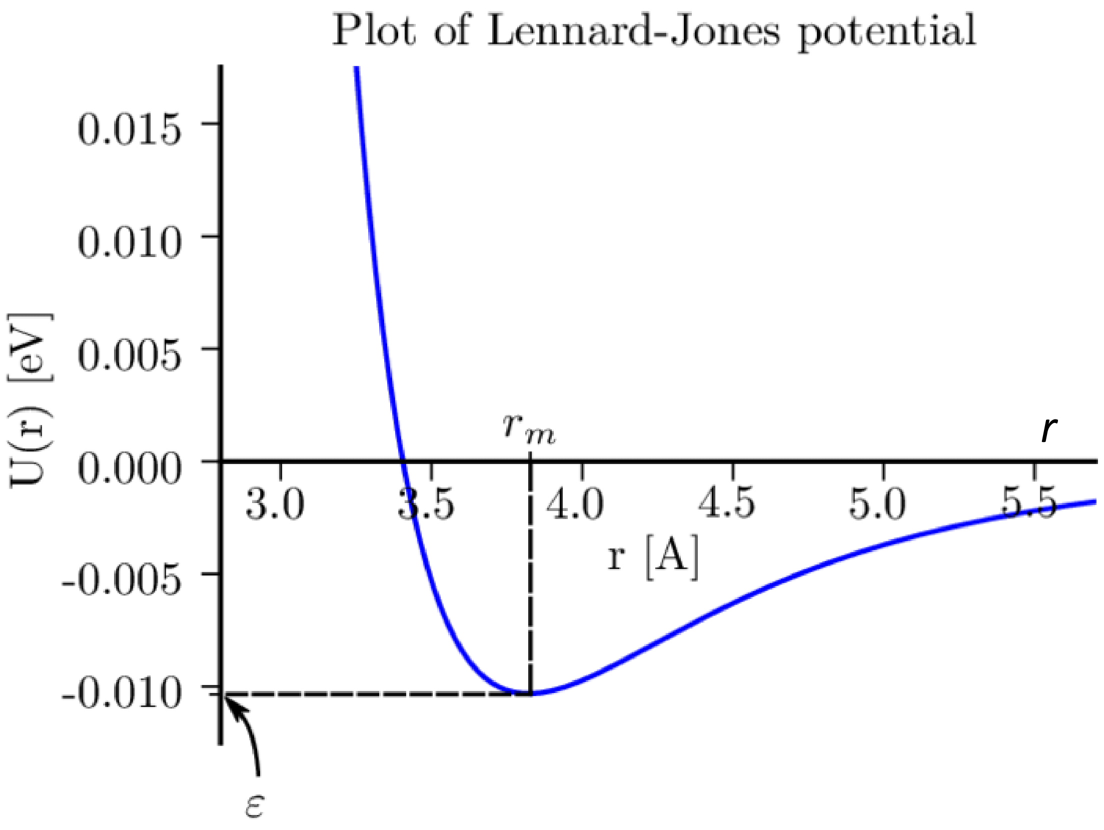
Typical behaviour: when the tip approaches a surface, there is an initial weak attractive force (mainly VdW attraction), followed by a steep change in slope leading to strong repulsion (electrostatic+Pauli repulsion, i.e. exchange interactions due to the overlap of the electronic orbitals at atomic distances). Orders of magnitude involved: **10^{-8}-10^{-9} N**

Well described by a Lennard-Jones potential e.g. 12-6

$$U(r) = 4e\left[\left(\frac{s}{r}\right)^{12} - \left(\frac{s}{r}\right)^{6}\right]$$

$$\vec{F}(r) = -\vec{\nabla}U(r) = -\frac{dU(r)}{dr}\hat{r} = 4\varepsilon\left[12\frac{\sigma^{12}}{r^{13}} - 6\frac{\sigma^{6}}{r^{7}}\right]\hat{r}$$

Empirical law: parameters and exponents to be adapted



Tip-surface forces 2/3

UNIVERSITÀ DI PISA

Brief recall: the attractive part of the Lennard-Jones potential can result from **dipole-dipole** (vdW) interactions

Note: both dipoles have an "instantaneous" character (an instantaneous polarization of an atom induces a polarization in nearby atoms, and therefore an attractive interaction)

$$U = -\vec{p}\cdot\vec{E} \quad \text{with} \quad \vec{E} = \vec{E}_{dip} = \frac{(3(\vec{p}\cdot\vec{r})\vec{r} - r^2\vec{p})}{r^5} \propto r^{-3}$$

Possible mechanisms at the base of the effect:

- Dipole orientation
- Dipole induction
- Dipole displacement

These lead to $\vec{p} \propto \vec{E} \quad \rightarrow \quad U \propto r^{-3} r^{-3} = r^{-6}$

