Introduction to
Scanning Probe Microscopy

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Overview

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What is a surface?

The definition of a "surface" in the microscopic world is not as trivial as in the macroscopic world (in a solid sample the atoms at "surface" are separated by distances much higher than the mean nuclear diameter, with the electrons surrounding the nuclei distributed in orbitals extending much beyond the intra-nuclear distances).

A practical definition is the one used for SPM: "*the locus of points traced by a "point-probe" while keeping constant the probe-sample interaction*"

The probe may be a "tip" (or an "electron beam" associated to a sensor detecting the effect of the beam interaction with the scanned surface, as in SEM).

Most SPM use a *piezoelectric scanner* to control the position (x,y) of the point-probe over the sample, and the probe-sample *distance* (z)

The probe signal is processed by a computer to build the 3D image $I(x,y,z)$

"3D topography" defines the height profile of the sample surface, i.e. the local measurement of the deviation from an ideal flat plane.

Images at nanometric resolution with real 3D topography may only be obtained using Scanning Probe Microscope.

Surface morphology techniques
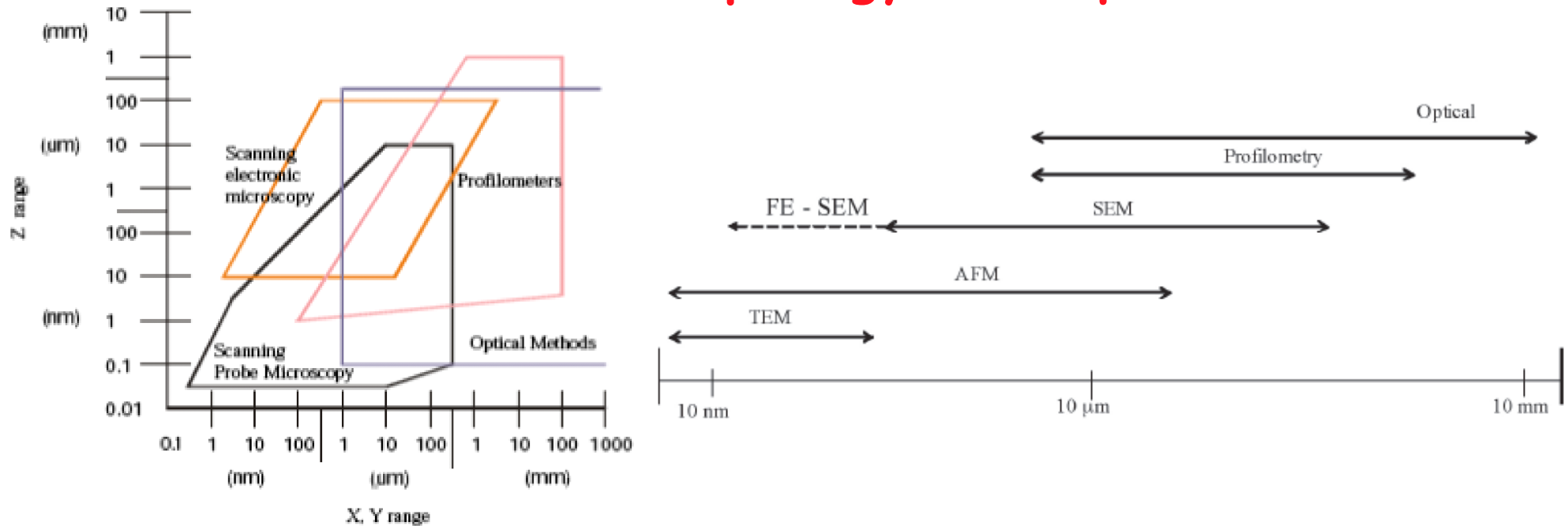
Comparison of optical, electronic and probe microscopes

	Optical	SEM	SPM
works in :	air, liquid, vacuum	vacuum	air, liquid, vacuum
Field depth :	small	large	medium
Resolution (x,y) :	1.0 µm	5nm	2-10nm (AFM) 0.1nm (STM)
Resolution (z) :	absent	absent	0.1nm (AFM) 0.01nm (STM)
Magnification :	$1X \rightarrow 2 \cdot 10^3 X$	$10X \rightarrow 10^6 X$	$5 \cdot 10^2 X \rightarrow 10^8 X$
Sample preparation	small	important	no
Needed sample features	not totally transparent	conducting, vacuum compatible	roughness not large than 10µm

Different types of SPM

Different kind of probe-sample interaction correspond to different types of SPM.

In **SEM-STEM** = Scanning (Transmission) Electron Microscope, the signals are produced by detectors that monitor the effect of electron collisions with the sample (*X-rays, reflected or transmitted electrons…*)

In **STM** =Scanning Tunneling Microscope, the signal is the *tunneling electron current*.

In **SFM** =Scanning Force Microscope, the signal is due to some *interactive force*
   (Van der Waals, magnetic, electric, friction,...).

In **SNOM**=Scanning Near Field Optical Microscope, the signal is the "*evanescent field*"
*of electromagnetic radiation* measured through a small aperture (usually an optical fiber).

Electron microscopes

Electron microscopes may be divided into two categories:

SEM, for Scanning (reflection) Electron Microscope
(S)TEM for (Scanning) Transmission Electron Microscope.

In both the image is normally obtained by detecting the signal produced by an electron beam at high energy (10÷500 keV), generated by an electron gun.

The electrons penetrate a region where suitable electrodes (electromagnetic lenses) create magnetic or electric fields that track the beam in a raster scanning of the sample surface (over an area of few mm^2).

Lab. Fis. Mat. G.Torzo
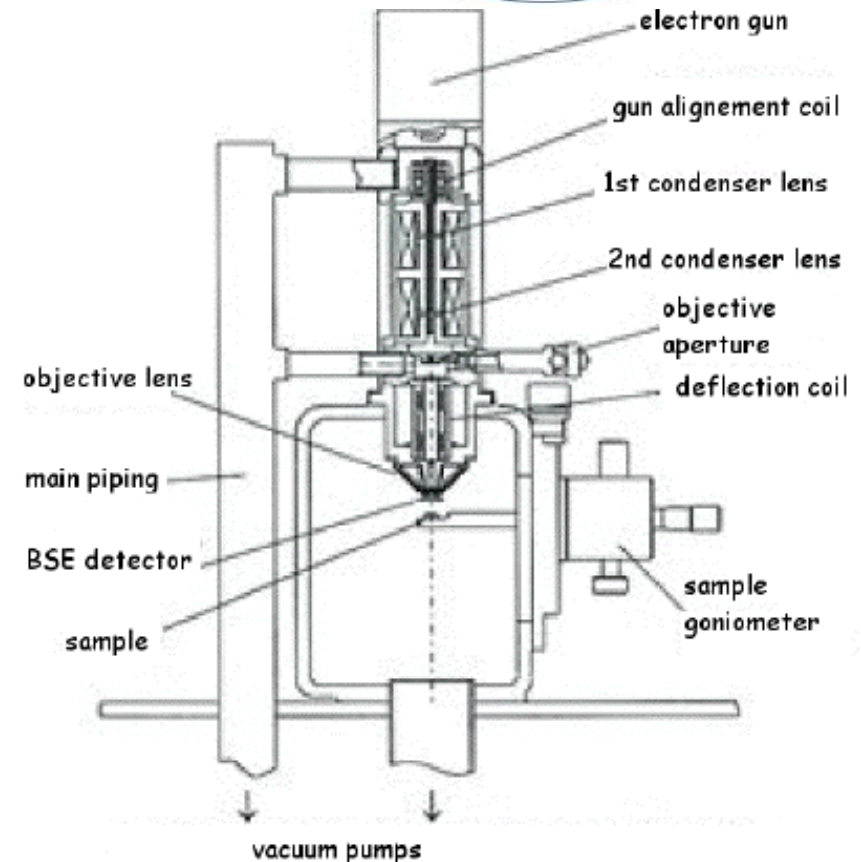
SEM

To avoid energy loss and diffusion, which would spoil the monochromatism and the collimation of the electron beam, one must keep the electron gun, lenses and sample inside a ultra-high vacuum chamber.($\approx 10^{-6}$ - 10^{-12} Pa).

The electron collisions with the sample produce emission of photons (or secondary-electrons) that, in the case of SEM, are collected by suitable photo-detectors generating an *image* of the swept area.

The electron beam interacts with the sample within a surface layer whose thickness ranges from few nm up to several μm. Also primary electrons (backscattered) and secondary electrons are emitted from the sample.

The secondary electrons (from the conduction band) are usually the signal used for morphologic SEM analysis. They have low energy (<50 eV) and only those produced in the first layers (few nm) may escape the surface to be detected by sensors.

Also primary electrons backscattered from deeper layers may produce secondary electrons close to the surface, but far from the entering beam spot.

SEM-TEM images

The image may be produced also collecting the *current* produced by the electron beam (EBIC = Electron Beam Induced Current), or the AUGER electrons, or X-rays, or cathode-luminescence electrons.

In STEM the image is created by electrons *transmitted* through the sample that is made extremely thin (z<1μm) in the investigated area.

A strong interaction is therefore unavoidable : the sample is always damaged.
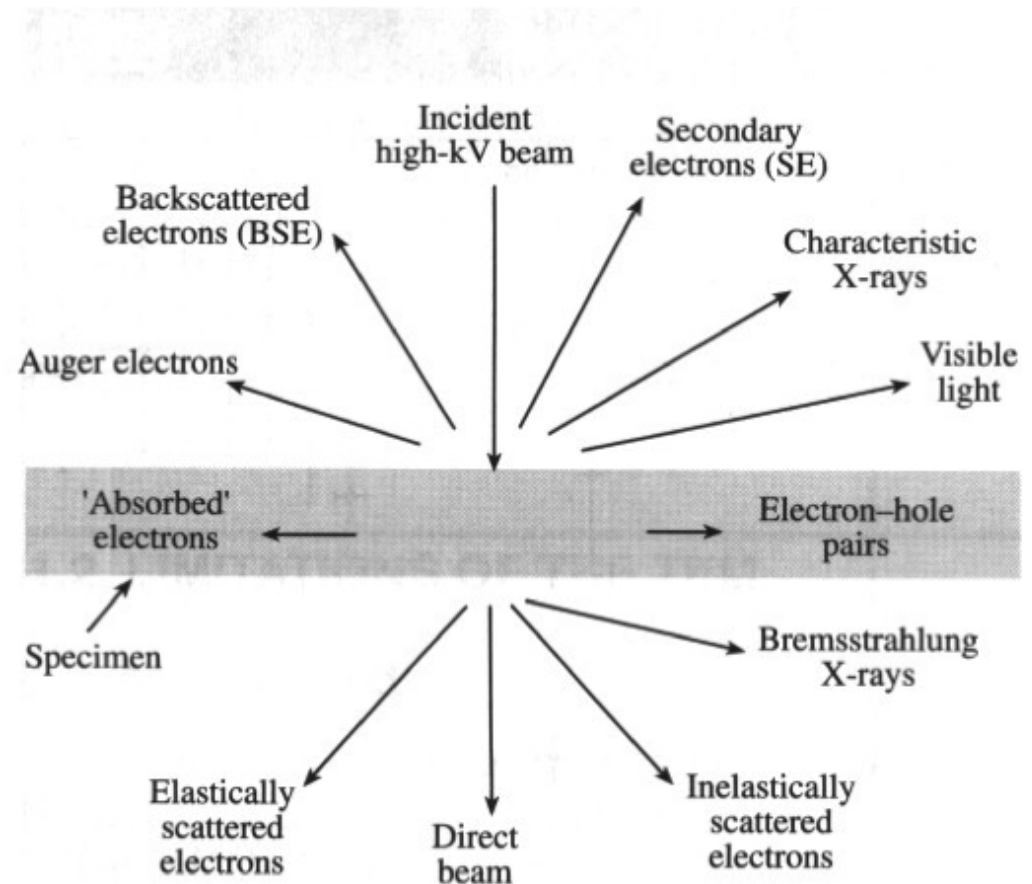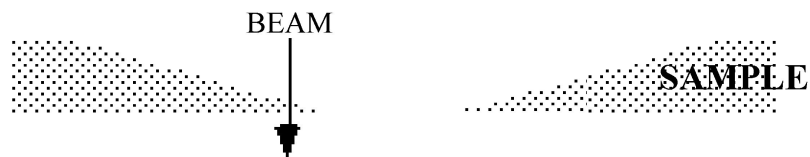
BEAM

SAMPLE

Comparing SEM-AFM

Any SEM or TEM image bears information produced by a sample layer (whose thickness depends on the *sample nature* and by the *beam energy*) which is quite thin, but never reaches the limit of a single atomic layer.

The SEM e TEM images give information on the *composition and microscopic structure* of the surface layer, *but not a real 3D topography*.

SEM-TEM require advanced skillness of the operator,

and are intrinsically very expensive devices (involve UH-Vacuum and high voltages)

Although the time required for making a measurement with the SEM image is typically less than an AFM, the amount of time required to get meaningful images is similar.
This is because the SEM/TEM often requires substantial time to prepare a sample.
With AFM no sample preparation is required.
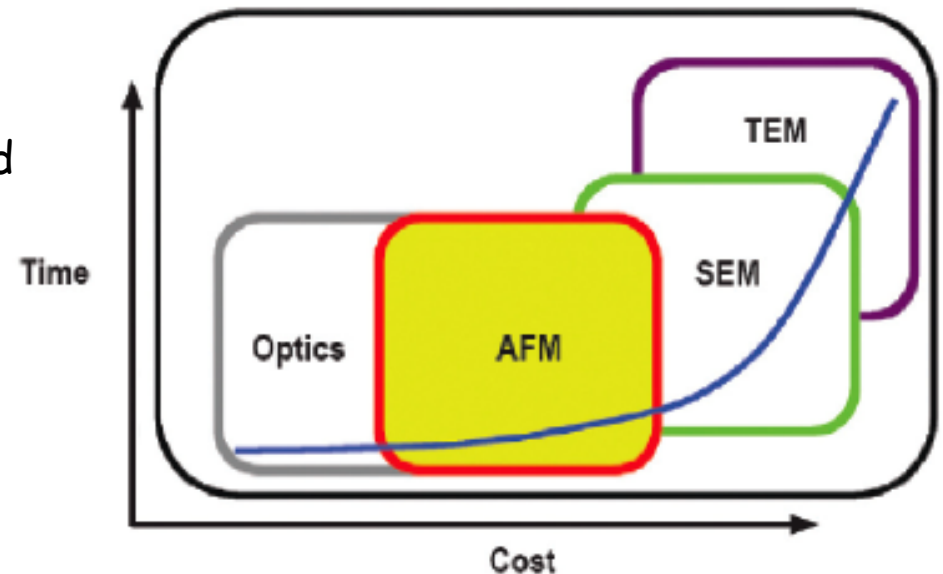
The precursor of STM

The "topografiner" was the first device using electron field emission to obtain surface topography of a sample
(electron field emission current between a sharp metal probe and a conductive surface is strongly dependent on the gap)
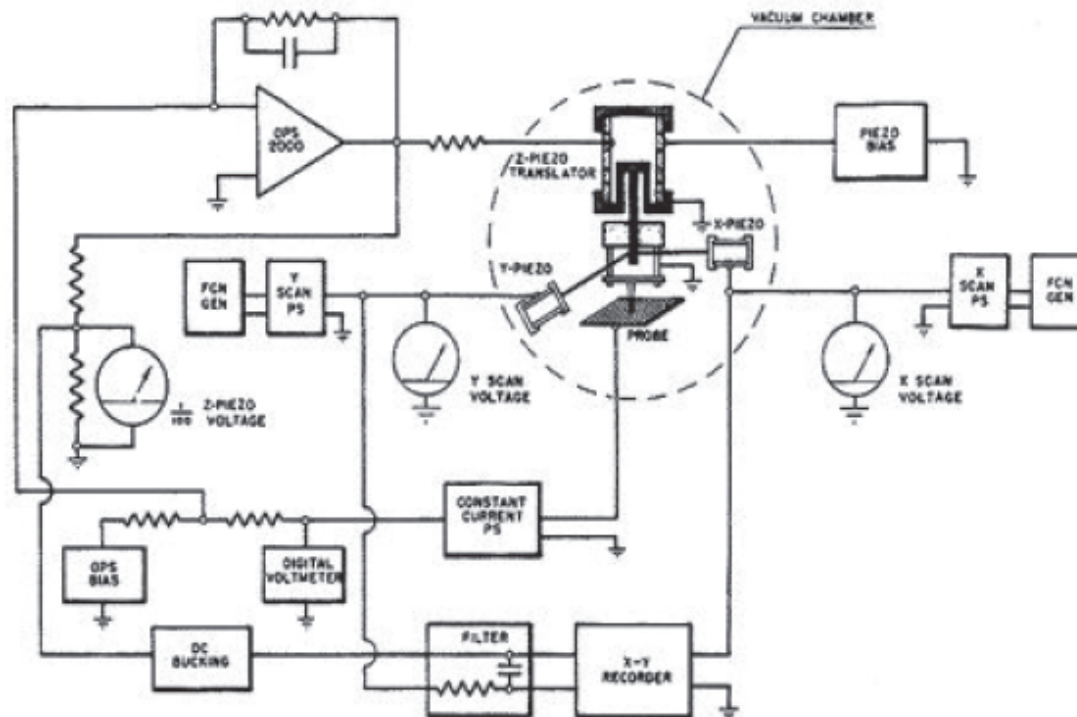
R. Young, J. Ward, F. Scire, *The Topografiner: An Instrument for Measuring Surface Microtopography*, Rev. Sci. Inst., Vol 43, No 7 (1971)

STM : Scanning Tunneling Microscope

The "tunneling" is a quantum effect: it describes a particle that crosses a potential barrier.

Considering charged particles (electrons) with total energy E_0, inside a box whose walls are a "shell" at potential E_1, with $E_1 > E_0$, within a classical model it is impossible that these particles escape the box.

To escape they should gain an energy larger than E_1.
Within quantum model a finite probability exist for a particle with energy $E_0 < E_1$ to cross the potential barrier: this crossing the barrier is named *tunnel* effect.
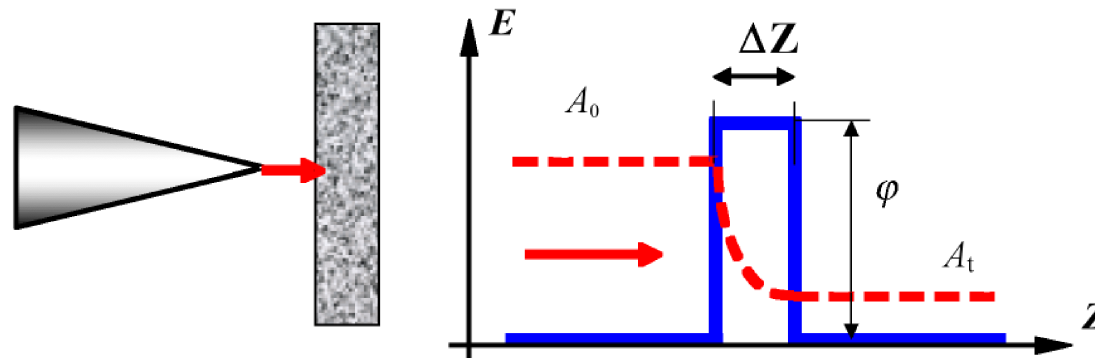
The working principle of STM is based on the electron tunneling through the narrow potential barrier between a metal tip and a conducting sample in an external electric field.

Tunneling probability

The probability W of electron tunneling (transmission coefficient) through one-dimensional rectangular barrier is :

$$W = \frac{|A_t|^2}{|A_0|^2} \cong e^{-k\Delta z}$$

where A_0 is the amplitude of electron wave function approaching the barrier; A_1 the amplitude of the transmitted electron wave function, k the attenuation coefficient of the wave function inside the potential barrier; Δz the barrier width.

In the case of tunneling between two metals, the coefficient k is:

$$k = \frac{4\pi\sqrt{2m\varphi}}{h}$$

where m is the electron mass, φ the average electron emission work function and h the Planck constant.

For small values of the bias voltage (eV< φ), the current density $j(z)$ can be approximated as:

$$j(z) = j_0(V)\, e^{-\frac{4\pi}{h}\sqrt{2m\varphi}\Delta z}$$

with jo(V) ≈ V/Δz

STM : vertical and lateral resolution

Assuming :

$$j \approx (V/z) \exp(-kz),$$

where V is the tip-sample bias voltage and, k is of the order of 10^{-10} m, by using the tunneling current as a measurement of the tip-sample distance (the barrier width z)
we may reach, with a current resolution of only 10%, the excellent vertical resolution of 0.01 nm .

If we model the tip with a paraboloid with curvature radius R at the apex, the current density j has a Gaussian profile along a plane parallel to the sample surface described by the function:

$$j(x) \approx (V/z) \exp(-kx^2/R),$$

giving a lateral resolution (for $R \approx 100$ nm) of the order of 2 nm.

Tip   Sample

V

Tip

Sample

I

Piezo x-y-z

Computer

electrons

barrier
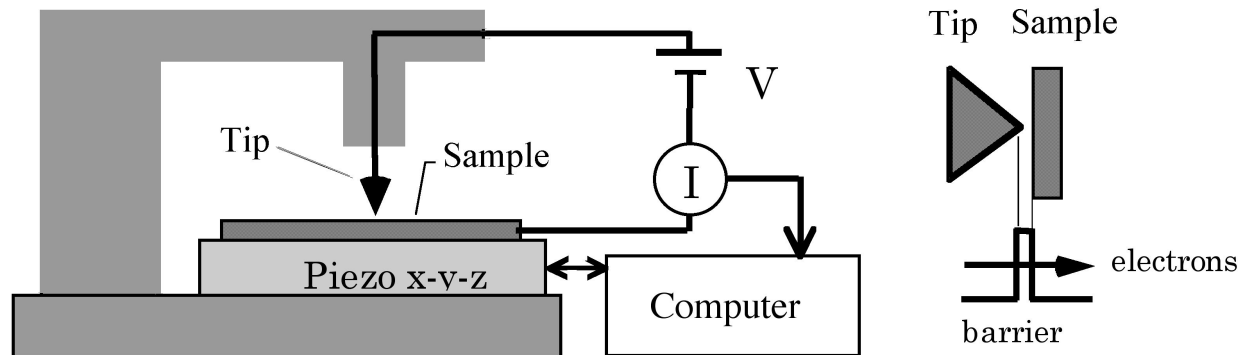
The feedback principle

An extremely accurate positioning of the tip with respect to the sample may be obtained using a piezo-transducer (PT) and a feedback control system (FS).
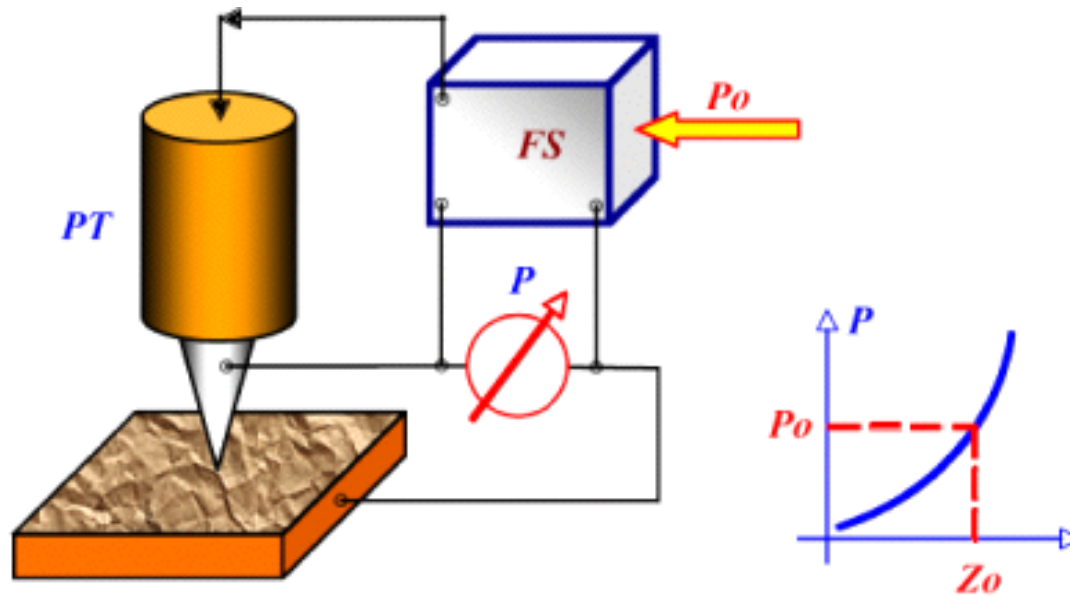
For a given function P(z) of a signal depending on the tip-sample distance z, the feedback system keeps constant the value of the parameter P (equal to the value Po, set by the operator).

If the tip-sample distance changes, the change in the parameter P is amplified and fed to the piezo transducer that controls the tip-sample separation, bringing it back to the preset value.   The measured value of the signal fed to the piezo gives the topographic z-value for each point (x,y) of the sample surface.

Tunneling Spectroscopy

The tunneling effect may be used for spectroscopy of electronic states by recording the dependence of the tunneling current on the local density of states (LDOS).

The STM allows to acquire the voltage-current characteristics (VCC) in any point of the surface and, therefore, to examine the local electric properties of the sample.

The expression for the tunneling current in the point r_0 on the sample surface is:

$$I(r_0, V) \propto \int_0^{eV} dE \rho_T(E) \rho_S(r_0, E) D(r_0, E)$$

where $D(r_0, E)$ is the barrier transparency, $r_T(E)$ is the density of the states connected with the tip, $r_S(r_0, E)$ is the density of the states in the r_0 point of the sample. The tunneling current is a convolution of density of states of the tip and of the sample, and with an assumption that the density of states near the Fermi level in the tip metal is *practically constant*, the equation for the current may be written as :

$$I(r_0, V) \propto \int_0^{eV} \rho_S(r_0, E) dE$$

The dependence of $I(r_0, V)$ on the voltage V is determined by the density of states of the sample. Practically, the $r_S(r_0, E)$ value is estimated by the voltage derivative of the tunneling current, taken at constant tip-sample distance.

$$\rho_s(eV) \propto \frac{\partial I}{\partial V}$$

Tunneling current spectra

Semiconductor samples have a complex structure of the electron energy spectrum. Presence of the energy band-gap and impurity levels in the semiconductor spectrum makes the VCC of the metal-semiconductor tunneling contact nonlinear. A contribution to the tunneling current is also given by surface states and energy levels due to foreign atoms absorbed on the surface. This is why examination of local tunneling spectra of semiconductor materials is performed in high vacuum.

Tunneling spectra allow to determine the arrangement of edges of the conducting and the valence bands with respect to the Fermi level as well as to identify the peaks connected with the impurity states inside the semiconductors band-gap.

The STM surface image depends on the value and polarity of the voltage applied to the tunneling contact. This is related to the features of electrons tunneling from the tip into the sample free states or from the sample filled states into the tip
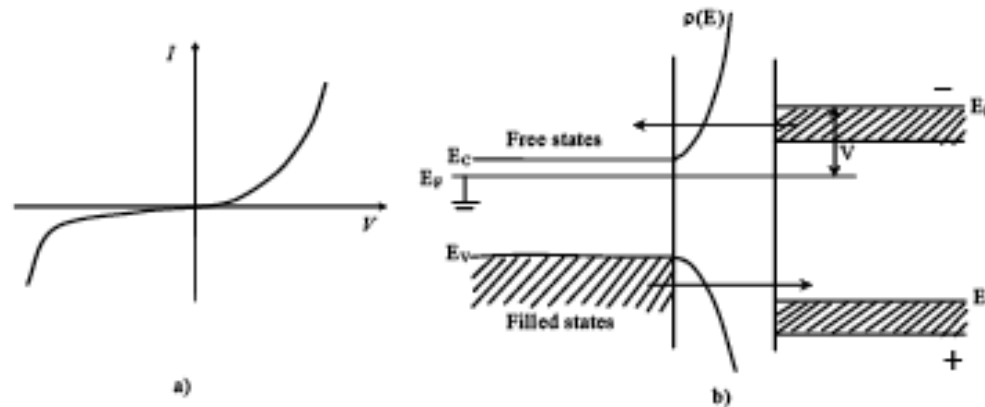
Tunneling current images with different bias voltage
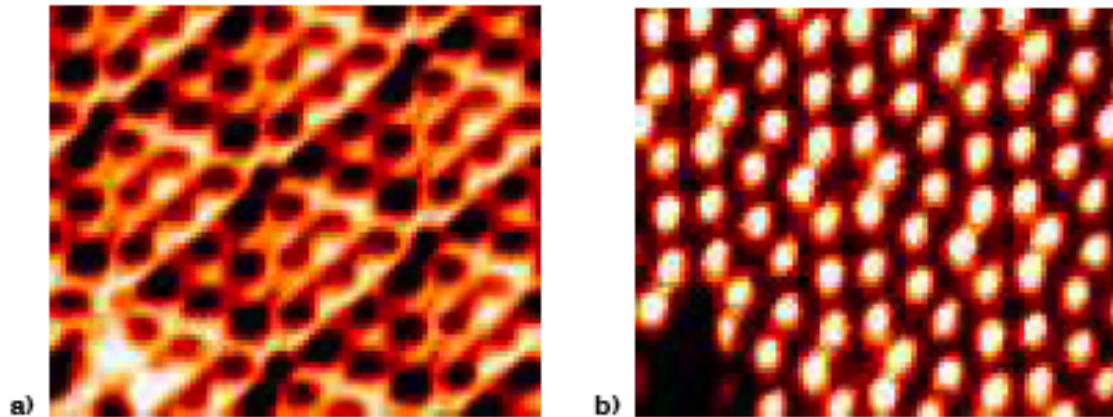
Two STM images of the Si (111)–(7x7) reconstructed with different voltages applied to the contact :
a) V=+0.4V   b) V=-1.4V

In the Voltage-modulation mode, besides the $V_=$ constant bias voltage, a $V_\sim$ small alternating voltage is applied to the tunneling contact. The resulting AC modulation of the tunneling current $I_\sim$ is :

$$I_\sim = \frac{dI}{dV} \propto \rho\left(X,Y,E_F + eV_=\right)$$

In the constant current mode, variations of the work function f may produce distortion of the topographic image. Those small distortions, (within a few angstroms for V change in the range 2 ÷ 5 V), may be taken into account by measuring f(X,Y). To do so, the tip-sample distance must be modulated at small amplitude d , and the AC component $I_\sim$ of the tunneling current is:

$$I_\sim = I_\tau \frac{\delta}{2} dI_\tau / dZ = I_\tau \frac{\delta}{\hbar} \sqrt{2m\varphi}$$

The piezo-scanner

It is necessary to control the tip-sample distance and to move the tip over the sample surface with high accuracy (at subnanometric level) in order to make the SPM properly working.

This performance can be achieved using scanners made of piezoceramics
(e.g. $Pb(ZrTi)O^3$ = lead zirconate titanate),
material that change size in an external electric field (inverse piezoelectric effect).

The piezoceramics is a polarized polycrystalline material, obtained by powder sintering from crystal ferroelectrics.

Polarization of ceramics is performed by heating above its Curie temperature T_c (≈300°C), and then slowly cooling in a strong electric field (≈3 kV/cm).

After cooling below Tc, piezoceramic retains the induced polarization and gets the ability to reversibly change its sizes under an applied electric field.

The inverse piezo-effect

The inverse piezo-effect consists in changes of the piezo-material geometry under the effect of an applied electric field.

In a simple model imitating the SiO_2 quartz structure, silicon positive ions in the lattice alternate with negative oxygen ions.

In the non-deformed cell the centers of positive and negative charges coincide.

If an external voltage is applied to the metal electrodes located on the opposite faces of the quartz crystal, the ions are shifted and this causes a deformation of the crystal lattice.
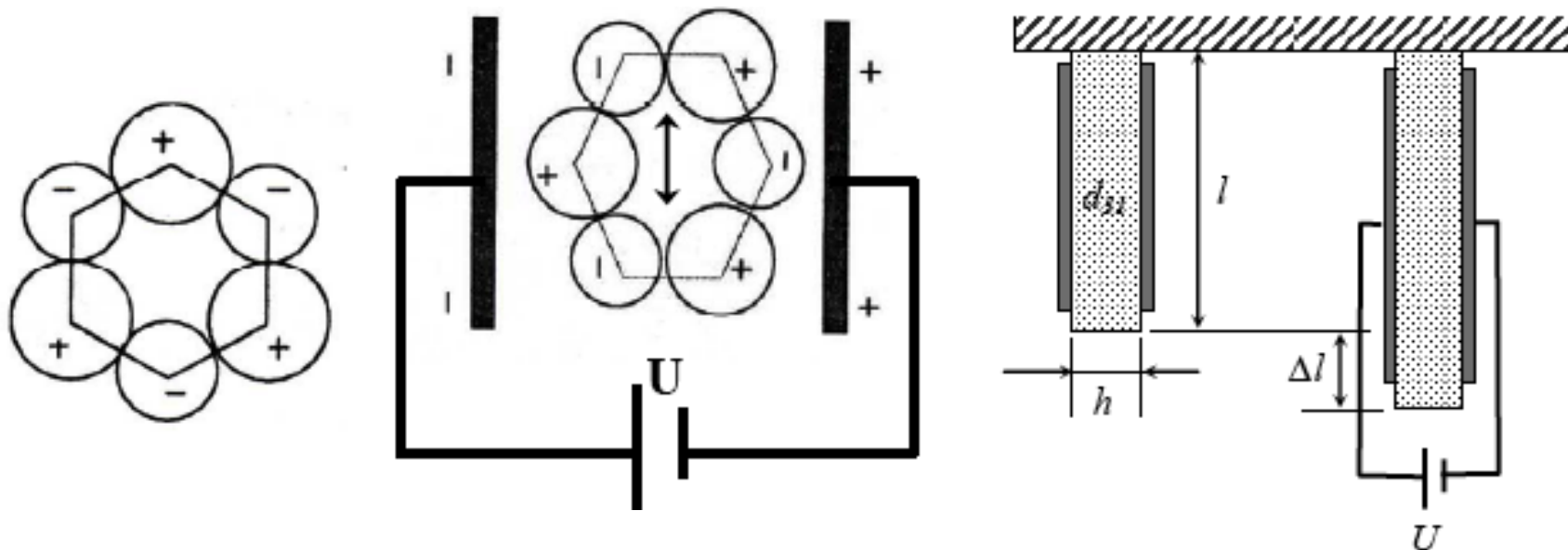
The piezo-scanner (2)

SPM piezo-scanners expand and contract proportionally to the applied voltage.

Whether they expand or contract depends upon the polarity of the applied voltage.

0 V

- V

+ V

No applied voltage

Extended

Contracted
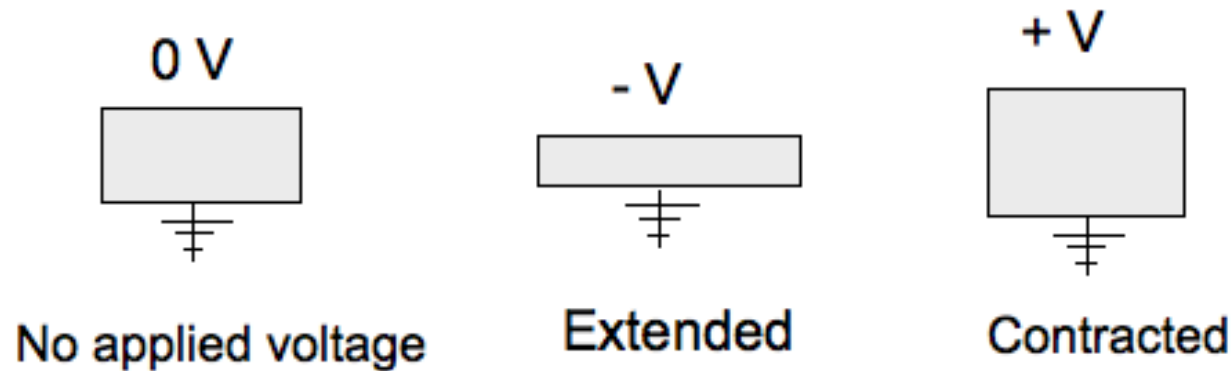
In some versions (scan by sample), the piezo tube moves the sample relative to the tip.
In other models (scan by tip), the sample is stationary while the scanner moves the tip.

