Central European Institute of Technology
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Nanobiotechnology

Scanning Probe Microscopies

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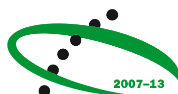
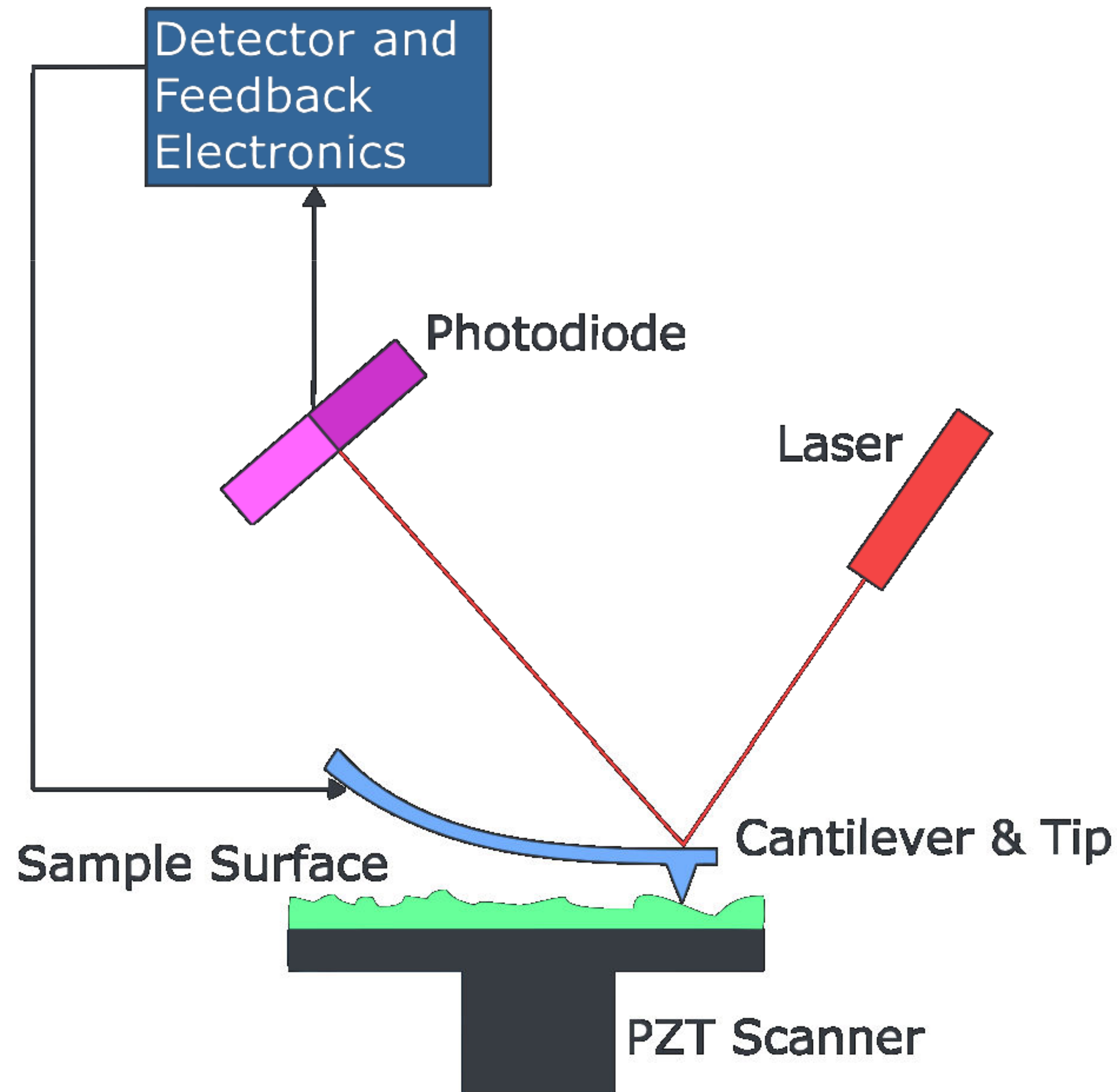