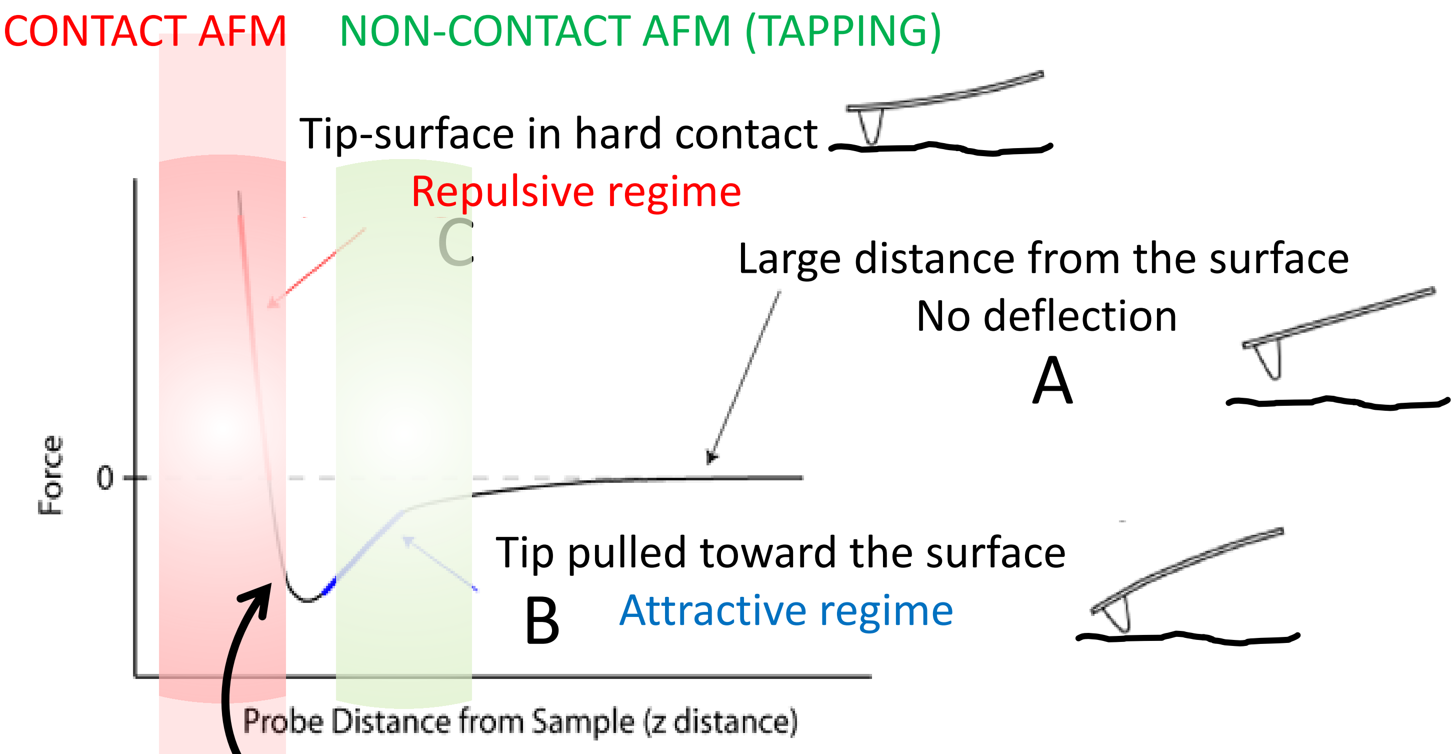
In the general case one has: $\vec{F} = -\vec{\nabla} U \propto -r^{-s}$ (attractive force)

with *s* depending on the actual geometry, materials, size, etc.

$$F\ \mu - d^{-2,\ -4}$$

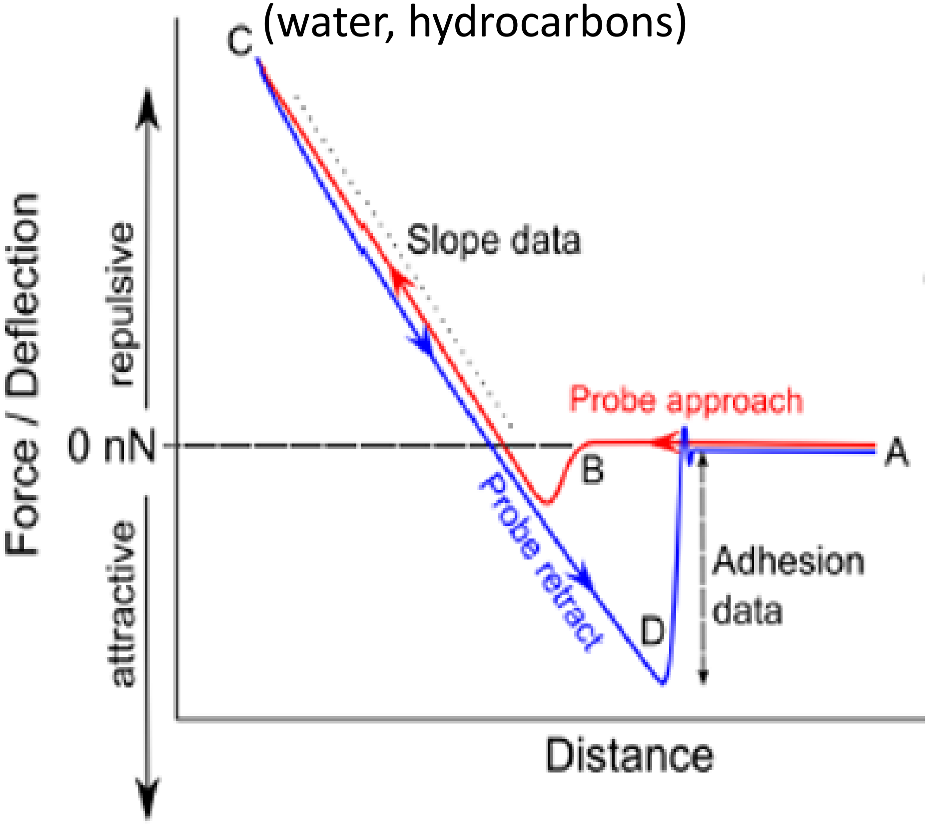
Tip-surface forces 3/3

UNIVERSITÀ DI PISA



Contact (roughly, not precisely defined at atomic scale)