Force sensor

x,y,z scanner

Scan by sample

x,y,z scanner

Force Sensor

Scan by tip
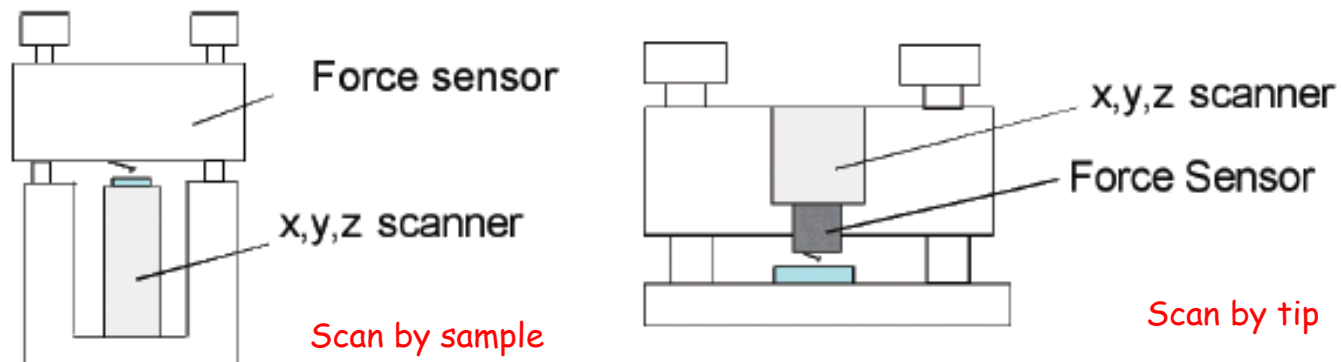
Tripod and Tubular scanners

A piezo-scanner may be made as a "tripod", i.e. 3 piezo-elements assembled along 3 orthogonal axes, or as a "tube" with one inner electrode and 4 outer electrodes.
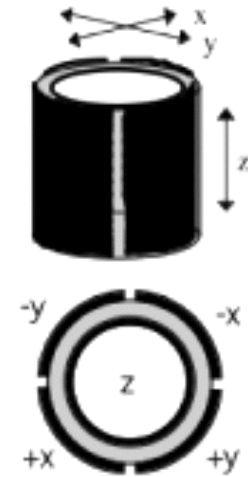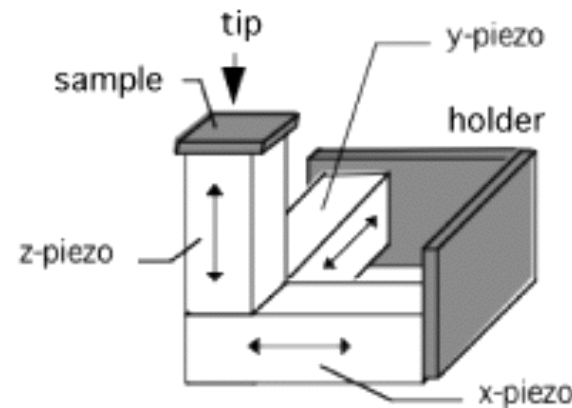
In the tripod the 3 piezo bars (x,y,z) are driven independently by applying voltages across each electrode pairs plated onto the lateral faces.

In the tubular scanner the lateral displacement (x,y) is obtained as deflection of the cylinder axis by applying opposite voltages to the outer electrodes, and the vertical displacement (z) by applying voltage to the central electrode.

The piezo-coefficient is in the range 0.1 to 300 nm/V.

If L, D and W(<<D) are the dimensions (in mm) of the tube height, diameter and wall thickness respectively, the typical displacements Δx, Δy, Δz (in nm/volt) are :
$$\Delta x, \approx \Delta y \approx 0.1 \, (L/D) \, (L/W), \quad and \quad \Delta z \approx 0.2 \, L / W.$$

For example with L≈D≈ 1 cm, W≈ 1 mm, the vertical sensitivity is about 2nm/volt.

Bimorph tripod scanner

Tripod scanners may be made also using bimorph cells.

Bimorph is made of two plates of piezoelectric material, which have been glued together with opposite polarization vectors .
If a voltage is applied to bimorph electrodes, one of the plates will extend, and the other one will be compressed, resulting in a bend of the whole element.
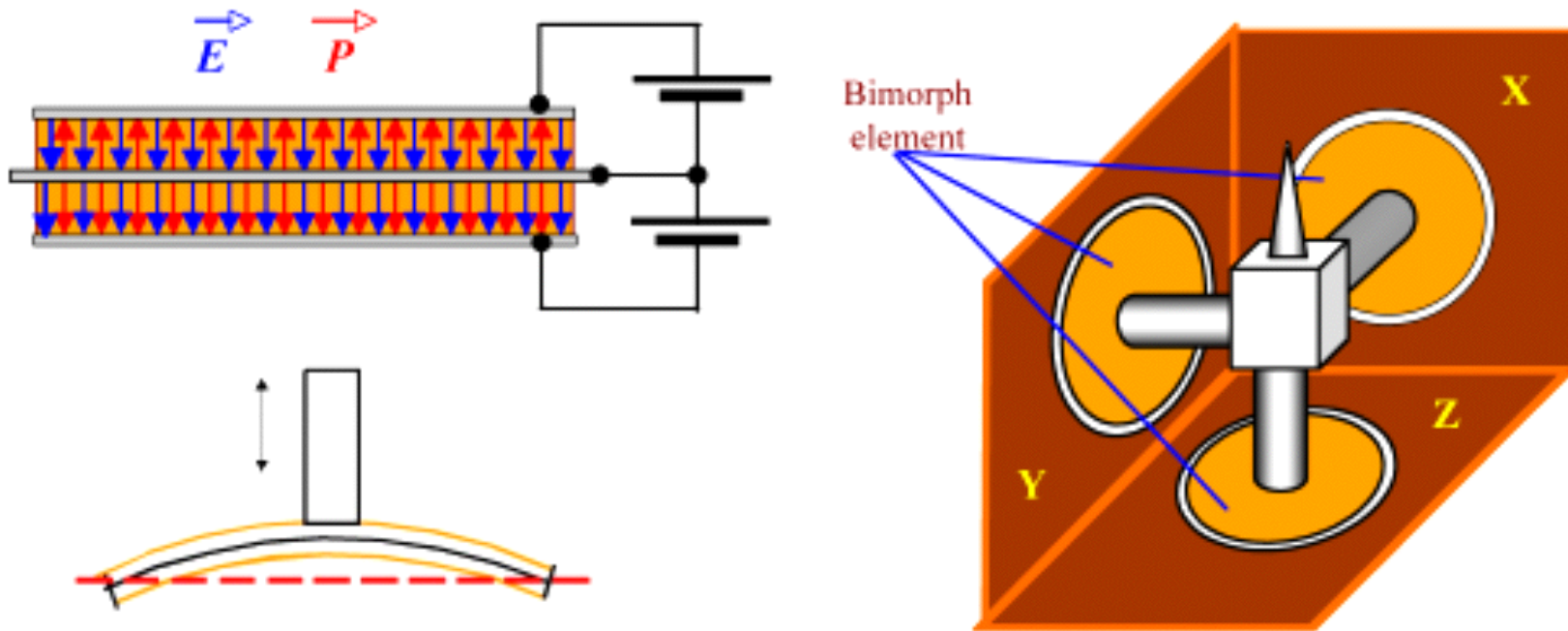
Flexure scanner

A flexure scanner operates by pushing on a flexure with a PZT.
In this design, the ceramic pushes against the flexure which then causes the stage to move.
There is a gain in the motion given by the ratio of L2/L1

$$\Delta r \; \alpha \; \frac{L_2}{L_1} \times V$$
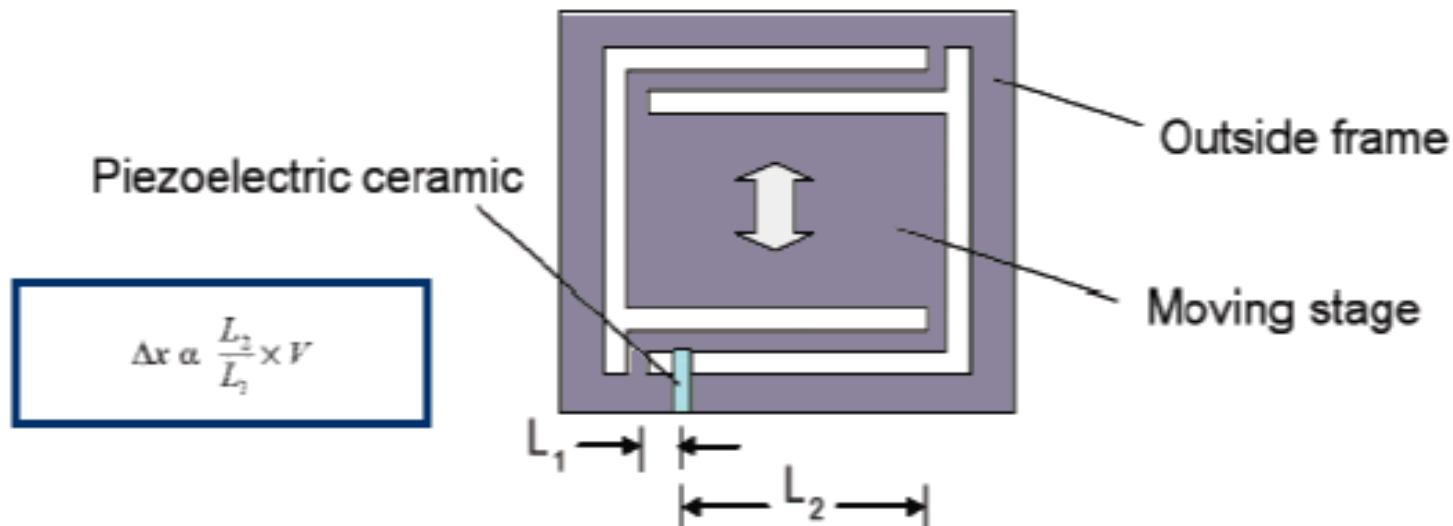
Piezoscanner properties

Electronically, piezoscanners act as capacitors and store charges on their surface.

Capacitances may be as large as 100 μ farads.
Once a charge is placed on the piezoceramic, the piezoceramic will stay charged until it is dissipated.
Electronic circuits used for driving the piezoceramics in an AFM must be designed to drive large capacitive loads.

All piezoceramics have a natural resonance frequency that depends on the size and shape of the ceramic.
Below the resonance frequency, the ceramic will follow an oscillating frequency, at resonance there is a 90 degree phase change, and above resonance there is a 180 degree phase change.
To a great extent, the resonance frequencies of the piezoelectric ceramics limit the scan rates of atomic force microscopes.

Tip-sample approach

Several techniques are exploited to obtain a precise and large movement, to position the tip close to the sample, starting from   macroscopic distance (needed to change sample or tip) :

stepping motors,
controlled by encoders or optical sensors,
or:

inertial stick-slip piezo movers
using "sawtooth" contraction/extension cycles
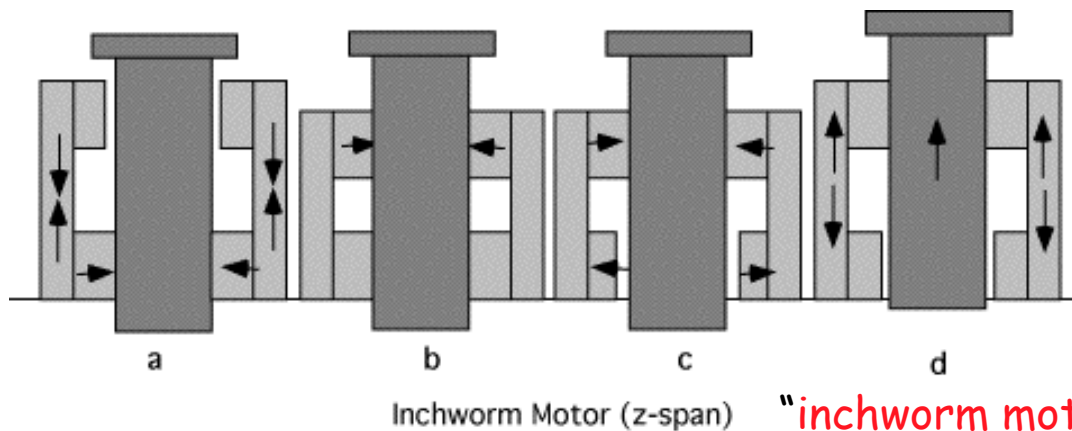
Inchworm Motor (z-span) "inchworm motors",
using contraction/extension cycles of coupled piezo.
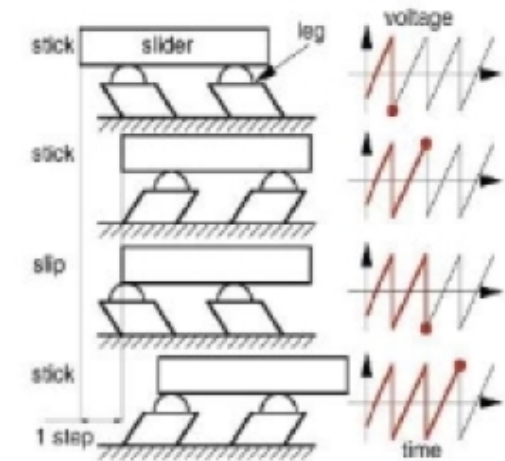
Both inertial and inchworm movers combine very high resolution with a very large range

Vibration insulation

A common feature of SPM is some device for vibration insulation :

active or passive pneumatic or piezo suspension tables,   hanging springs,
magnetic levitation,   acoustic enclosures…
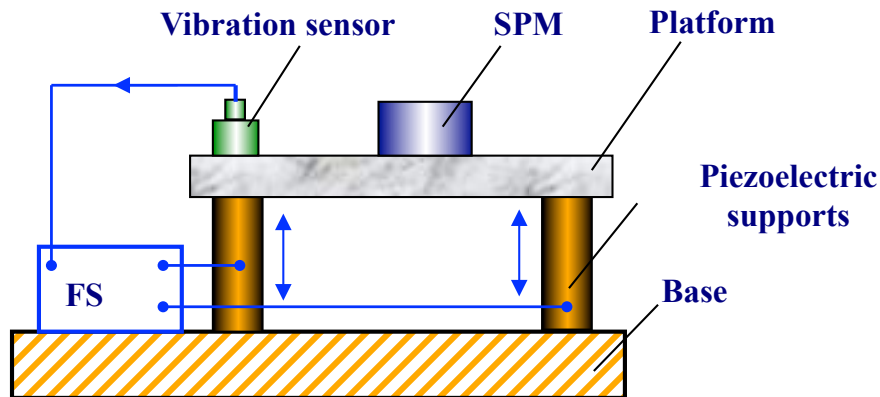
Active piezo suspension table
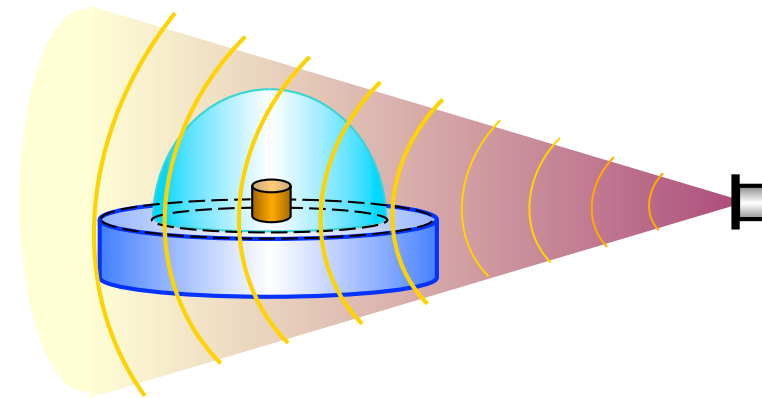
Acoustic insulation enclosure

The mechanical loop in an AFM includes all of the structural elements that are
required to hold the probe at a fixed distance from the sample.
If this loop is not rigid, then the probe can vibrate out of phase
from the sample, and noise is introduced into images.
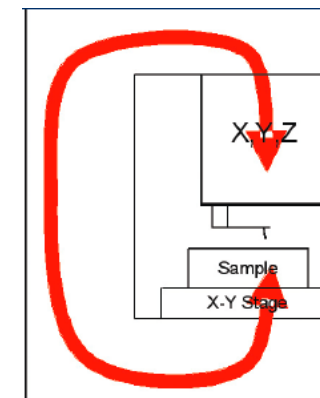
The "reflected beam" force sensor

The most commonly used force sensor in SFM is a micro-cantilever, with the tip attached to the free end, and a thin light beam, produced by a laser diode which is reflected by the cantilever end, and detected by a split photo diode.
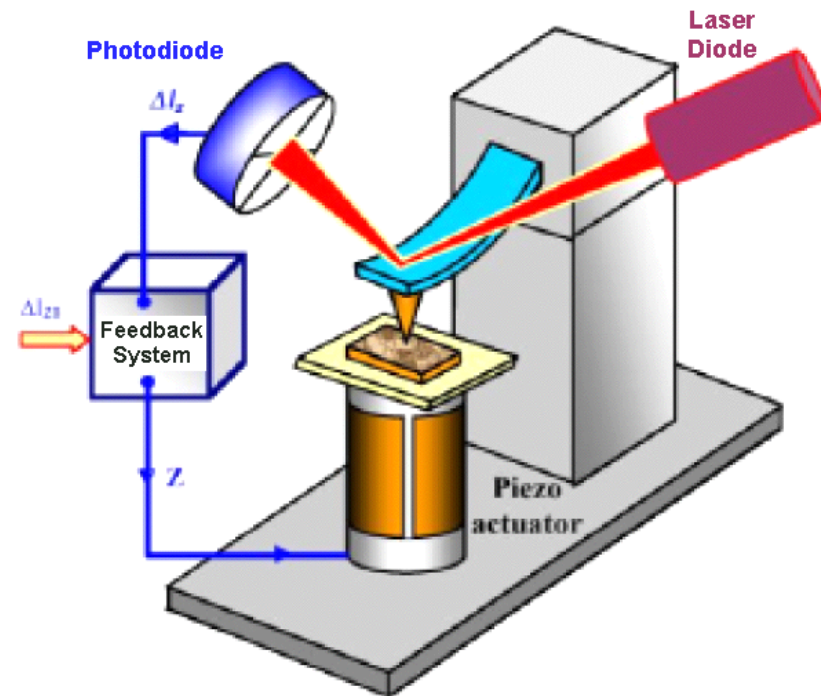
The interactive force between the atoms of the tip and of the sample deflects the cantilever and deviates the reflected laser beam.
The different illumination of the photodiode sectors produces a differential photocurrent signal that measures the interactive force.
This optical detection technique of the tip-sample interaction force is commonly named "**Beam bounce**".

Different methods for detection of the cantilever deflection

STM current modulation,   interferometry,   capacitive bridge,
piezo strain gauges or piezo-actuators

Electron tunneling

STM   Lever
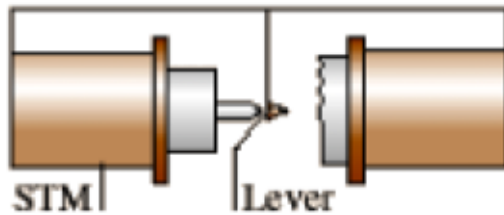
Optical interferometry

Lense

Capacitance method

Electrode

PIEZOTUBE
oscillation amplitude
TIP
V
driving voltage
Current/Voltage
Phase Detector

strain gauges
sample

Contact
Metallization
Contact pad
L_1
L_2
b
w
Substrate
Piezoresistor
Oxide
Metall
Oxide
Intrinsic silicon
Amorphous silicon
Substrate
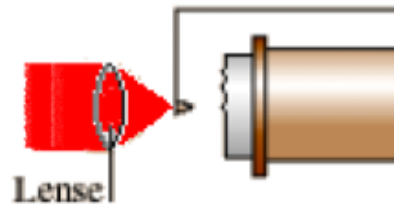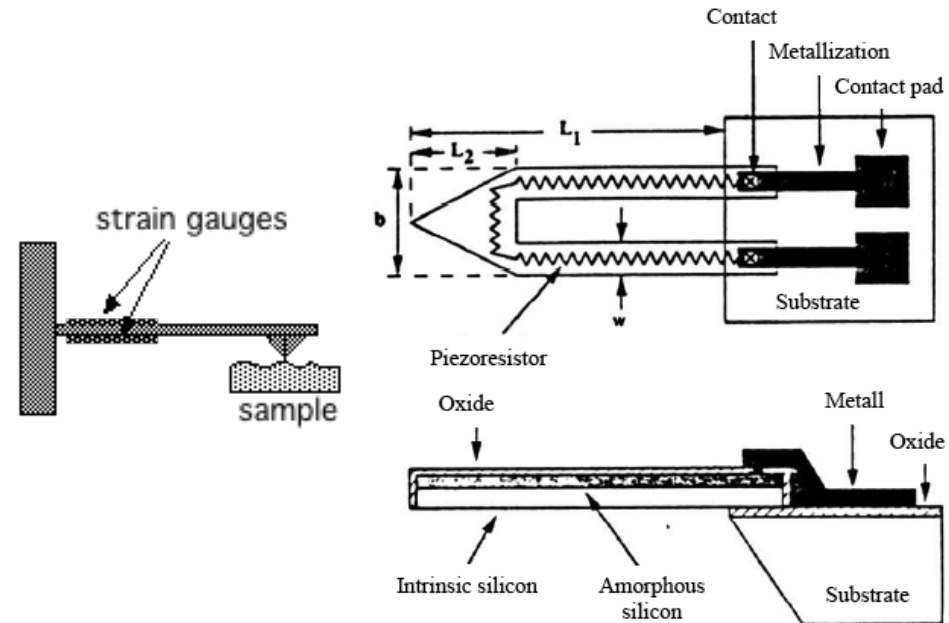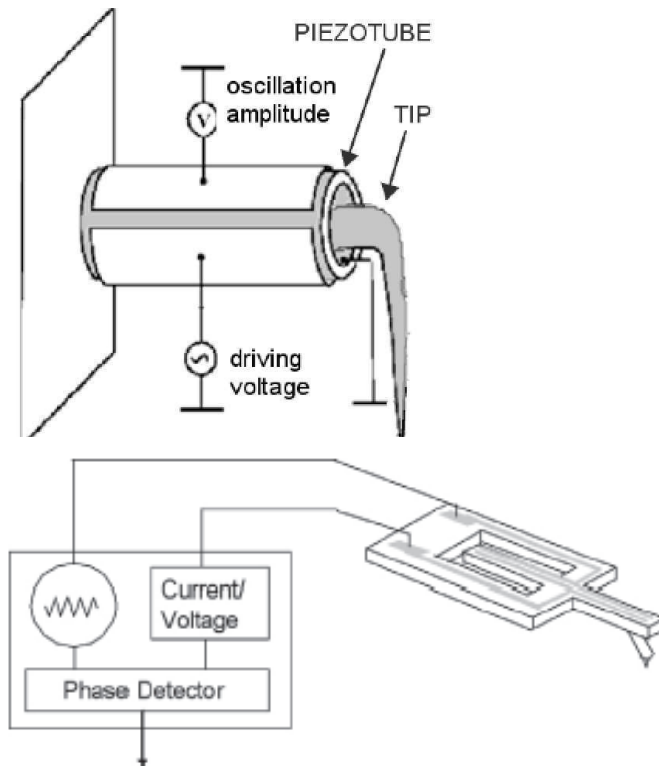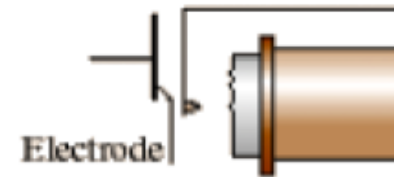
Image acquisition at "Constant Height in Contact Mode".

The mapping of the interactive force may be obtained by sweeping in a raster scanning the sample area, and by recording in a bidimensional matrix the values V_i $(x_i y_j)$. of the photodiode output, for each position $x_i y_j$ of the tip (i.e. for each pair of voltage values fed to the scanner in order to bring the tip over the point $x_i y_j$).

For example when the tip meets a relief increasing the repulsive force, the cantilever deflects causing the reflected beam to displace its spot on the split photodiode with a consequent change of the photodiode output.

The matrix of the output values may be displayed on the computer screen using a grayscale representation of $V_i(x_i y_j)$: this is the map of the interactive force over the scanned area of the sample.

Pixel by pixel mapping of the sampled area
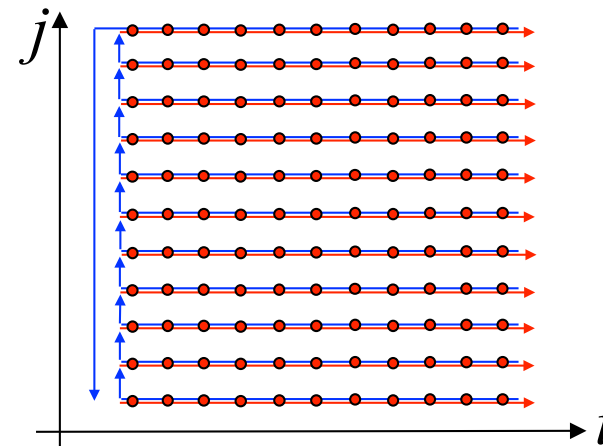
Contact Mode at "Constant Height" or "Constant force"

The operating mode that records the force F [i.e. the photodiode output $V_i(x_iy_j)$] is named "Constant Height Mode" , because no modulation of the tip-sample distance (z coordinate of scanner) is made during acquisition.
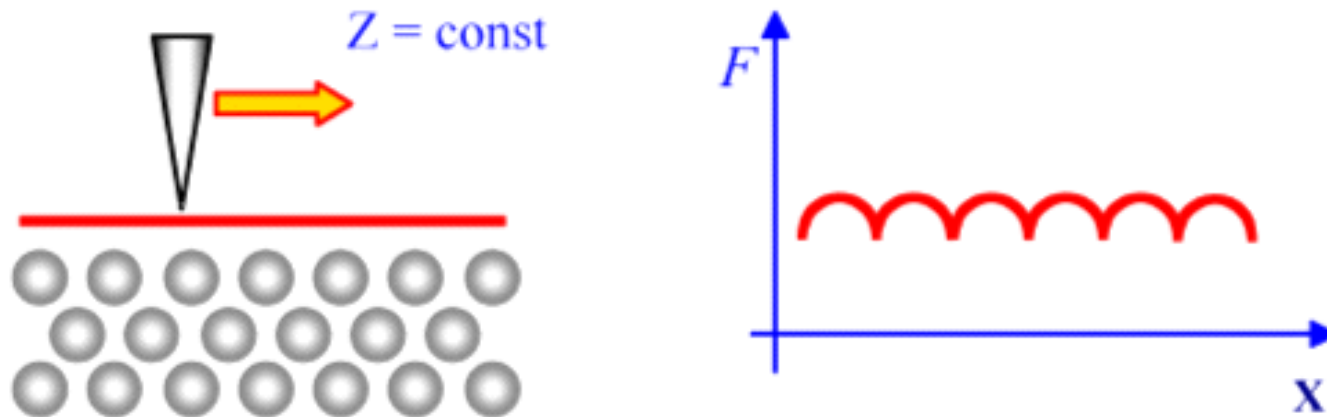
The non-linearity and the limited range of the V(z) response function makes possible this procedure only for sample with very small roughness , and with very small tilt of the scanned sample plane ($x,y, z = z_0$) with respect to the sample surface plane.

Acquisition at "Constant Force".

Using the photodiode output to control (with a feedback system) the z value, thus keeping the output signal always at a constant value (i.e. keeping constant the interaction force) we increase both the linearity and the dynamic range.
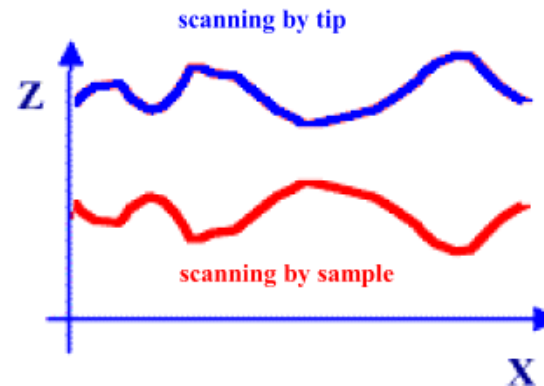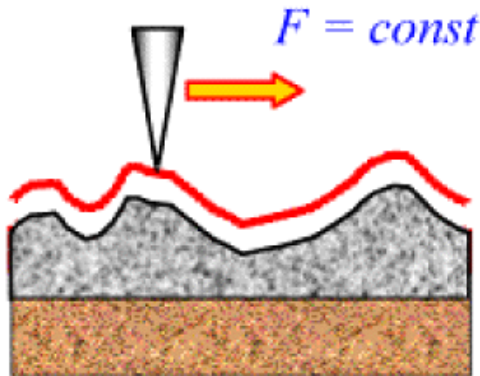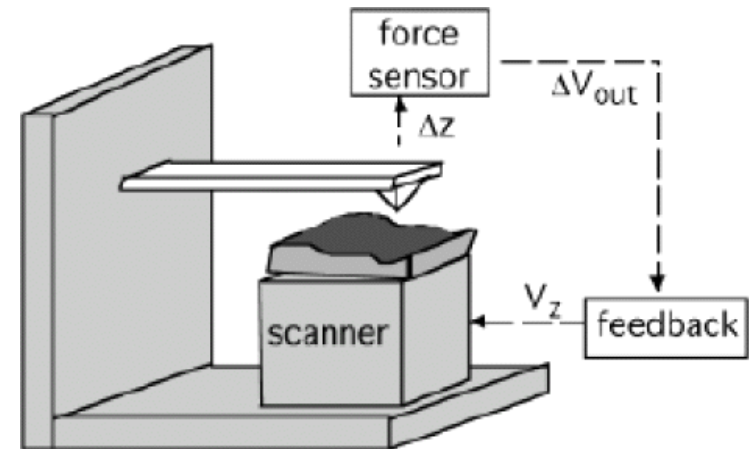
In this case the computer records the matrix $V_z(x_i y_j)$ of the voltage values fed to the scanner to keep constant the force: this matrix is a real topographic image of the scanned area.

The constant force mode is the technique normally used in SFM

Structure of SPM for constant force feedback

Laser

Photodiode

PA

Operator
Force set

DAC - Set

ADC

Z-out
signal

U U_0

DA

"Z"

SW

A4   DAC - X

Raster
scan

A5   DAC - Y

A2

DAC - Z

Tilt correction

Switch SW = feedback on
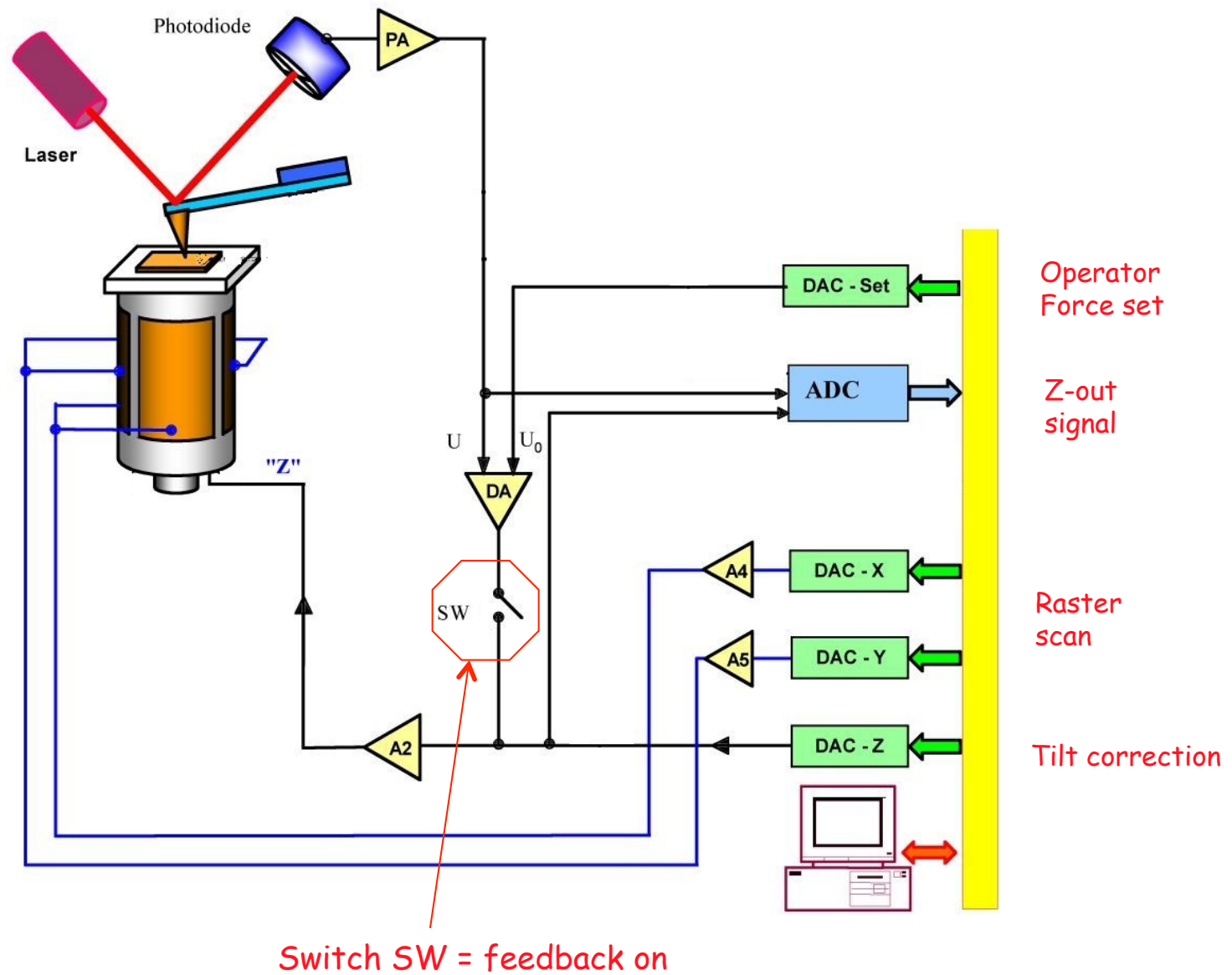
Top-view and 3D-view images

Once recorded the matrix $Z(x_i, y_i)$, with the height values z_i, we may display it as a grayscale image (2D, or Top view) or as three-dimensional (3D) view seen from a user-defined view-angle, eventually expanding the z axis to enhance the sample details not visible in a proportional representation .

SPM frames are normally square matrixes (typically 256x256, 512x512 or 1024x1024).
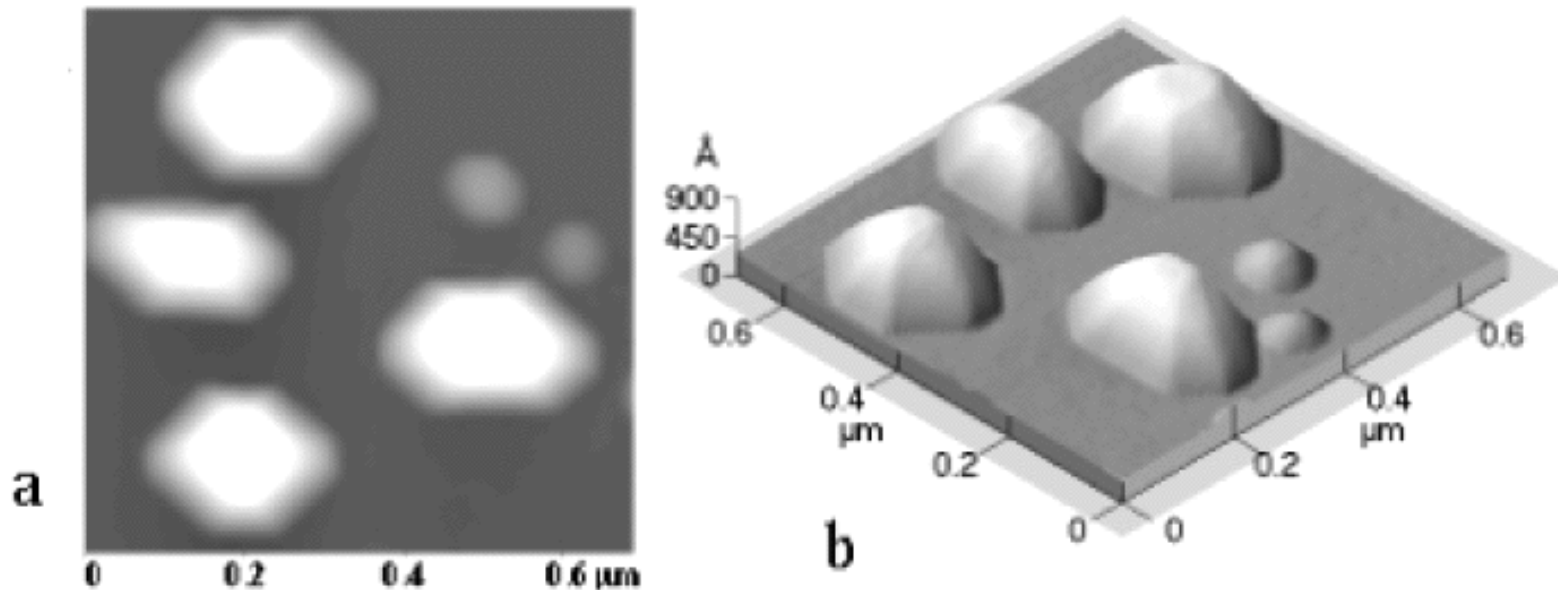
Image of nanostructures of InP grown on a substrate of GaAs
Matrix of 256 x 256 dots:
a) Top (2D) view ,   b) Axonometric (3D) view

Tip-sample interaction: short- and long-range forces

In the Contact Modes (both at constant height and at constant force) we exploit the steepness of the F(z) curve slope in the repulsive region of the tip-sample interactive force.

At short distances in fact prevails the "hard sphere" repulsion due to the Pauli exclusion principle, that forbids the overlap of the electron wave functions of the outer atomic orbitals.

At large distances (some nm from the sample surface) the van der Waals polarization prevails and the force becomes attractive, with a much smaller gradient.

