AFM @ NIS: what can it be used for?

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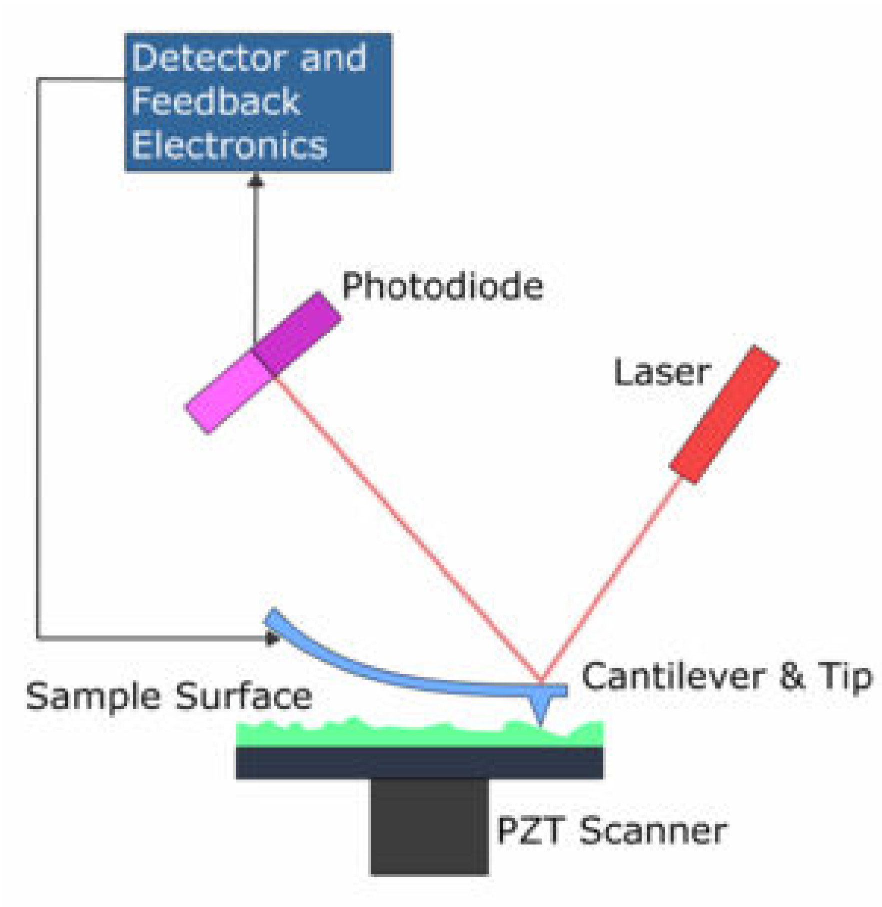
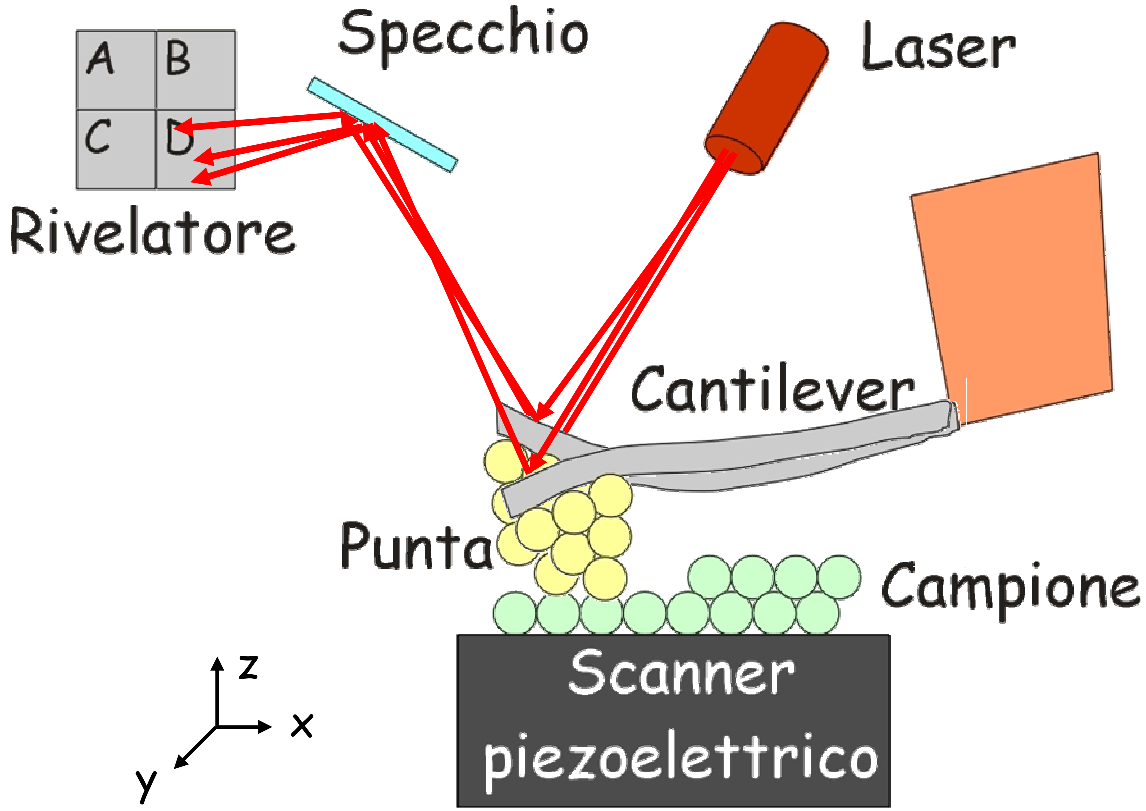
AFM: block scheme



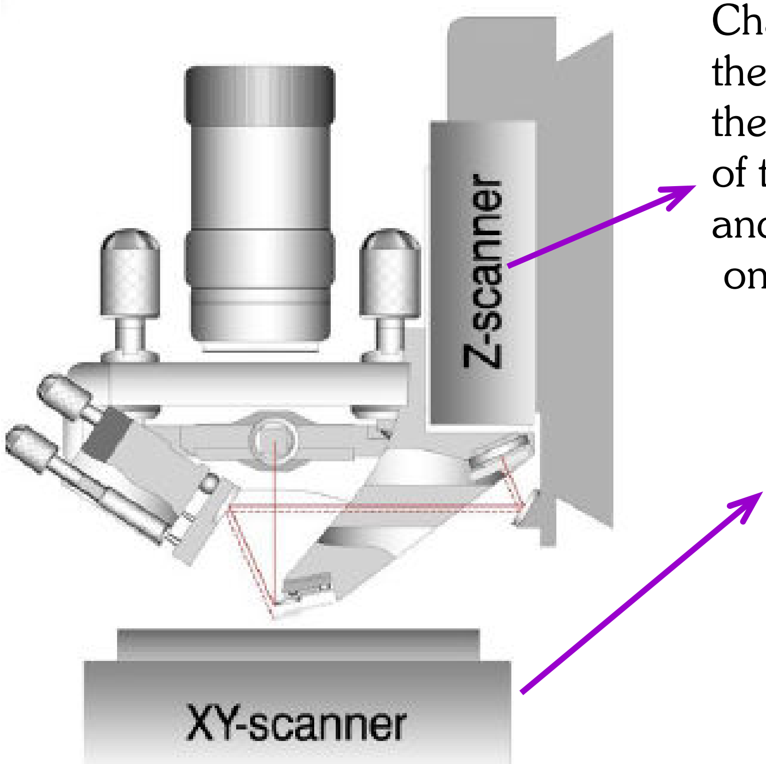
In an **Atomic Force Microscope (AFM)**
a micrometric tip
attached to a cantilever
with a low spring constant
(comparable to that
of a group of atoms in a solid)
is brought near the sample surface
at a distance ranging from few
to a hundred of Å).
The tip is subjected
to attractive or repulsive forces
depending on tip-sample distance.
The PSPD detector collects
the upward and downward
movements of the tip.