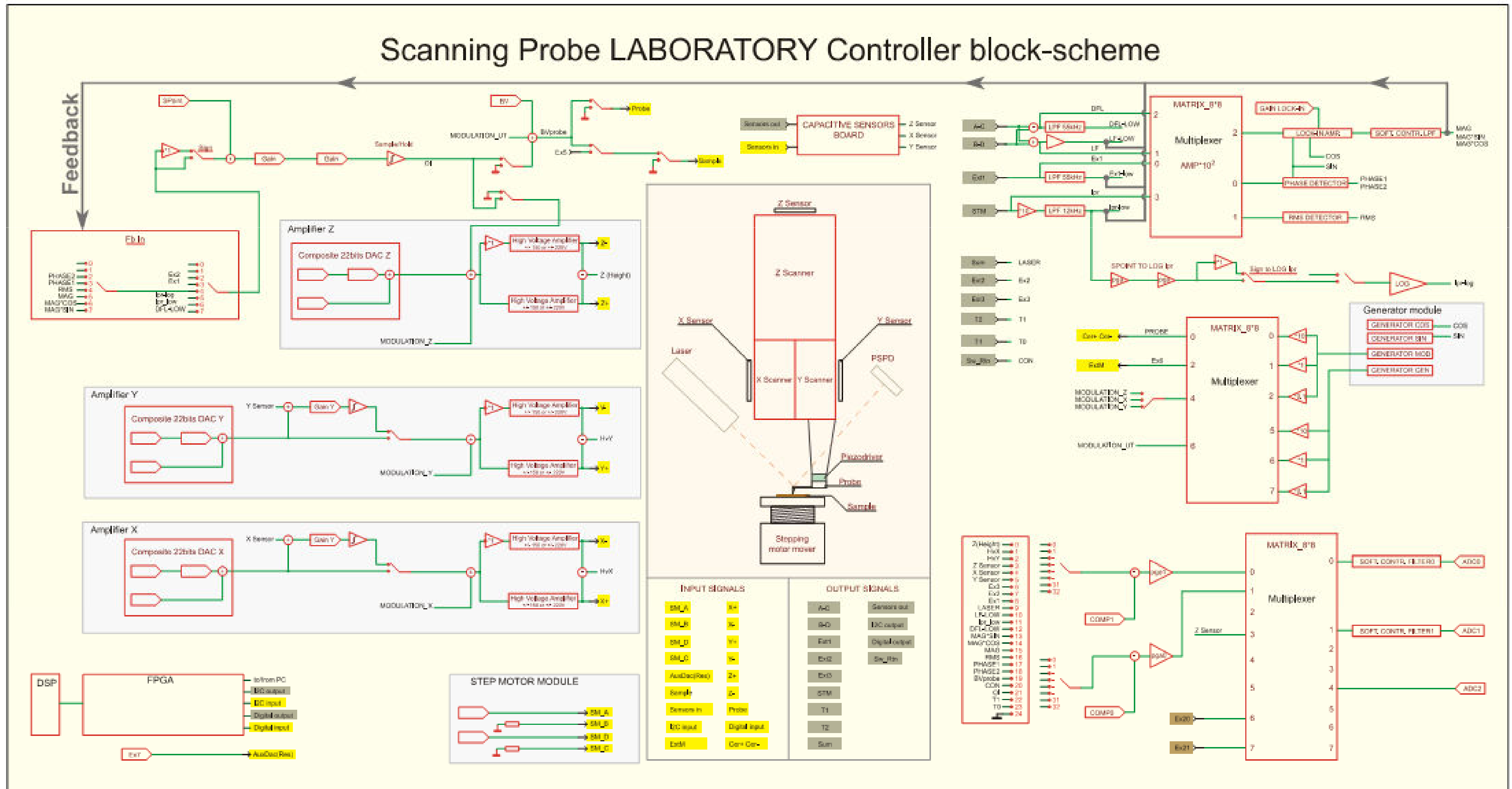
OP Research and
Development for Innovation