$F_{Hertz} \sim (-h)^{3/2}$

$F_{VdV} \sim -1/h^2$

"semicontact" mode

contact mode

non-contact mode

h= conventional tip-sample distance (surface at negative isoforce)
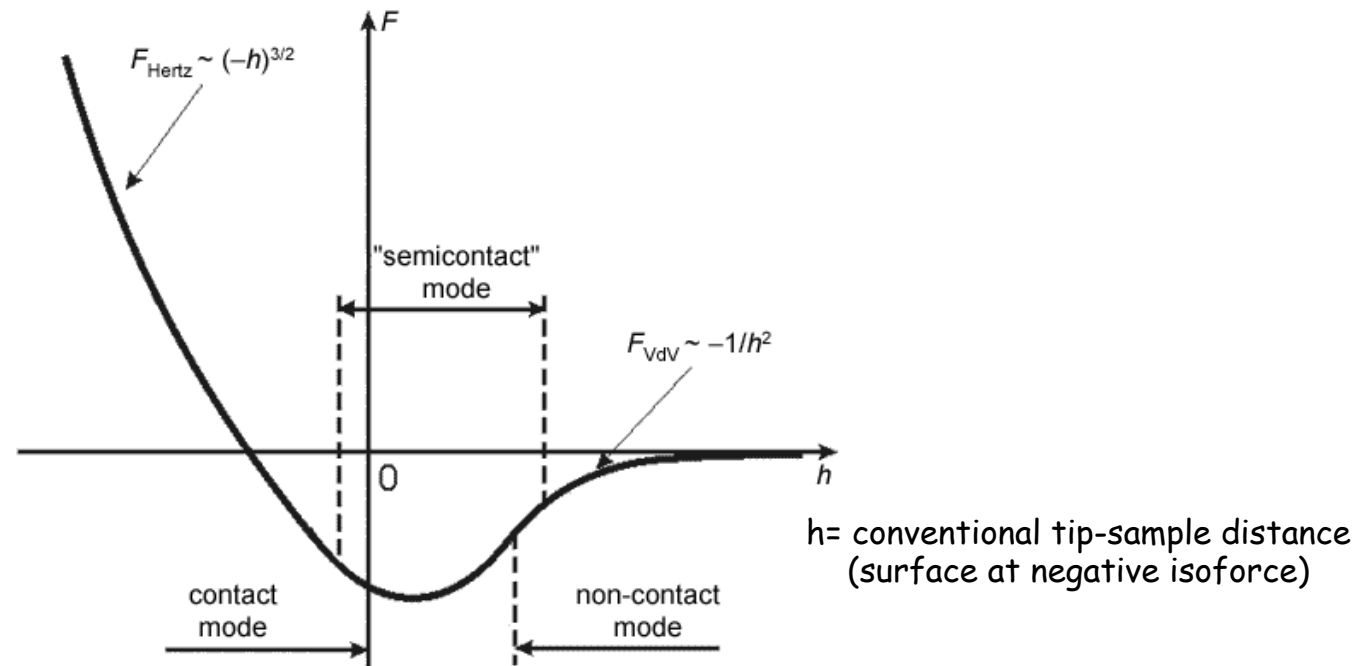
Contact and Non-Contact

The slope of F(z) changes sign at some z value and goes to zero for z≈∞

Also the force gradient ∂F/∂z changes sign:

it is negative and large at short distance (contact region)

and positive and small at large distance (non-contact region).

F

Contact region
$\partial F/\partial z < 0$

F>0

z= conventional tip-sample distance
(surface at zero isoforce)

z

F<0

Non-Contact region

$\partial F/\partial z > 0$
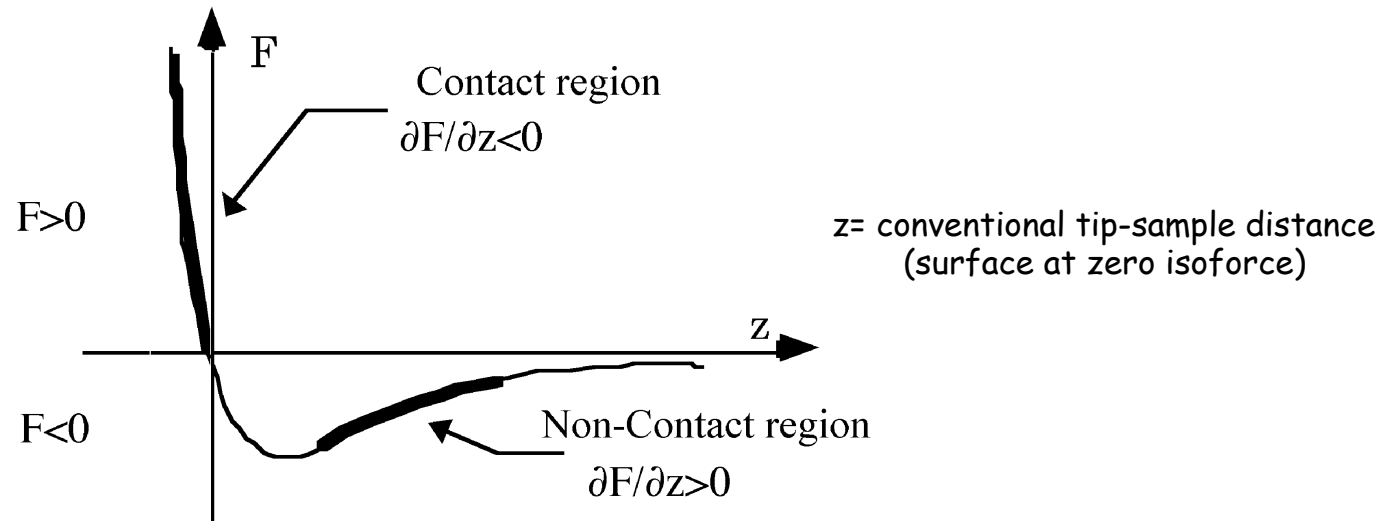
In Non-Contact (F<0 e ∂F/∂z>0) the cantilever *deflects toward* the surface producing the (small) output signal, suitable for a feedback.

The cantilever elastic constant in this case must be chosen large enough to avoid *positive* feedback (when the attractive force is larger than the Hooke's restoring force).
This further decreases the force probe sensitivity in Non-Contact Mode.

"Non–Contact": working principle

To increase the system sensitivity a great improvement in the signal/noise ratio must be provided: a lock-in technique is needed.
The cantilever is forced into vibration at its resonant frequency ω_0, and the changes in oscillation amplitude A due to changes in the force gradient, are used as feedback signal.
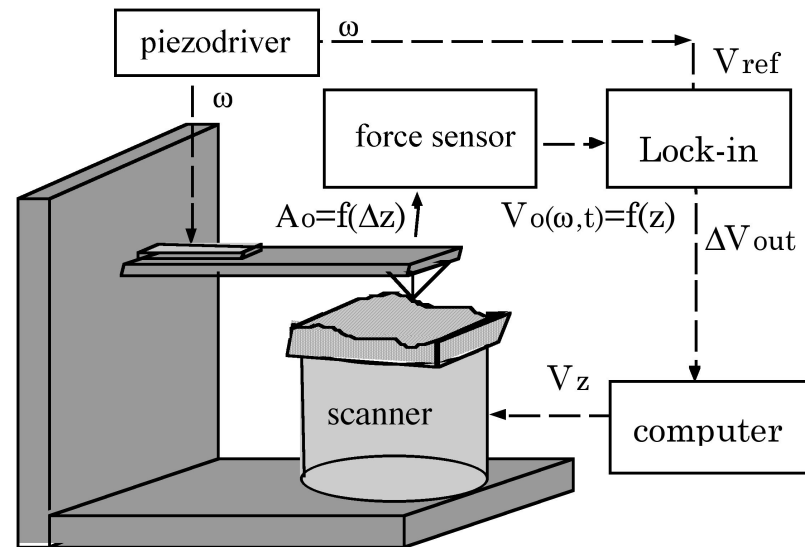
A synchronous detector (lock-in), locked to the reference signal, excites the cantilever into oscillation through a piezo-driver.
The increased dissipation near the sample surface broadens the resonance curve and the force gradient produces a shift of the resonant frequency: both the effects *reduce the oscillation amplitude*.

Structure of SPM for Non-Contact Mode

PD

PA

Laser

PV

Vin

"Y"

Vref

SD

U   U$_0$

DA

"X"   "Z"

VCO

SW4

A2

DAC-Set

ADC

Output Z-signal

A4   DAC - X

A5   DAC - Y

DAC - O

DAC - Z

Operator
Force set

Raster
Scan

Oscillation driver

Tilt correction

PV: cantilever piezo-driver   SD: synchronous detector
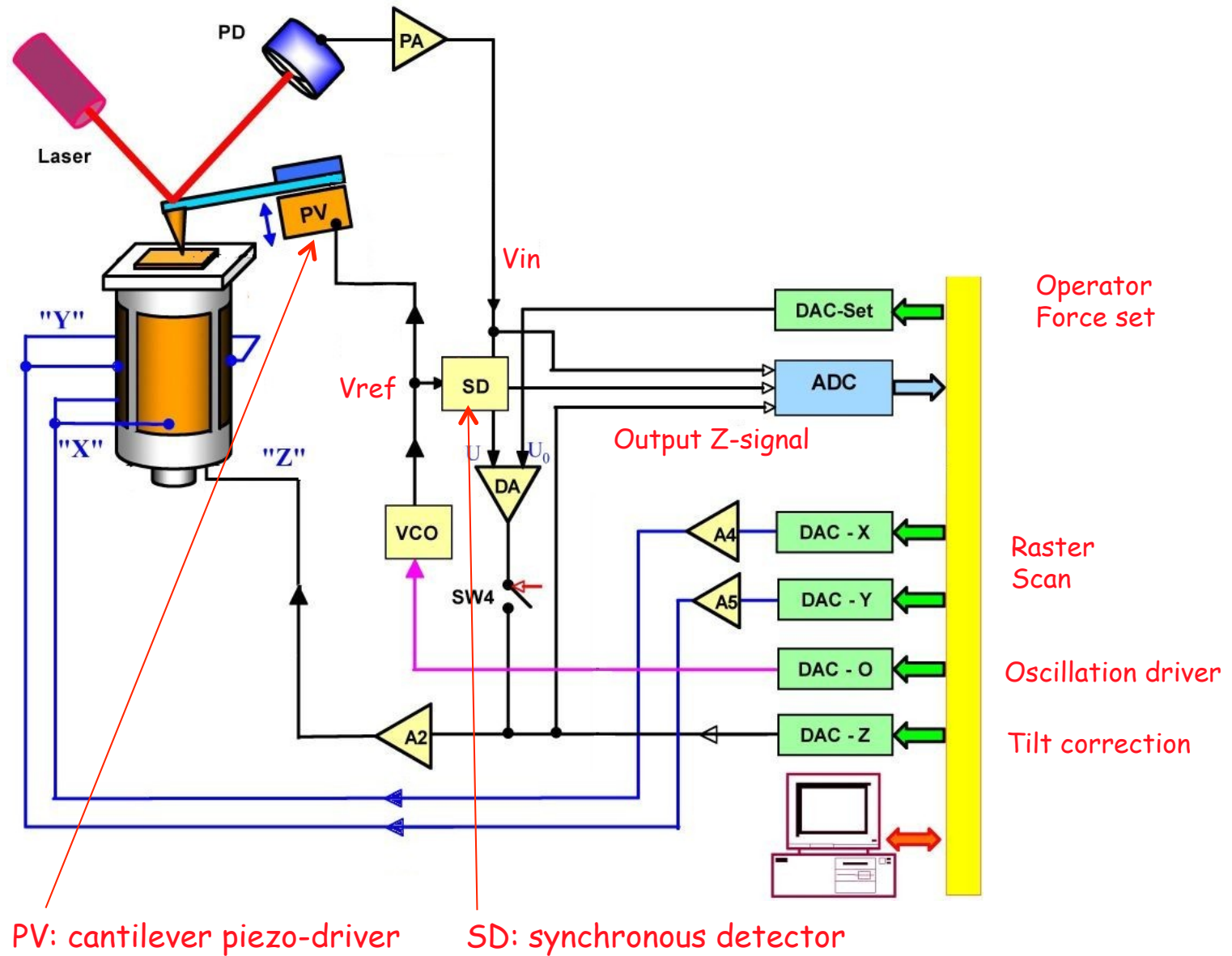
Electronic Blocks for SPM
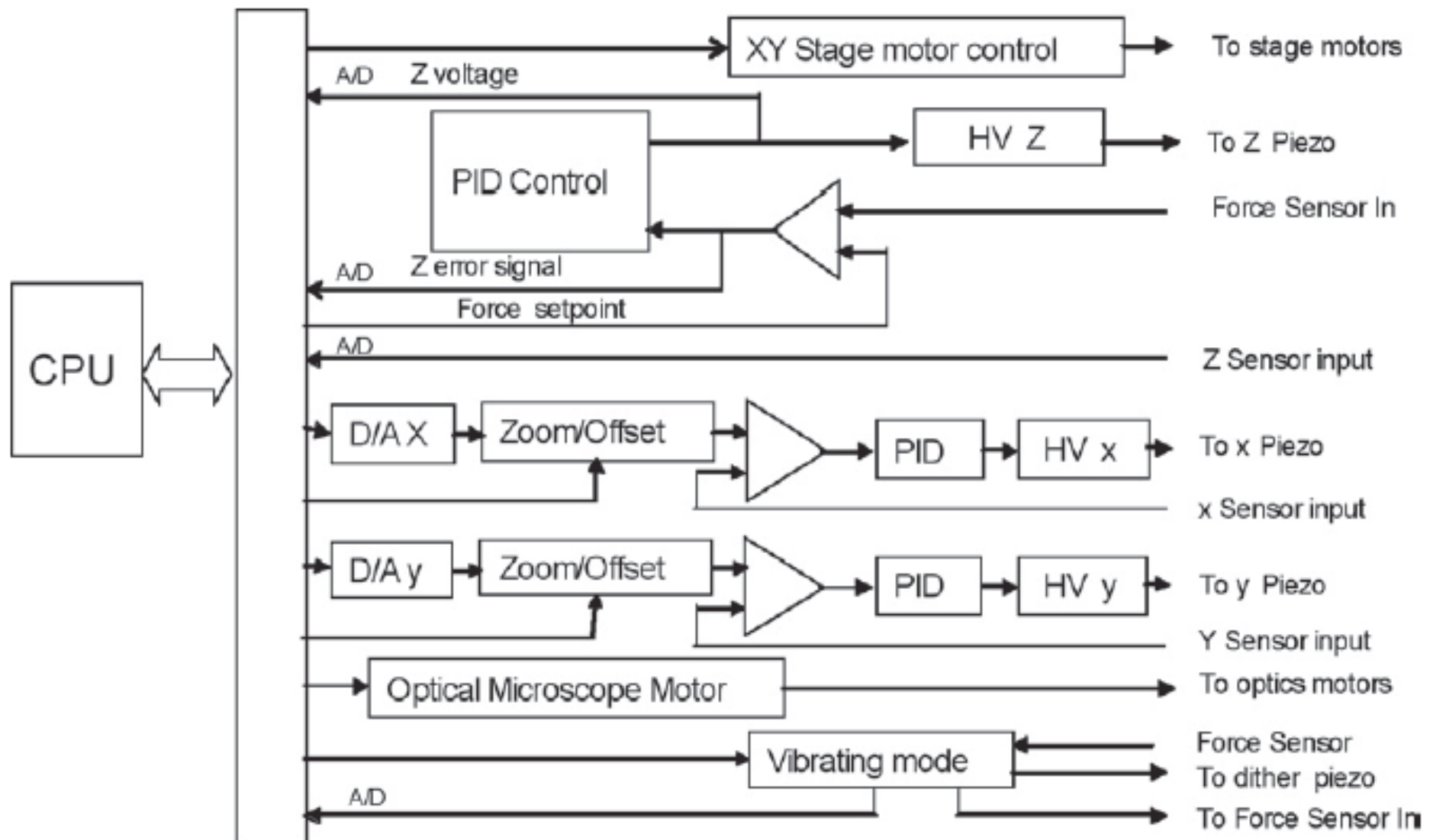
A/D = analog to digital converter   D/A = digital to analog converter   PID= Proportional, Integral, Derivative filter

Lab. Fis. Mat.   G.Torzo

How the force gradient changes the resonance curve

The cantilever may be considered a spring obeying to the Hooke's law :
$F_0 = -k\Delta z$, where k is the elastic constant.
In presence of a force gradient $\partial F/\partial z$, in a linear approximation:

$F \approx F_0 + (\partial F/\partial z)\Delta z = -k\Delta z + (\partial F/\partial z)\Delta z = -(k-\partial F/\partial z)\Delta z = -k'\Delta z.$

the effective elastic constant k' decreases approaching the surface due to the positive force gradient:

$$k' = k - \partial F/\partial z$$

The resonant frequency ω_o may be written : $\qquad \omega_o = \sqrt{k/m}$
where m is the effective oscillating mass of the cantilever.

Due to the force gradient the resonant frequency shifts
(in a linear approximation), to a lower value ω_o^* :

$$\omega_0^* \approx \sqrt{k/m}\left(1 - \frac{\partial F/\partial z}{2k}\right) = \omega_0\left(1 - \frac{\partial F/\partial z}{2k}\right)$$

Frequency, Amplitude and Phase dependence on the interactive force

For small frequency shift: $\Delta\omega_o/\omega_o = (\omega'_o - \omega_o)/\omega_o \ll 1$, we get :
$$\Delta\omega_o/\omega_o \approx -(\partial F/\partial z)/2k$$

The relative amplitude change $\Delta A/A$ may be evaluated in terms of the quality factor $Q = \omega_o/(\omega_2 - \omega_1)$:
$$\Delta A/A \approx Q\,\Delta\omega_o/\omega_o = -Q(\partial F/\partial z)/2k$$

A third feedback signal may be taken from the phase Φ between excitation and response signals.
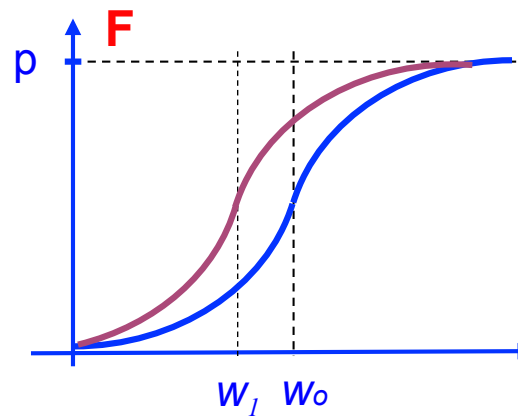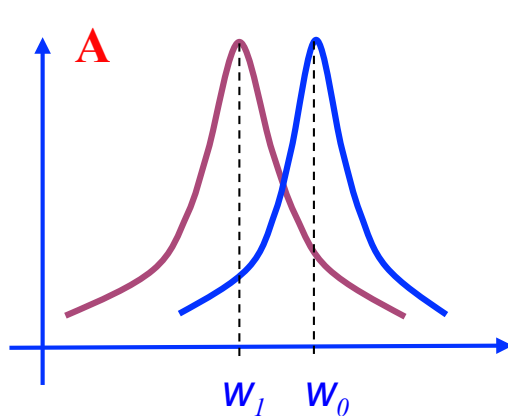
Blue= large distance
Violet= small distance

Phase and Amplitude plots

The phase relation between excitation and response, near the resonant frequency ω_o, changes rapidly from ~0 to ~180 degrees

$$\phi(\omega) = \arctan\left[\frac{\omega\,\omega_0}{\left\{\omega_0^2 - \omega_0 - (\partial F / \partial z)/m\right\}}\right]$$

The amplitude changes depend strongly on the quality factor Q

$$A(\omega) = \frac{u_0\,\omega_0^2}{\sqrt{\left[\omega_0^2 - \omega^2 - (\partial F / \partial z)/m\right]^2 + \omega^2\omega_0^2 / Q^2}}$$
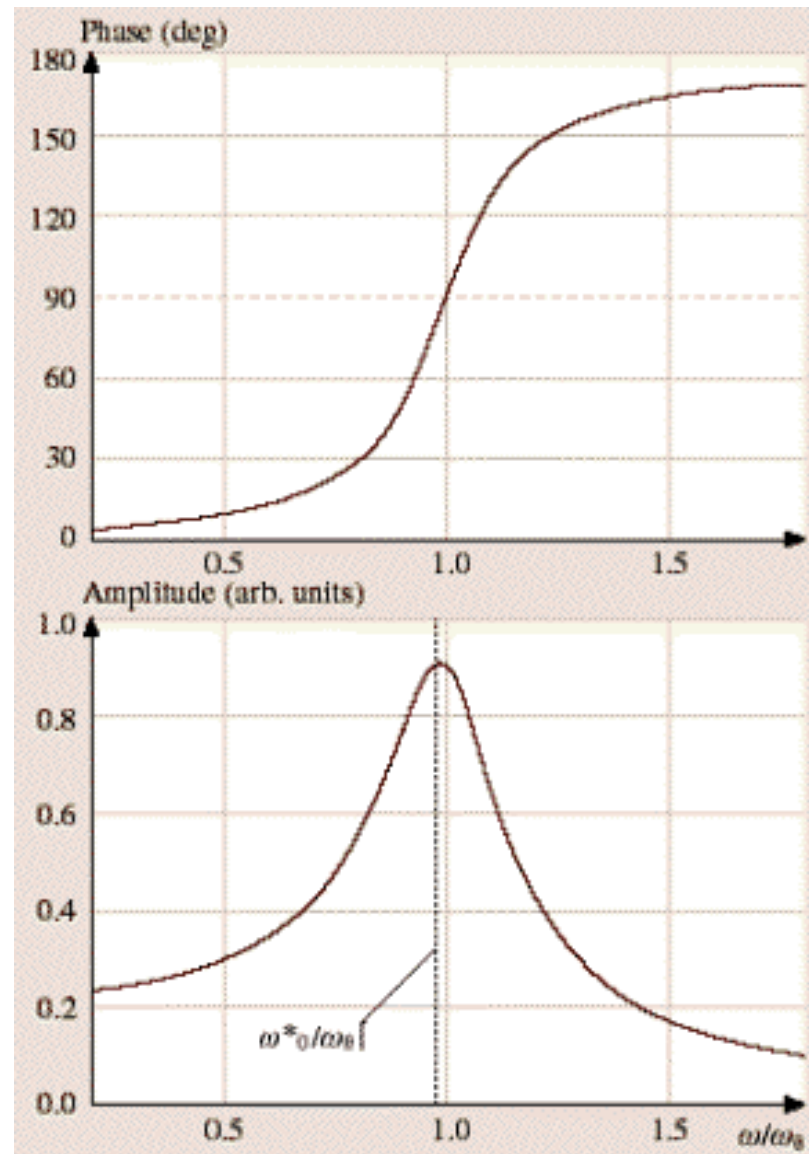
The water film

Working in air (normal condition for SFM) a water film (one or more monolayers) covers the sample producing a capillary force of the order of 10^{-8} N:
this introduces an hysteresis in the force-distance cur
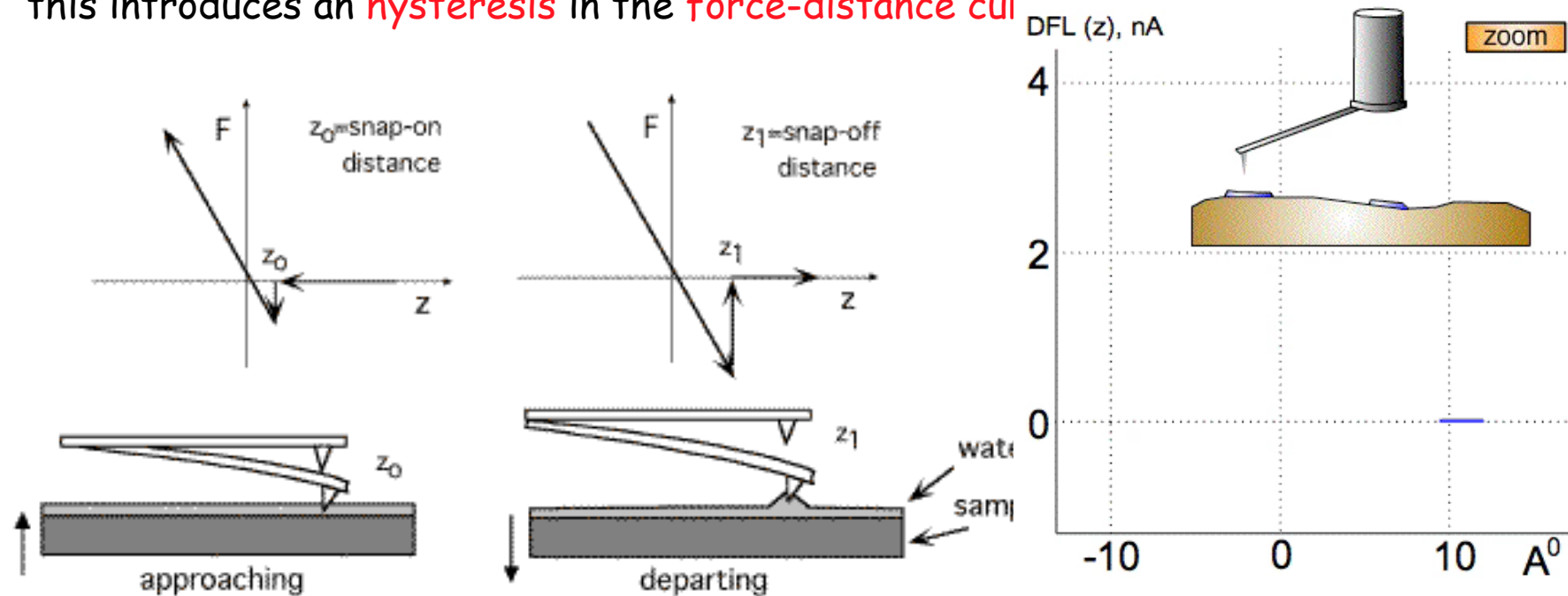
When the tip approaches the surface, the module $|\partial F / \partial z|$ of the force gradient increases until it becomes greater than the cantilever elastic constant k.

At this point (snap-on distance z_0) the cantilever increases its deflection until it touches the water film at the surface .

When the scanner retracts the capillarity effect impedes the cantilever detachment until the coordinate z reaches a larger value ($z_1 > z_0$ snap-off distance).

Contact vs Non Contact: Advantages and Disadvantages

Contact Mode

» Advantages:

- High scan speeds
- "atomic resolution" images
- Easier scans in rough samples with extreme changes in topography

» Disadvantages:

- Lateral (shear) forces can may give distorted images
- Higher tip-sample interaction
- The combination of lateral forces and high normal forces may reduce spatial resolution and may damage soft samples

Non Contact Mode

» Advantages:

- Allows "Lift Mode" techniques and "Phase Contrast" imaging
- Lower forces and less damage to soft samples

» Disadvantages:

- Slightly lower scan speed

"Tapping-Mode"

In "**Tapping Mode**" also named "**Periodic Contact Mode**" or "**Semicontact Mode**" or "**Intermittent Contact Mode** " the cantilever oscillates with an amplitude between 20 and 100 nm . In case of meassurement in air , this is sufficient to make the tip to enter and exit the liquid water layer at each period.
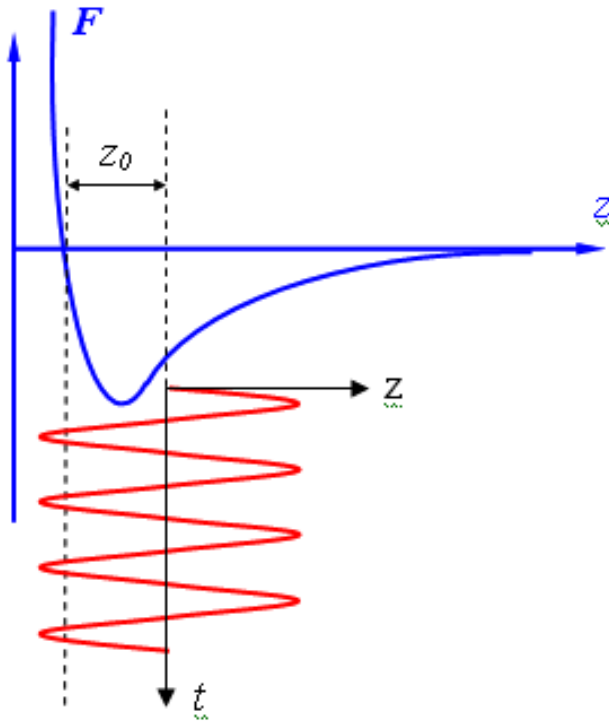
Tapping mode may also be used while measuring in liquid
(the whole cantilever submerged)
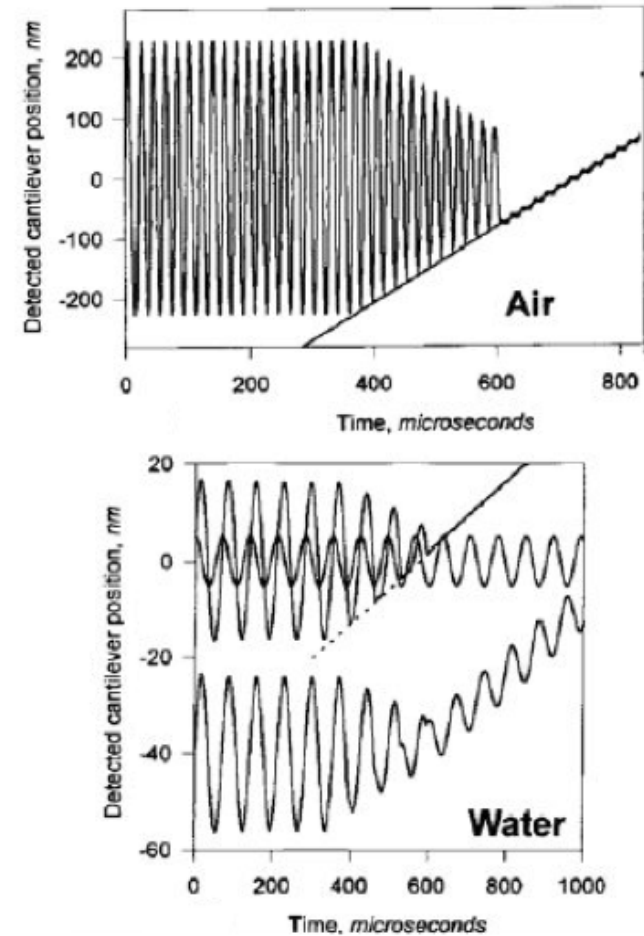
taken from "How does a tip tap?" by N.A. Burnham et al.

Phase contrast images

The phase-image is a map of the changes of the cantilever oscillation phase that may give information on the surface properties (composition, adhesion, friction).
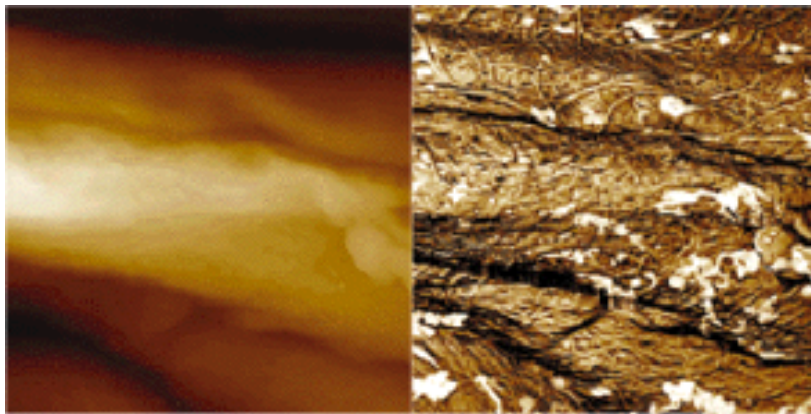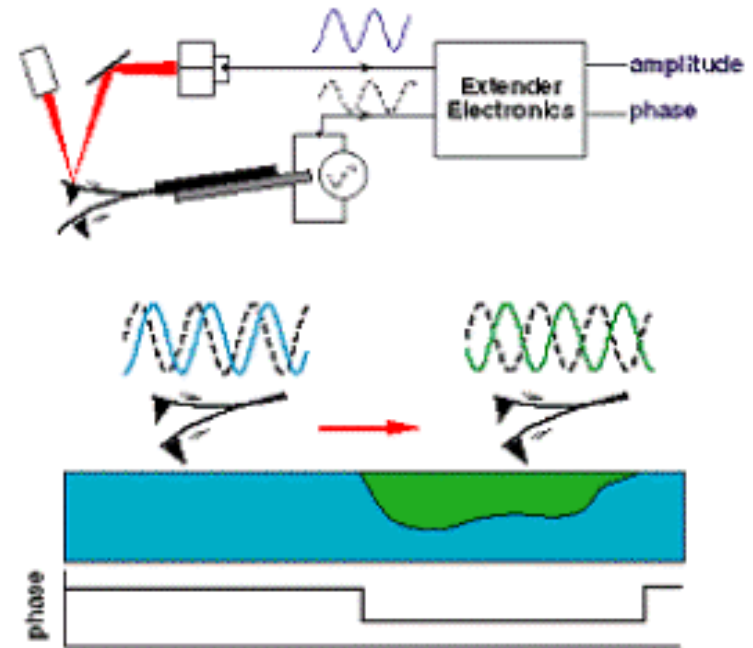
Topography and Phase-image of a sample of wood: cellulose microfibers appear more sharply defined in the phase image
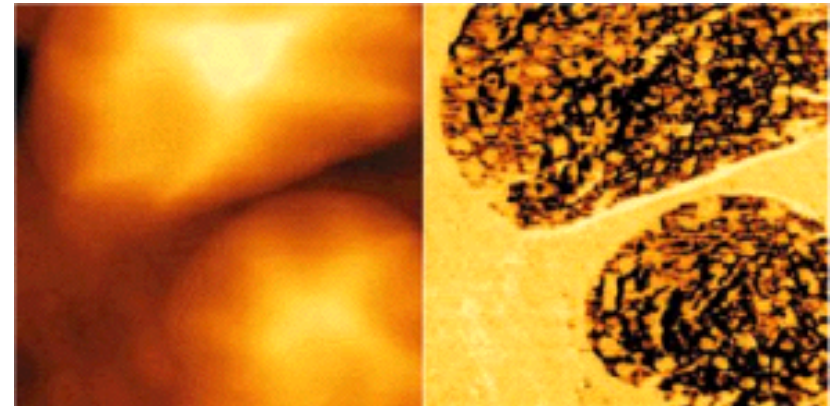
Topography and Phase-Image of a polymer composite

Non-Contact: more details

The oscillation amplitude is kept very small (of the order of 0.1 nm)
while the tip-sample distance is relatively large (from 10 to 100 nm).

The larger tip-sample distance corresponds to a smaller slope ∂F/∂z of the force-
distance curve and therefore the probe sensitivity is smaller.

Moreover at larger distance the interaction involves more atoms and this may
decreases the lateral resolution.
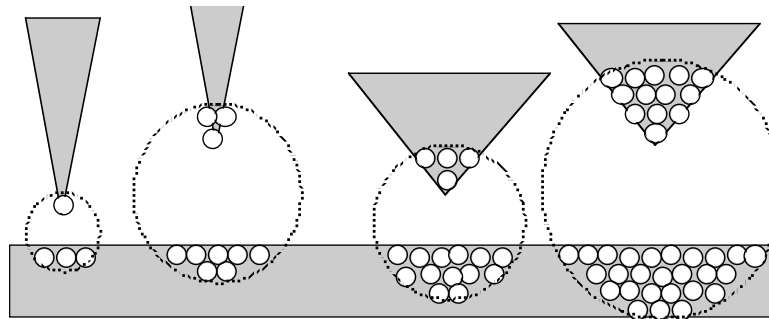
The lateral resolution may be increased either by decreasing the tip-sample
distance or by decreasing the curvature radius of the tip apex

LFM

The microscopic friction forces may be mapped using the "**Lateral Force Mode**" (**LFM**).
A signal proportional to the lateral force originated during the relative tip-sample movement in contact (due to differences in the local friction coefficient) may be obtained by detecting the cantilever torsion that produces a lateral displacement of the reflected light beam.
The lateral displacement is detected using a four quadrant split photodiode by measuring both the difference and the average value in the light intensity in adjacent quadrants).
This system allows to resolve lateral forces of the order of 10^{-11}N.

lateral beam deflection

B   A   vertical beam deflection
D   C

X scan direction

torsion
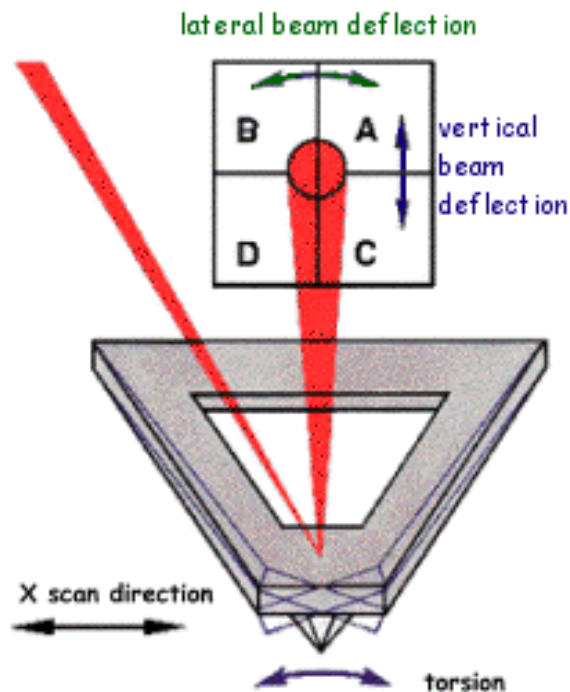
In the geometry shown here, the cantilever moves along X and the friction signal is calculated as (A + C) — (B + D), while the topographic signal is calculated as (A + B) — (C +D).