AFM operating principle



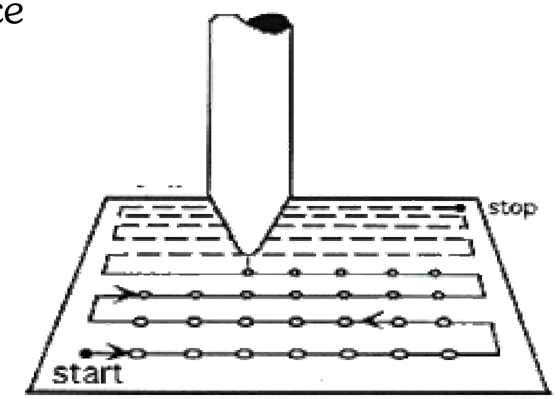
Basic operation modes: features

XE-100 is equipped with two separate scanners: XY and Z



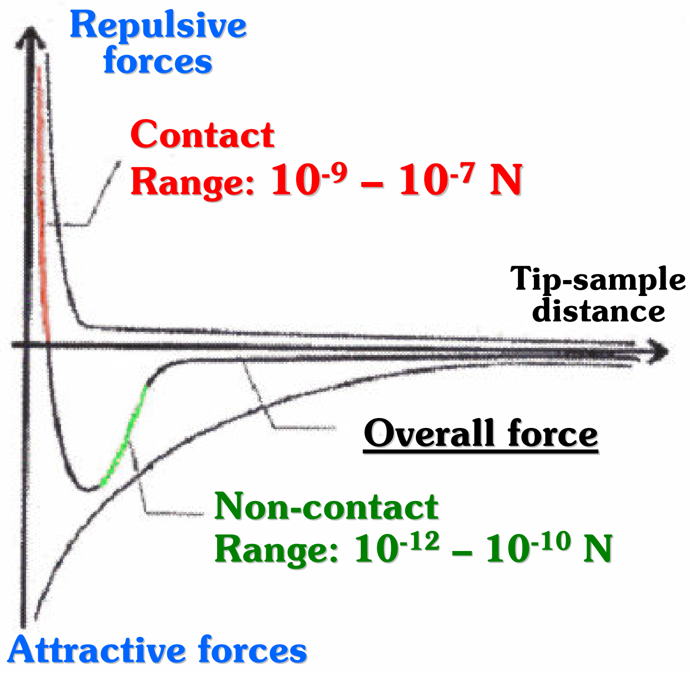
Changes the **position** of the **tip** according to the measured **tip/surface interaction force**; the PSPD detector registers the deflection of the cantilever and transforms this value in information on the topography (Z) of the examined sample.

Performes a **raster** onto the sample, allowing to obtain a topographic **map** of the surface



XE-100: basic operation modes

- Contact, Non contact and Intermittent

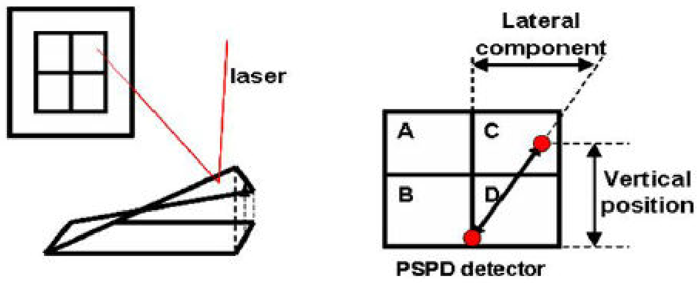


Contact: tip brought into contact with sample surface
Topographic image is "directly" determined from tip movement

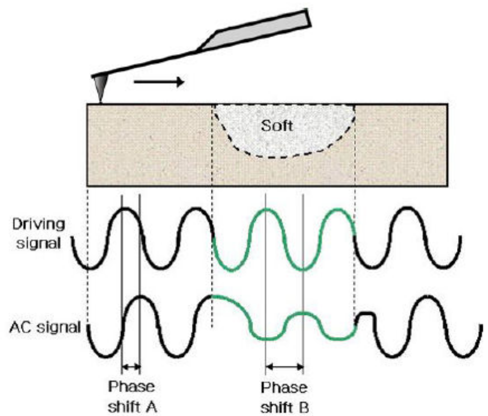
Non contact & intermittent: interaction forces are too small to work like in contact mode
Tip oscillates…
…NOT in contact with the surface (NC-AFM)
…or TAPPING the surface at each oscillation (intermittent)
Topographic image is determined from the changes in the oscillation amplitude of the tip

Information from basic operation modes

- Contact mode: topography and surface friction (from lateral force values)
  Hard,
  wear-resistant samples



- Non contact & intermittent contact modes: topography and phase imaging
  Soft samples

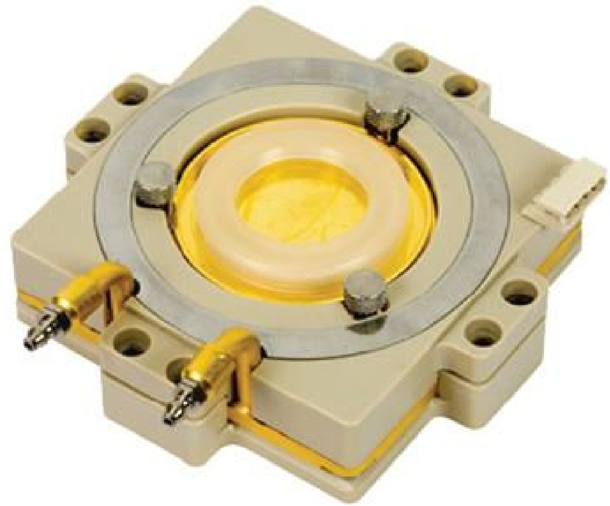


AFM imaging in liquid

- Universal liquid cell
- Working conditions: open and closed

- Open:

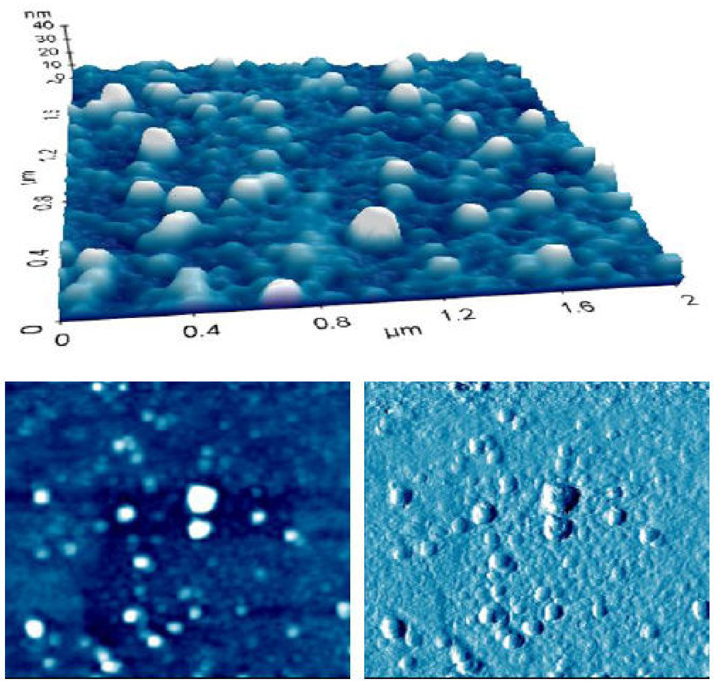
C–AFM and NC–AFM
No possibilities of liquid circulation