Hysteresis – adhesion effects
In ambient conditions, meniscus forces (also called capillary forces) are present, due to adhesion layers on tip and sample (water, hydrocarbons)



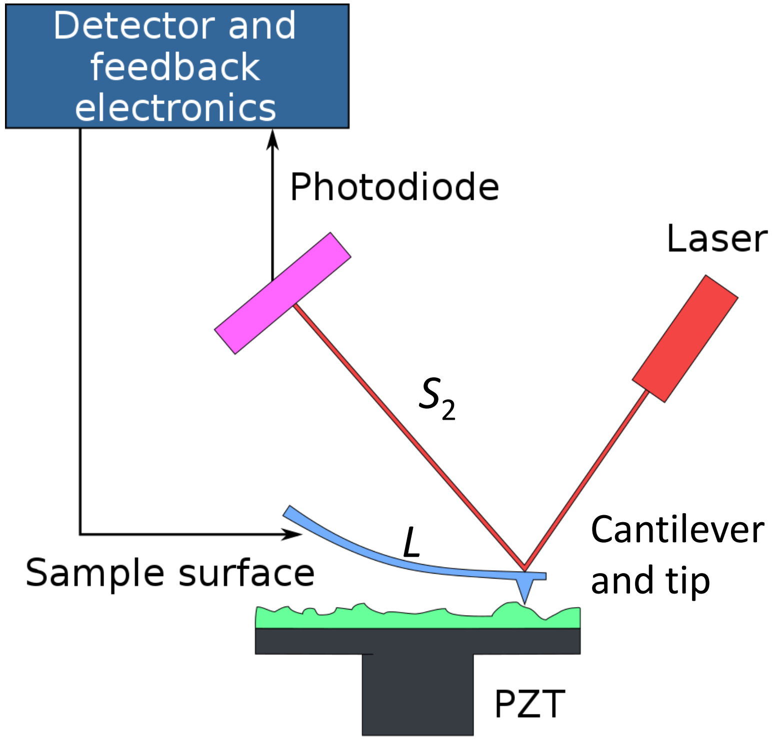
A: No detectable interaction force

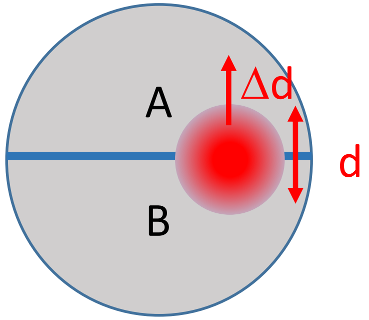
B: Gradient of the tip-surface interaction > restoring force of the cantilever

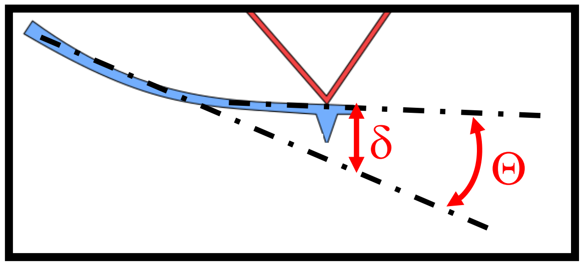
C: Positive cantilever deflection (if indentation occurs, can be used for measuring mechanical properties at nanoscale)

Detection: optical lever

Optical lever: device to magnify a small displacement and thus to make possible an accurate measurement of the displacement.







Let's suppose to have a photodiode divided in two regions, A and B: vertical displacement of the reflected laser spot on the photodiode: Δd

Δd is proportional to δ:

$\Delta d = 2 \sin\Theta \times S_2 \cong 2\Theta \times S_2 = 3S_2 \times \delta / L$

where we used:

$\sin\Theta \approx \Theta$ (small angles);

the 2 prefactor before $\sin\Theta \times S_2$ comes from our geometry (if the reflecting cantilever deflects by Θ, the angle between the reflection point and the laser spot on the photodioded changes by 2 Θ;

$\Theta = 3\delta/2L$ (this can be demonstrated by considering an elastic beam with a given Young modulus, the factor 3/2 is not really important here…)

* in principle. But, see next page…

<u>Lever-due amplification</u>

<u>$\Delta d/\delta = 3S_2/L$ (Huge!)</u>

(0.01 nm accessible)*

If P is the total power measured by the photodiode, one has (roughly):

$P_A = (P/2) \times [(d+\Delta d)/d]$ (here, d is the spot diameter; $0 \leq \Delta d \leq d$)

$P_B = (P/2) \times [(d-\Delta d)/d]$

The ratio of the difference and the sum signal is:

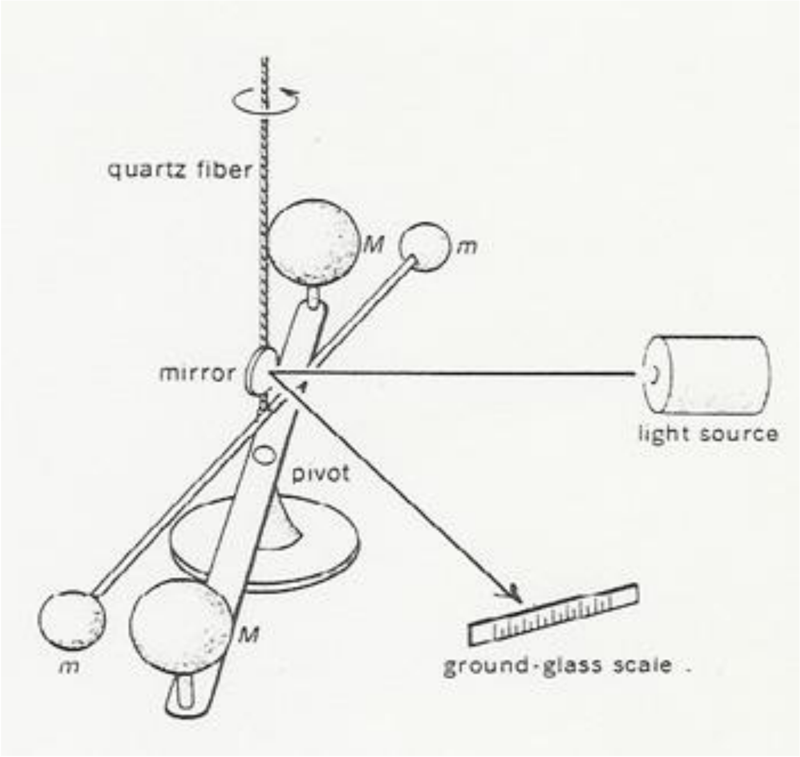
$(P_A - P_B) / (P_A + P_B) = 3S_2 \times \delta / Ld$

The optical lever: an old concept….

1679: Law of universal gravitation (Newton)

1798: first measurement of the constant G (Cavendish)

G=(6.75 $\pm$ 0.05) N×m²/kg²



* A more reliable estimation of the **resolution limit in z:**

Sub-Angstrom resolution possible only at low temperature. Thermal noise on the tip of elastic constant k:

$< \frac{1}{2} k\delta^2 > \cong \frac{1}{2} k_B T$

Some numbers:

$k_B T = 4\times10^{-21}$ J (room temperature)

k = 1 N/m

→ Best vertical resolution achievable = 0.5 nm

The tapping mode

UNIVERSITÀ DI PISA

Preventing the contact between the tip and the sample is advantageous to not damage the sample. However, this method requires higher sensitivity to sense weakly changing attractive forces

This is achieved by applying a controlled tapping displacement to the tip. <u>While the tip is oscillated at about the resonance frequency (50-500 kHz), the amplitude of the oscillation is kept constant by a proper feedback mechanism that acts on the vertical position of the piezoelectric element controlling the sample-tip distance.</u>

During the AFM operation one can look at:

- tapping oscillation **amplitude**
- tapping oscillation **phase**
- tapping oscillation **resonance frequency**

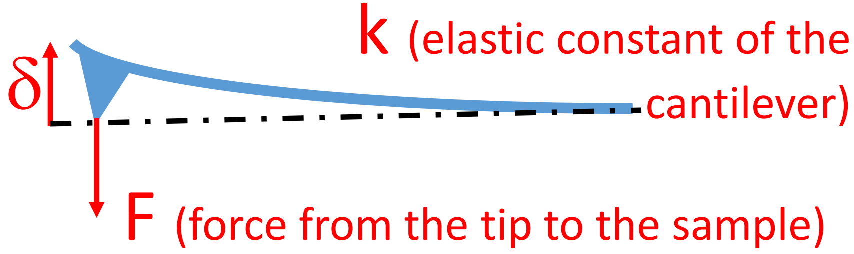
One immediatedly sees that oscillation amplitude $\propto$ F :