LFM may be useful to enhance contrast at the edge of relief or at the boundary of different compounds.

Reversing scan direction, the LFM signal due to friction change sign.
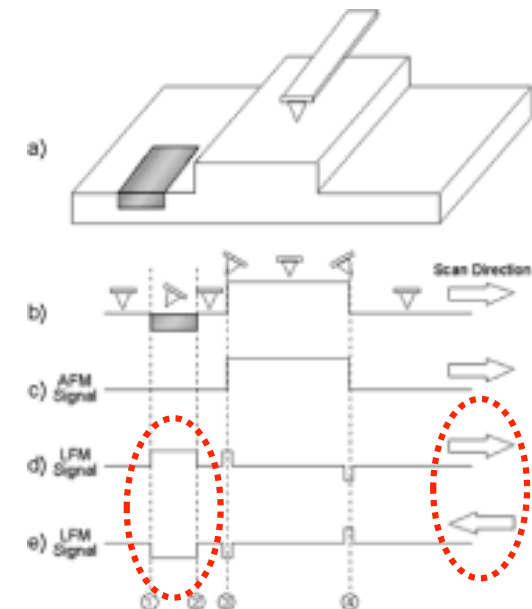
LFM (2)
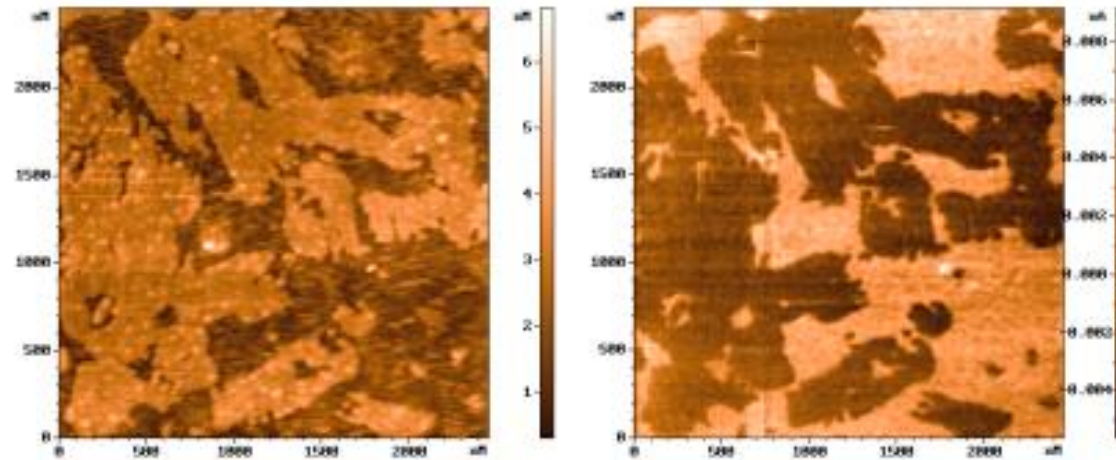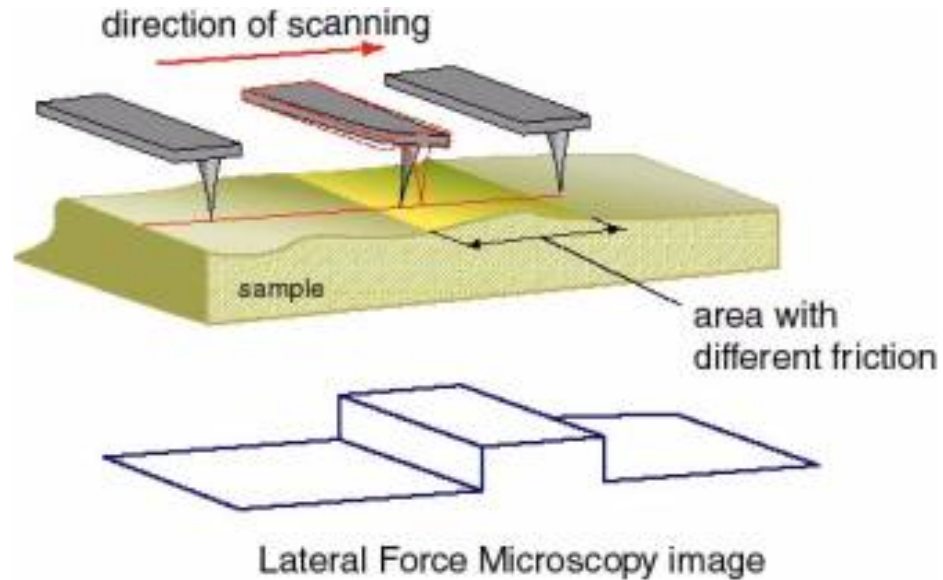
The sample is one monolayer Langmuir-Blodgett (LB) film deposited on silicon surface.
Fast scanning direction: in the image at left -> in the image at right : <-

a. Forth scan results.

b. Back scan results.

c. Half-difference and average of two scan results.

49
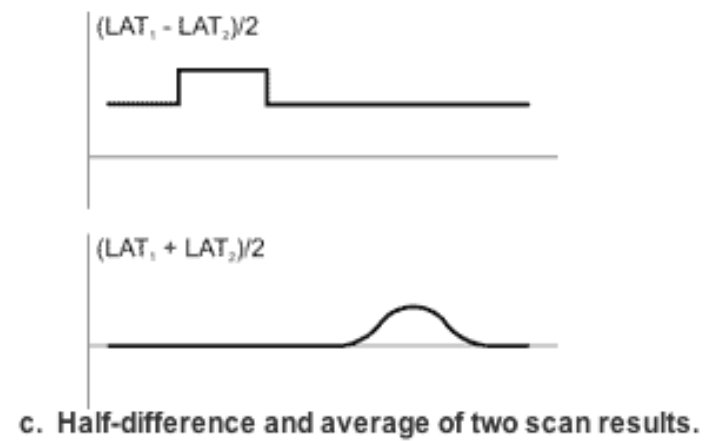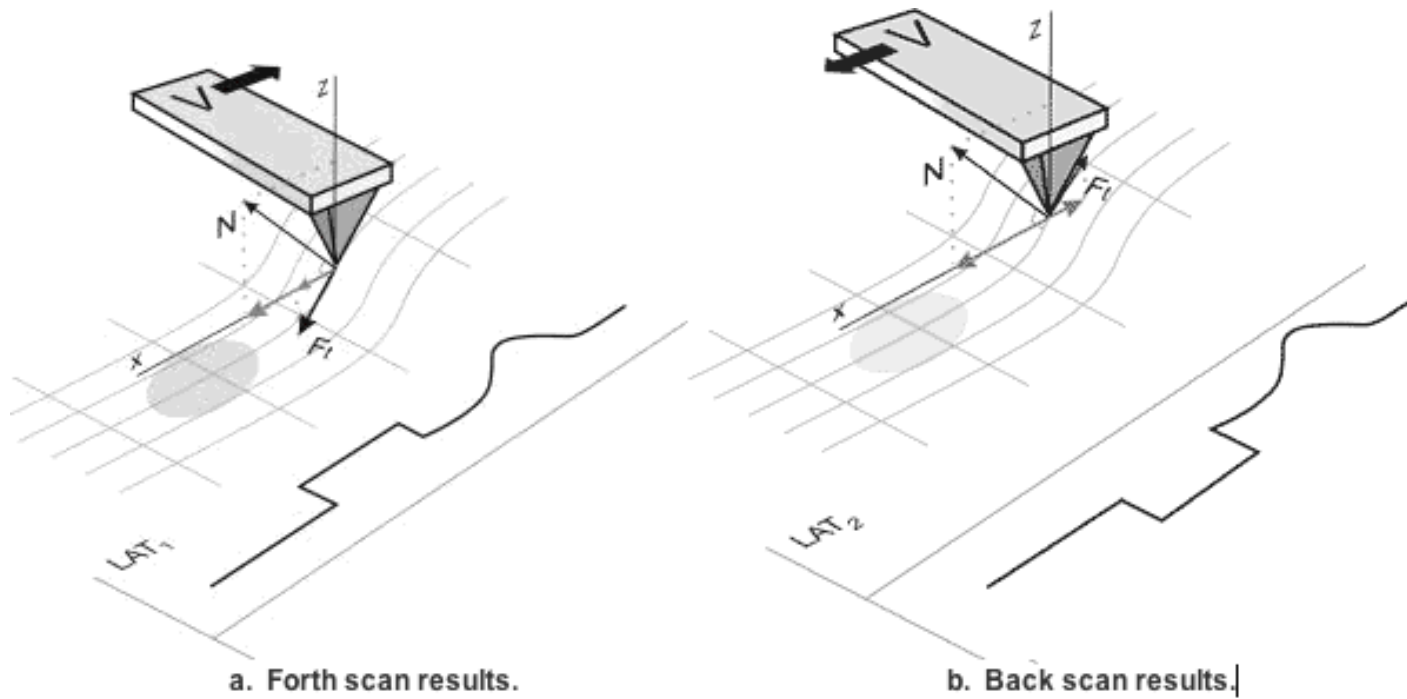
SFM : which force does it measures ?

The SFM measures the tip-sample interaction force.
Which force ?

The tip-sample interaction may be due to many kinds of different forces, depending both on the type of tip and sample material, on the value of the tip-sample separation, on the possible presence of electric field between tip and sample, on the possible presence of magnetic field (either produced by the tip or by the sample)….

It is a task of the user to choose the particular force he wants to measure, and consequently to choose the particular technique that best fit his goal.

Using different tip-sample separations allows to make negligible either the short-distance forces or the long-distance forces,

Using a voltage applied to a conductive tip or to a conductive sample allows to make the electric force prevailing over other interatomic forces,

Using a magnetized tip allows to investigate the magnetic force…

The two-pass or Lift Mode

2nd pass

1st pass

Magnetic or Electric
Field Source

Lift Height

Topography

Non-contact force
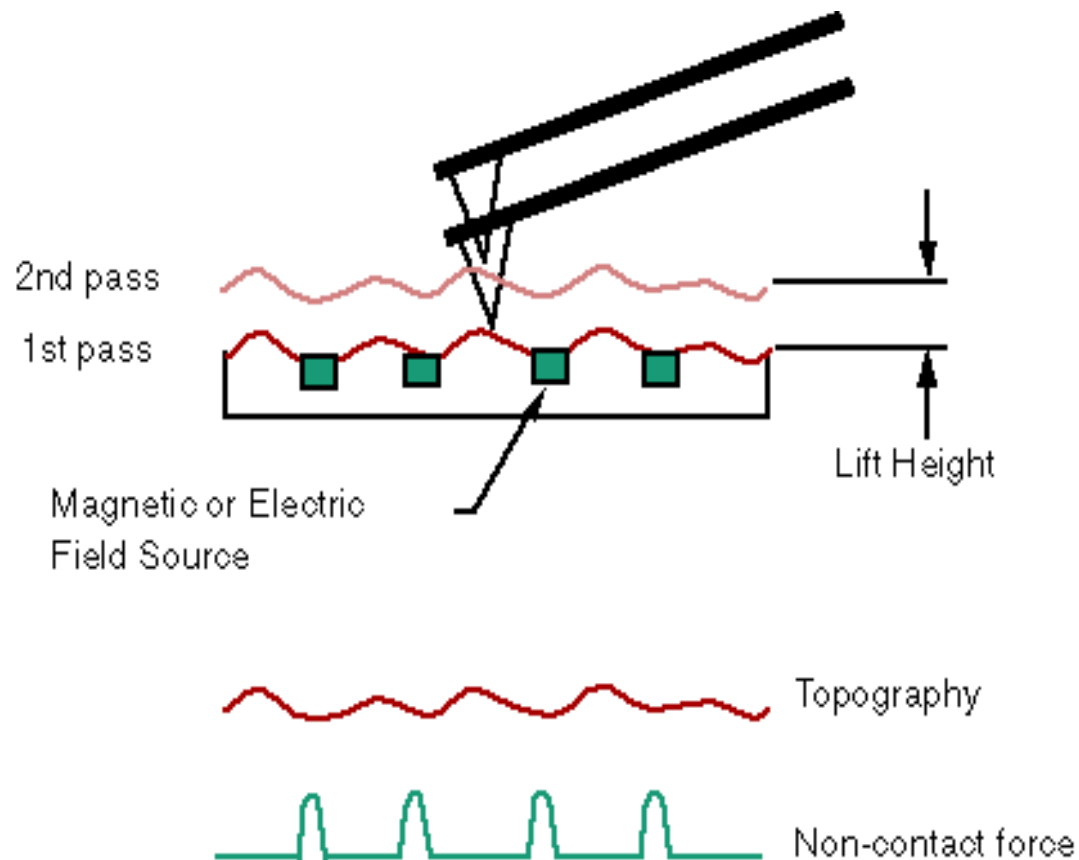
Two passes are made over the sample. The first measures topography
while the second measures a material property (magnetic, electric, etc.)

MFM (magnetic force microscopy)

The MFM technique gives a spatial distribution of the parameters that describe the tip-sample magnetic interaction.
The cantilever is coated by a magnetic film (e.g. cobalt).
The MFM image is obtained by separating the contribution due to magnetic force from that due to the sample topography, e.g. using the two-pass method (or Lift Mode).
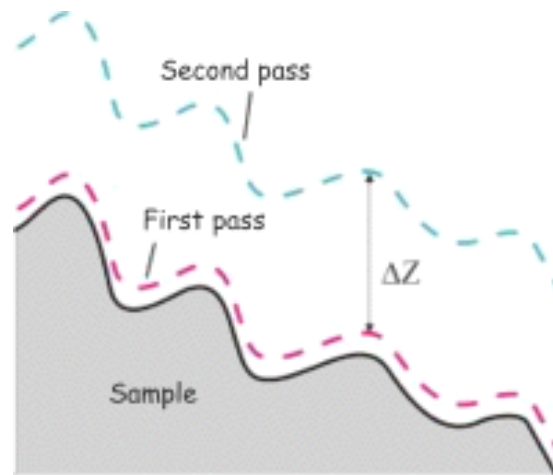
In the first scan the topographic profile is recorded in contact (or tapping) mode.

In the second scan (lifted) the cantilever is moved at a preset distance Δz from the sample surface and the feedback loop is broken (the line scan is controlled by the previous recorded profile).
The separation Δz (5-50 nm) must be large enough to make negligible the Van der Waals force, leaving only the magnetic term (that decays at larger distance).

MFM: various techniques

Different techniques are used in the MFM two-pass method:
1) if the cantilever is not excited into oscillation during the second pass, we get a DC MFM image, where the recorded force is between the tip magnetic dipole m and the magnetic field produced by the sample surface $$F = (\vec{m} \bullet \vec{\nabla}) \vec{H}$$
2) if the cantilever is excited at its resonant frequency during the second pass two types of images AC MFM may be obtained : by recording the phase shift or the amplitude changes.
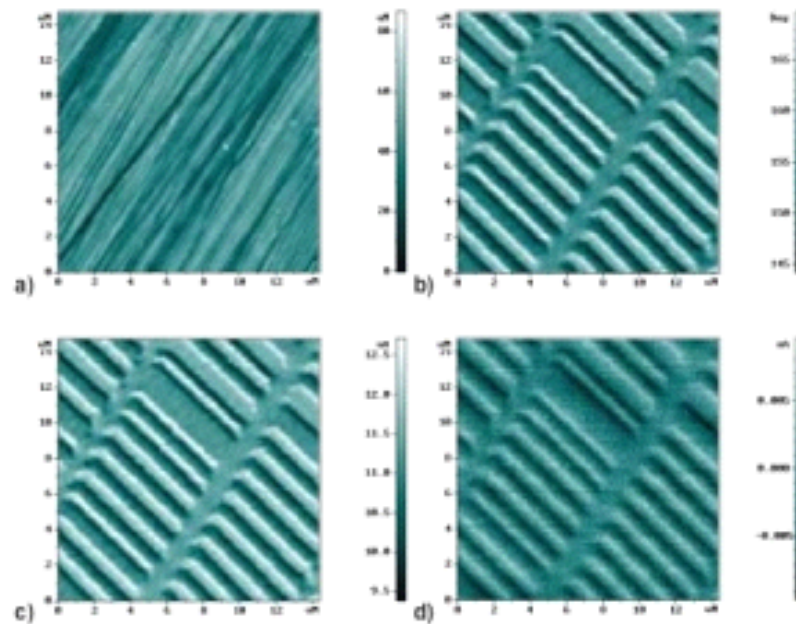
Various SPM images on Hard Disk:
a) Topography, b) Phase ACMFM   c) Amplitude ACMFM, d) DC MFM

DC-MFM (Lift Mode)

In the first pass the topography is determined in Semicontact mode (Tapping).

In the second pass the magnetized cantilever is lifted to a selected height for each scan line and scanned using the stored topography (without feedback);

the tip-sample separation during second pass is therefore kept constant
and it is large enough to make negligible the Van der Waals' force
so that the cantilever is affected mostly by long-range magnetic force.

1-st pass. Sample profile acquisition.

Copyright © NT-MDT, 2002   www.ntmdt.com
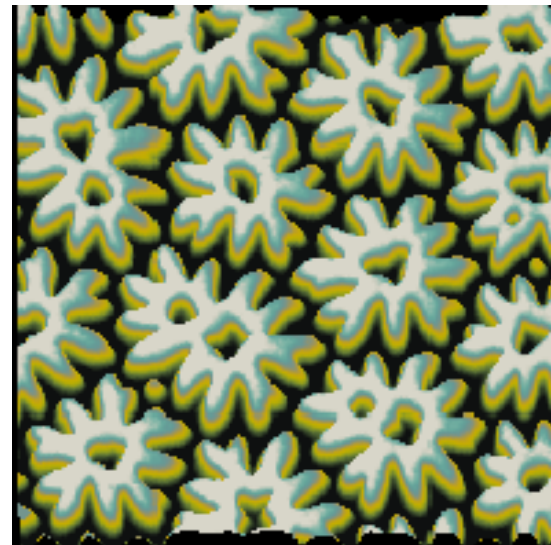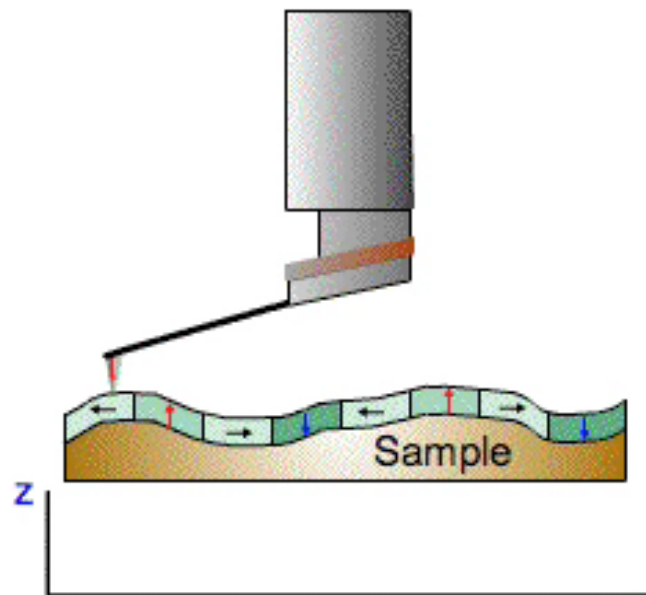
Domains in a 80 micron garnet film

AC-MFM (Phase Shift Image)

1-st pass. Sample profile acquisition
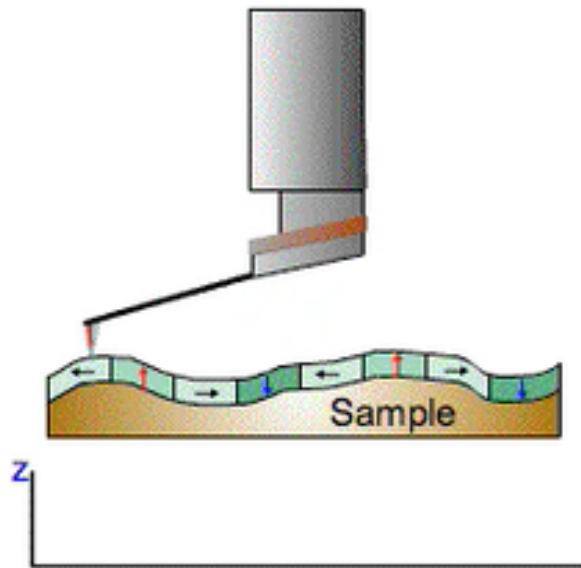
In the first pass the topography is determined in Semicontact mode
In the second pass the cantilever is driven at its resonant frequency;
the phase shift are recorded.

Magneto-optical disc
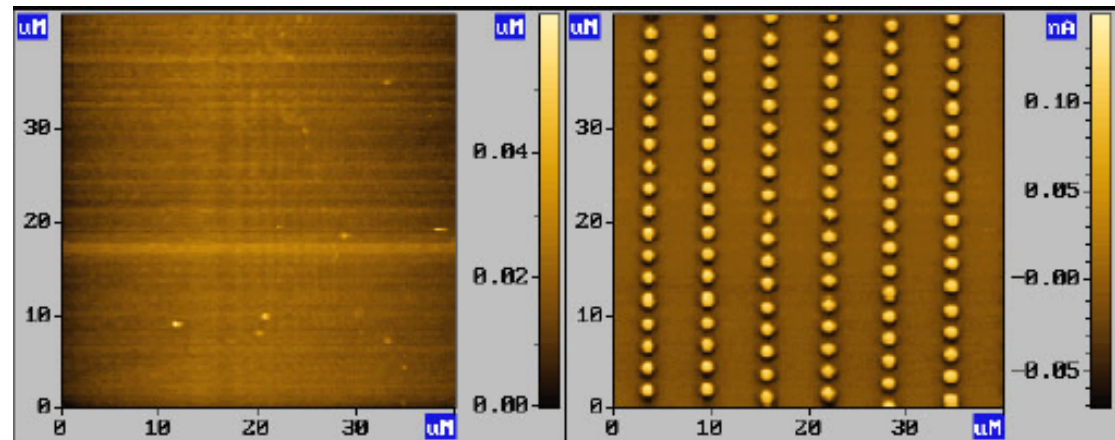
AFM topography

MFM image

EFM (electric force microscopy)

Also EFM images are obtained using the two-pass method (Lift Mode).

During the second scan the (conducting) cantilever is forced at resonance and biased with a constant voltage.

The tip-sample capacitive induction produces a modulation of the resonant frequency (and consequently changes in the oscillation phase and amplitude).
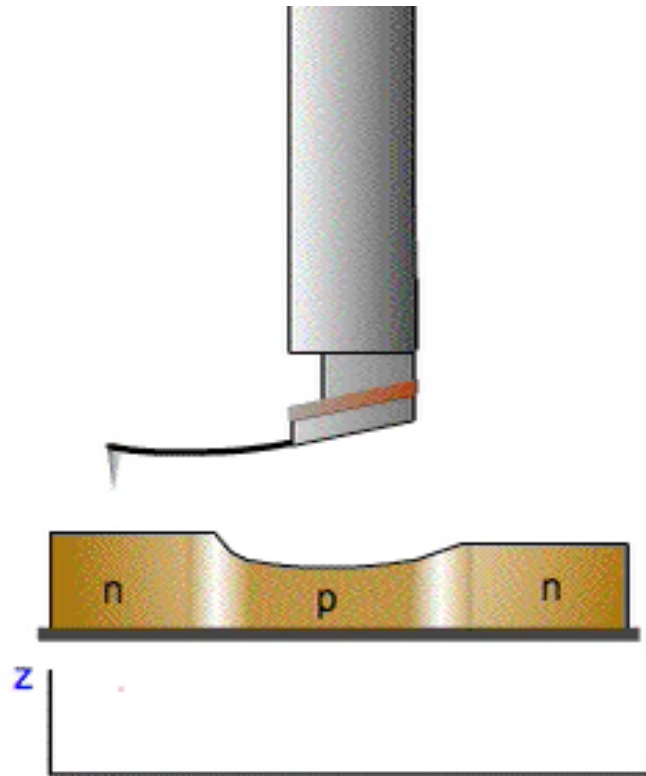
Scanning Capacitance Microscopy

The **SCM** images are a particular kind of **EFM** images
In **SCM** the cantilever is biased with a modulated voltage: $V_{tip}=V_{dc}+V_{ac}\sin(\omega t)$ And the sample is biased at V_s
The tip-sample capacitive force $F_c(z)$ is:

$$F_c(z) =(1/2) (V_{tip} - V_s)^2(dC/dz)$$

Where the tip-sample capacity $C(z)$ depends on the tip and sample geometry and on the distance **z**.
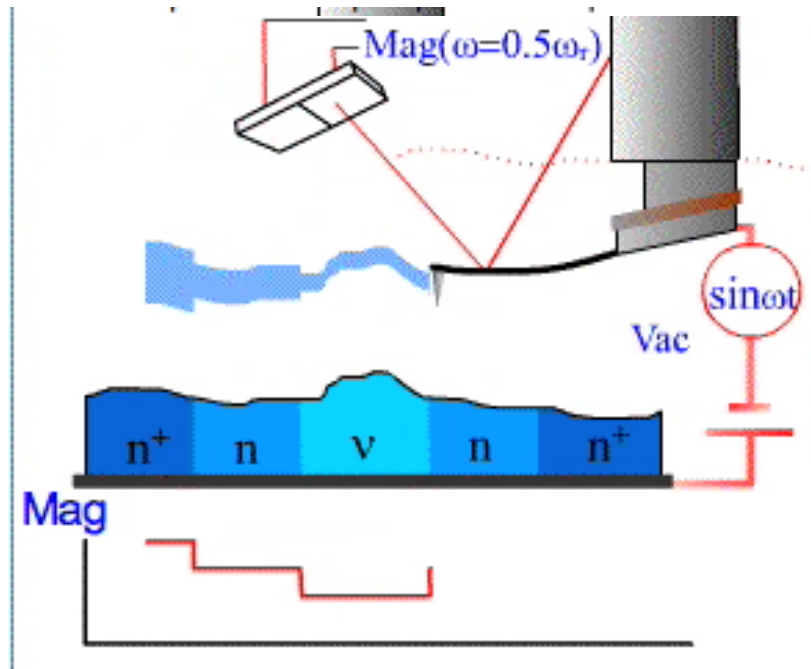
The second harmonic $F_{c2}(z)$ depends only on (dC/dz) and V_{ac}

$$F_{c2w}(z) =(1/2)(dC/dz) V_{ac}^2\sin(2\omega t)$$

To optimize the second harmonic signal the driving frequency is chosen at half the resonant frequency of the cantilever.

SCM 2

Scanning Capacitance Microscopy of nonuniformly boron doped silicon. Scan size: 9 x 9 um
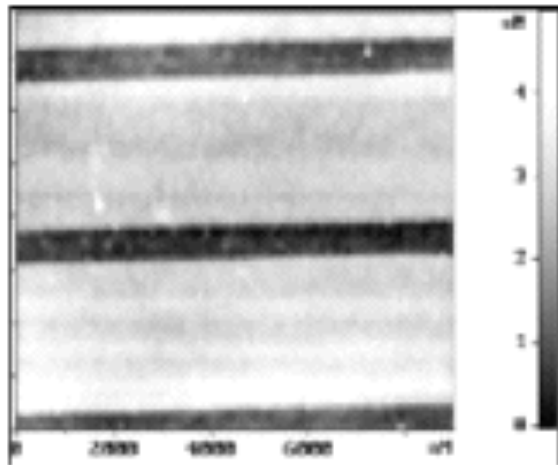
topography image of silicon surface, strip doping regions are well visible.
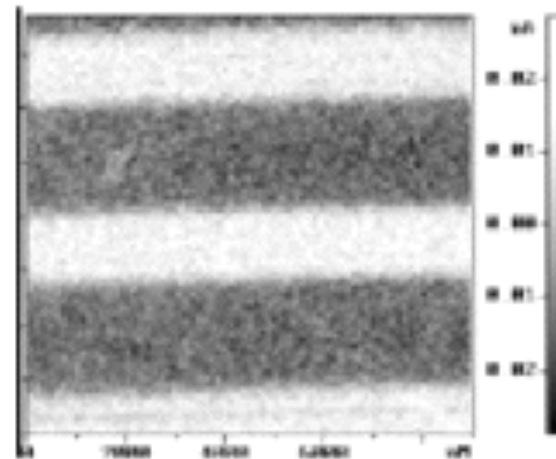
SCM image, resulting distribution of dopant impurity.

UHF SCM

The electrical properties in semiconductors (e.g. charge. density) may be modified by doping (without any topographic signature).
To measure the local distribution of the surface density of the charge carriers two methods are available: **SKPM** or **UHF SCM**
In **UHF-SCM** a "ultra-high-frequency" resonant capacitive sensor is connected to the (conductive) tip by means of a tuned line. When the tip touches the surface, the sample becomes part of the resonant circuit.
The tip-sample capacity loads the line, changing the resonant frequency. The sensitivity may be very high, achieving a resolution of the attofarad (10^{-18} farads)
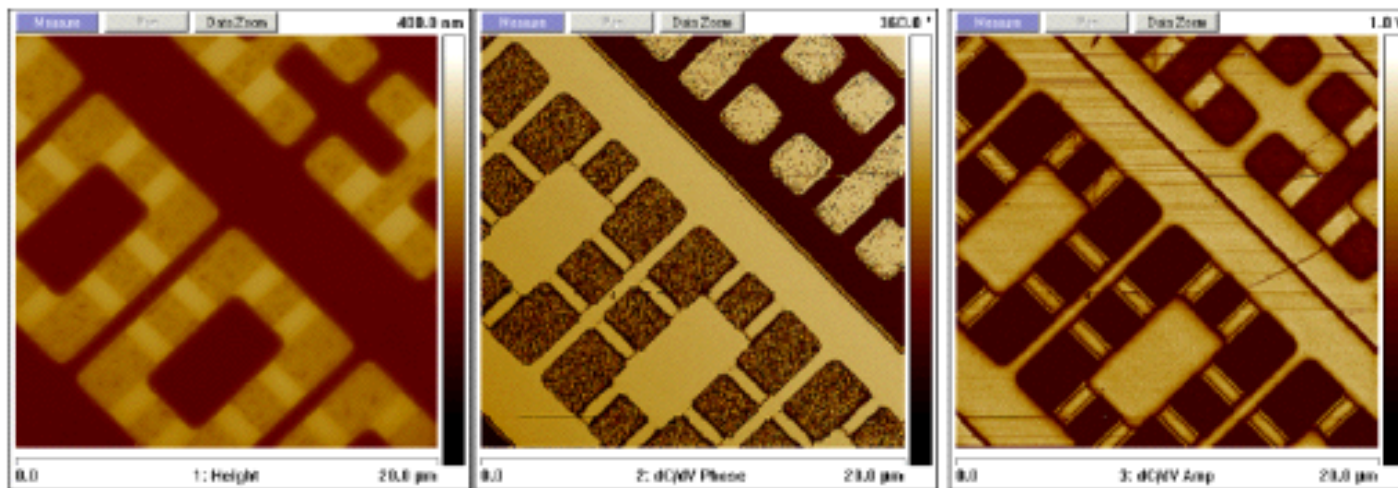
sample: silicon DRAM

Charge displacement in UHF SCM

By applying a low-frequency bias voltage (a few kHz) changes are induced in the capacitor, made of tip and sample separated by the (dielectric) surface oxide film
The free charges in the semiconductor, close to the contact, moves under the applied a.c. voltage and the junction behaves as a variable capacitor.
Capacity changes are due to : 1) applied voltage amplitude; 2) thickness and type of dielectric film;, 3) free charge density.
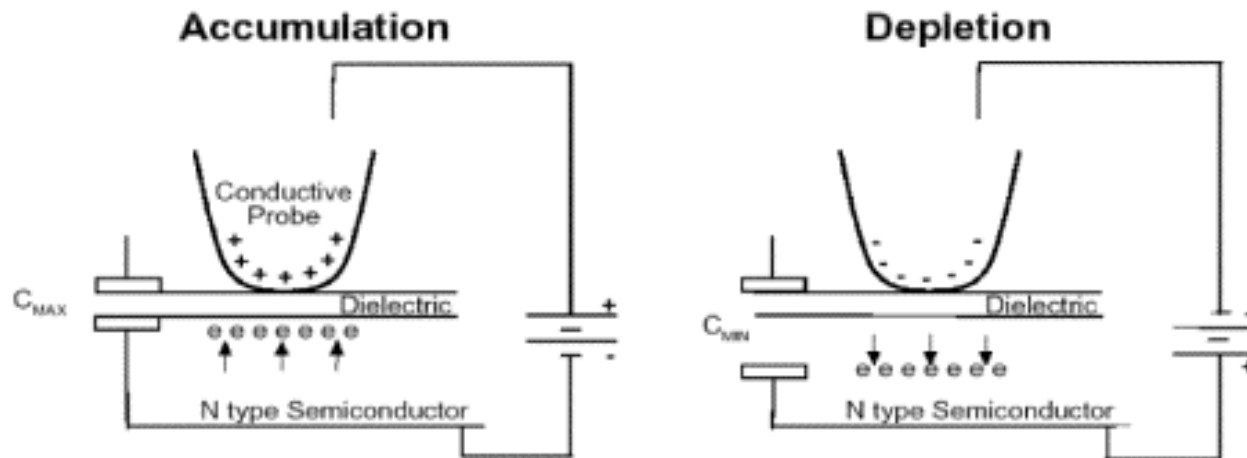
The free charges screen the electric field : the higher is the field (or the lesser is the charge density, or the thinner is the film) the deeper is the field penetration into the semiconductor (= smaller capacity).
E.g. in a sample with uniform doping, with variable oxide thickness, the fields penetrates deeper in the thinner oxide regions.
In a sample with variable charge density, for the same applied voltage, the field penetration is deeper were the charge density is smaller.

UHF SCM working principle
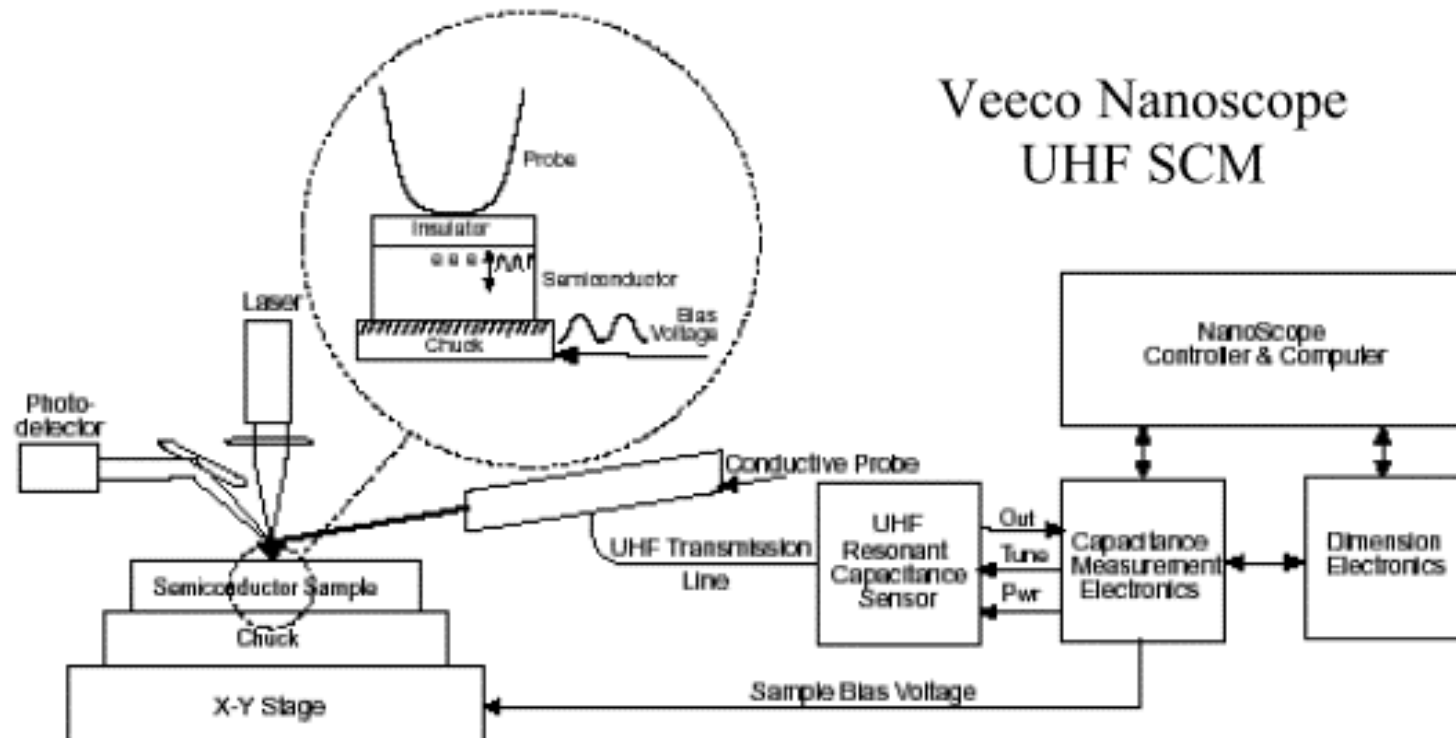
The UHV SCM technique measures the contact capacity changes as a function of the applied field changes . The quantity dC/dV is inversely è proportional to the free charge density, and to the oxide thickness.
The signal used to measure dC/dV is a high frequency constant-amplitude sine wave (≈900 MHz), adjusted close to the resonance for the circuit including the tip-sample junction.
A lock-in detects the amplitude changes produced by the capacity changes.

dV/dC in UHF SCM

N-type CV Curve

Depletion — High concentration — Accumulation

High Carrier Concentration Sample Location

High Doping ΔC

Low Doping ΔC

Low concentration

Low Carrier Concentration Sample Location

Contact Mode topography (left) and SCM dC/dV images of a cross-sectioned transistor in a Pentium-II chip. 1.25µm scans.