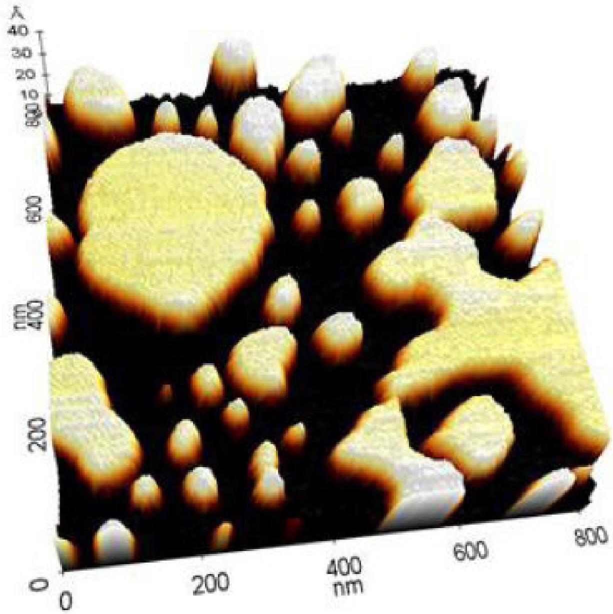
- Closed:

C–AFM only (NC–AFM cannot be performed due to tip mounting)
Liquid (and gas) circulation & heating
“Controlled” atmosphere conditions available

AFM imaging in liquid



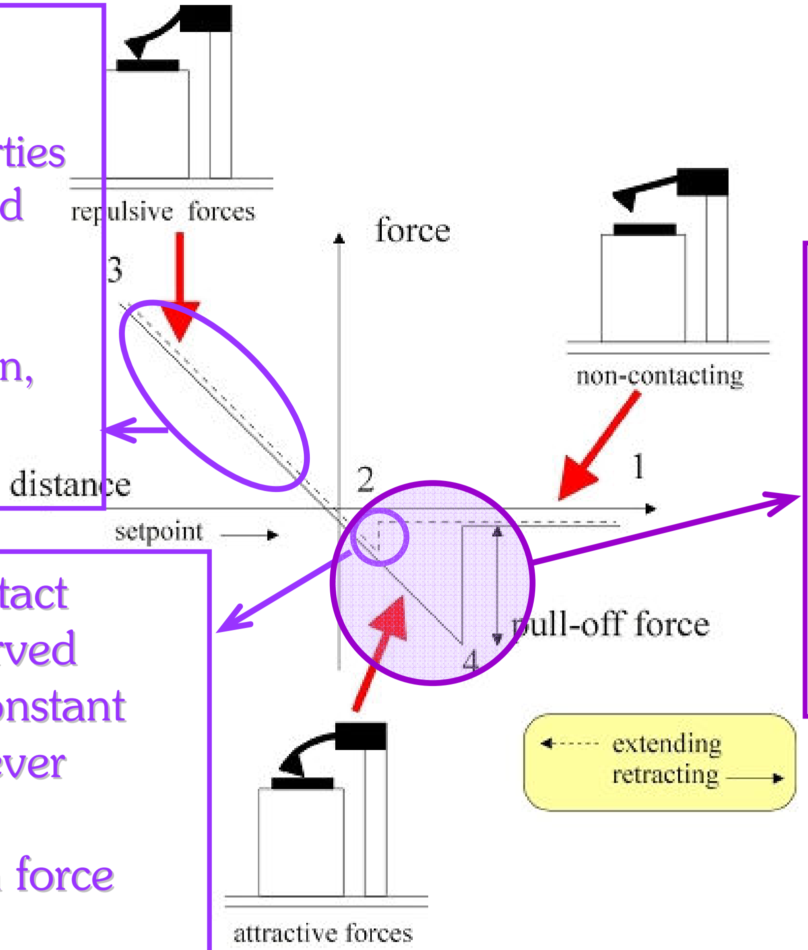
3D, topography and error images
of liposomes in liquid.
2 µm scan. NC-AFM.



Lipid vesicles and bilayer in liquid.
0.8 µm scan. NC-AFM.

Force/distance spectroscopy – 1

Slope is related
to hardness
and to elastic properties
of both sample and
cantilever
In case
of elastic interaction,
the two curves
are coincident

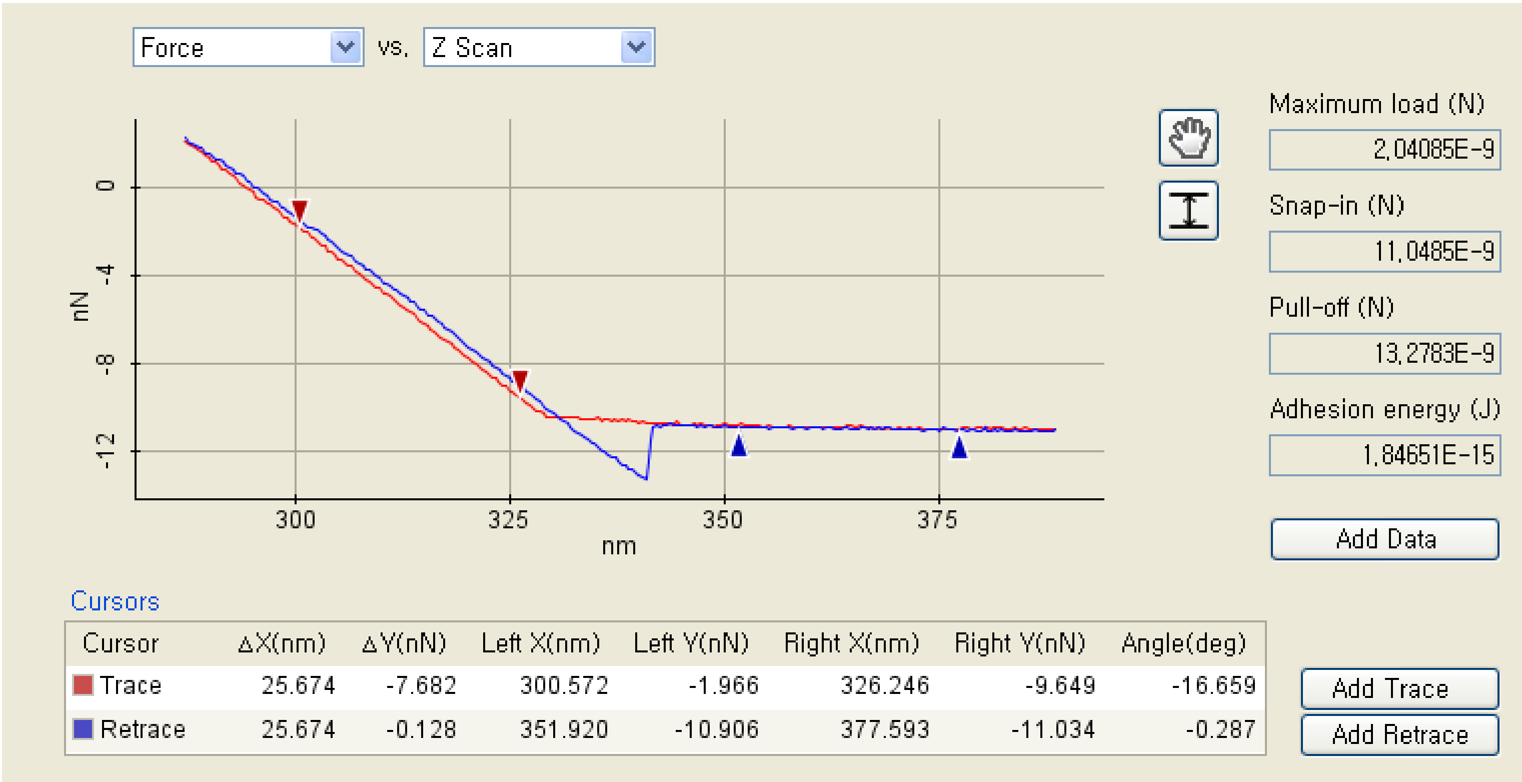


Jump-in contact
may be observed
only if spring constant
of the cantilever
is smaller
than maximum force
gradient

Hysteresis
can be related
to plasticity
or capillary effects
(presence
of a water adlayer
onto both sample
and surface)