Resonance frequency $f_R = (1/2\pi \times \sqrt{(k/m)})$

$F=-k\times\delta$

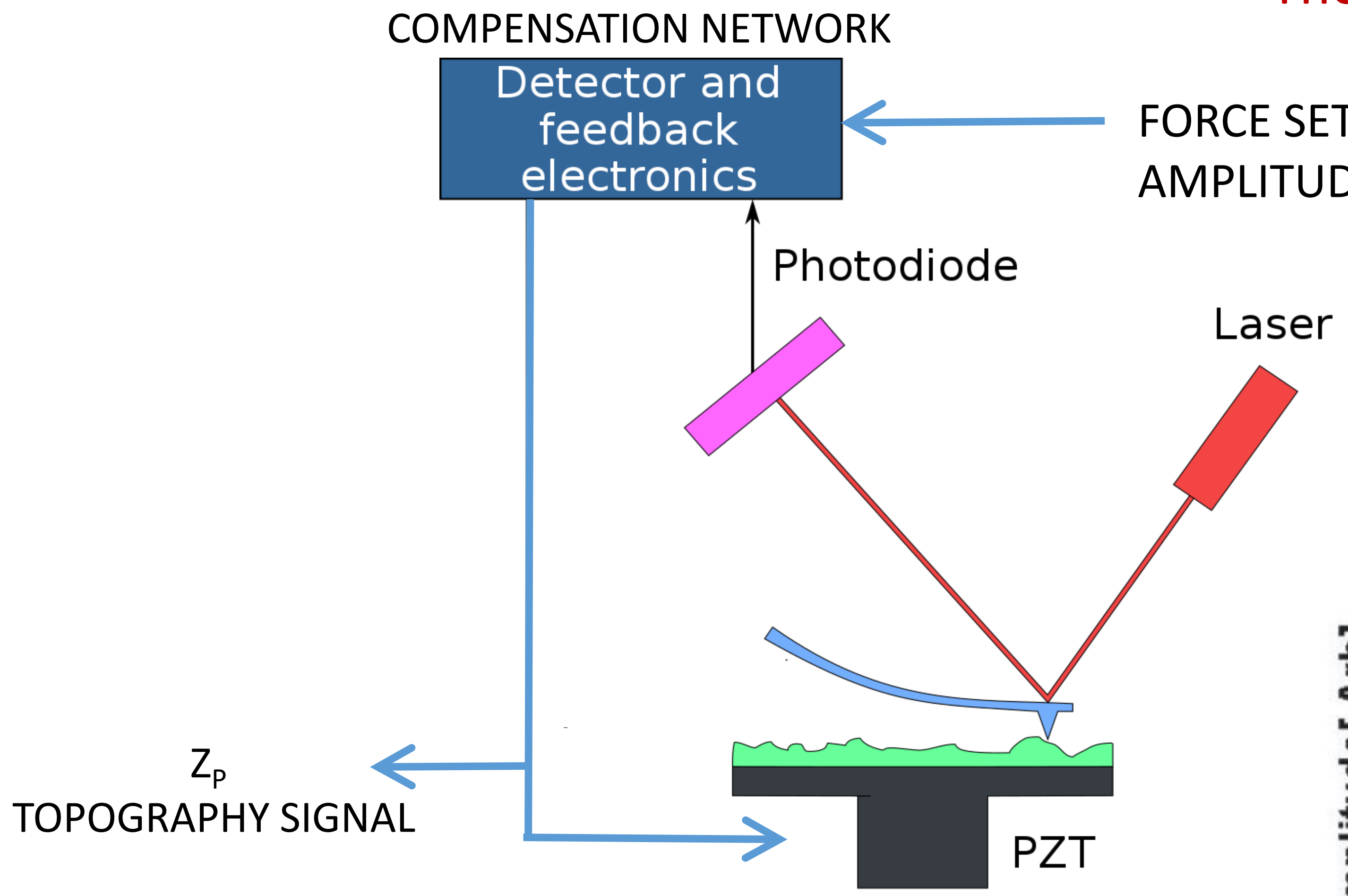
k: 0.005-40 N/m

Shorter/ticker cantilever → higher k



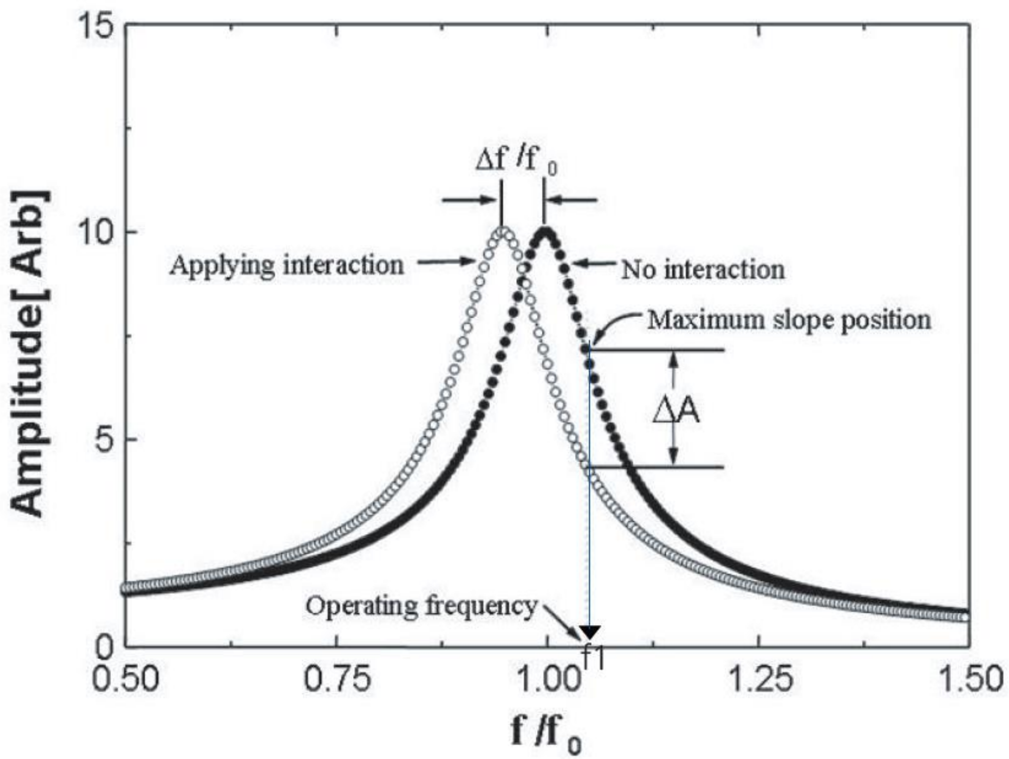
Y. Martin, C.C. Williams, and H.K Wickramsinghe, *J. Appl. Phys.* **61**, 4723-4729 (1987): AFM with a vibrating cantilever