AFM

AFM microscope basic scheme

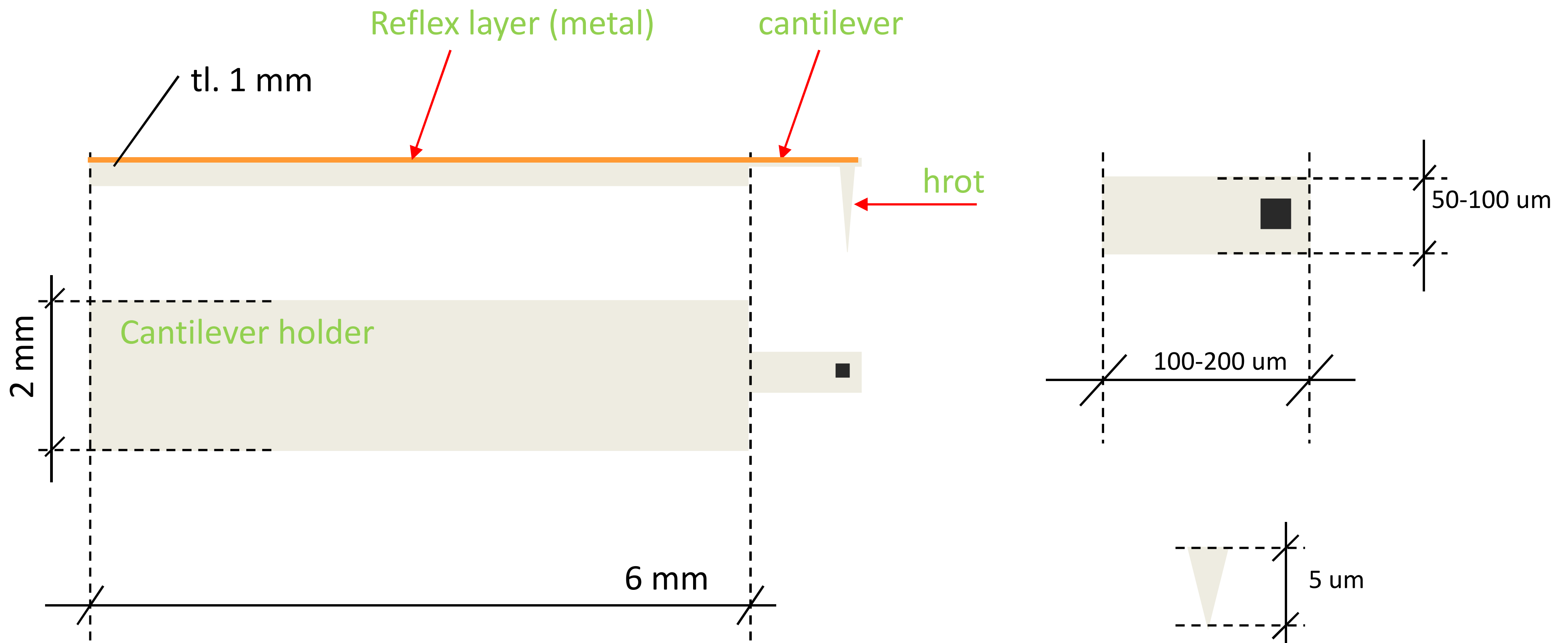


AFM microscope block scheme



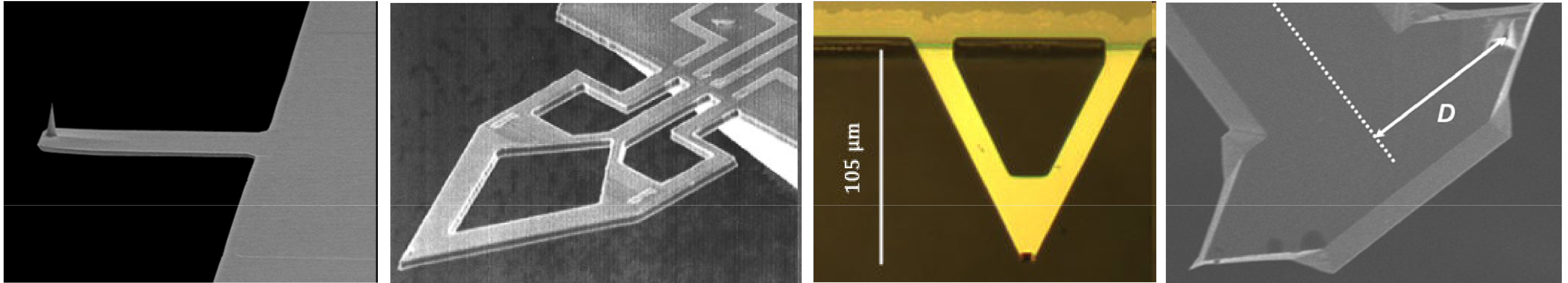
Tip and cantilever

Cantilever and tip



- Cantilever holder is quite universal