(Dimension 3100 Veeco)

V_{AC}

Capacitance — Voltage
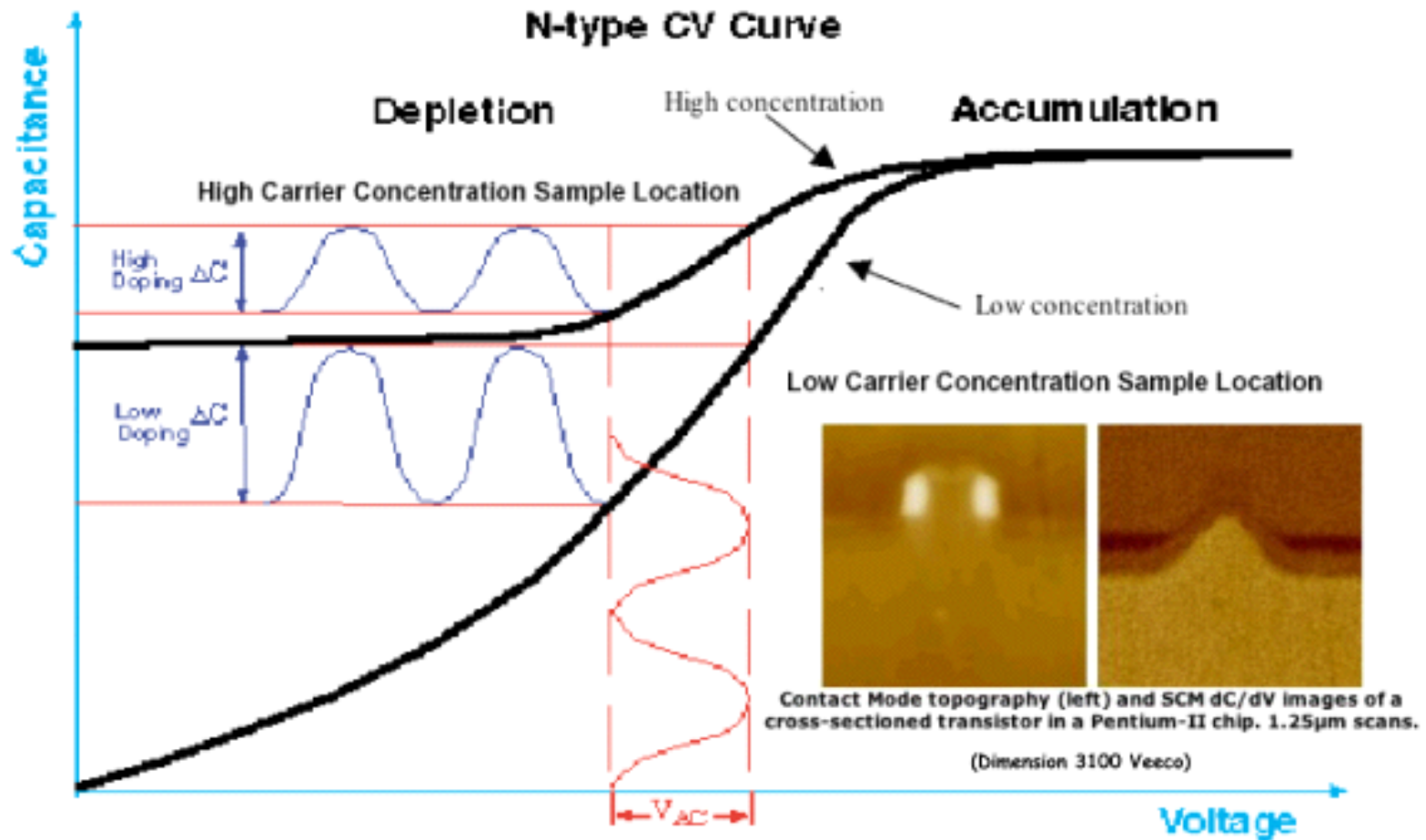
A DC voltage may be added to the AC signal, to adjust the working point.

SKPM (Kelvin Mode of Scanning Probe Microscopy)

SKPM images offer a mapping of the tip-sample work function F(x).

The first pass records the line-profile.

The second pass is made (in Non-Contact) with the cantilever retracted and excited by a voltage Vtip with a.c. and d.c. components :
Vtip = Vdc + Vac sin(ωt).

1-st pass. Sample profile acquisition.
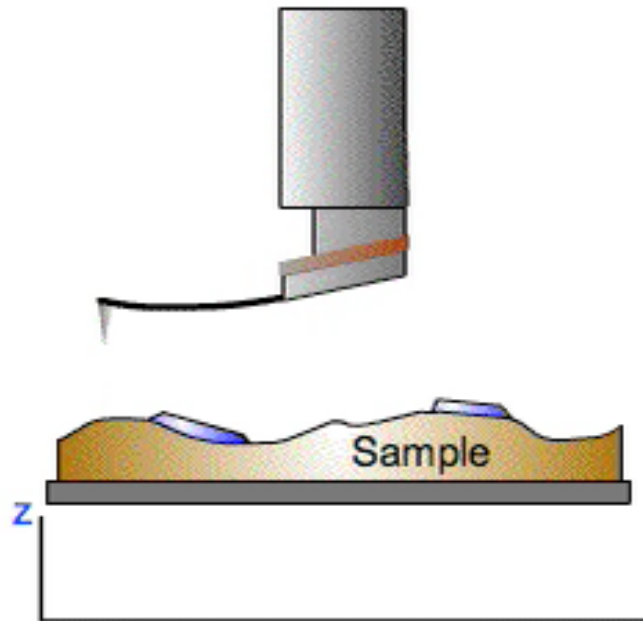
The electric force due to the tip-sample capacitive induction Fcap is:

$$F_{cap} = (1/2) (V_{tip} - F(x))^2 (dC/dz)$$

whose first harmonic

$$F_{cap}(\omega t) = (V_{dc} - F(x) (dC/dz) V_{ac} \sin(\omega t)$$

drives the oscillation.

The feedback system controls the voltage Vdc in order to bring to zero the oscillation amplitude, letting Vdc(x,y) = F(x,y).

The SKPM image is the matrix Vdc(x,y)

Sample

z
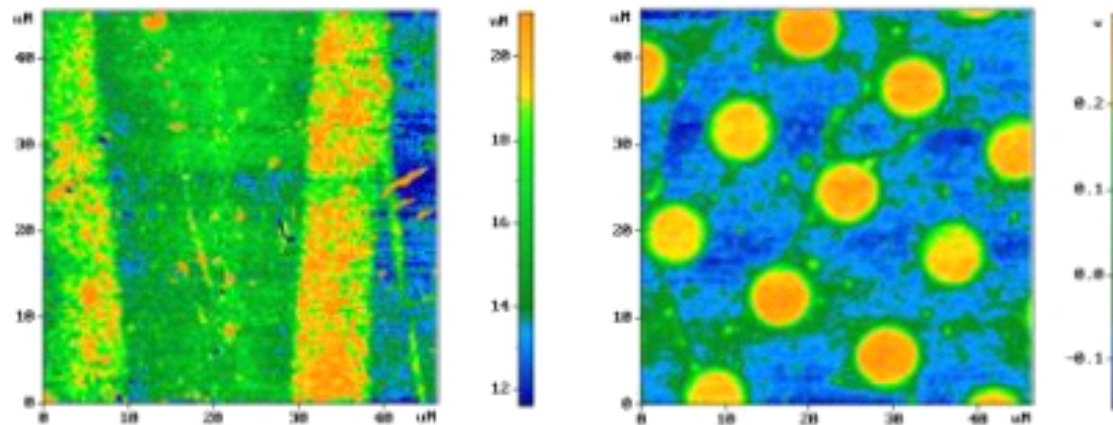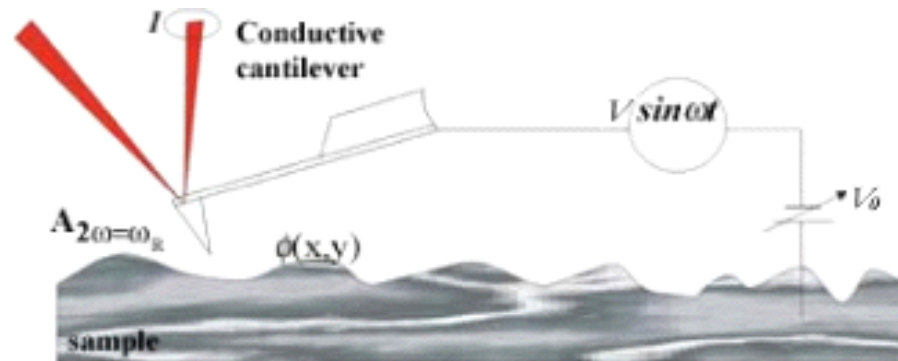
Self assembled monolayer of azobenzene containing thiols.The regions with higher potential (round spots) are dodecanethiol(A). Surrounding monolayer is 4-trifluoromethyl-4'-(10-mercapto-decyloxy) azobenzene(B).
The SKPM contrast is provided by strong dipole moment of the molecules of B whereas the dipole moment of A is low.
Sample courtesy of Dr. P. Karageorgiev and Dr. B. Stiller (University of Potsdam, Germany)

(SolverPro NT-MDT)

FFM (Force Modulation Microscopy)

Vibrating the scanner which holds the sample and measuring the amplitude response of the cantilever maps the differences in surface stiffness or elasticity.

Force modulation technique is useful for detecting soft and stiff areas on substrates which exhibit overall uniform topography.
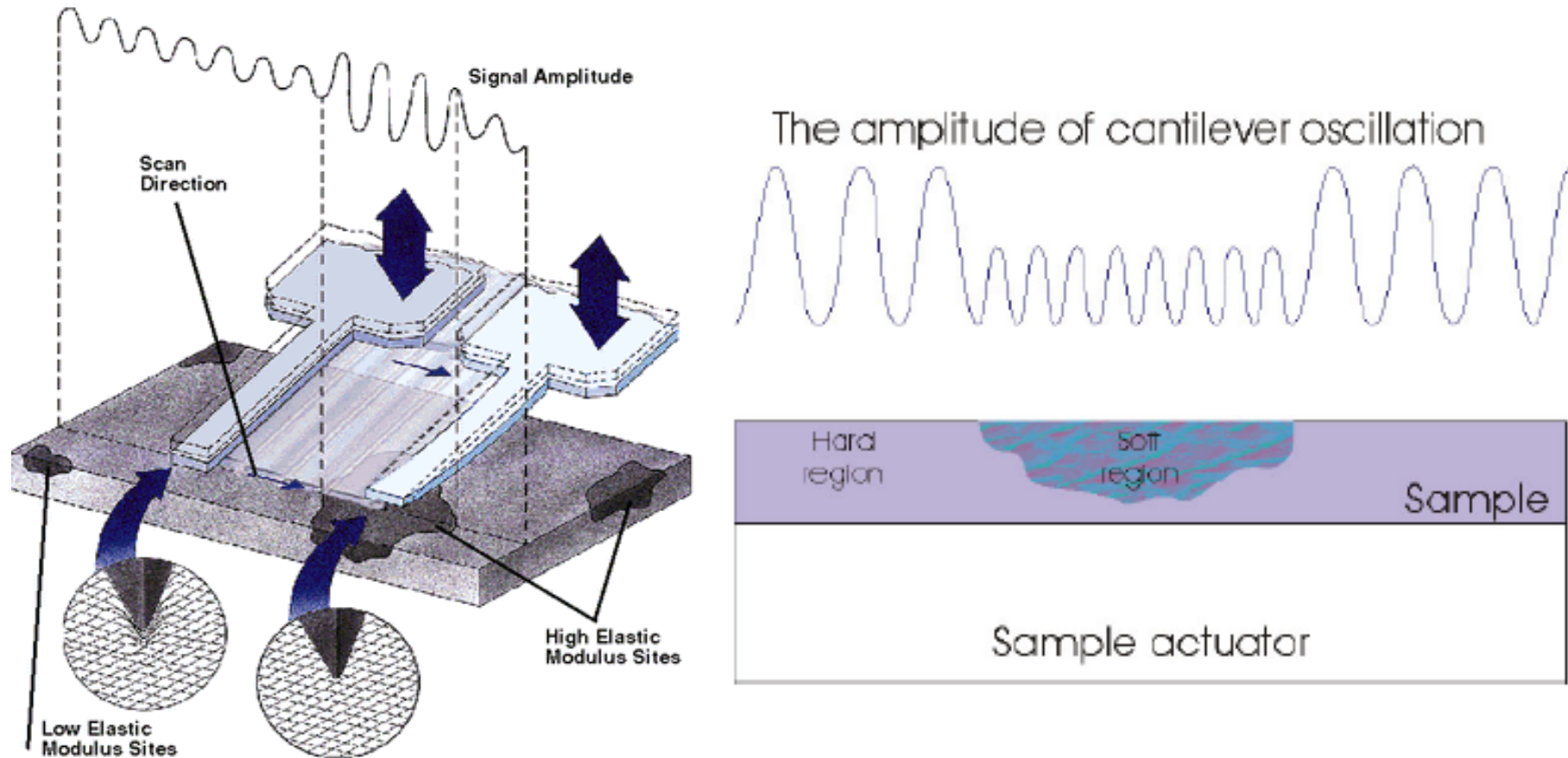
FMM (2)

In FMM the sample is scanned in Contact and the cantilever is periodically pushed (by an auxiliary piezo) against the sample-
The indenting depth produced by the tip depends on the local stiffness of the sample.
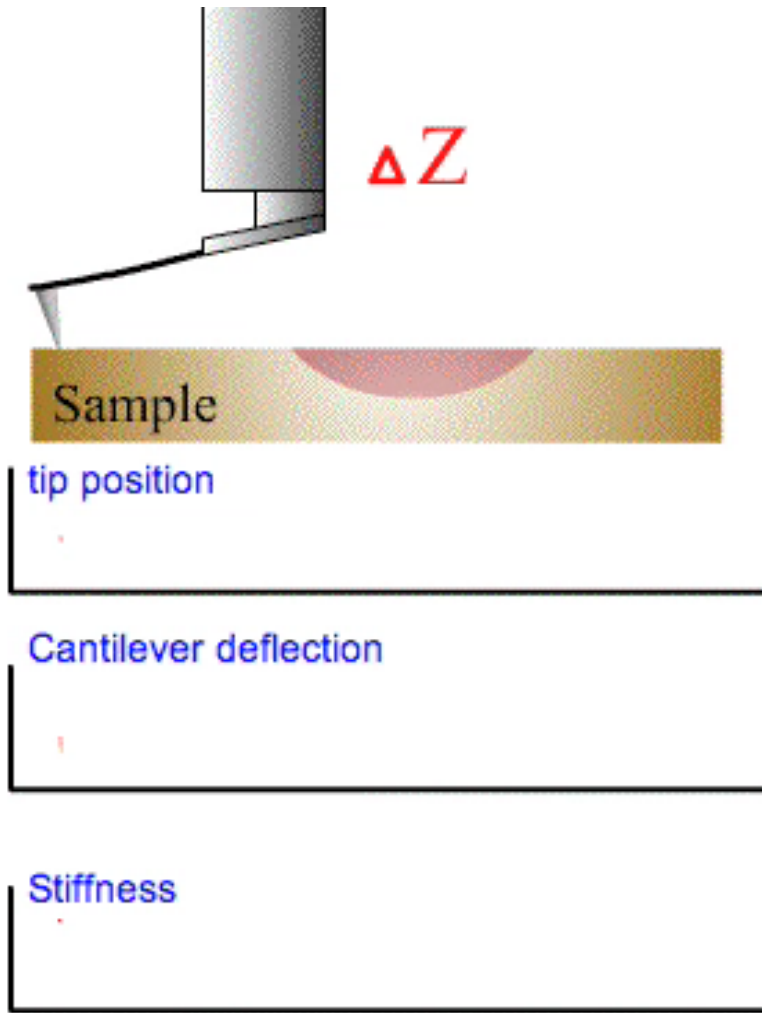
On the stiffer regions the cantilever deflection is larger than in the softer regions .
The opposite holds for the indenting depth D.

If Dz is the scanner displacement, the vertical cantilever deflection is Dz-D.

ΔZ

Sample

tip position

Cantilever deflection

Stiffness

Once known the cantilever elastic constant k_c,
the local elastic constant k_s
of the sample
is obtained by the relation:

$$k_s = k_c \cdot (Dz/D - 1)$$

GaN Buffer

AlGaN$_{(7nm)}$/GaN$_{(2nm)}$ SL

0   200   nM
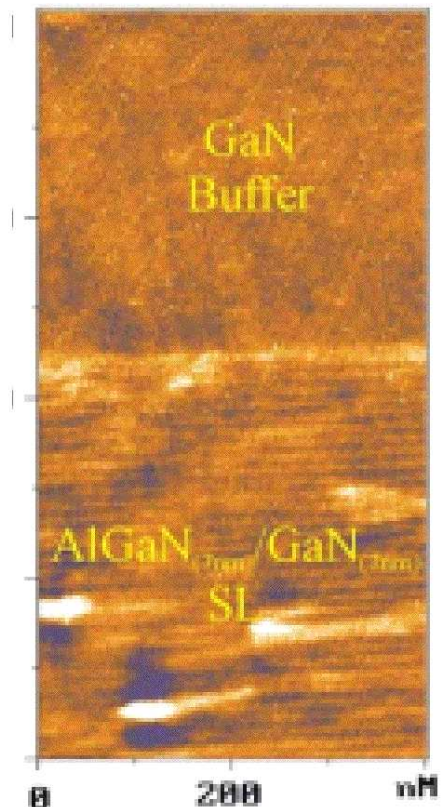
AFAM (Atomic Force Acoustic Microscopy)

The AFAM images map the cantilever oscillation amplitude by exciting the sample through an auxiliary piezo.

The acoustic wave produced by the piezo-actuator drives the sample surface into vibration and couple with the cantilever through the tip in contact.

The cantilever vibration modes (frequencies) depend on the stiffness of the tip-sample contact on the contact area size, (both functions of the Young module of tip E_t and sample E_s, of the tip curvature radius R, on the effective load F_n, and on the surface topography) .

The AFAM spectroscopy is performed by sweeping the piezo-actuator frequency

PZT film   Scan size: 2,6 x 2,6 um

AFAM   Topography

Cantilever deflection

Stiffness

Sample

Piezo

$\sin \omega t$
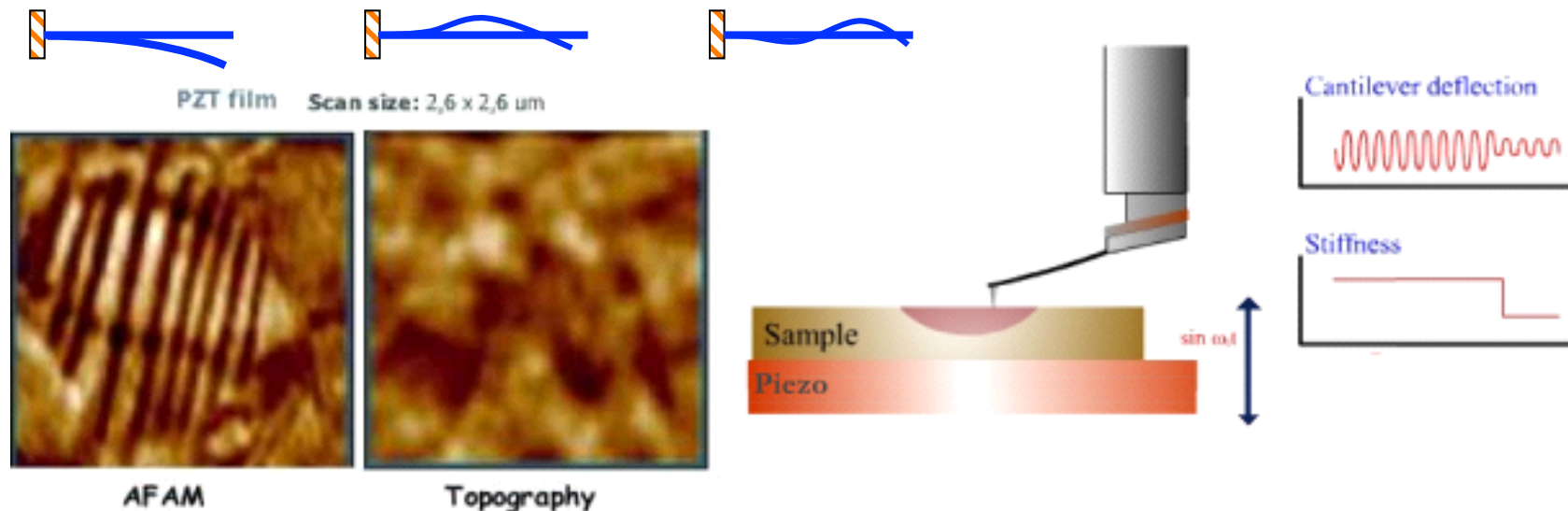
Stripe ferroelectric domain structure are made visible by the difference in local Young's moduli of domains of different polarization. (Sample courtesy of Prof. W. Arnold, Fraunhofer Institute for Nondestructive Testing, Saarbruecken, Germany)

SSRM (Scanning Spreading Resistance Microscopy)

The SSRM images map the local surface resistance by using a conducting tip and a logarithmic current amplifier (dynamic range from 100m☒A to 10 pA).

The sample is scanned in Contact with a biased tip: the resistance is calculated as the ratio between the applied voltage V and the measured current I
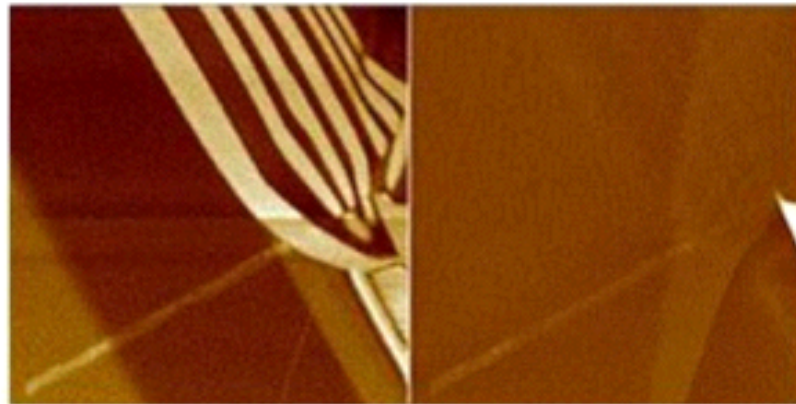
SSRM (left) and contact mode topography (right) scans of an InP-based heterostructure. 7mm scans. Sample courtesy Lucent Technologies. The contrast in the SSRM image shows the different regions of the heterostructure: alternating Zn-doped p-type and S-doped n-type layers.

(Veeco Dimension 3100)

TUNA (Tunneling AFM)

An extension of SSRM technique to samples covered by a thin insulating oxide film is the TUNA an STM were very small current are measured (from 100 pA down to 100fA).
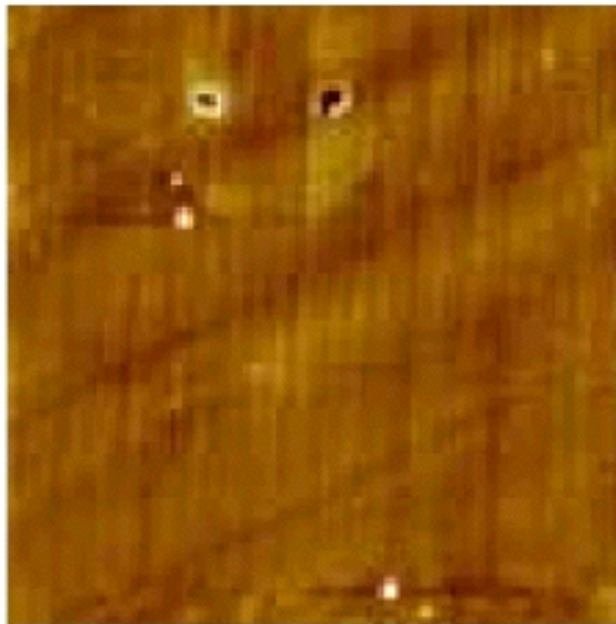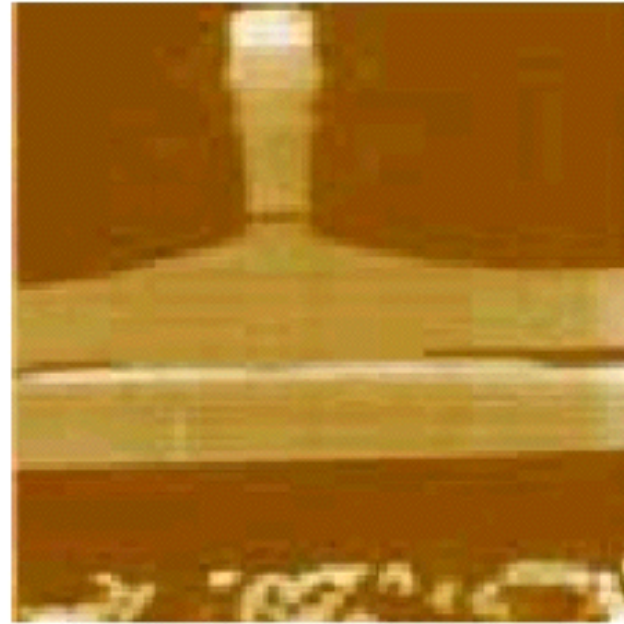
topography   TUNA

Magnetic recorder head : scan size 12 um

SThM (Scanning Thermal Microscopy)

SThM images map the local thermal conductivity of the sample surface
It requires special tips (micro-thermocouples).

Micropipette
Wire

contacts

glass

gold coating
platinum wire

cantilever height h

d

D
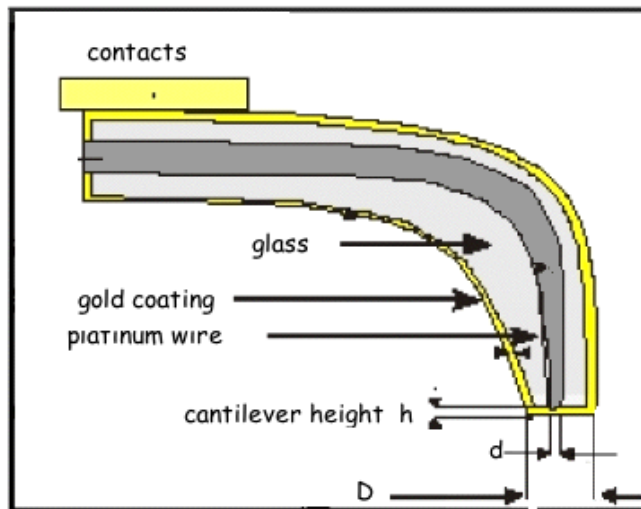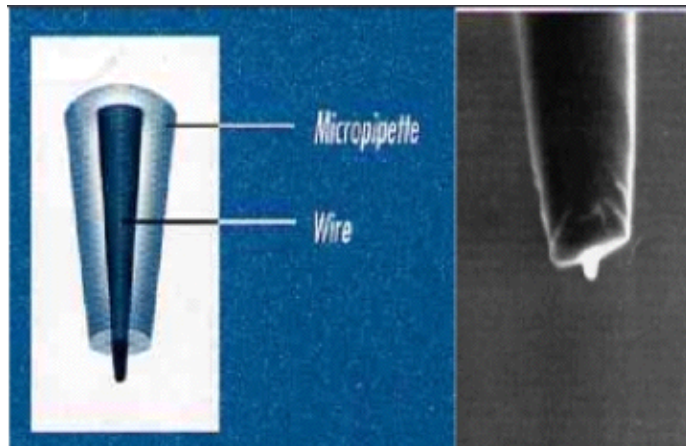
Diagrammatic representation of a single
channel thermal cantilevered glass probe.
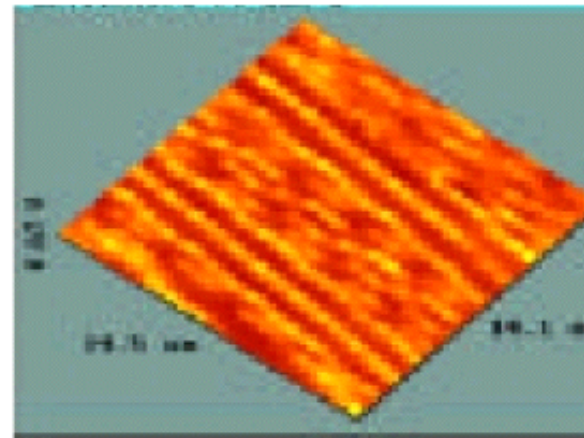
Thermal conductivity image

Micro-thermocouples may be obtained as gold
coated micropipettes with metallic core

SPM-Lithography types

Depending on the operating mode, lithography can be done in different ways:
by applying voltage pulses between the probe and the sample when working in STM mode
or with conductive cantilevers in AFM mode;
or by "scratching" , or "engraving" the surface with the probe when working in SFM mode;
or by using the probe (and applied voltage) to displace atoms on a smooth sample surface.

- STM lithography
- AFM anodic-oxidation lithography
- SFM force lithography
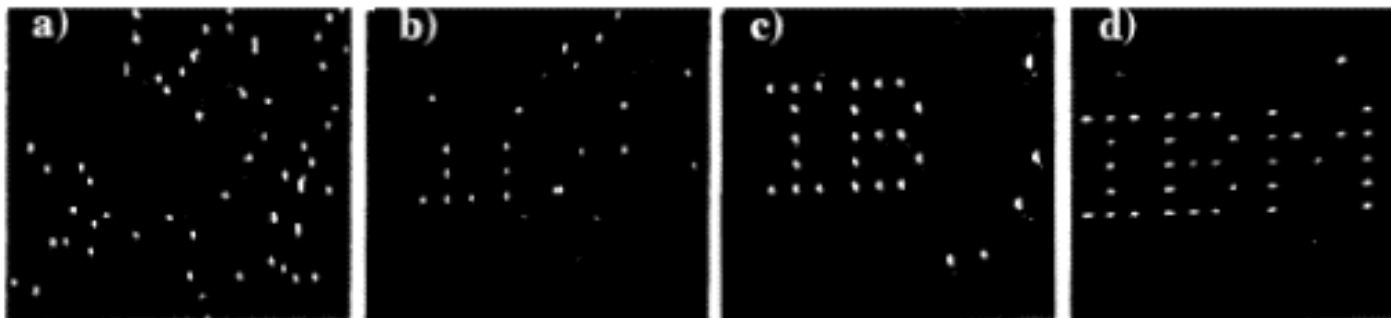
An example of STM nanomanipulation. IBM company name is composed of
Xenon atoms precipitated on the nickel surface (picked-up by applying
voltage, then shaked-off after positioning).

Anodic Lithography

Anodic AFM lithography consists in a local modification of the metallic sample surface
by a voltage applied through a conductive tip

E.g. a thin Titanium layer on a Silicon substrate may be oxidized even during a non-contact scan:
the water film adsorbed onto the sample provides the electric contact by capillarity.
The negative biased tip behaves as a cathode and on the sample surface anodic oxidation occurs.
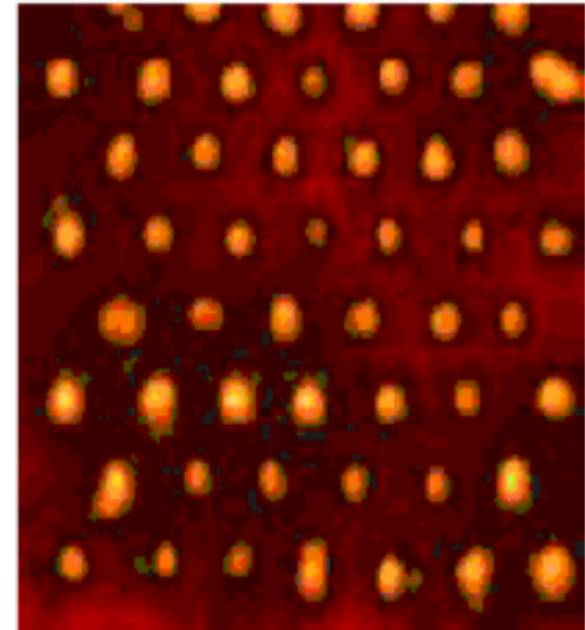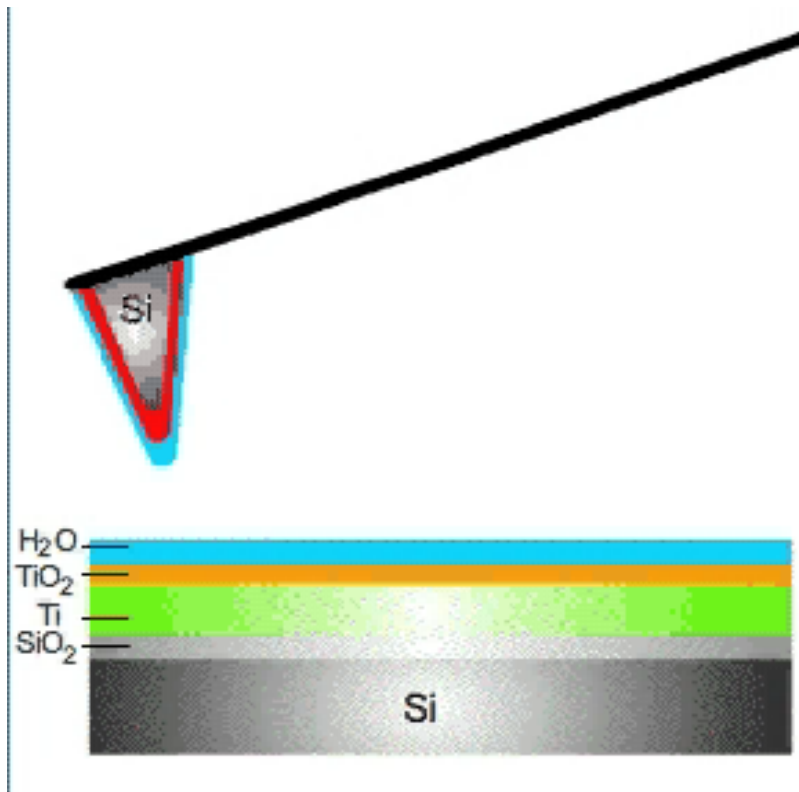
Image (size 200x200 nm^2) of titanium film
on silicon surface, oxidized in the preset points

Force lithography
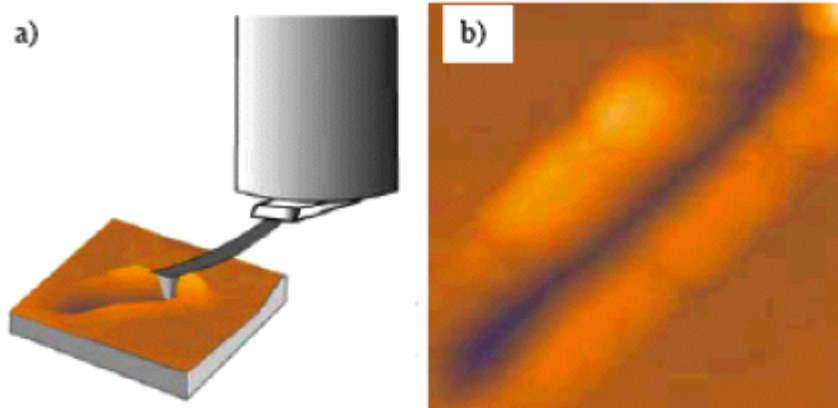
(a) Schematic diagram of static force lithography
(b) image of Al surface (1,6x1,6 ⌦m²) with a scratch
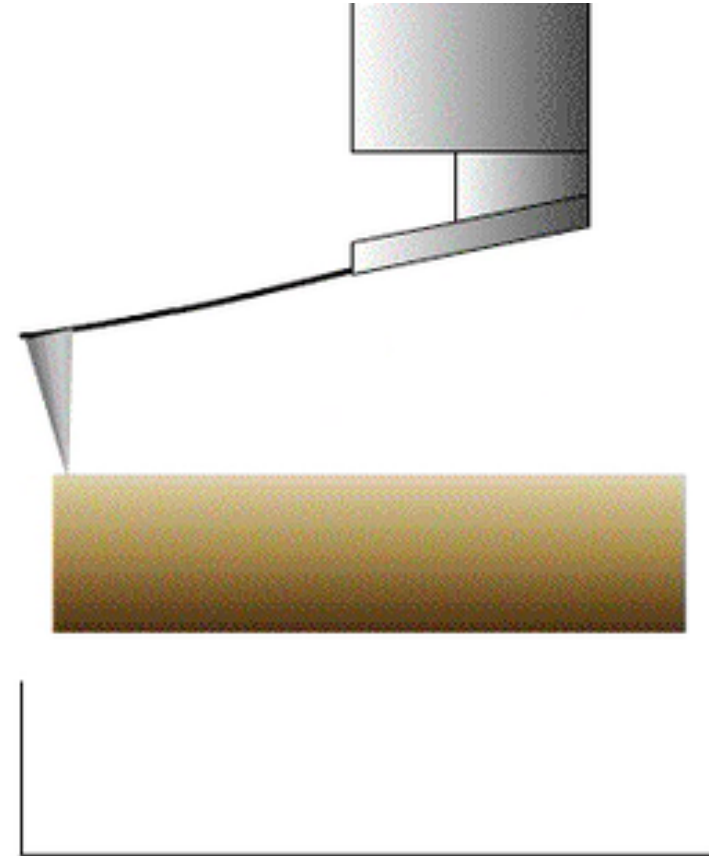
In vector dynamic force lithography (nanoembossing) the surface is modified due to indentations formed by oscillating tip in semicontact mode:

(a) regular array of pits (220x220 nm²)
(b) raster lithography (2,5x2,6 ⌦m²)
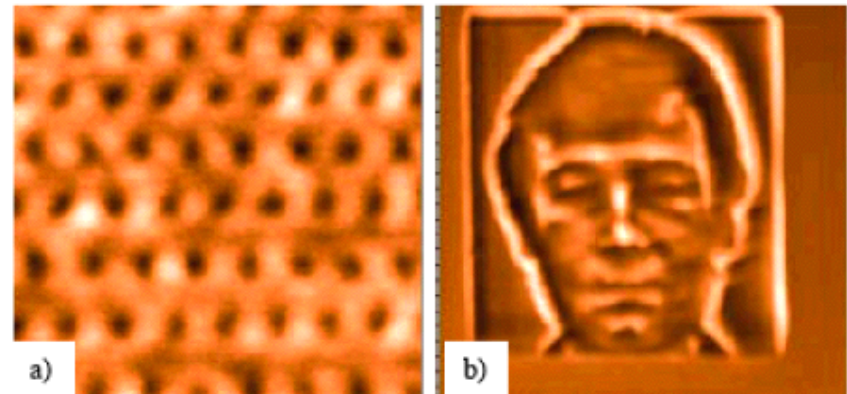
STM Lithography

Using a current pulse the sample surface under the tip can be melted.
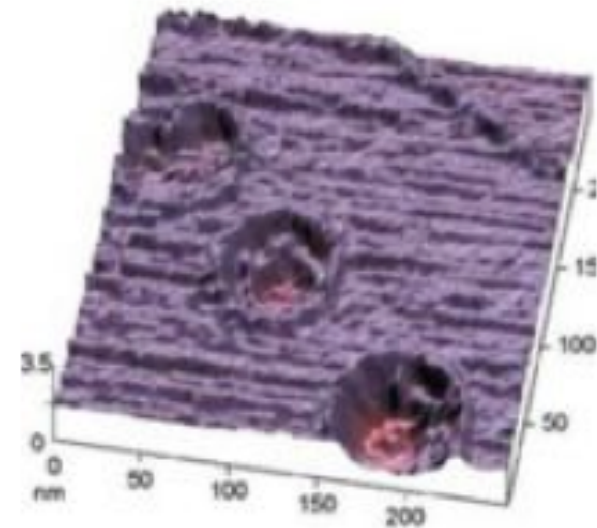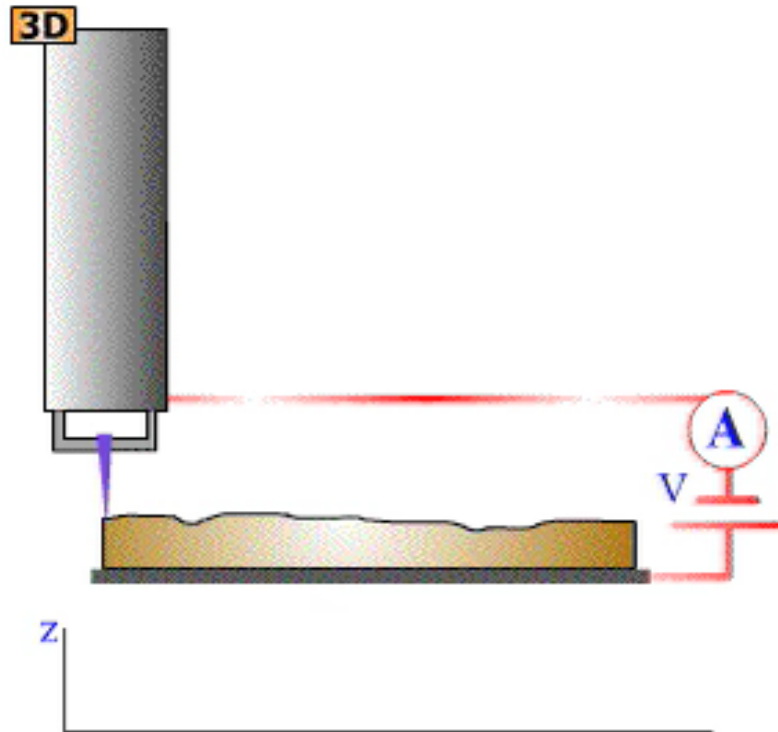
STM Lithography on LB film.

three monolayers of conducting LB film after exposure to three electric pulses.