The tapping mode



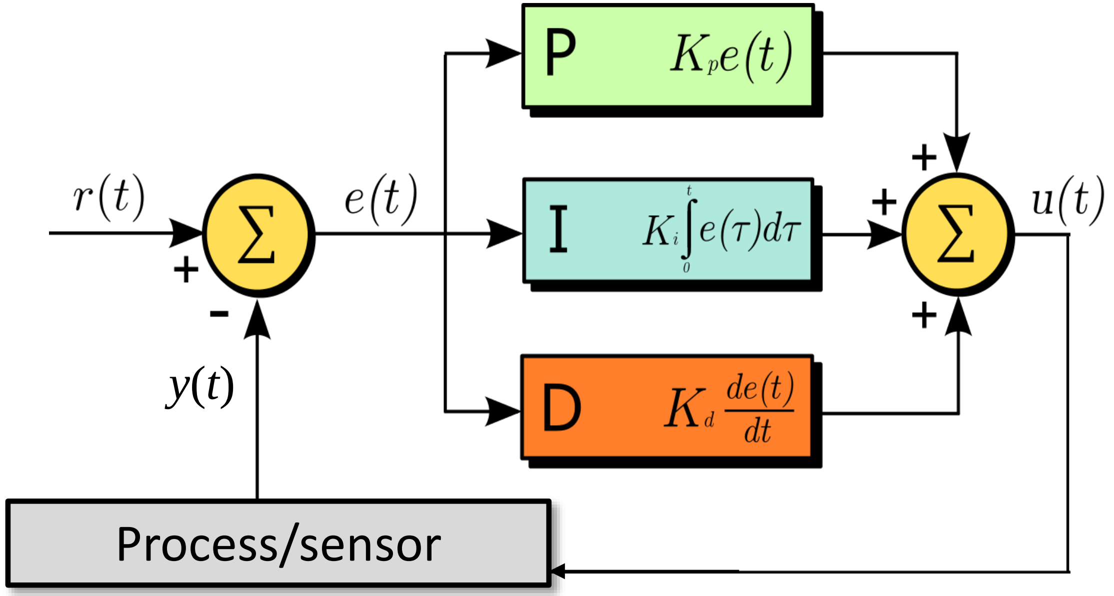
FORCE SET-POINT (or, equivalently, AMPLITUDE SET-POINT)

PID FEEDBACK ON THE Z_P POSITION OF THE PIEZO TO RE-ESTABLISH THE DESIRED AMPLITUDE



https://parksystems.com/images/menu/afm_technology/Non-Contact-AFM-in-Ambient-Atmosphere.pdf

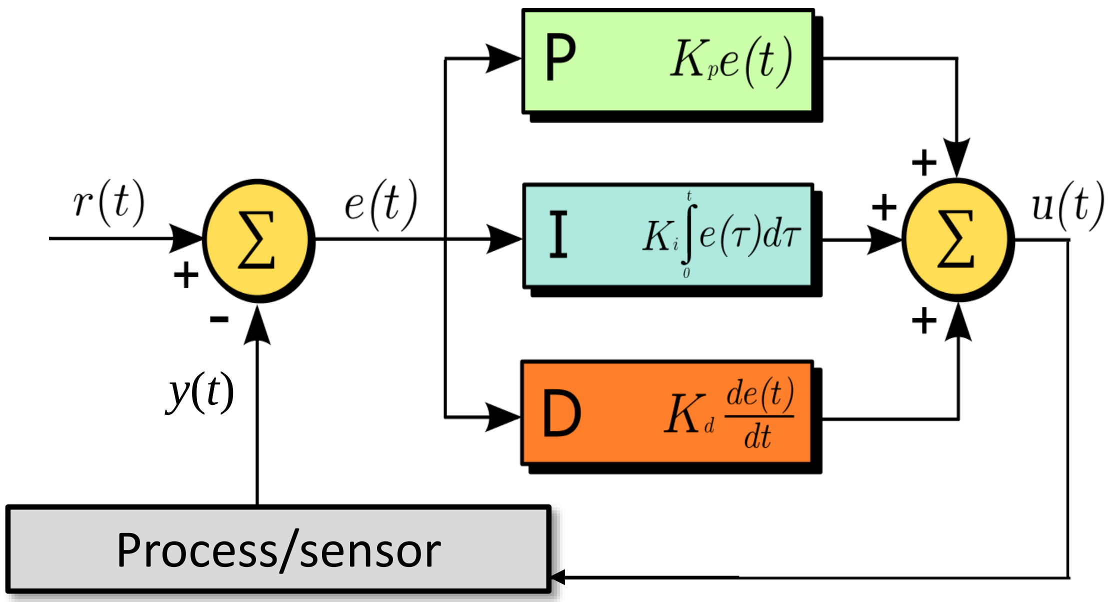
PID controller



r(*t*): Set-point. Here, fixed amplitude
y(*t*): Measured value of the experimental variable. Here, deflection amplitude, through the photodiode
e(*t*): error = *r*(*t*) - *y*(*t*)
u(*t*): correction value, to the process (here, increasing or decreasing the *z* vertical coordinate of the piezo)