Force/distance spectroscopy – 2

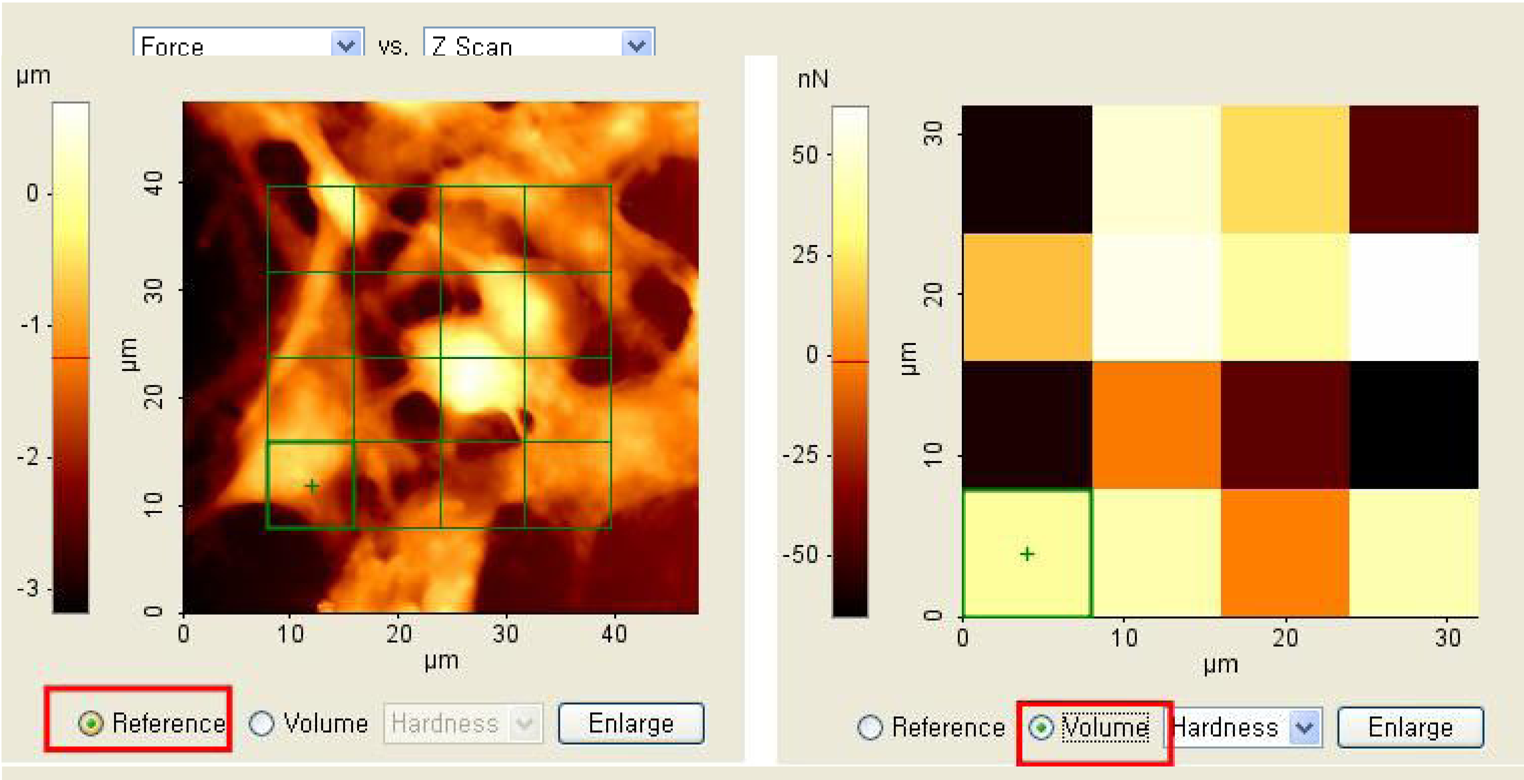
- Analysis options: random points



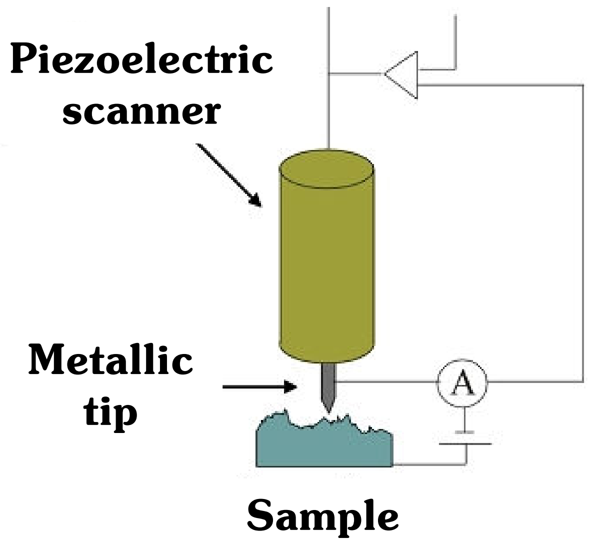
Cursor	ΔX(nm)	ΔY(nN)	Left X(nm)	Left Y(nN)	Right X(nm)	Right Y(nN)	Angle(deg)
Trace	25.674	-7.682	300.572	-1.966	326.246	-9.649	-16.659
Retrace	25.674	-0.128	351.920	-10.906	377.593	-11.034	-0.287

Force/distance spectroscopy – 3

- Analysis options: grid



Scanning tunneling microscopy (STM) – 1

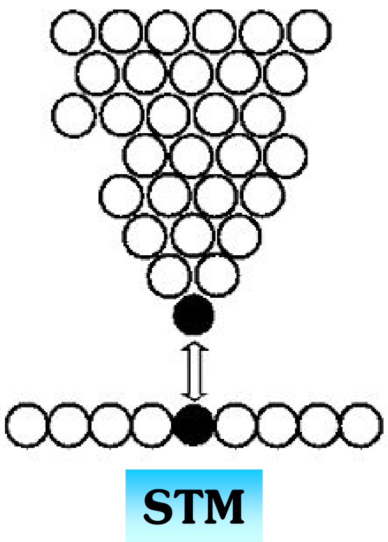


$\mathbf{I} = A \exp(-bd)$

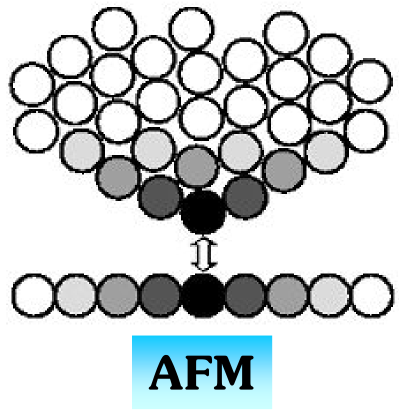
where d is the distance between tip and sample

- **Physical observable**: **tunneling current** between tip and sample (current resolution down to pA) (due to tip mounting, bias is applied to the sample with respect to the tip)
- The **piezoelectric scanner** moves up and down the tip in order to **keep constant** the tunneling **current**
- The **raster** of the **XY scanner** allows to **map** the surface (as in AFM)