- Cantilever and tip – a variety of various types

Cantilevers



Material properties

– Stiffness **Force Constant [N/m]**

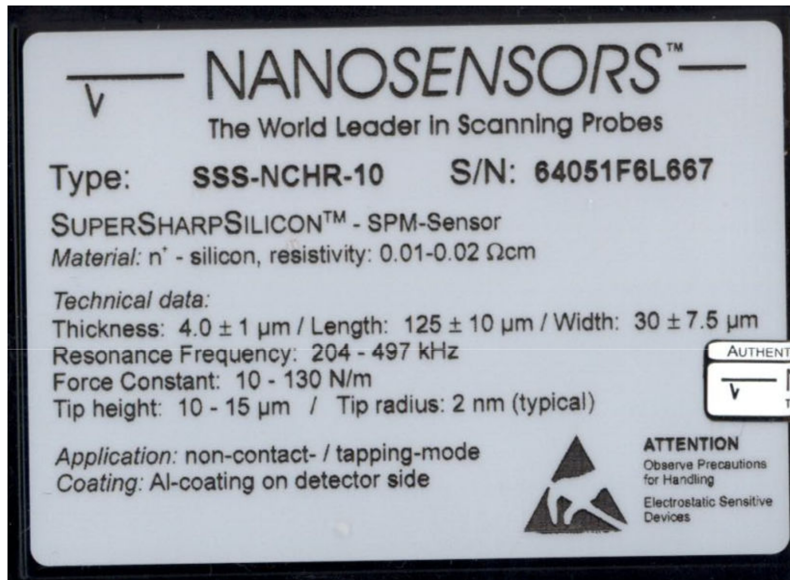
Force const.[N/m]	10-130	1-10	0.1-1.0	0.005-0.1