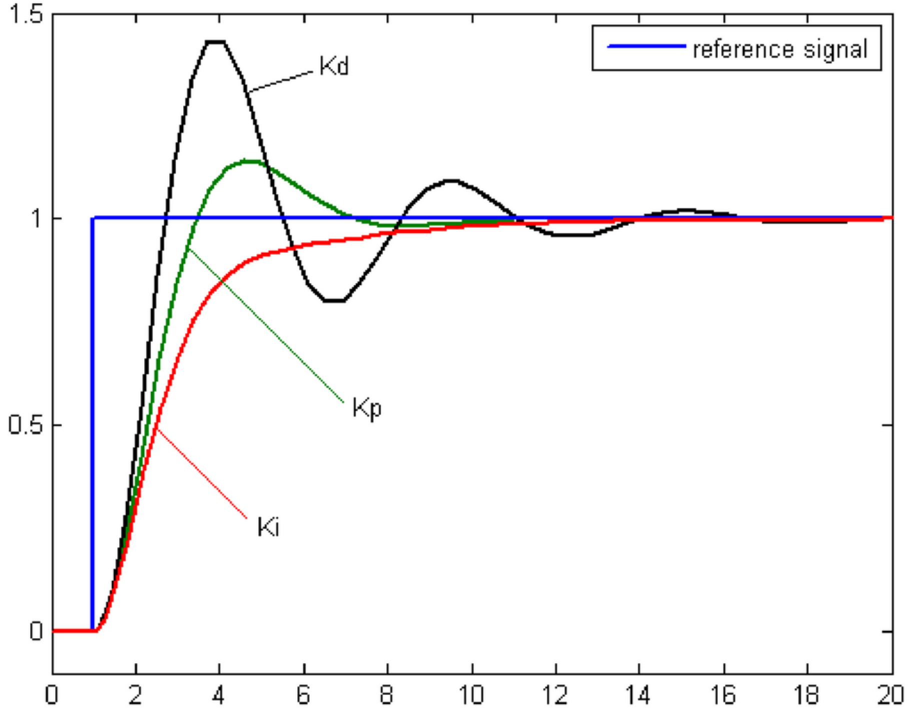
P: proportional to the current value of the error *e*(*t*). It needs $e \neq 0$ to apply a corrective response.
I: accounts for past values of the error (history), integrates them over time to produce the corrective response. For example, if there is a residual SP – PV error after the application of proportional control, the integral term seeks to eliminate the residual error by adding a control effect due to the historic cumulative value of the error. The proportional effect diminishing as the error decreases, is somehow compensated for and refined by the growing integral effect.
D is a best estimate of the future trend of the error, based on its current rate of change (called "anticipatory control“). The more rapid the change of *e*(*t*), the greater the dampening effect.

PID controller



UNIVERSITÀ DI PISA

r(*t*): Set-point. Here, fixed amplitude
y(*t*): Measured value of the experimental variable. Here, deflection amplitude, through the photodiode
e(*t*): error = *r*(*t*) - *y*(*t*)
u(*t*): correction value, to the process (here, increasing or decreasing the *z* vertical coordinate of the piezo)



AFM modes

Contact mode: the deflection of cantilever is normally kept constant
Non-contact mode: amplitude of the oscillation (i.e. the force between the cantilever and the tapped surface) kept constant, set at about 100% of "Free" amplitude;
Tapping mode: amplitude of the oscillation (i.e. the force between the cantilever and the tapped surface) kept constant, set at about 50 -60% of "Free" amplitude. Resolution by tapping is higher than resolution by non-contact;
What information from phase? Phase lag between the signal that drives the cantilever to oscillate and the cantilever oscillation output signal. In Tapping-Mode AFM, the cantilever is excited into resonance oscillation with a piezoelectric driver, and phase imaging is used to map variations in surface properties such as elasticity, adhesion, friction, different compositions, contaminations etc., which all may cause the phase lag.

In all the modes above, the topography image is generated from the piezo-scanner z-motion*. The scanning speed is thus limited by the response time of the feedback circuit (we'll see this in the Lab!).

* Another method for the contact-mode could be keeping constant the height (of the piezo-scanner): in this mode, the spatial variation of the cantilever deflection is used directly to generate the topographic data. Constant-height mode is sometimes used for collecting atomic-scale images of atomically flat surfaces, where the cantilever deflections (i.e. force variations) are very small. Constant-height mode is also useful for recording real-time images of changing (dynamic) surfaces, where high scan speed is crucial.

AFM modes

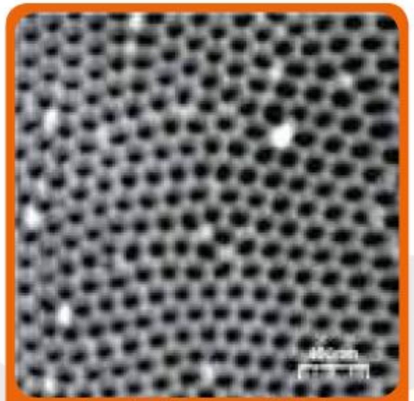
Lateral force microscope (LFM): surface friction. LFM measures lateral deflections (twisting) of the cantilever that arise from forces on the cantilever parallel to the plane of the sample surface.