Scanning tunneling microscopy (STM) - 2

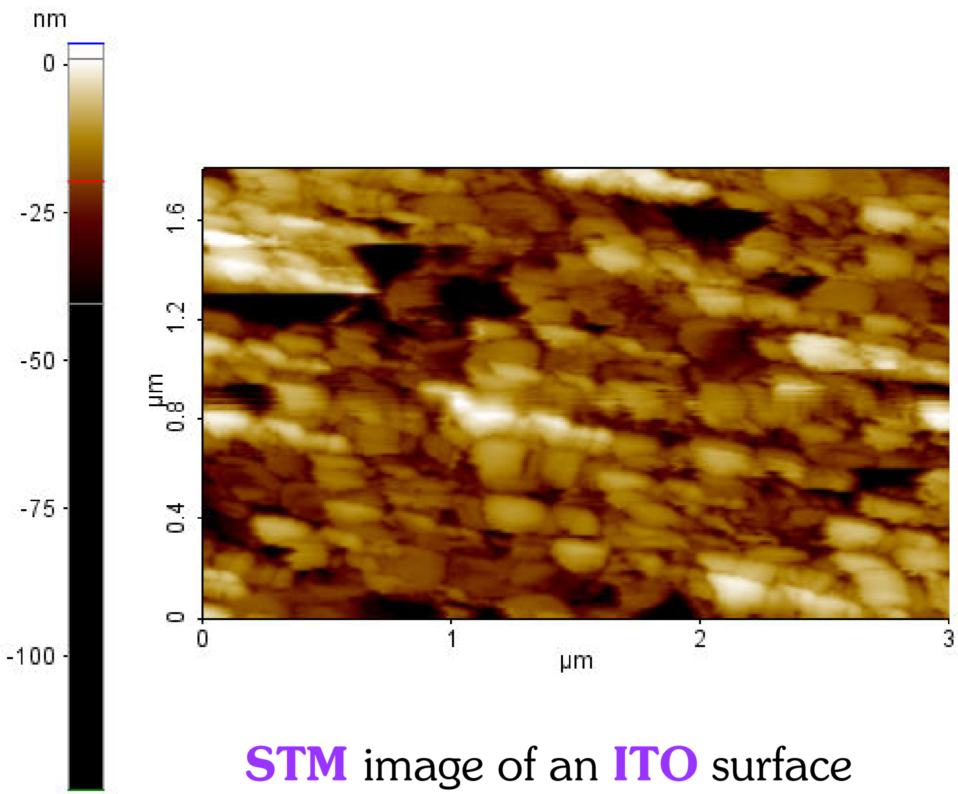


- **Better resolution** with respect to **AFM**



- **Imaging** of **conductive** or **semiconductive** samples **only**
  - Allows **I/V spectroscopy**

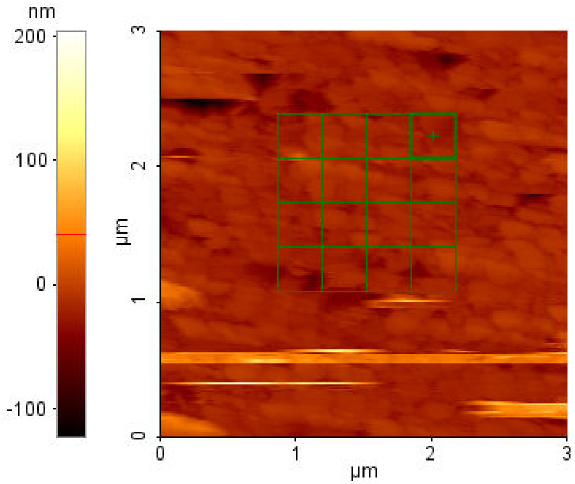
Scanning tunneling microscopy (STM) - 3

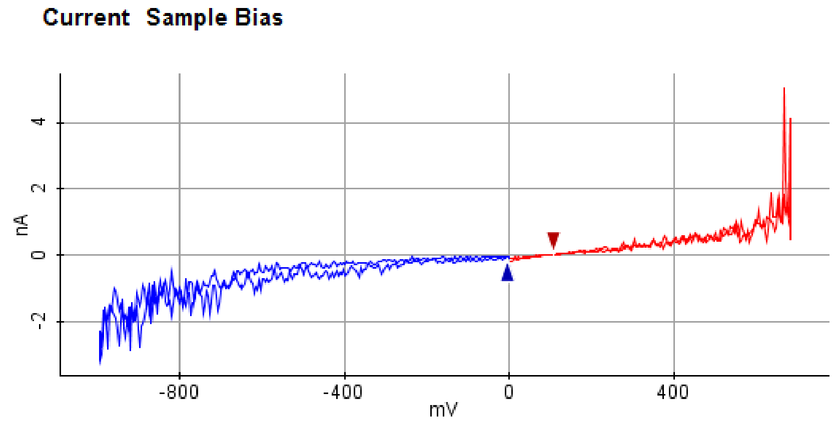


STM image of an **ITO** surface

I/V spectroscopy

I/V spectroscopy on an ITO surface



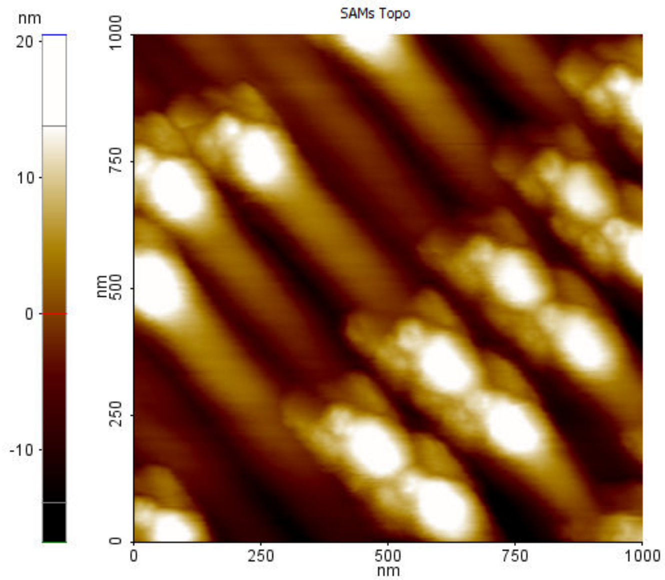


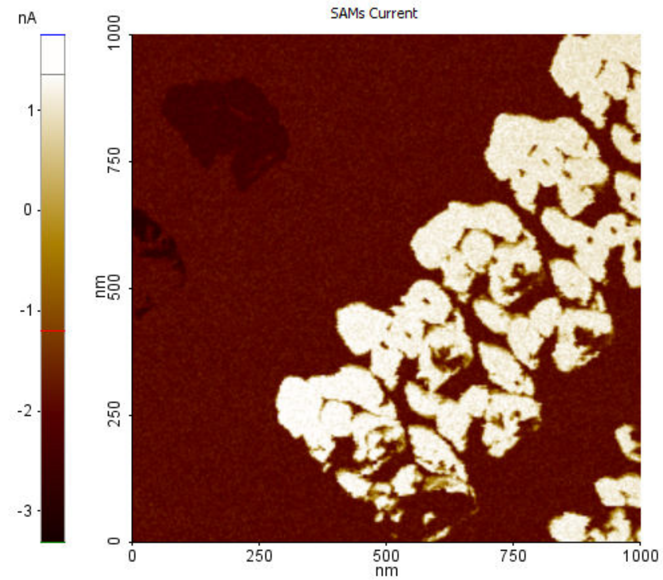
Cursors

Cursor	ΔX(nV)	ΔY(pA)	Left X(mV)	Left Y(pA)	Right X(mV)	Right Y(pA)	Angle(deg)
Trace	0.000	0.000	108.337	5.141	108.337	5.141	N/A
Retrace	0.000	0.000	-2.136	-99.487	-2.136	-99.487	N/A

- **Allows to obtain I/V spectroscopy curves,**
i.e. I/V characteristics of different points onto the surface (current resolution: 1 pA)
- **Points can be randomly selected or arranged in a grid (this allows to obtain a volume figure as for F/d spectroscopy)**