Cantilevers

The cantilever elastic constant k must allow not-negligible deflection under forces of the order of $10^{-8} \div 10^{-11}$ N: typical values are in the range $k \approx 0.1 \div 100$ N/m.

For a rectangular bar (width = W, length = L, thickness = t), with a Young modulus E we have $k = E(t/L)^3 W/4$.

Assuming $E \approx 10^{11}$ N/m^2 (Silicon or Silicon Nitride), we get $k \approx 0.5$ N/m with $W \approx 20$ μm, $L \approx 100$ μm, and $t \approx 1$ μm.

The most flexible cantilevers (used for Contact Mode) are obtained in a rectangular geometry that offers high sensitivity to "lateral force"

More stiff cantilever (e.g. for Non-Contact Modes) are "V"shaped and insensitive to "lateral force"
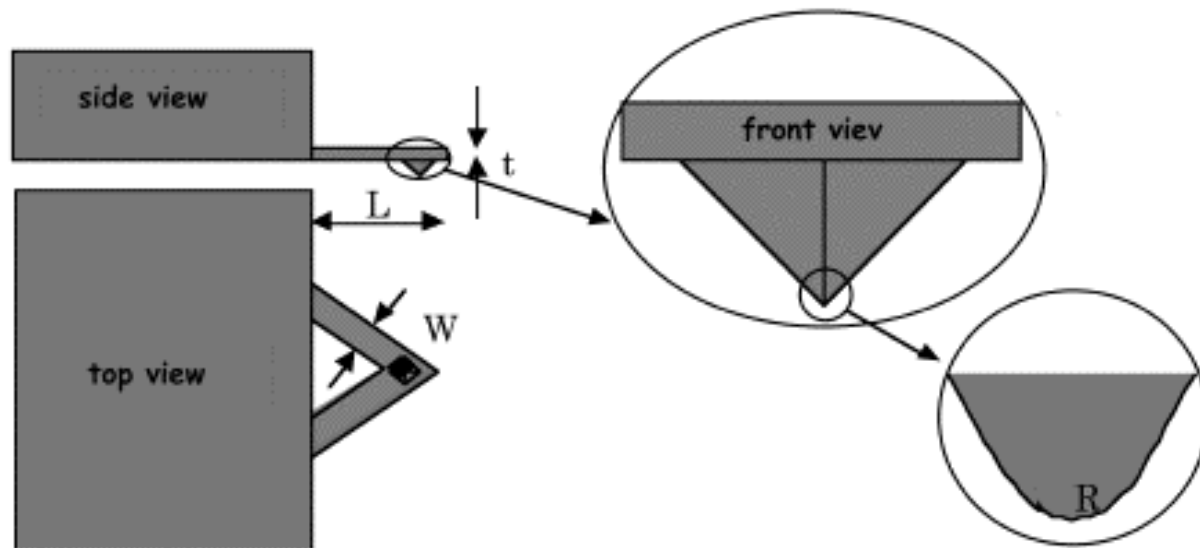
Cantilevers (2)

The mechanical resonant frequency f_r of a cantilever with one end blocked is proportional to $\sqrt{(E/\rho)}\, t/L^2$, where ρ is the density and E the Young modulus.

To increase the frequency f_r while keeping constant the stiffness k, the cantilever must be compact.

High resonant frequency allows faster scans : typically the maximum scan rate f_s (=$1/T_s$, where T_s is the time interval between two signal acquisitions) is chosen $f_s = f_r/100$.

Commercial cantilevers offer f_r ranging from a few kHz to fraction of MHz.
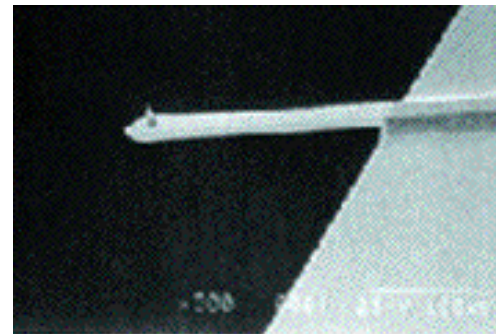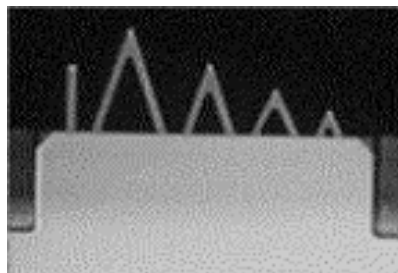
Tip-Sample interaction

Dependence on tip shape anf on tip-sample distance

1. **Parabolic or spherical probe.**

$\pi\rho^2 \sim 2Rx$
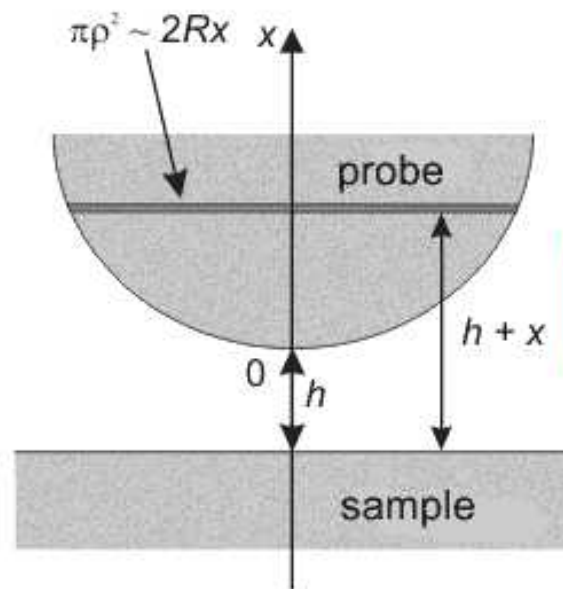
$A_3 \approx 10^{-78}\,\mathrm{J\cdot m^6}$

$$F = \frac{\pi^2 n_1 n_2 A_3 R}{6h^2}$$

probes with a spherical tip at small tip-sample separations $R \gg h$.
The same result is obtained generally for the parabolic tip.

at $R = 10\,\mathrm{nm}$, $h = 0.1\,\mathrm{nm}$ $F \approx 3.3\cdot 10^{-9}\,\mathrm{N}$

2. **Conical probe.**

$\pi(x\,\mathrm{tg}\varphi)^2$
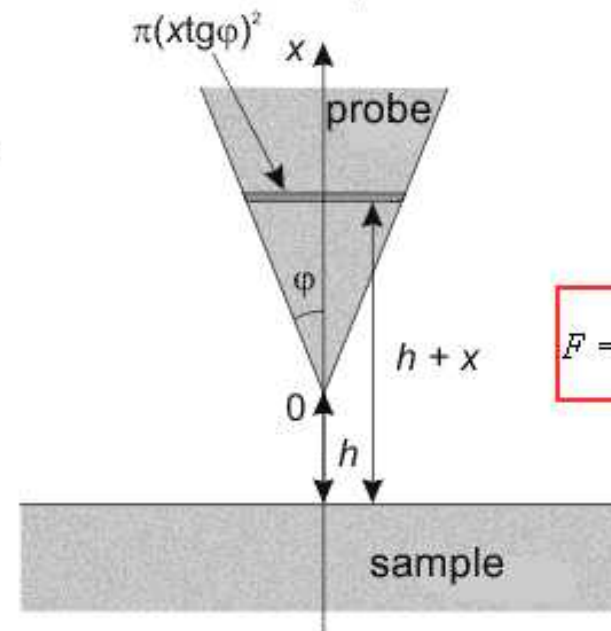
$$F = \frac{\pi^2 n_1 n_2 A_3\,\mathrm{tg}^2\varphi}{6h}$$
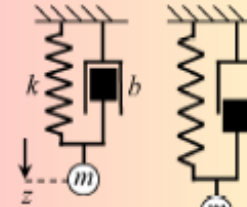
when tip curvature radius can be neglected ($R \ll h$).

at $h = 100\,\mathrm{nm}$, $\varphi = 11°$, $F \approx 1.3\cdot 10^{-15}\,\mathrm{N}$

Cantilevers Formulas (from Asylum Research)

The Physics of Atomic Force Microscopy

Simple Harmonic Oscillator

$$F = -kz \qquad T = \frac{2\pi}{\omega_0}$$
$$\omega_0 = 2\pi f_0 = \sqrt{\frac{k}{m}} \qquad A \propto \exp(-t/\tau)$$
$$Q = \frac{k}{\omega_0 b} = \frac{\pi\tau}{T} = \frac{A_{max}}{A_{drive}} \equiv \frac{\omega_0}{\Delta\omega}$$
$$m\frac{d^2 z}{dt^2} = F_{drive} - b\frac{dz}{dt} - kz + F_{ext}$$
$$F_{drive} = kA_{drive}\cos\omega t$$
$$A = \frac{A_{drive}\omega_0^2}{\sqrt{(\omega_0^2 - \omega^2)^2 + (\omega\omega_0/Q)^2}}$$
$$\tan\theta = \frac{\omega\omega_0/Q}{\omega_0^2 - \omega^2}$$

Attractive / Repulsive
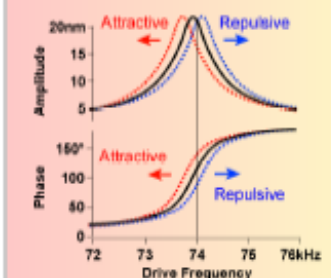
$$\delta A_{(max\ slope)} = \frac{2QA_0}{3\sqrt{3}(k - F')}F'$$
$$\delta\omega_0 \approx -\frac{\omega_0 F'}{2k} \qquad \delta\theta_{res} = \frac{Q}{k}F'$$
$$\overline{P}_{tip} = \frac{1}{2}\frac{kA^2\omega}{Q}\left[\frac{QA_{drive}\sin\theta}{A} - \frac{\omega}{\omega_0}\right]$$

Cantilever Beam Theory

$$k = 3\frac{EI}{l^3} \qquad f_0 = \frac{C}{2\pi}\sqrt{\frac{k}{m_t + 0.24m_c}} \qquad m_c = \rho A l$$

$C = 1$

$$d \qquad I = \frac{\pi d^4}{64}$$

$C = 6.25$

$$d_o \quad d_i \qquad I = \frac{\pi}{64}(d_o^4 - d_i^4)$$

$C = 17.5$

$$t \qquad I_z = \frac{wt^3}{12}$$

$C = 34.4$

$$t_o \quad t_i \qquad I_z = \frac{w_o t_o^3 - w_i t_i^3}{12}$$

Cantilever Shapes

$$h_{end}(x) = \frac{3Lx^2 - x^3}{2L^3}$$
$$h_{dist}(x) = G(\cosh\kappa x - \cos\kappa x) + H(\sinh\kappa x - \sin\kappa x)$$
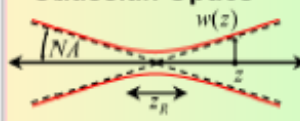
Added Mass Correction

$$m_t = m_w\left(\frac{L - \Delta L}{L}\right)^3$$

Reference Spring

$$k = k_{ref}\left(\frac{\Delta z_{ref}}{\Delta z} - 1\right)$$

Shot Noise

$$\delta V_{rms} = R\sqrt{2e\eta P_0 B}$$

Johnson Noise

$$\delta V_{rms} = \sqrt{4k_B TRB}$$

Thermal Noise

on resonance
$$\delta z_{rms} = \sqrt{\frac{4k_B TBQ}{k\omega_0}}$$

off resonance
$$\delta z_{rms} = \sqrt{\frac{4k_B TB}{Qk\omega_0}}$$

Energies

$k_B T = 4$ pNnm

ATP hydrolysis
12-21 k_BT
Hydrogen Bond
4-18 k_BT
Covalent Bond
100-200 k_BT
Protein Stability
6-20 k_BT

Forces

Covalent Bonds
2-5 nN
Unzipping DNA
20 pN (GC)/10 pN (AT)
1 mW of light reflecting
off of a surface
7 pN

Distances

Covalent Bonds
0.1nm
Hydrogen Bond
0.25 nm
Tip Radius of Curvature
2-50 nm
DNA diameter
2nm

Stiffness

Covalent Bond
10N/m
Cantilevers
0.01 -100N/m

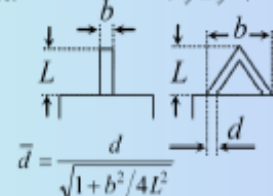|	Young's Modulus E (GPa)	Density ρ (kg/m³)	Speed of Sound (m/s)	Thermal Expansion (um/mK)	Thermal Conductivity λ (W/mK)	Heat Capacity c_p (J/gK)
Stainless	200	7760	5100	10-13	12-25	.5
Steel	210	7850	5200	11	35	.46
Ti	116	4500	5100	8.9	17	.528
Al	70	2700	5100	24	190	.88
Invar	148	8050	4300	1.3	10.15	.515
SiN	260-320	3100	~9800	3	30	.71
Si	179	2330	8800	2.6	150	.7
Cu	117	8900	3600	17	383-391	.385
W	400	19300	4500	4.4	163	.134
Granite	20-60	2500-2700	~5000	3.7-11	1.4-4.2	.21-.35
Pyrex	61	2250	5500	4.4	163	.134

Thermal Method
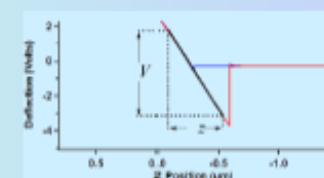
$$\chi = \frac{InVOLS_{free}}{InVOLS_{end}}$$
$$InVOLS_{end} = \Delta z/\Delta V\ (nm/volt)$$
$$k = \frac{k_B T}{\langle\delta V^2\rangle\chi^2 InVOLS^2}$$
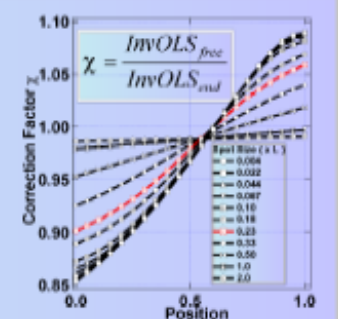
Sader Method

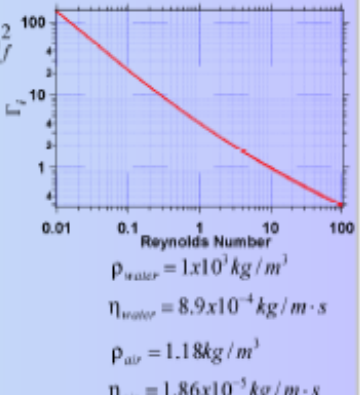$$k_{rect} = 0.1906 b L^2 \rho_f Q_f \Gamma_i(Re)\omega_f^2$$
$$\overline{d} = \frac{d}{\sqrt{1 + b^2/4L^2}}$$
$$D_0 = k_{rect}\frac{4L^3}{b} \qquad Re = \frac{\rho\,\omega b^2}{4\eta}$$
$$k_{tri} = \frac{D_0\overline{d}}{2L^3}\left(1 + \frac{4\overline{d}^3}{b^3}\right)^{-1}$$

$$\rho_{water} = 1 \times 10^3\ kg/m^3$$
$$\eta_{water} = 8.9 \times 10^{-4}\ kg/m\cdot s$$
$$\rho_{air} = 1.18\ kg/m^3$$
$$\eta_{air} = 1.86 \times 10^{-5}\ kg/m\cdot s$$

Gaussian Optics

$$w(z) = w_0\sqrt{1 + \left(\frac{\lambda z}{\pi w_0^2}\right)^2}$$
$$z_R = \frac{\pi w_0^2}{\lambda} \qquad N.A. = \frac{\lambda}{\pi w_0}$$

Coherence Length

$$\Delta l = c\Delta t \cong \frac{\overline{\lambda_0}^2}{\Delta\lambda_0}$$

Thin Lens

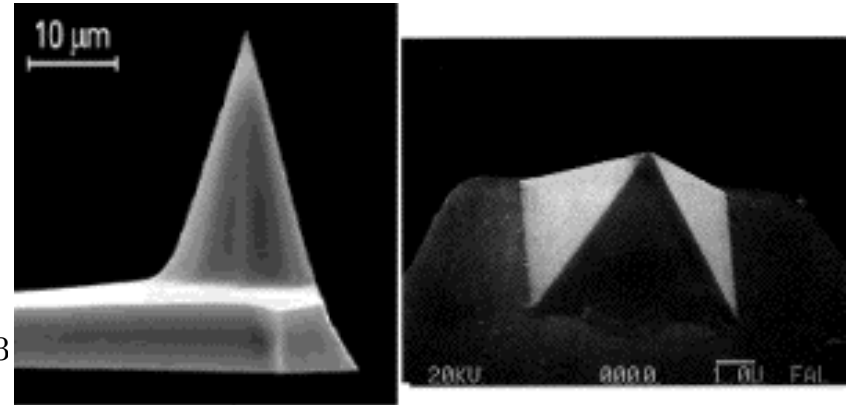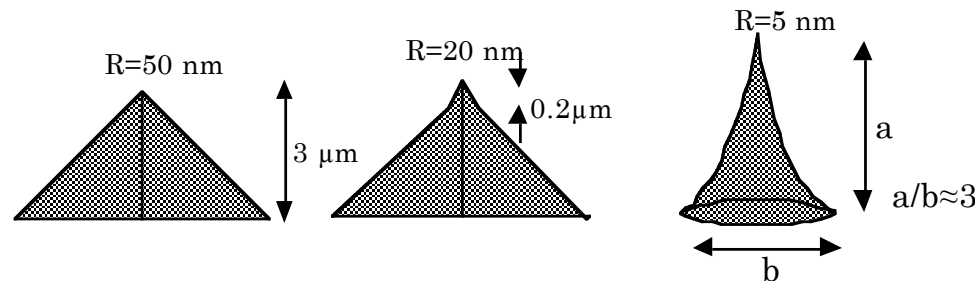$$\frac{1}{s} + \frac{1}{s'} = \frac{1}{f}$$

Lab. Fis. Mat. G.Torzo

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Tips

Different commercial tips are available with different values of the apex curvature radius R :
typically

Pyramidal R ≈ 500 Å
Sharpened pyramidal R ≈ 200 Å
Conical (*ultratip*) R ≈ 50÷100 Å

Pyramidal tips are obtained by photolithography: through a squared hole in a mask evaporated onto a silicon wafer, a pyramidal pit is produced by anisotropic etching of [001] plane; then a Silicon Nitride film is deposited and glued onto a glass plate. By chemical etching the silicon is removed leaving the Silicon Nitride cantilever with pyramidal tip.

Conical tip are grown by SEM deposition in "dirty" atmosphere, or ion-milling (FIB Focused Ion Beam)
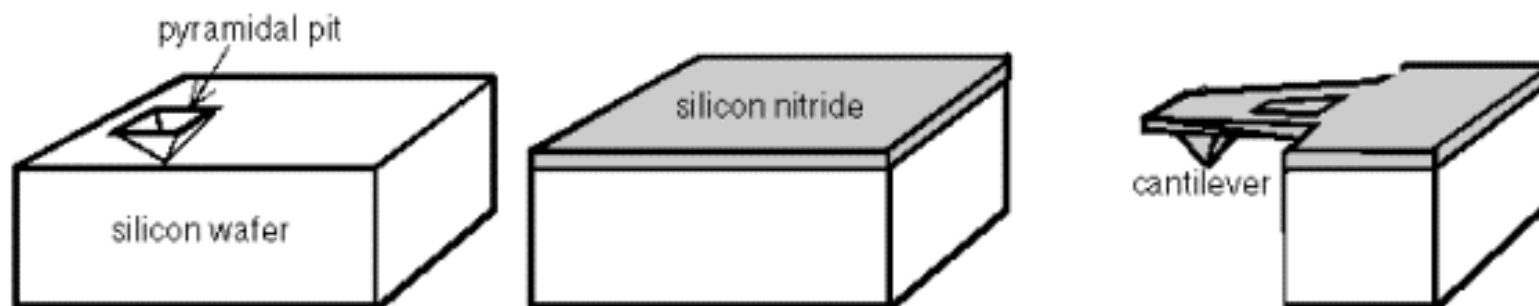
Modified Tips

Special tips may be functionalized through physical or chemical modifications

Chemical or biological coatings can be applied to particle probes or off-the-shelf tipped AFM cantilevers.

Gold, Platinum, Diamond, Cobalt coatings are available

Chemically & Biologically Coated AFM Tips

Particle AFM Probes

Custom AFM Probe Modifications

Chip thickness	0.4mm
Reflective side	Au
Spring number	2
Aspect ratio	3:1
Cone angle f	<=22°
Curvature radius of a tip	typical 10nm
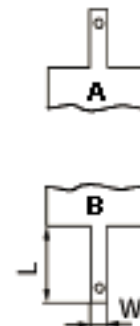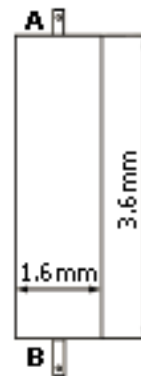
Available Cantilever series	Spring	Cantilever lenght, L ±5µm	Cantilever width, W ±3µm	Cantilever thickness, µm			Resonant frequency, kHz			Force constant, N/m		
				min	typical	max	min	typical	max	min	typical	max
DCP11	A	100	35	1.7	2.0	2.3	190	255	325	5.5	11.5	22.5
	B	130	35	1.7	2.0	2.3	115	150	190	2.5	5.5	10

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Etalon Tips

polysilicon levers with silicon high resolution tips
reproducible parameters (typical dispersion �10% / ⎷20%)
high aspect ratio (cone angle 22°)
 typical curvature radius of 10nm.

Special shape allows minimizing a tip base increasing the tip aspect ratio

Silicon tip — Polysilicon lever — SiO₂ — Reflective coating - Au

Specification for HA_NC probes

Chip thickness	0.45mm
Reflective side	Au
Spring number	2
Tip height h1	5-10 µm
Tip base height h2	4-6 µm
Ratio h1/h2	>1
Tip aspect ratio	5:1
Cone angle ц	<=22°
Curvature radius of a tip	typical 10nm

Special frequency stabilizer
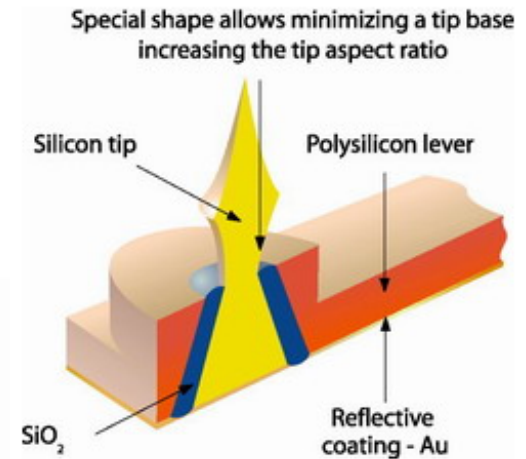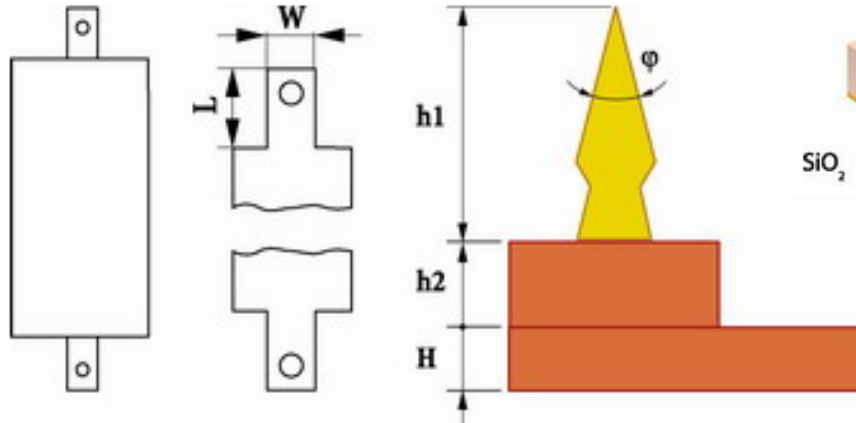
Cantilever series	Spring	Cantilever lenght, L ±2µm	Cantilever width, W ±3µm	Cantilever thickness, µm			Resonant frequency, kHz		Force constant, N/m	
				min	typical	max	Nominal	Typical dispersion	Nominal	Typical dispersion
HA_NC	A	87	32	1.7	1.75	1.8	200	±10%	5.8	±20%
	B	117	32	1.7	1.75	1.8	120	±10%	3.4	±20%
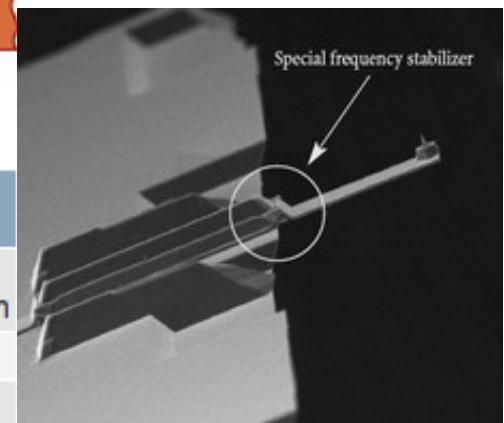
Aspect Ratio and Curvature Radius

An important parameter is the aspect ratio (ratio between tip height and base)
The larger is the aspect ratio the better is resolved the SPM image.

Alternatively the tip quality is defined by the sidewall angle, that for a pyramidal tip is 45° or 27.5° (edge or face of the tetrahedron) and for conical tips varies from base to apex reaching a minimum of about 22°.

FIB tips

10 μm
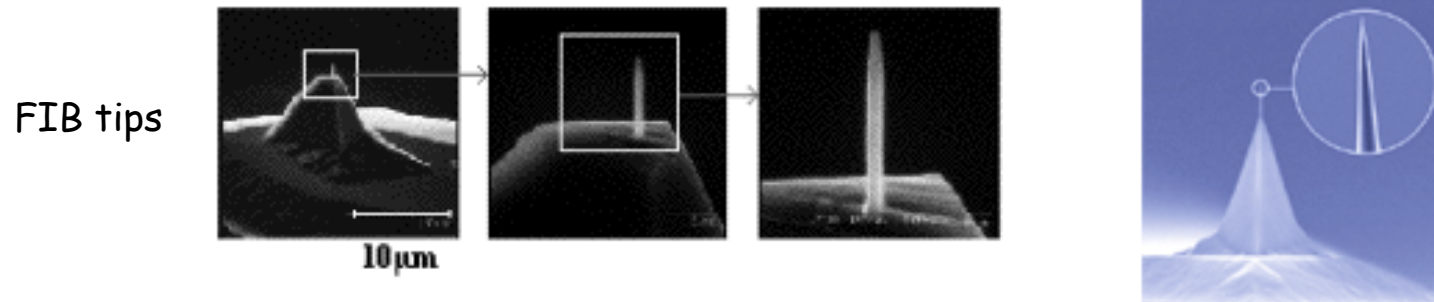
Very high Aspect Ratio tips may be obtained from nanotubes

__ 200 nm
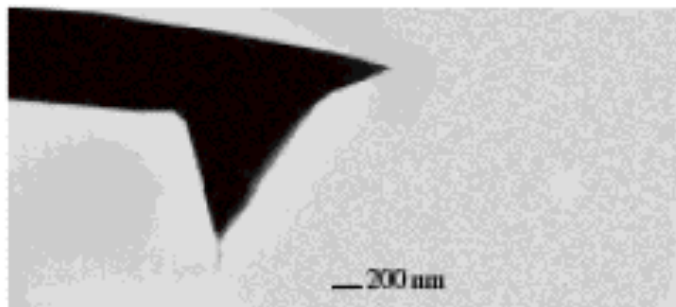
SEM micrograph of a multi-walled carbon nanotube (MWNT) tip physically attached on the single-crystal silicon, square-pyramidal tip (Courtesy Piezomax Technologies, Inc.)

Regular Si₃N₄ Tip

MWNT___

1 μm
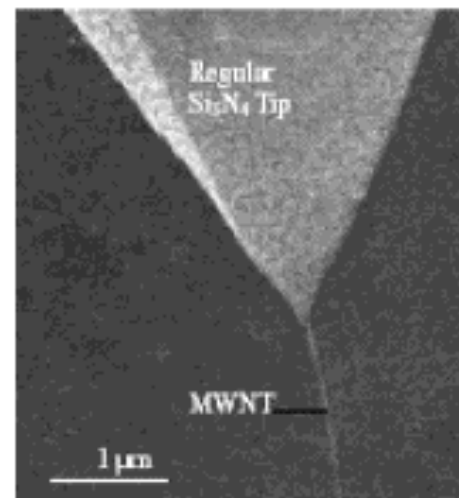
Scanning electron microscopy image of a carbon nanotube (MWNT) mounted onto a regular ceramic tip as a probe for atomic force microscopy

Nanotube tips: high aspect ratio
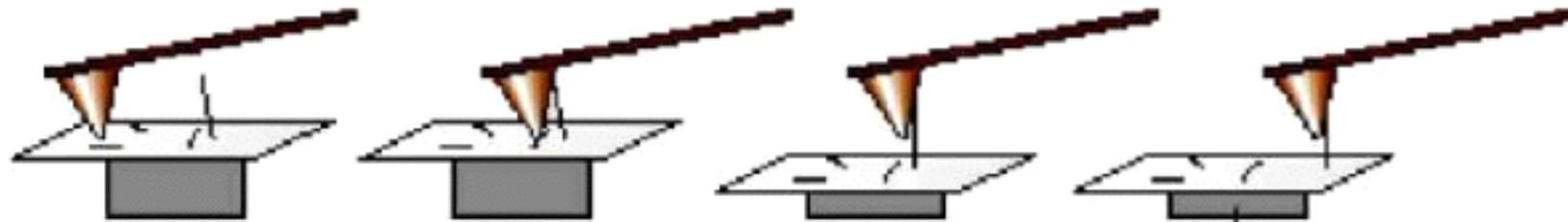
Pick-up tip assembly of nanotube probes.

Z (50 nm/div)

Time (10 ms/div)
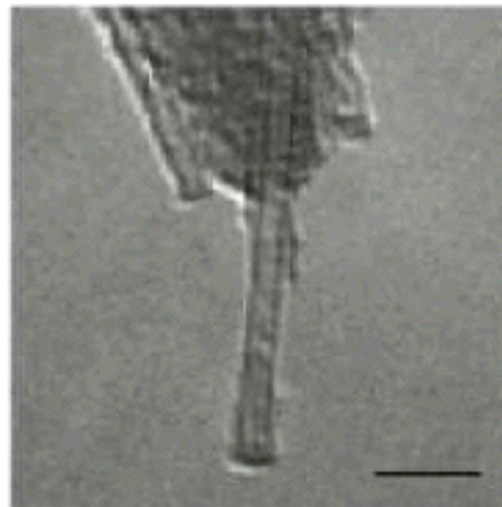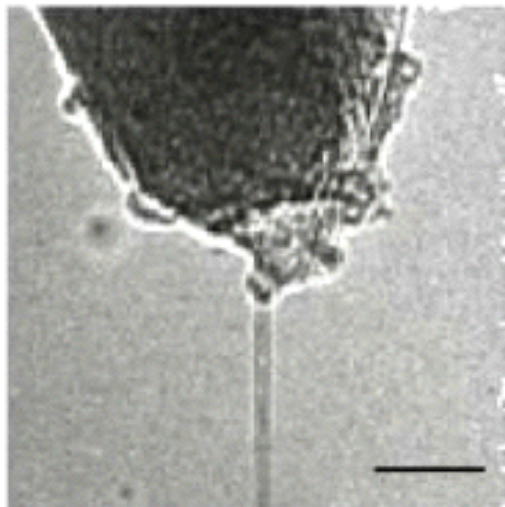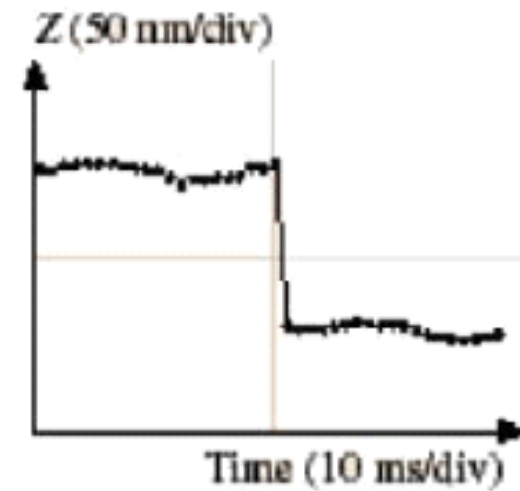
trace of the Z-position during a pick-up event.