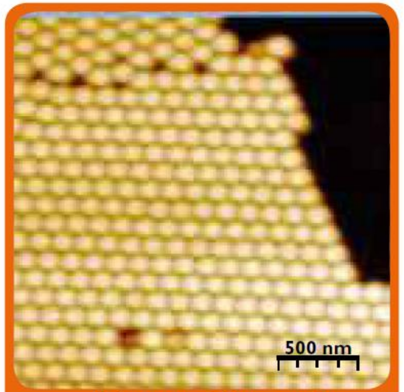
AFM nanolithography: controlled scratching of the probed surface, or controlled deposition/growth of molecules onto the probes surface

Comparison with other microscopy techniques

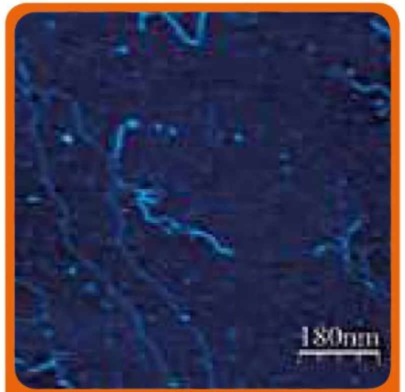
- Optical and electron microscopes easily generate 2D images of a sample surface, with a magnification as large as 1000× for an optical microscope (limited by the used light wavelength and the numerical aperture of the optical set-up), and a few hundreds thousands 100000× for an electron microscope (limited by electron scattering).
- However, by these microscopy technique measuring the vertical dimension (z-direction) of the sample, the height (e.g. particles) or depth (e.g. holes) of the surface features, is very difficult (everything within the depth of focus of the used beam is generally imaged in 2D).
- AFM, using a sharp tip to probe the surface features by raster scanning + feedback on the vertical piezo, can image the surface topography with extremely high magnification, up to 1000000×, comparable or even better than electronic microscopes.
- The measurement of an AFM is made in 3D, the horizontal X-Y plane and the vertical Z dimension. Resolution (magnification) at Z-direction is normally higher than X-Y.

Exemplary AFM images, from the Nanomagnetics ezAFM+ brochure:

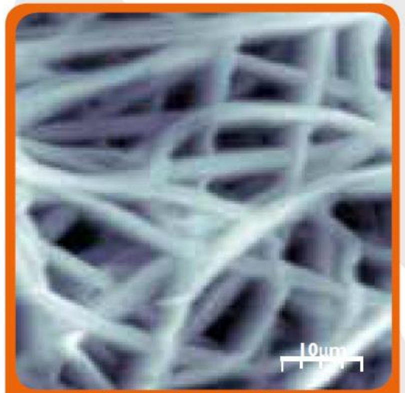

Anodic Aluminum Oxide[1]



Polystyrene Spheres on Si



DNA

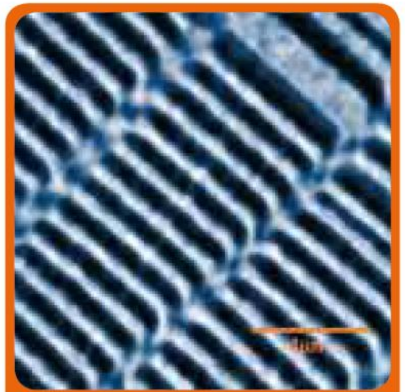


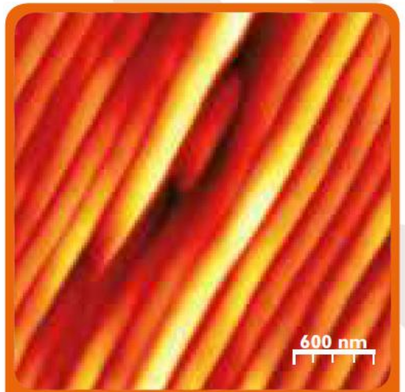
Polycaprolactone Fibers[3]

PS-b-PMMA Phase Image[4]

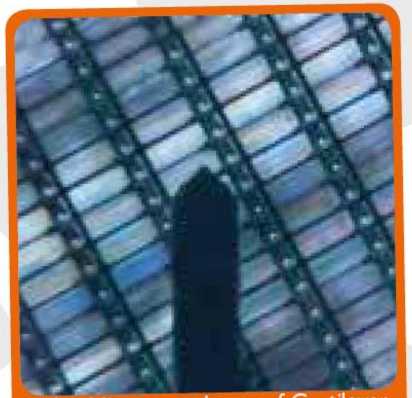
Organic Single Crystal[2]

Red Blood Cells


MFM Image of HDD



Nanostructured Substrate[5]


Video Microscope Image of Cantilever

(1) Sample courtesy of Dr. Fatih Buyukserin, TOBB University (2) Sample courtesy of Dr. Yasuo Nakayama, Chiba University (3) Sample courtesy of Dr. Aylin Sendemir, Ege University (4) Sample courtesy of Dr. Serdar Onses, Erciyes University (5) Sample courtesy of Dr. Francesco Buatier, Genova University

https://www.nanomagnetics-inst.com/en/home/
See also videos at https://www.nanomagnetics-inst.com/en/products/ambient-spms/ezafm-plus/

https://youtu.be/YQL1u101kwE

https://youtu.be/gK_MFXWojVE