Internal conductive AFM





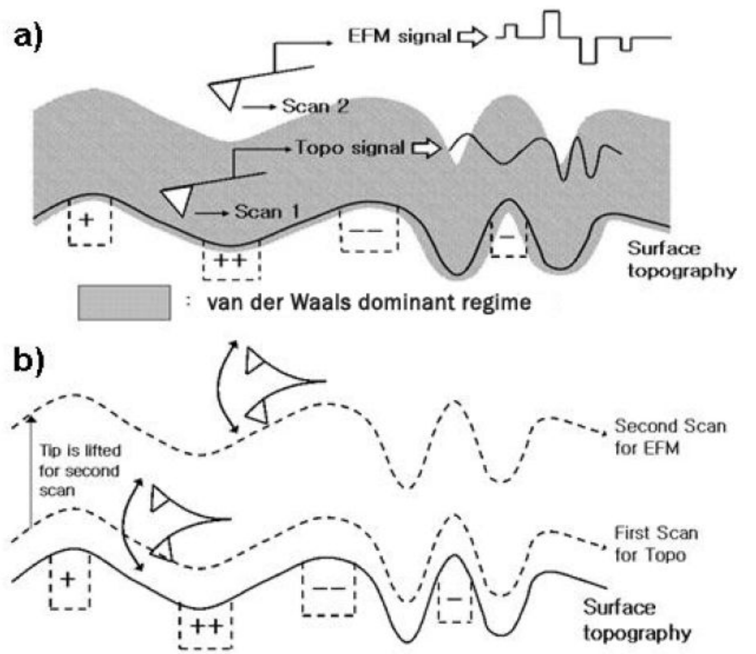
- **Contact mode using a conductive AFM tip**
- **Applied bias between tip and sample (also from tip side)**
- **Instrument measures topography (tip deflection) and surface conductivity (current flow) at the same time**
- **Current detection down to pA**
- **I/V spectroscopy**

Electrostatic Force Microscopy

- Probes the **electric properties** of a surface by measuring the **electrostatic force** between the **surface** and a **biased AFM cantilever**
- **Van der Waals forces** are proportional to $1/r^6$, while **electrostatic** ones to $1/r^2$
- In **standard EFM**, surface is probed **two times** in both **EFM** and **C-EFM**

1st pass: topography
2nd pass: electrostatic properties

- **Internal amplifier** (inside the controller)



Enhanced Electrostatic Force Microscopy – 1

- Probes the **electric properties** of a surface by measuring the **electrostatic force** between the **surface** and a **biased AFM cantilever**
- **AC bias** of given frequency ω (from lock-in amplifier) in addition to **DC bias** from electronics
- All signals detected at one time (topography and EFM), no changes in the distance between tip and sample
- **Electrostatic contribution** to **signal** of **tip deflection** is **extracted** from the **overall signal** by the lock-in amplifier and **decomposed** into different **parts** analysed **separately:**

ω component

2 ω component

Surface potential → V_s

$$V(t) = V_{dc} - V_s + V_{ac}\sin(\omega t) \qquad (1)$$

$$F = q \times E = q \times V/d = C \times V^2/d \qquad (2)$$

$$F(t) = (C/d) \times V(t)^2$$

$$= (C/d) \times [(V_{dc} - V_s)^2 + \frac{1}{2}V_{ac}^2] \qquad (a)$$

$$+ 2 \times (C/d) \times (V_{dc} - V_s) \times V_{ac}\sin(\omega t) \qquad (b)$$

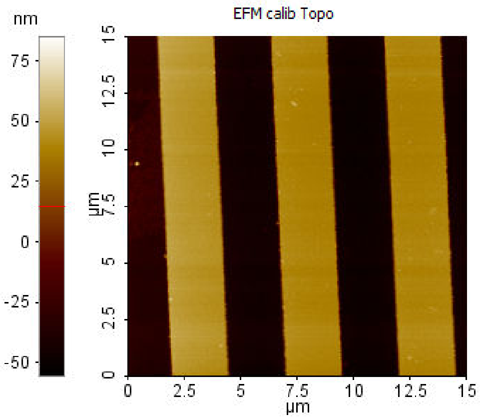
$$- \frac{1}{2}(C/d) \times V_{ac}^2 \cos(2\omega t) \qquad (c)$$

Enhanced Electrostatic Force Microscopy – 2

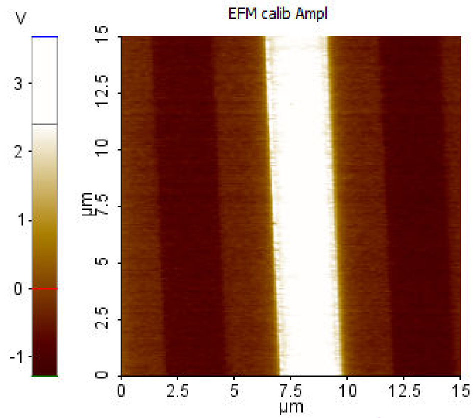
- **EFM(Ext):**

Non contact mode based
Conductive materials
Measures surface potential (surface charge cannot be detected, as it flows into the material)

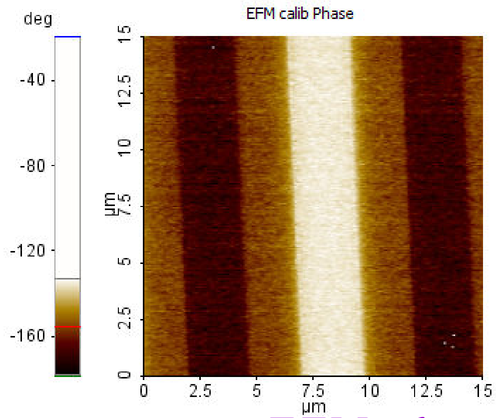
EFM calibration grid



Topography



EFM amplitude
refers to the intensity of the surface potential



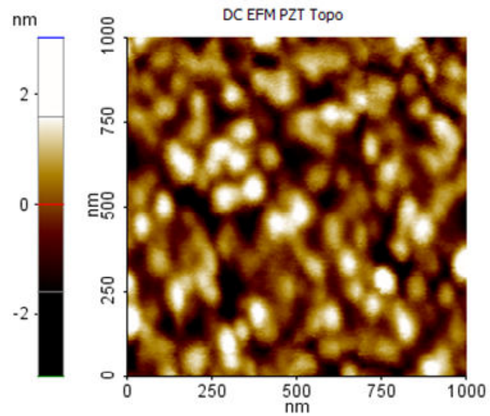
EFM phase
refers to the sign of the surface potential

Enhanced Electrostatic Force Microscopy – 3

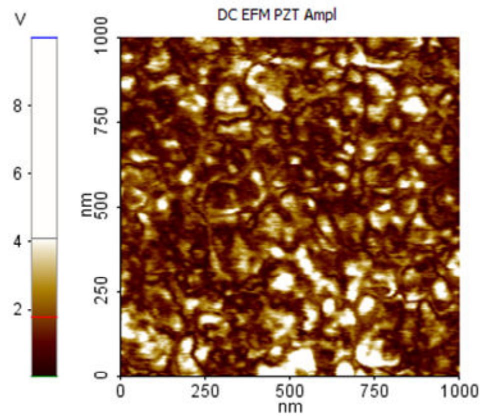
•**DC – EFM:**

Contact mode based
Insulating materials
Measures surface charge (NOT surface potential)
Detection of surface hardness allowed (via lateral force)

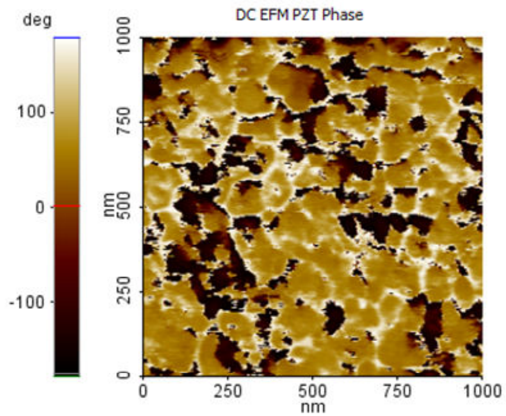
PZT surface sample



Topography



EFM amplitude
refers to the intensity of the surface charge



EFM phase
refers to the sign of the surface charge

Scanning Kelvin Probe Microscopy (SKPM) – 1

It works in EFM(Ext) or DC – EFM conditions (depending on sample characteristics)