(diameters 0.9 nm and 2.8 nm)

Whisker tips: high aspect ratio

170 nm

530 nm
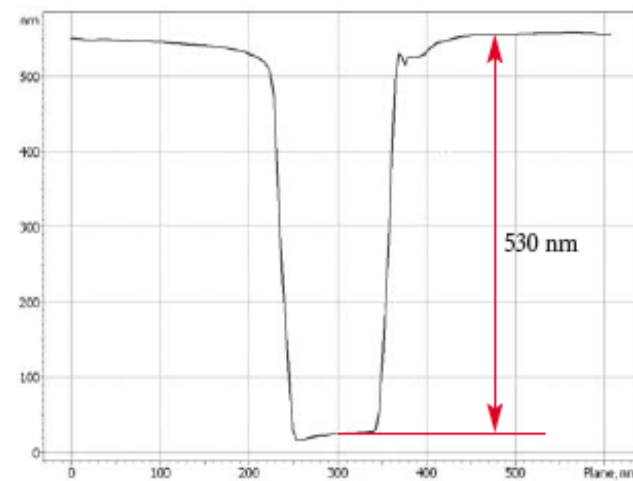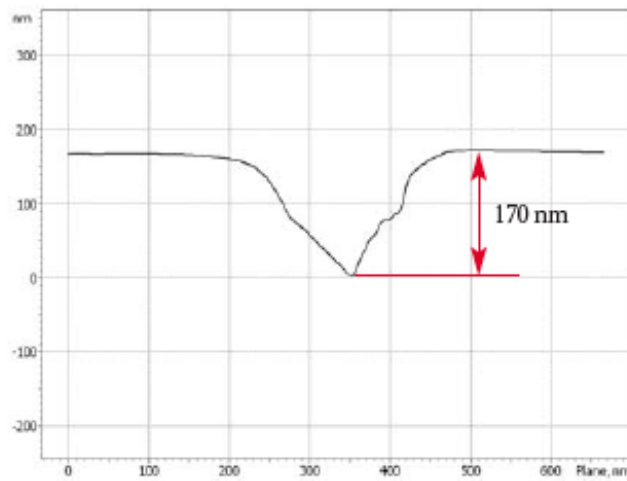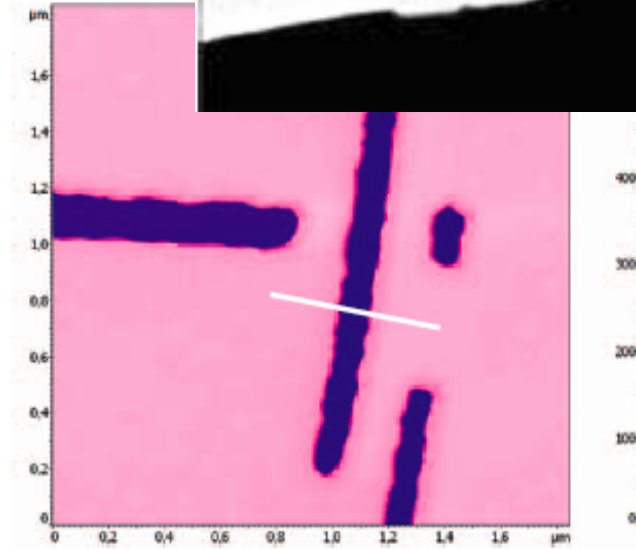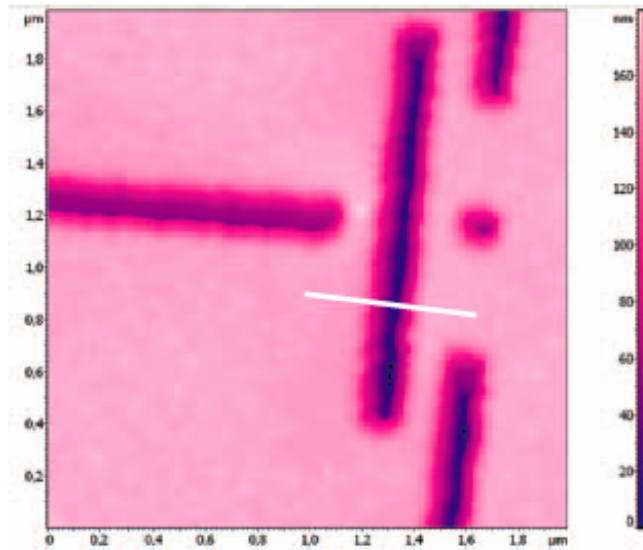
Sources of Image artifacts

Blunt tip Multiple tip → Tip replicas, multiple features

Hardware noises → Blur

Tilted sample surface → Tilt

Valid signal

Instability of Tip - Sample contact → Glitches Streaks

Scanner imperfections → Creep , Distortion

Acoustical or optical interference → Ghost sinusoidal pattern
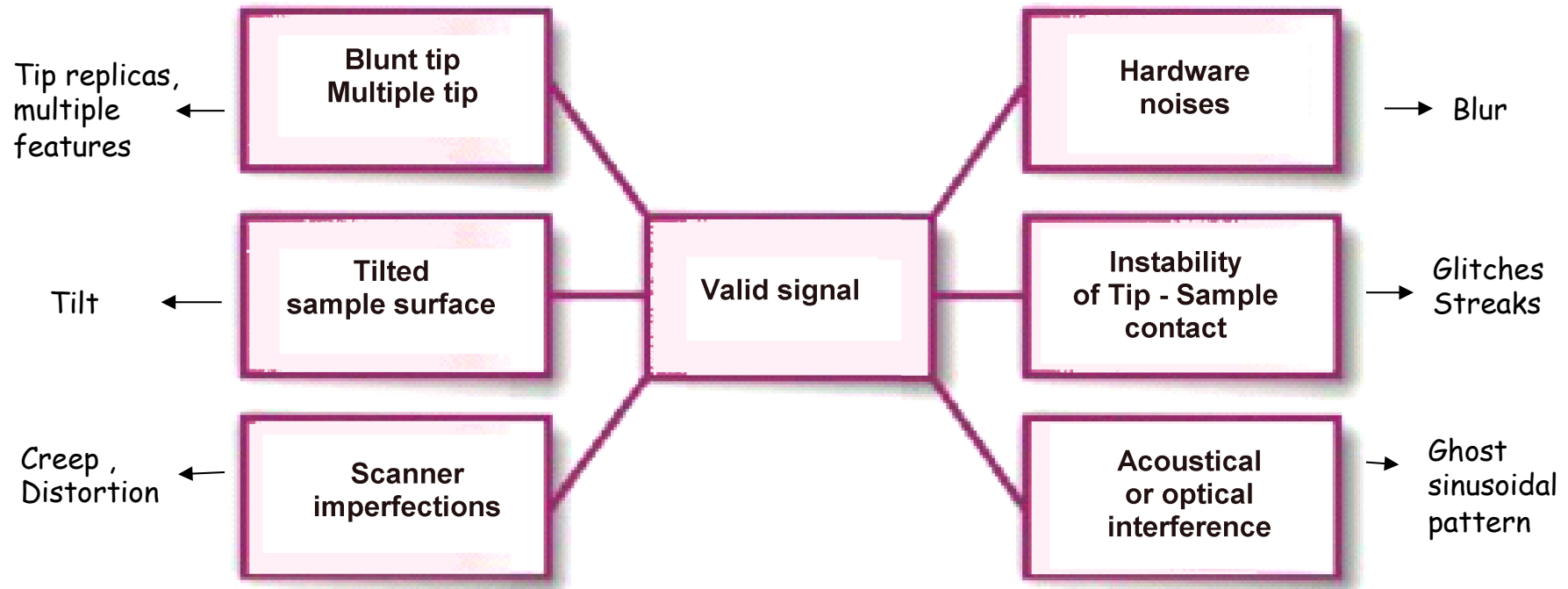
Z offset - Tilt - Non-linearity & Drift - Blur - Glitches

Scanner non-ideality

Typical scanner imperfections are:
Non-linearity
Creep
Hysteresis
Aging
Thermal drift
Cross-coupling among x-y-z displacements

Non-linearity   hysteresis   Creep
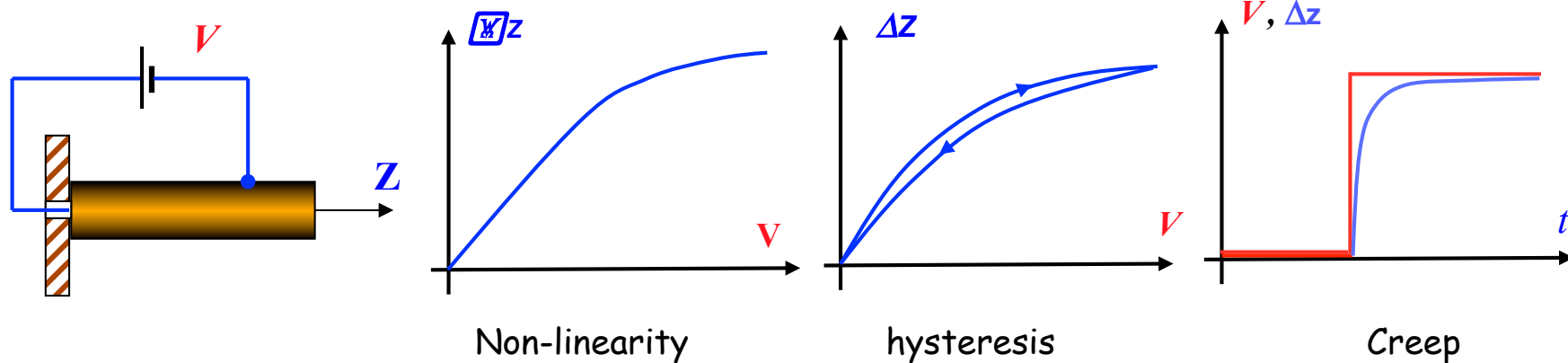
Scanner non-linearity introduces image distortions:
to improve linearity secondary position detectors (optical, capacitive…) may be used,
or a correction through non-linear transfer function (LUT =Look-Up-Table) may be
calculated through scanner calibration, measuring the applied voltages (V_x,V_y,V_z)
corresponding to known values of displacements (x,y,z) on a standard calibration grating.

X-Y-Z cross coupling

In tubular scanners the sample makes a rotation during x-y scan due to *cross-coupling* among horizontal and vertical displacements.

As a result the tip-sample contact moves on a spherical surface (whose curvature radius is the scanner length) introducing in to the image a concave distortion,

scan movement

tubular scanner

flat sample

concave image
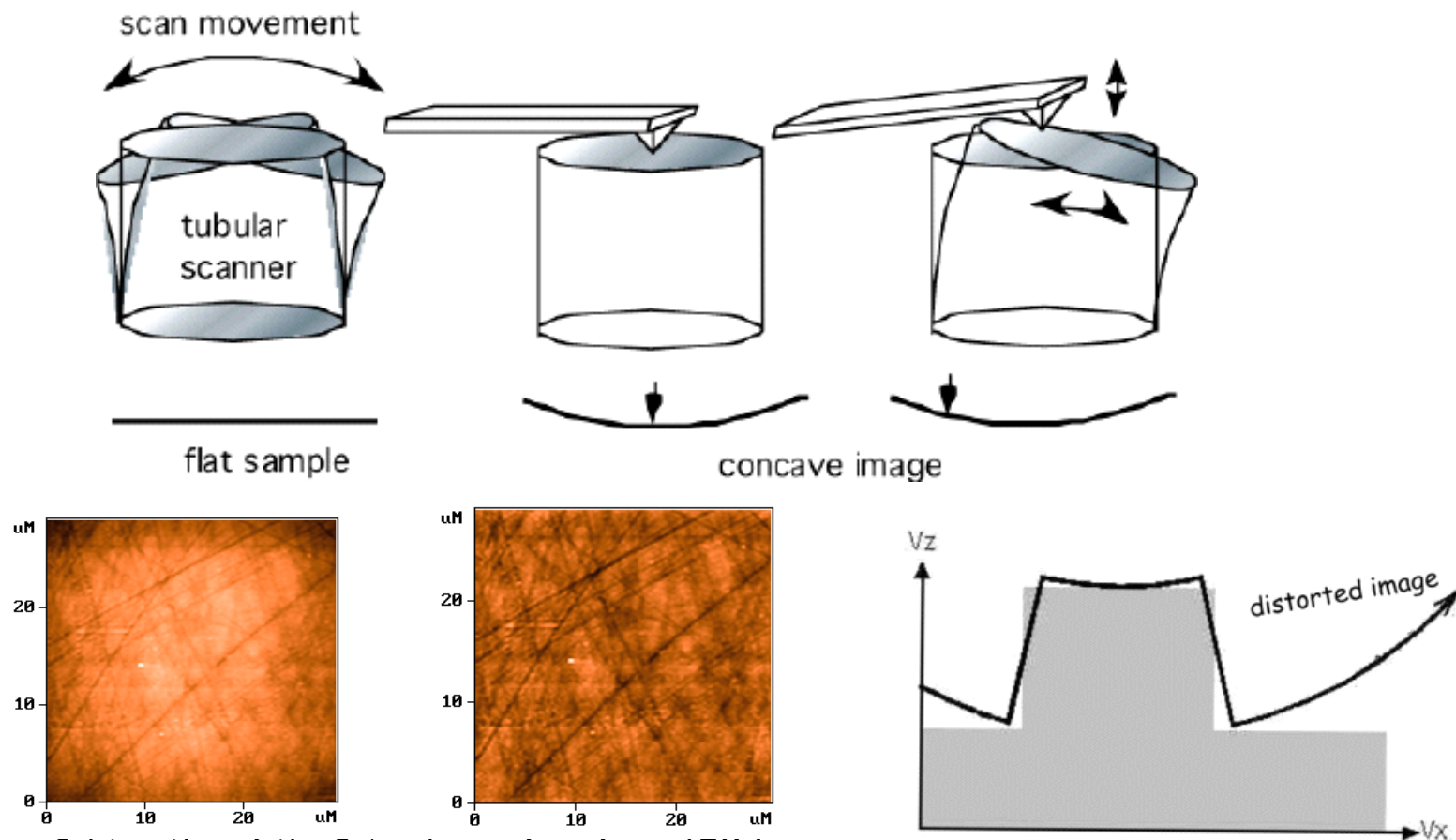
Subtraction of the 2nd order surface from AFM image

87

Scanner aging

The piezoceramics is a polycrystalline material obtained by powder sintering from crystal ferroelectrics.

The ceramic is heated above its Curie temperature $T_c(>200°C)$, and then it is slowly cooled in a strong electric field (about 3 kV/cm).

After cooling below T_c, piezoceramic retains the induced polarization and gets the ability to change its sizes under an applied electric field).

At room temperature the number of dipoles loosing alignment increases in time due to thermal energy, (scanner aging): the scanner must be periodically polarized.
Using the scanner help keeping the polarization
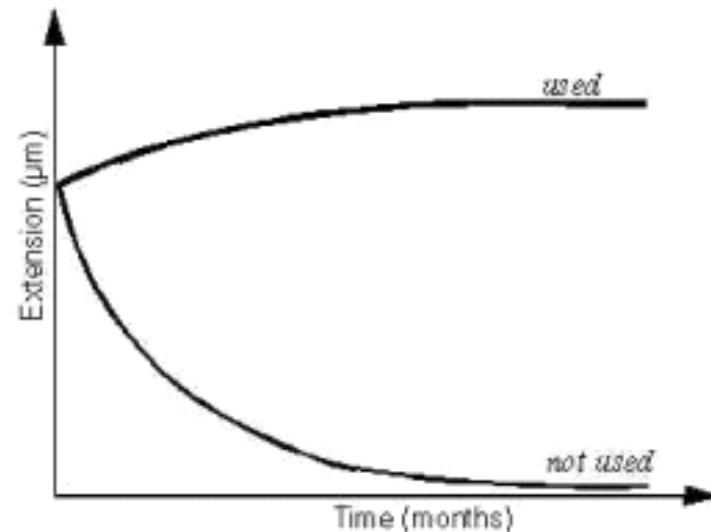
Creep

The creep is due to the *finite response time of the piezo-transducer*.

A step voltage applied to a piezo produces a deformation in a finite time interval.

Most of the deformation is achieved in a short time (a few µs), the full deformation is achieved after a long time (from 10 to 100 seconds).

Typical creep values : 1% -20%.

Creep introduces not negligible image artifacts when the sample topography has large and steep relief or when the investigated sample area is suddenly changed.
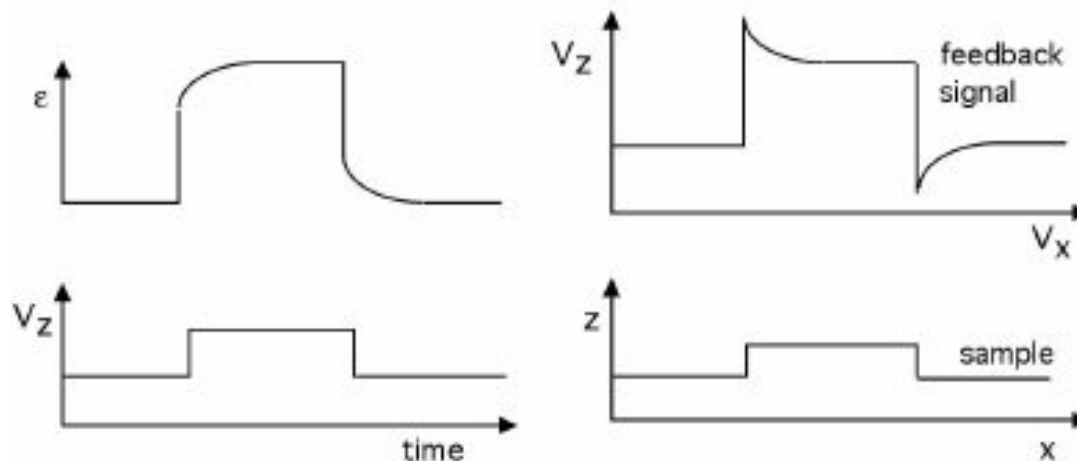
Tip replicas

False images may be obtained using blunt tips.

A test for such effect may be made by rotating the sample: microscopic features should rotate correspondly.

If not they are inverted replicas of a blunted tip.

tip imaging
90 degree rotation of sample

true imaging
90 degree rotation of sample
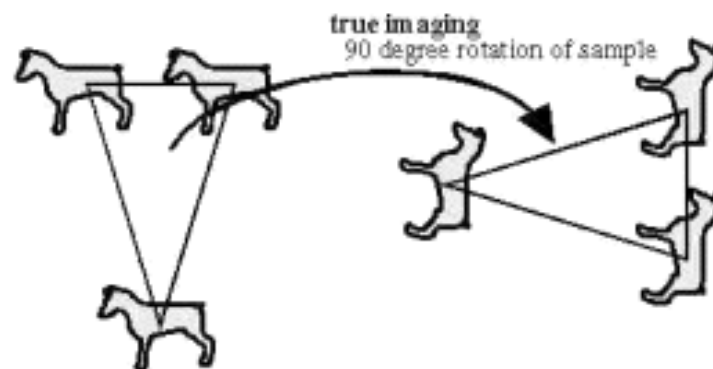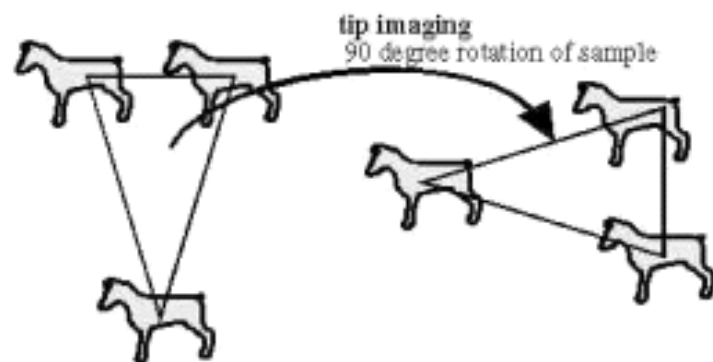
Lateral resolution

The force measured by the cantilever is the sum of the interactions among all the atoms of the tip apex and of the sample in front of it .
Therefore the image is a convolution of the tip shape and of the sample topography.
The sharper is the tip (and the shorter is the tip-sample distance), the smaller is the number of interacting atoms , and the more accurate is the resulting image.

The best commercial tips (conical) give a lateral resolution of about 10-20 Å, taken as Rayleigh criterion for adjacent peaks separation (19% valley between peaks)
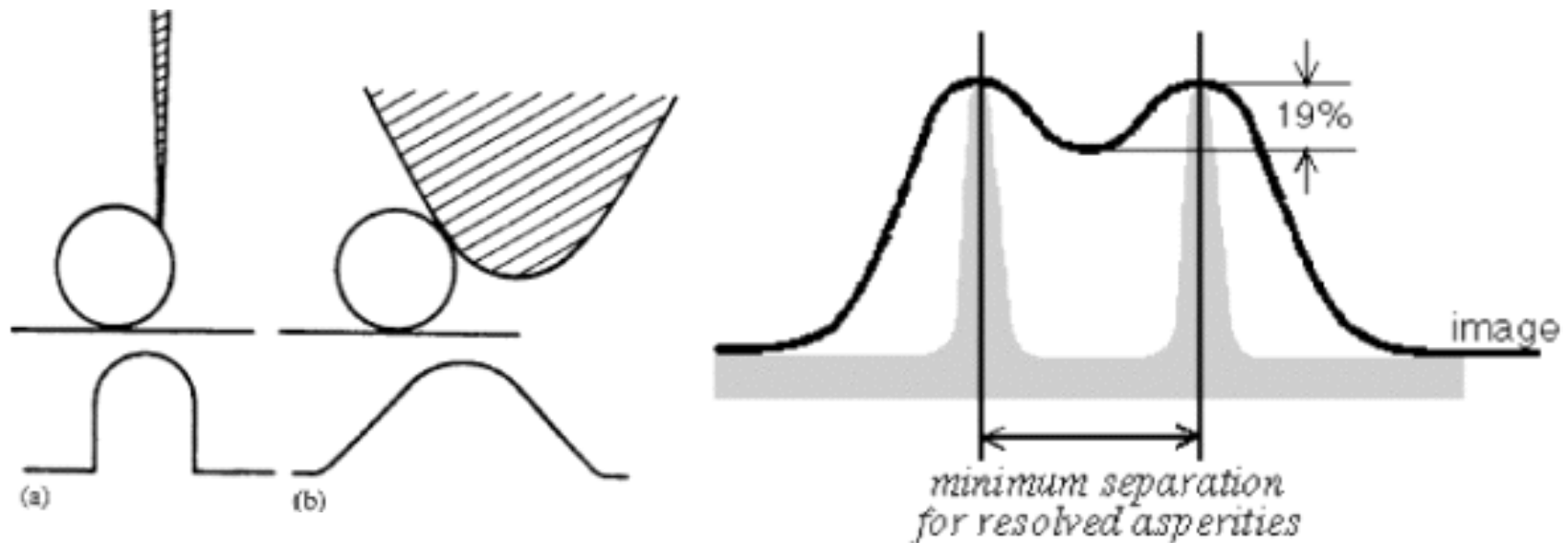
Only in STM this limit is exceeded because only the closest atom is responsible of the current tunneling.
As a comparison: in SEM the maximum resolution is about 100 nm.

Lateral resolution (2)

a Scan direction
Tip
R
Δz
Sample spikes
$\leftarrow d \rightarrow$

b Scan direction
Tip
R
Inverted tip surfaces
Δh
Δz
$\leftarrow d \rightarrow$
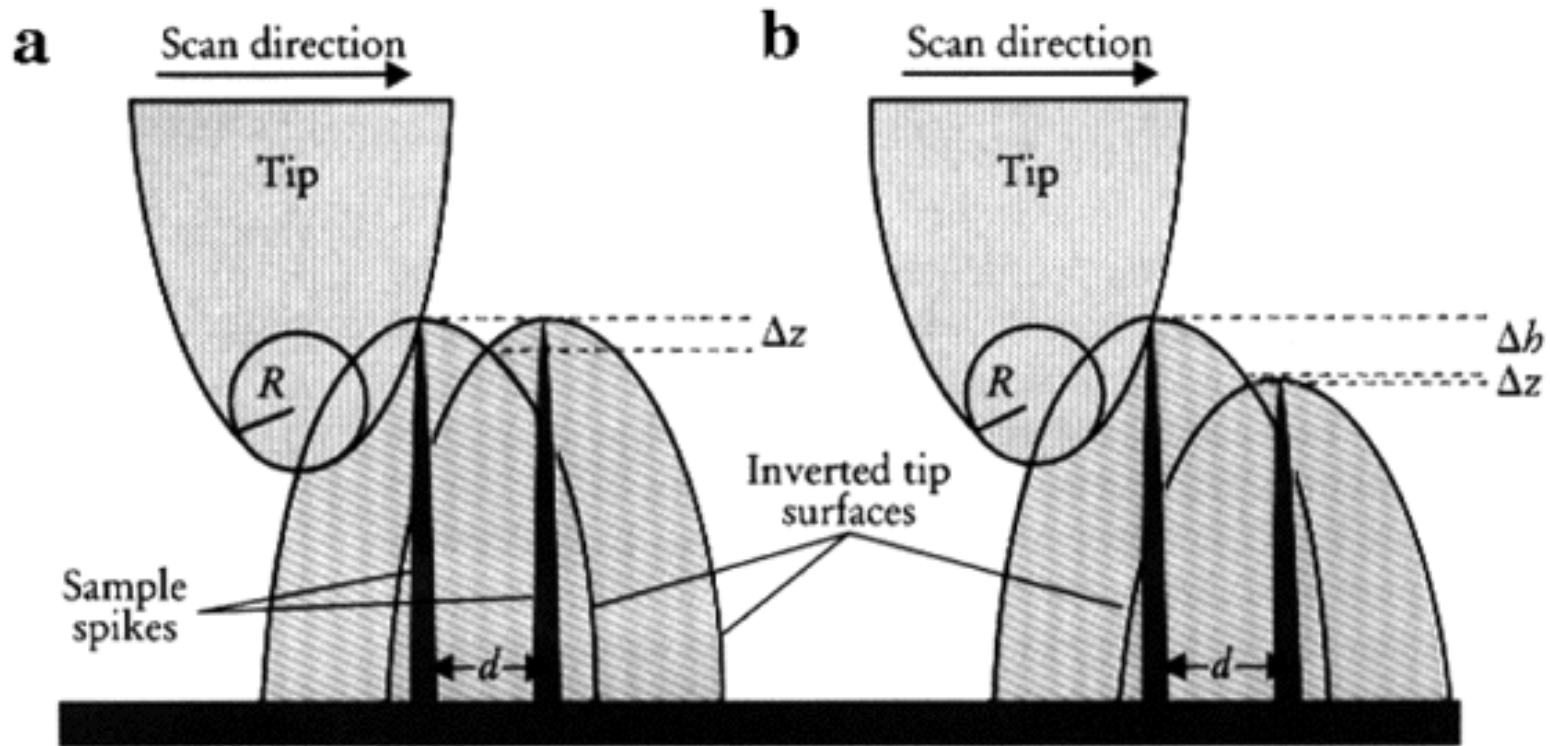
Minimum resolved distance between two spikes as a function of curvature radius R, peak-valley difference Δz and spike-height difference Δh

$$d = \sqrt{2R} \cdot \left(\sqrt{\Delta z} + \sqrt{\Delta z + \Delta h} \right)$$

Tip convolution effects

The relevance of tip convolution effect increases with the aspect ratio of the sample reliefs and decreases with the aspect ratio of the tip.

a/b=aspect ratio
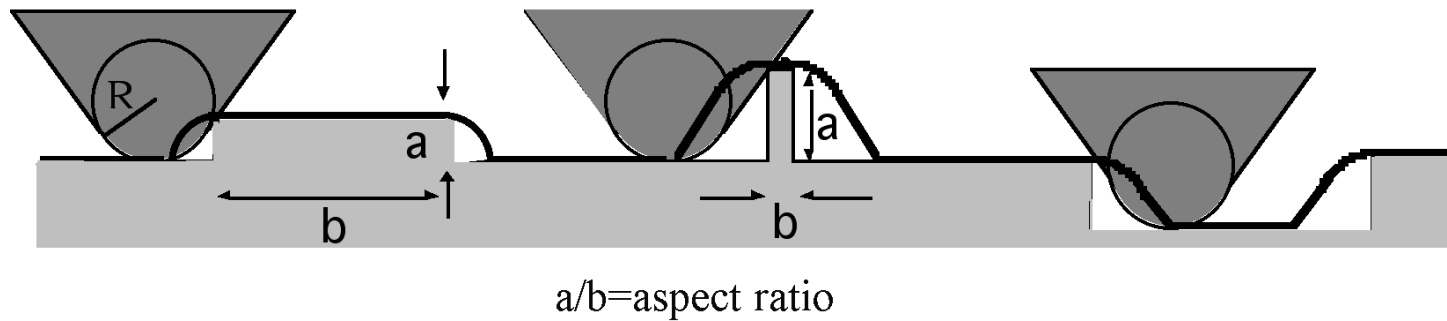
The size of the recorded image of a relief is in excess.

If the sample surface has holes or cracks, the size of the deformed image is in defect.

Atomic resolution

The typical SFM lateral resolution of 10 - 20 Å seems to contradict the suggestion given by images with atomic resolution published by SFM producers. We must distinguish between real atomic resolution and images at atomic scale of periodical structures
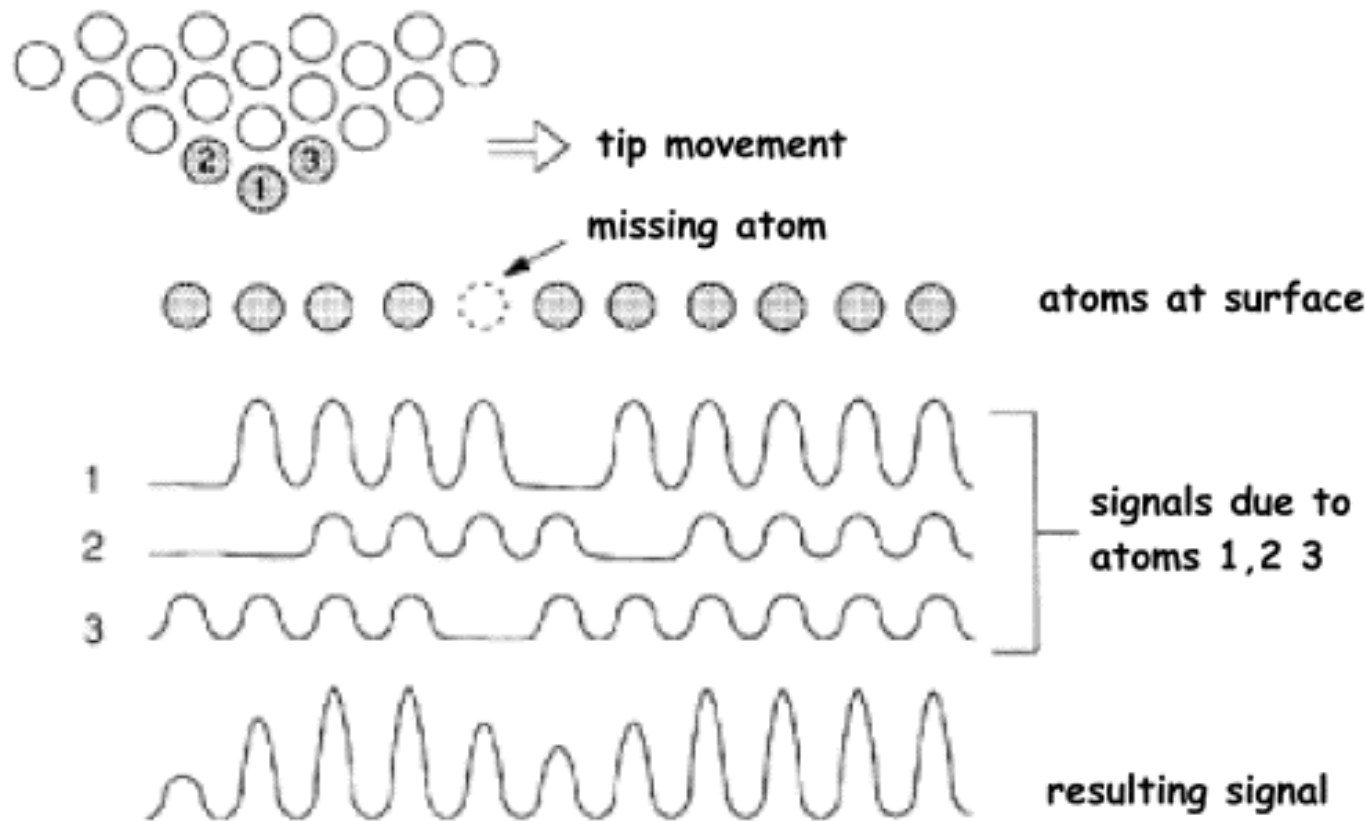
AFM may well reproduce the periodicity of a lattice even without real atomic resolution.

Atomic resolution (2)

Examples of AFM images with atomic resolution

(a) STM and
(b) NC-AFM images
of SrTiO$_3$(100) surface
(c) A proposed model
of the SrTiO$_3$(100)-($\sqrt{5}\times\sqrt{5}$)R26.6°
surface reconstruction.