Allows to determine the actual surface potential

DC bias is controlled by feedback loop to set to zero the ω term

ω signal can be expressed by the following equation

$$2 \times (C/d) \times (V_{dc} - V_s) \times V_{ac} \sin(\omega t)$$

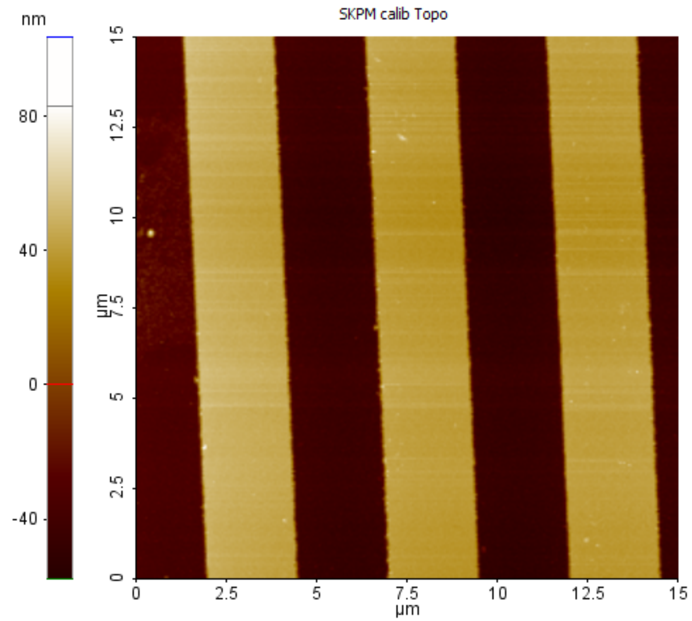
The amplitude of the ω signal is zero when Vdc = Vs, or when the DC offset potential matches the surface potential

The feedback loop varies the DC bias in order to keep ω signal equal to zero

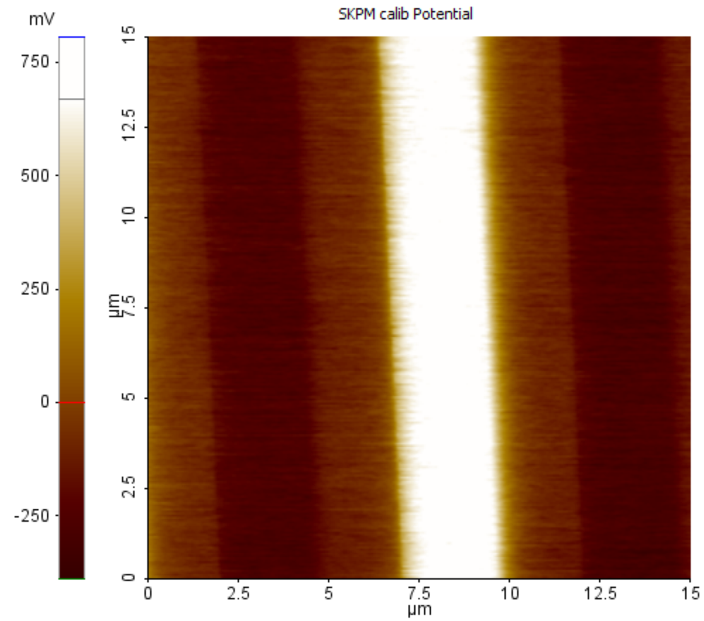
Images obtained in this way represent the absolute value of surface potential

Scanning Kelvin Probe Microscopy (SKPM) – 2

EFM calibration grid



Topography



Surface potential

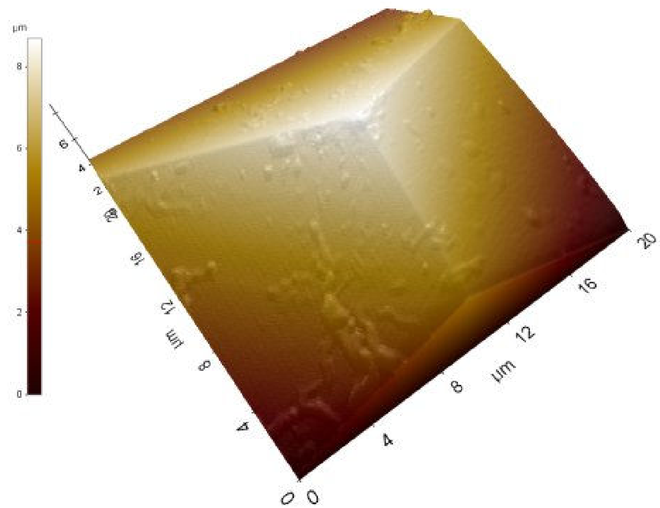
Nanoindentation – 1

- Uses a **Berkovich probe**, that is a **metallic** cantilever with a **diamond tip**, spring constant **100 N/m** (double value with respect to NC cantilevers)

- Allows to probe **hardness** and **elasticity** at the nanoscale

- Can be performed in terms of force exerted on the surface or Z scanner displacement

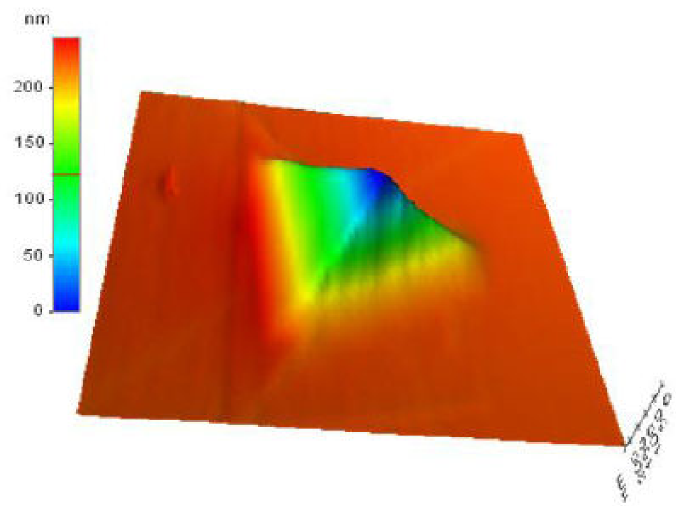
- Different **load cycles** allow to dissipate **heat** and **electrical charges** produced by indentation process

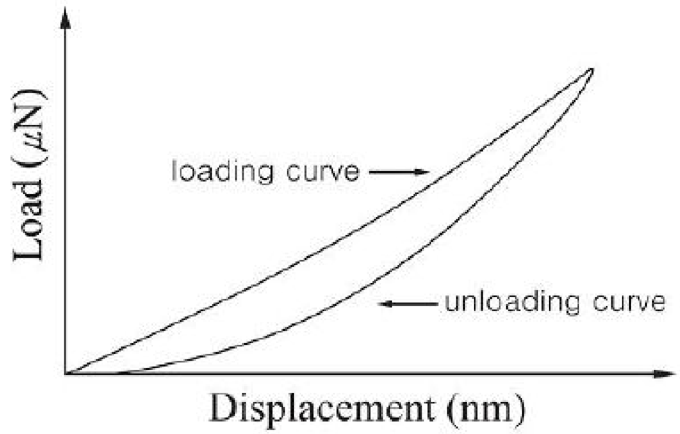


AFM image of a Berkovich tip

Nanoindentation – 2

Hardness of the material
can be calculated
by dividing the **loading force**
by the projected
residual **area of indentation**

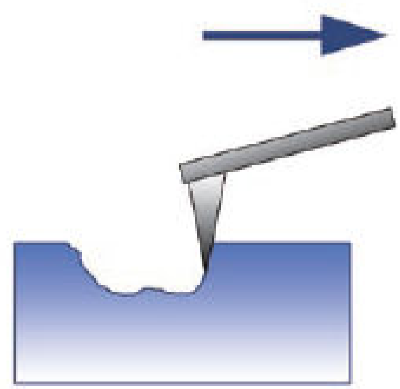




Young modulus of the material
can be obtained
from the **slope** of the **unloading curve**
(in this case,
hysteresis indicates
a partially inelastic deformation)

Nanolithography – 1

- **Allows to deliberately scratch and modify the surface of a material**
- **Working conditions:**



•Z scanner mode
(the control is on Z scanner movement)
•Setpoint (the control is on applied force)
• No special tip needed (usually, NCHR with soft samples)

Setpoint is better than Z scanner mode because scratches depth can be controlled more easily)

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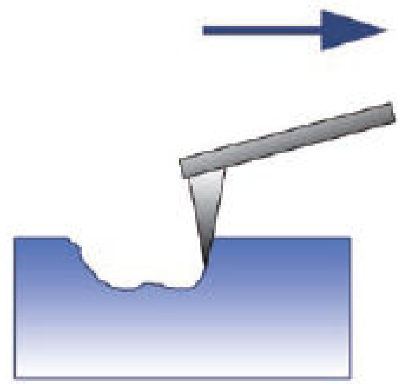

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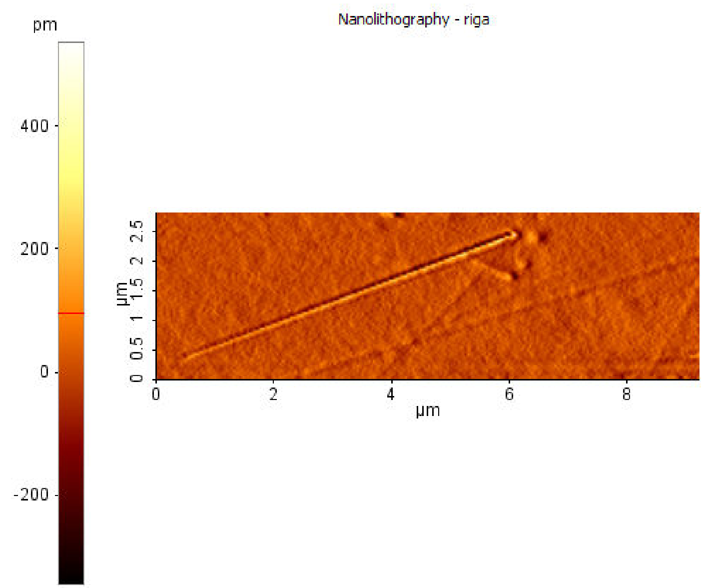
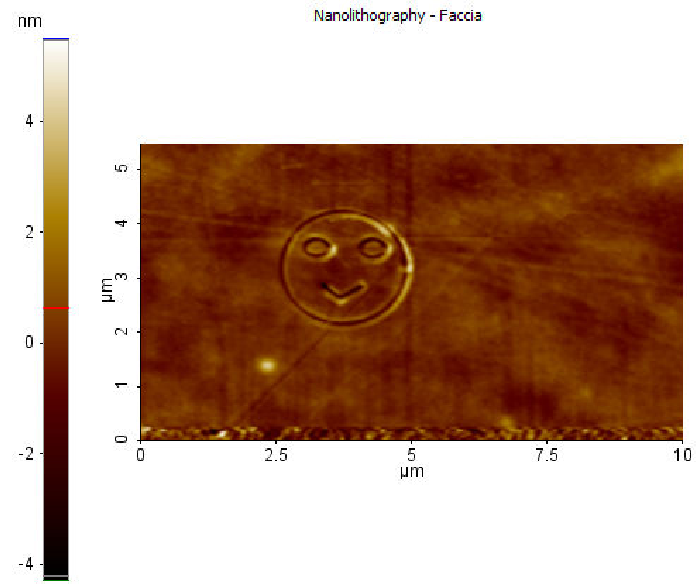
Nanolithography – 2



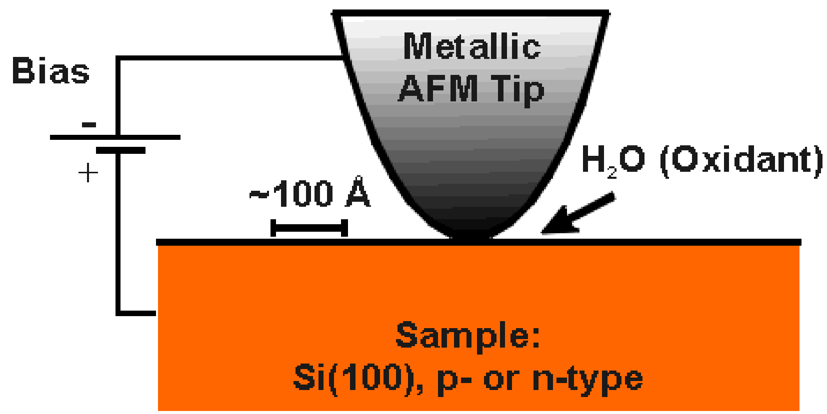


NCHR probe tip onto Cu surface

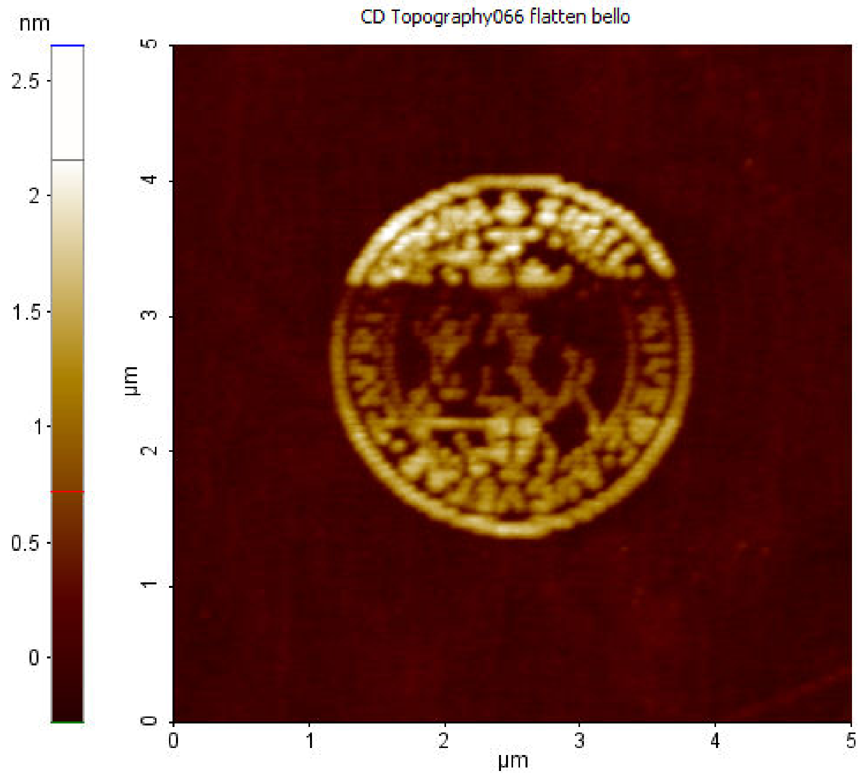
- **Lithography software allows to create images and to import bitmap images and CAD files**

Nanolithography – 3

- 3rd working condition: **Local anodic oxidation**
- Sample surface is **oxidated** by **negatively biasing** the tip with respect to the substrate in **humid air** conditions
- **Ambient humidity** has to be **high enough** to ensure the **presence** of a **drop** of **water** between tip and sample (otherwise, **no reaction** and **no modification** of the surface **occur**!)
- In HV Lithography, applied bias > 10 V
- Contact – AFM based technique
- Raster



Nanolithography – 4



- **Torino University logo (Local anodic oxidation on Si surface)**

Booking & operating information

- Online calendar: http://www.my.calendars.net/afm_nis
  (for booking modifications and deletion,
  ask me or Dominique Scalarone)
- System is equipped with the newest available software
  (XEP version 1.7.54 and XEI version 1.7.1)
- Common purpose (C-, NC-, EFM and liquid environment)
  AFM tips are already available

- **New users** are **STRONGLY RECOMMENDED**
  to follow a short training
  BEFORE beginning their own measurements

Thanks to…

Park Systems International User' Training
KANC, Suwon, South Korea, August 2008