[010]   ● Sr atom
[001]   ◎ Ti atom   ○ O atom
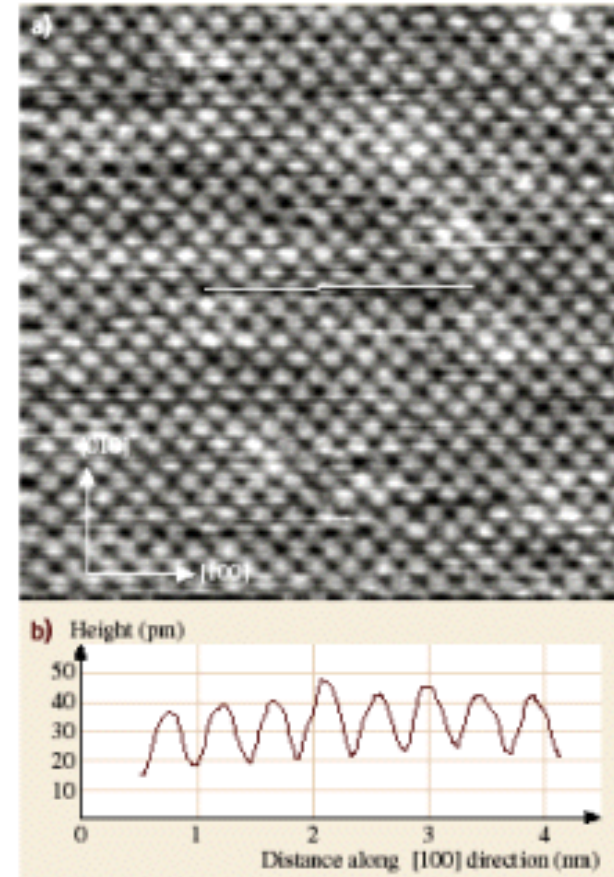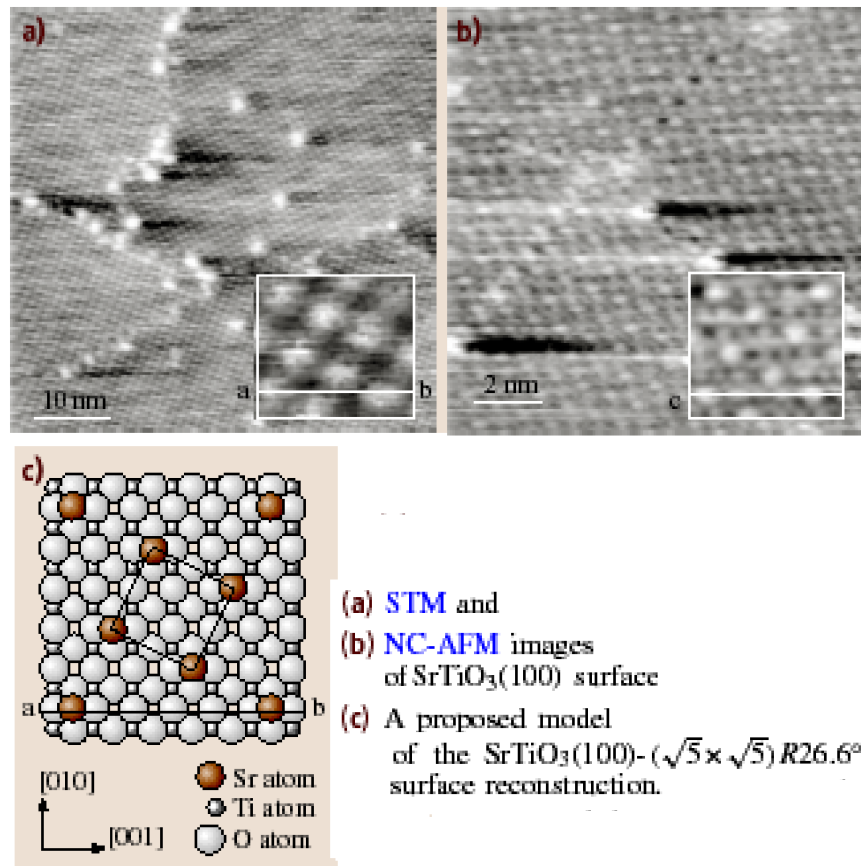
an atomically resolved image of a NiO(001)
surface with a ferromagnetic Fe-coated tip

Atomic resolution (3)
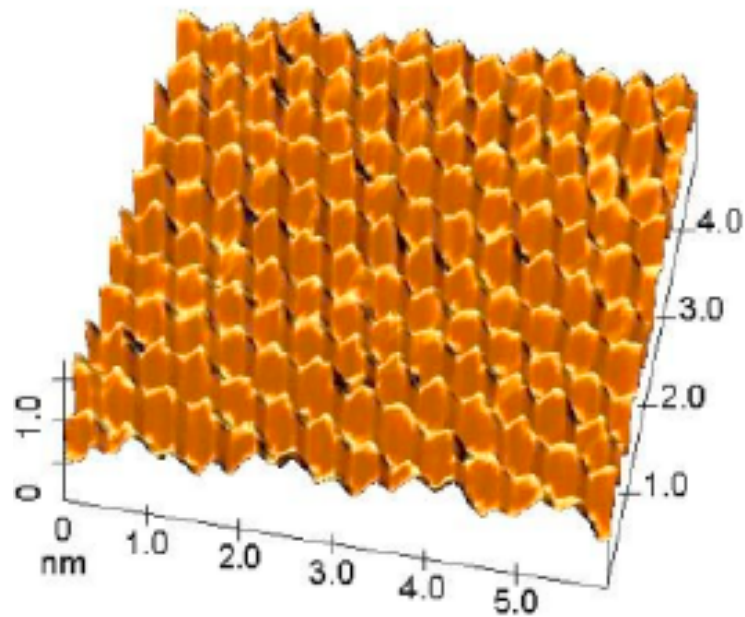
AFM High-Resolution Contact Mode Image
of Mica
from A.Belyayev, State Research Institute of
Physical Problems & NT-MDT, Moscow, Russia.
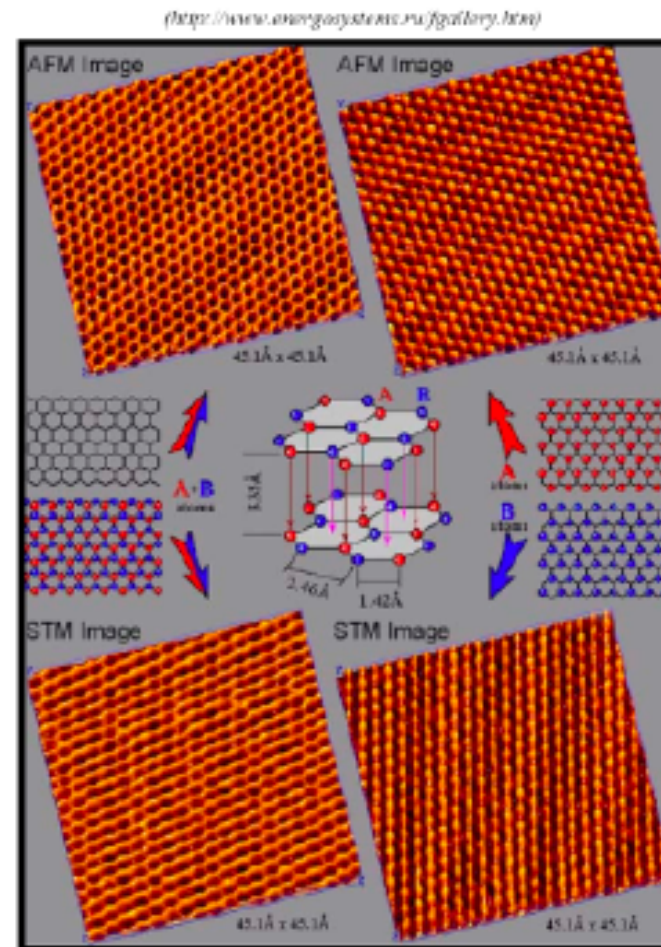
High-Resolution Images of HOPG

Resolution of mapping

Besides the lateral resolution (defined by the tip radius) and the vertical resolution (defined by signal/noise ratio and feedback gain), also the mapping resolution must be accounted for.
This is defined by the distance between the closest recorded points.
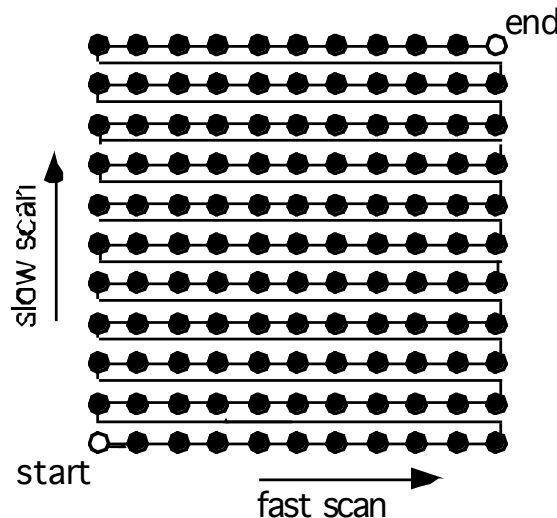
A typical SPM image with (512 ☒ 512) pixel covering an area (1μm x 1μm) should allows a max theoretical resolution of 20Å (1μm /512).
The best commercial tips have curvature radius of 50Å, giving a lateral resolution of 10 - 20Å.

However images covering larger areas have a resolution that is limited by the number of pixel.
(e.g. 25μm x 25μm image taken with 256 x 256 pixels has a resolution of about 100 nm)

Larger matrix requires more computer memory and longer acquisition time
(to give an order of magnitude: with 256x256 pixel, 1 line/s, one image requires a few minutes).

Image Post-processing
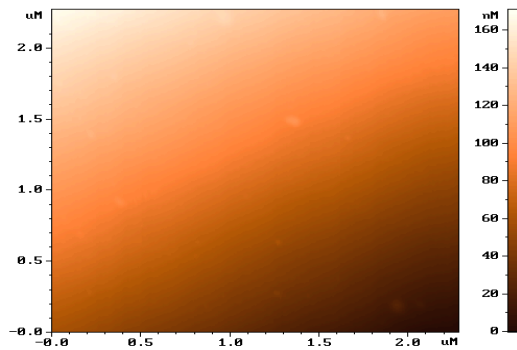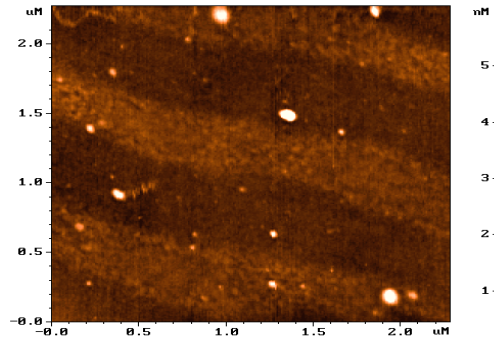
Tilt correction
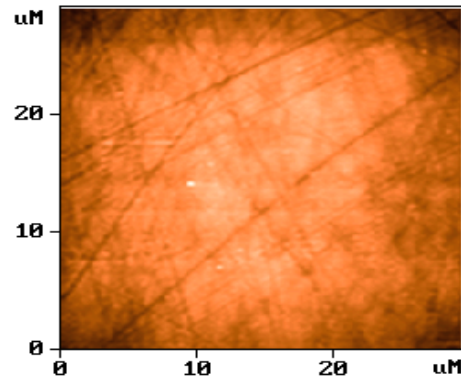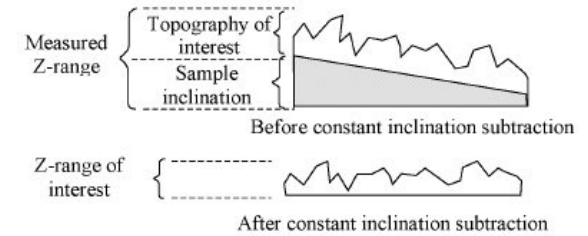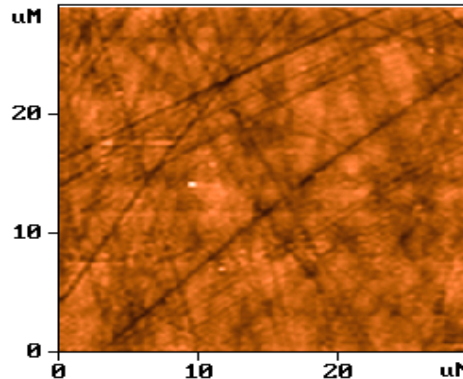
Second-order surface subtraction
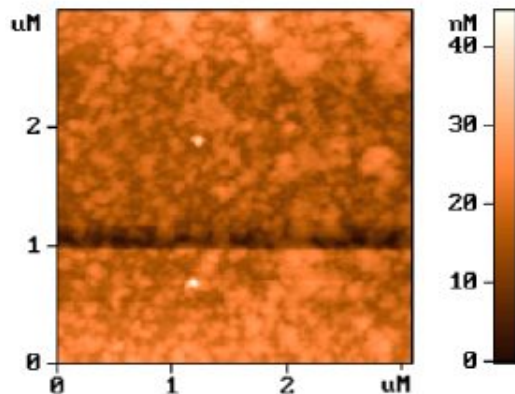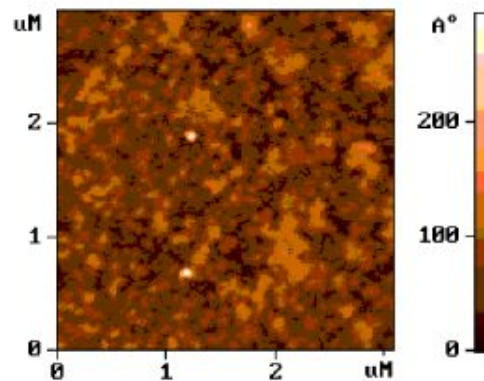
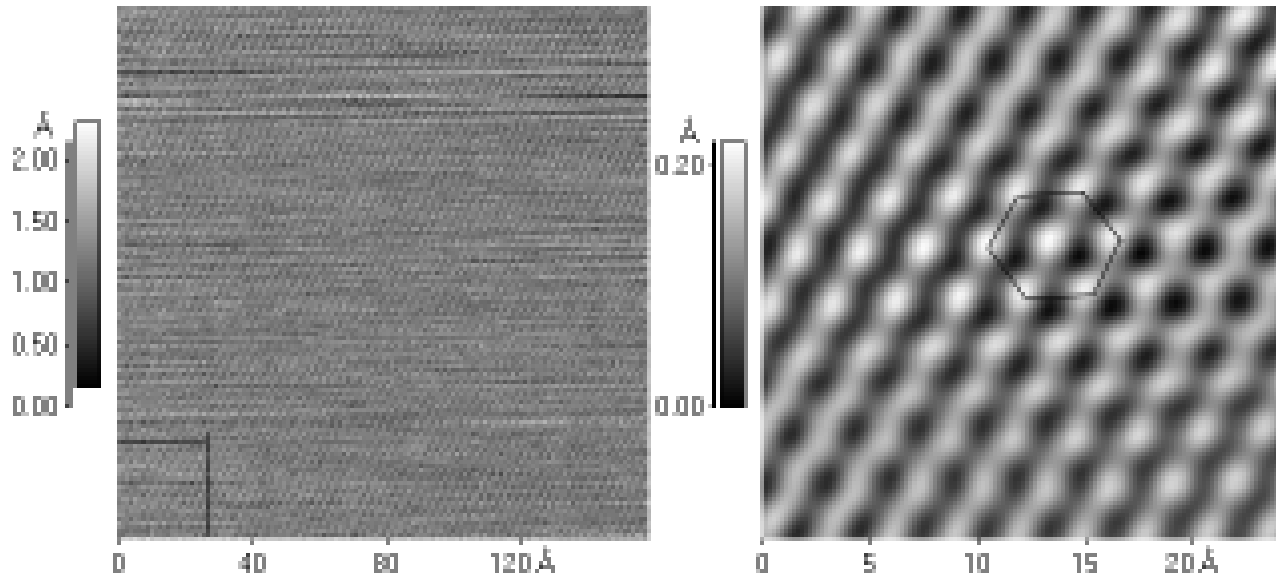Line averaging

Spatial filtering: Discrete Fourier Transform
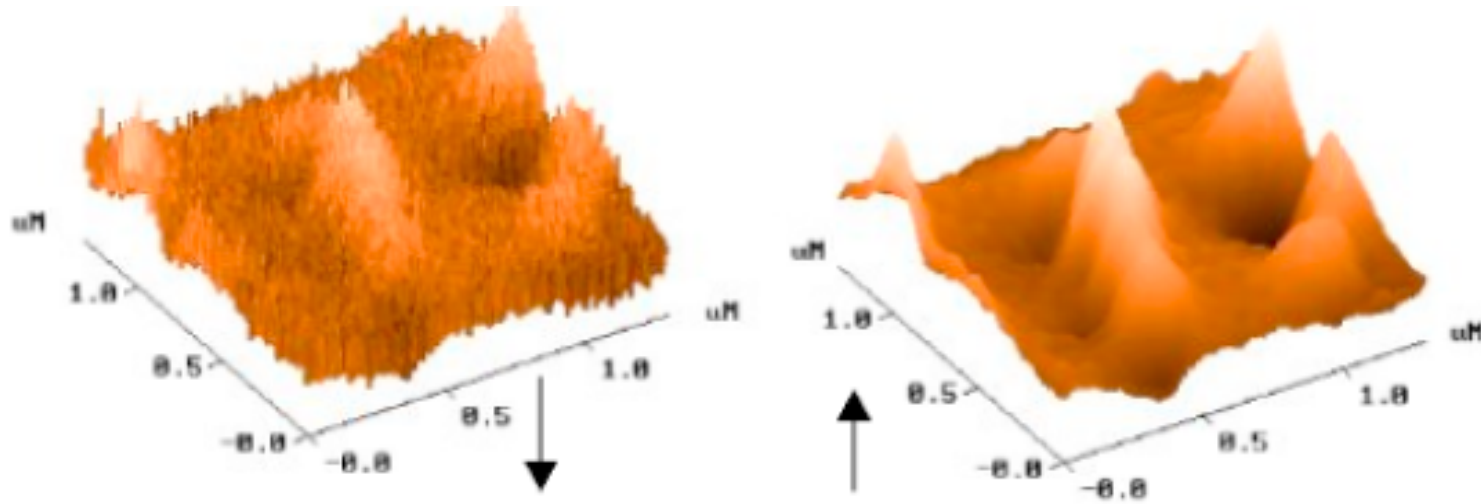
$$F_{\alpha\beta} = \frac{1}{N^2} \sum_{ij} Z_{ij} \exp\left[2\pi v(\frac{\alpha \cdot i}{N} + \frac{\beta \cdot j}{N}) \right]$$

$$Z_{ij} = \sum_{\alpha\beta} F_{\alpha\beta} \exp\left[-2\pi v(\frac{\alpha \cdot i}{N} + \frac{\beta \cdot j}{N}) \right]$$
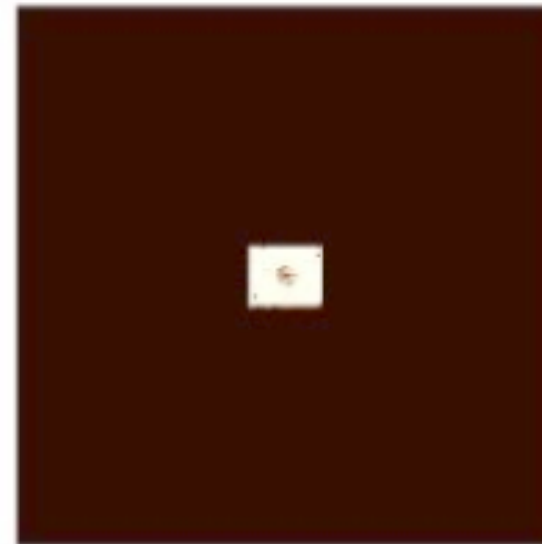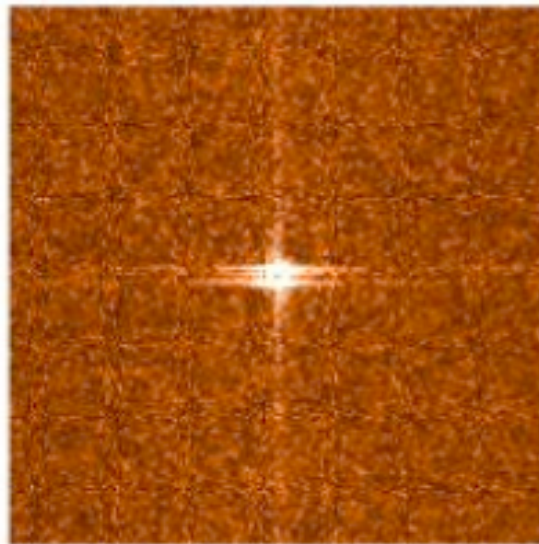
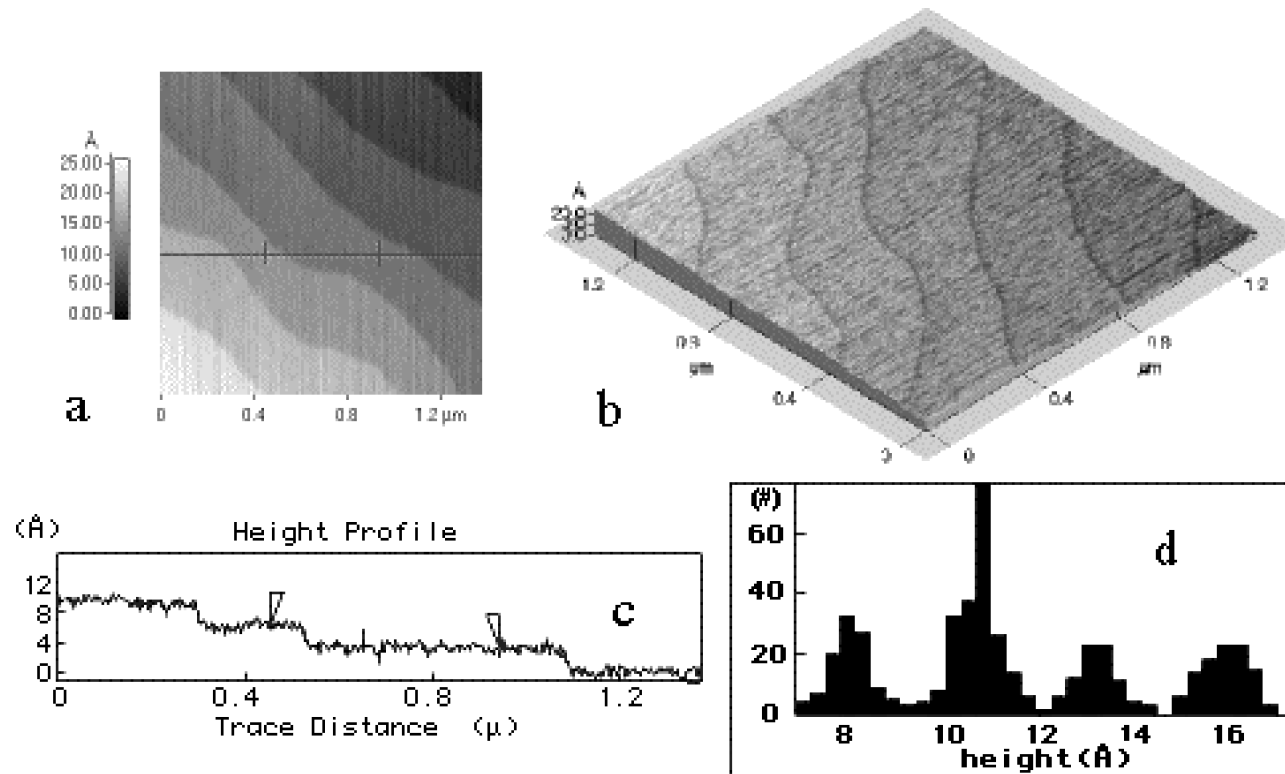$$F'_{\alpha\beta} = F_{\alpha\beta} \cdot H_{\alpha\beta}$$

DFT Filtering example

HOPG (Highly Oriented Pyrolitic Graphite)
AFM original image (left) and filtered (right)

The DFT filtered image is taken from the square region marked in the lower-left corner.
(Image taken in contact mode with Park CP - INFM, Padova)

Atomic layers

GalliumArsenide <001>. a) "top view", b) 3D view, c) line profile, d) histogram of height.

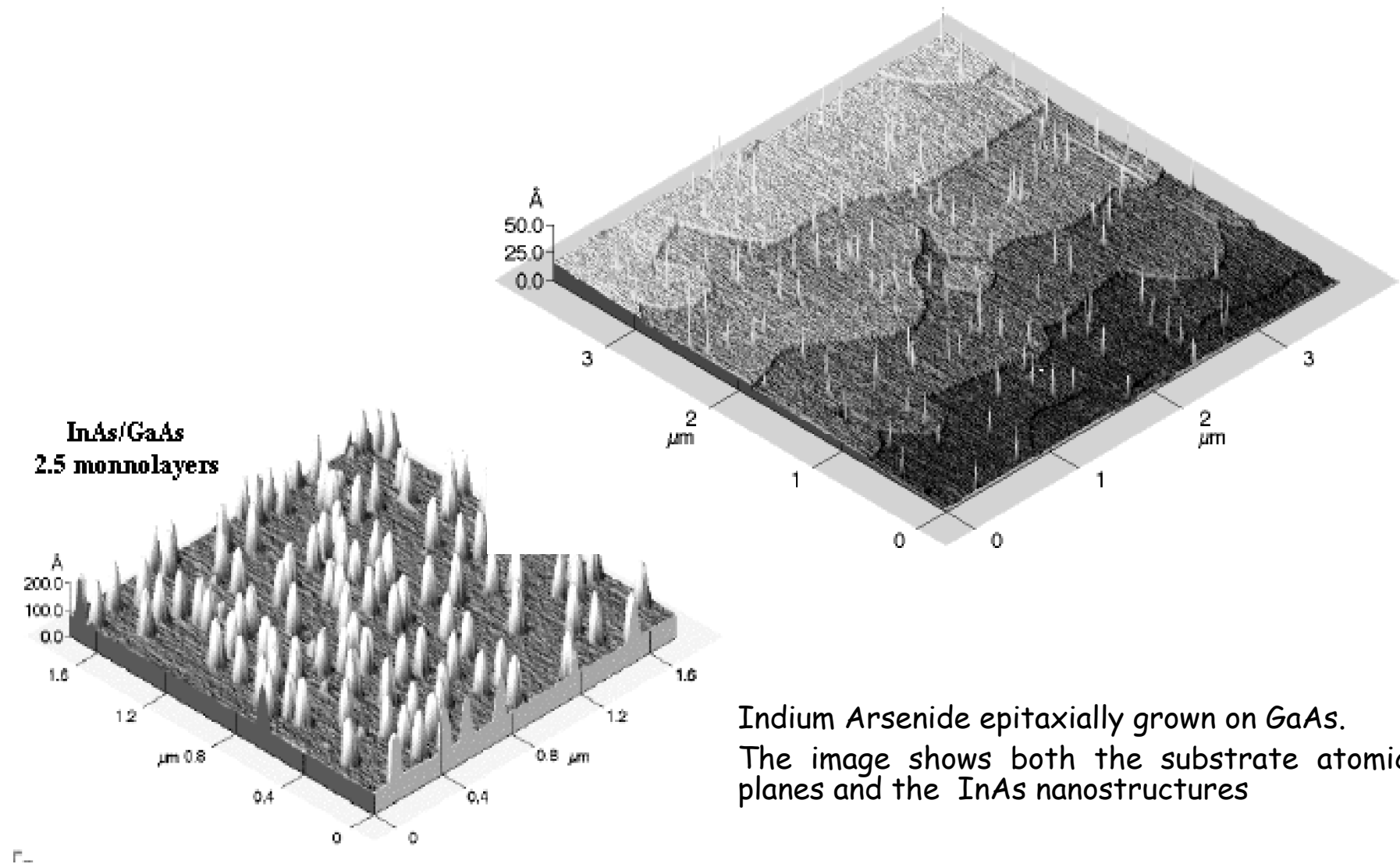The steps (⊠z≈0.29 nm) between planes are half lattice spacing

(Image taken in contact mode with Park CP - INFM, Padova)

Sub monolayer film
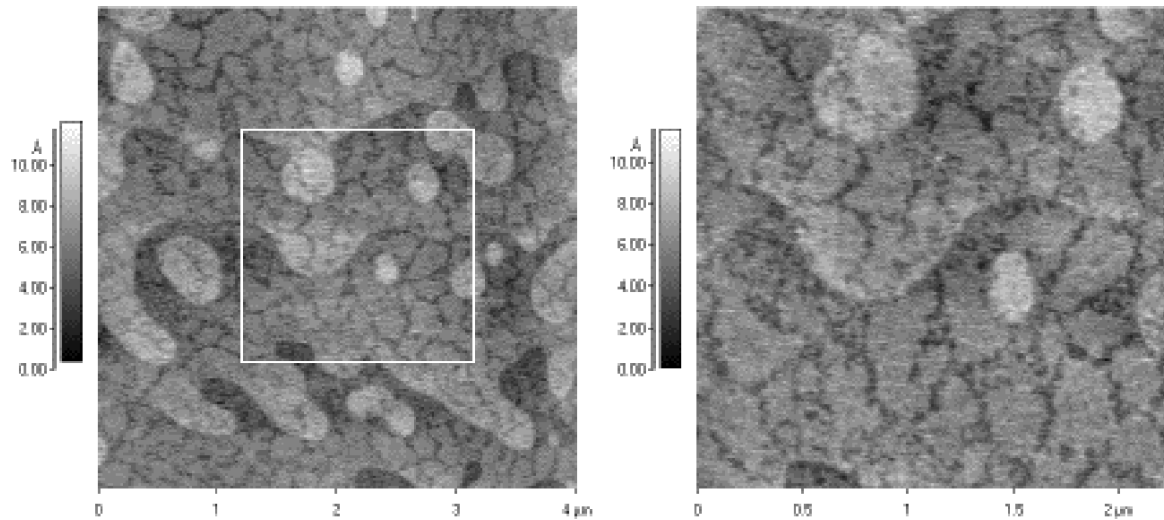
1.7 Monolayer of InP on GaAs. The film, grown layer-by-layer does not complete the first atomic layer before starting the next
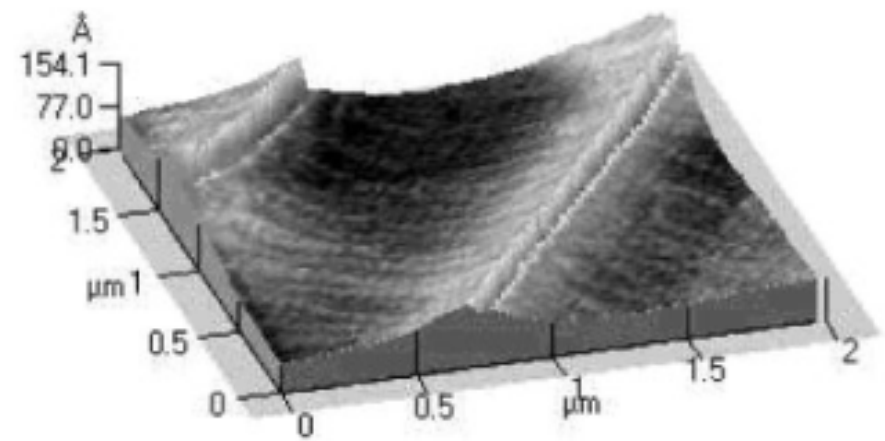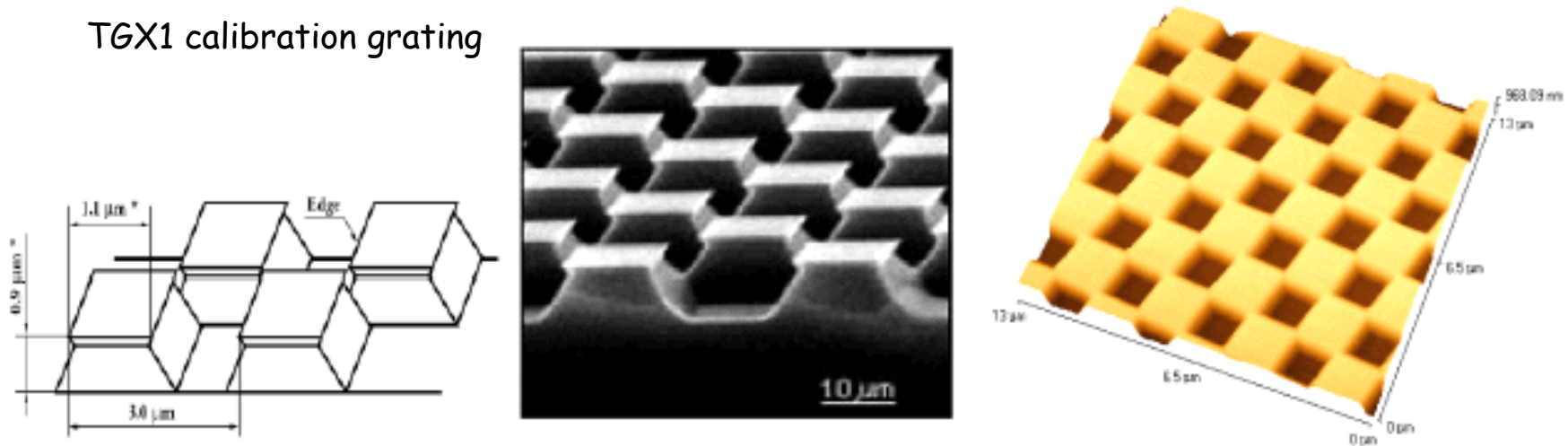
Nanostructures

InAs/GaAs
2.5 monnolayers

Indium Arsenide epitaxially grown on GaAs.

The image shows both the substrate atomic planes and the InAs nanostructures

Cracks in InGaAs/InP films
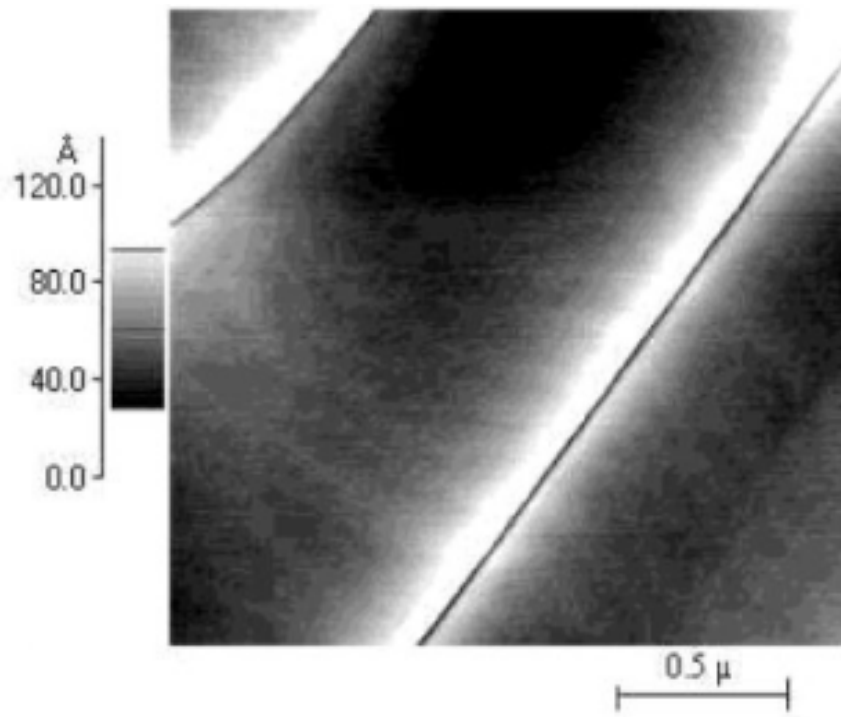
Top view
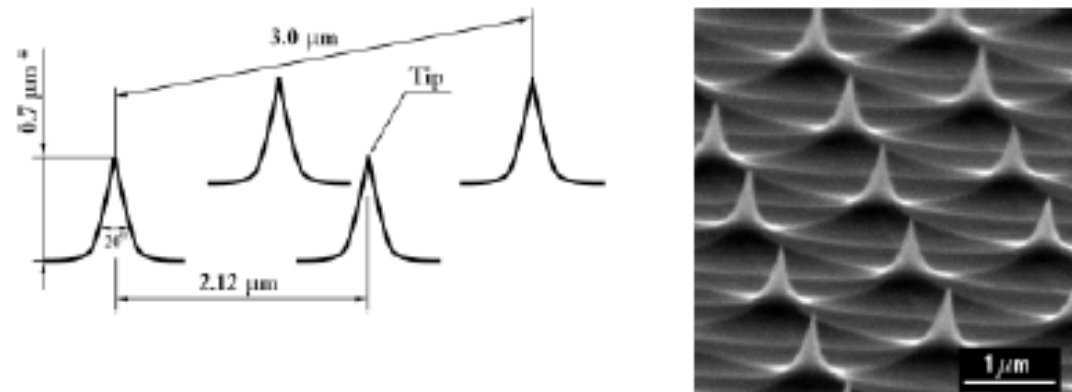

3D view

(note the different scale for x,y and z axes)
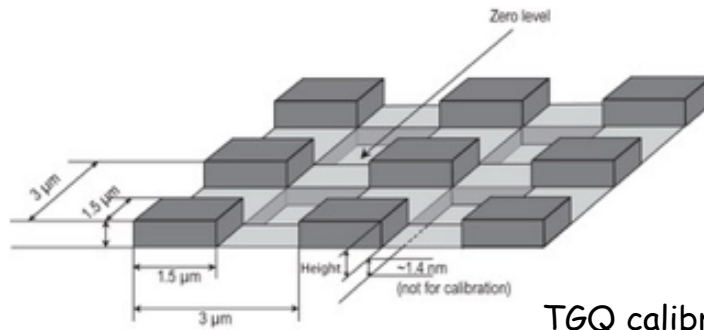
Calibration grating

TGX1 calibration grating

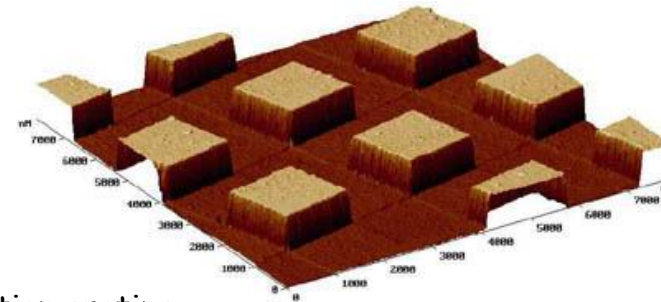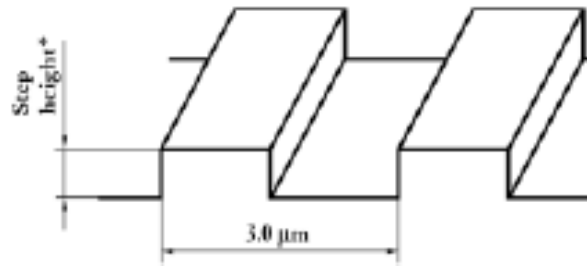SEM image (left) and SFM image acquired using NanoEducator (right).

TGT calibration grating

Calibration grating 2

Zero level

3 μm

1.5 μm
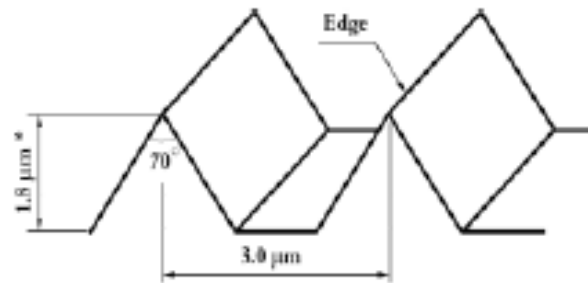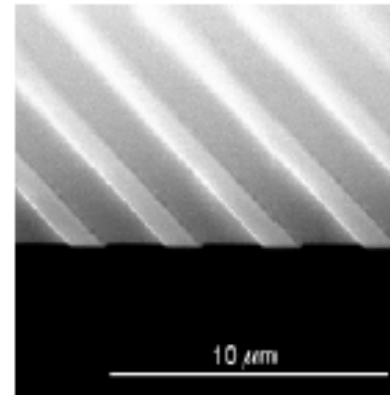
1.5 μm

Height ~1.4 nm
(not for calibration)

3 μm

TGQ calibration grating

TGQ1

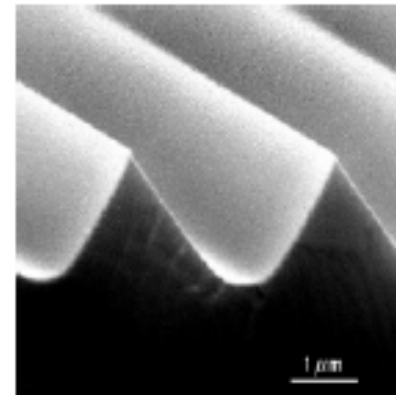Step height⁺

3.0 μm

TGZ calibration grating

10 μm

Edge

1.8 μm ⁺

70°

3.0 μm

TGG calibration grating

1 μm

Calibration grating producers

www.vlsistandards.com   VLSI Standards
www.vlsistandards.com/products/dimensional

www.2spi.com   StructureProbesInc -SPI
www.2spi.com/catalog/mounts/holographic-diffraction-grating-calibration-SEM-AFM.shtml

www.pacificnanotech.com   Pacific nanotechnology

www.ntt-at.com   NTT Advanced technology
www.ntt-at.com/products_e/nanopattern/index.html

www.spmtips.com   Micromash
http://www.spmtips.com/test_structures

www.emsdiasum.com ElectronMicroscopy Standards
www.emsdiasum.com/microscopy/products/standards/xray.aspx

[WWW.NT-mdt.com](WWW.NT-mdt.com)   NT-MDT
www.ntmdt-tips.com