Material	cryst. silicon	pol. silicon	glass	Si_3N_4

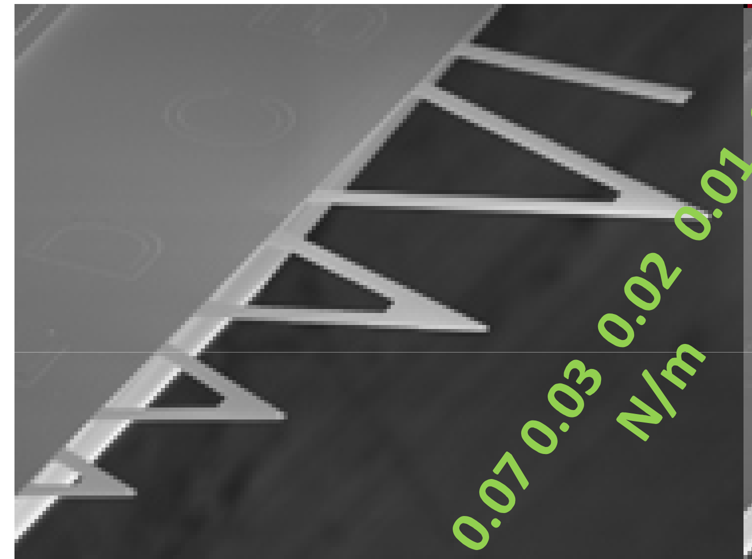
Res. f. [kHz]	200-500	100-200	15-100	1-20

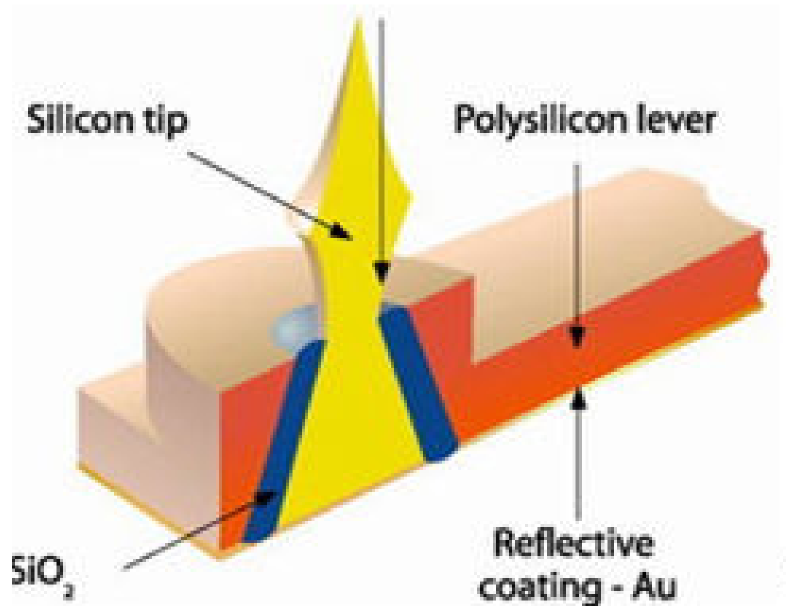
Special applications – conductive, colloid, magnetic, tip less, …

Cantilever
characterization you may find on box



Cantilever field
choose the one you like/need

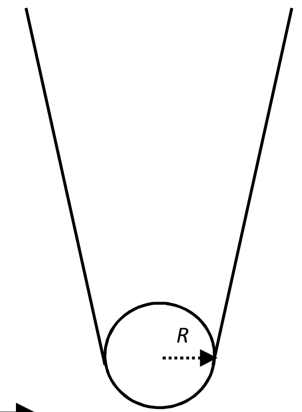




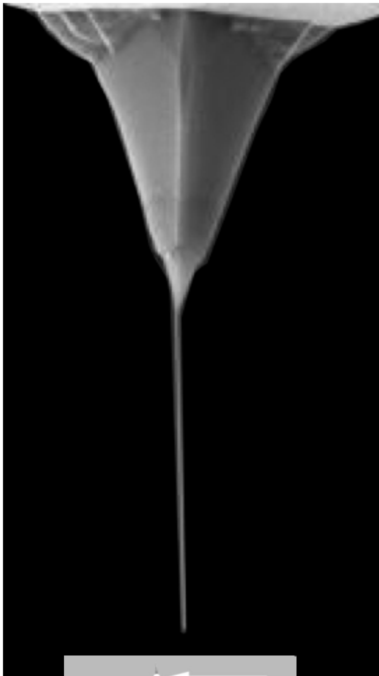
AFM probes (**micro)fabrication** is quite complex

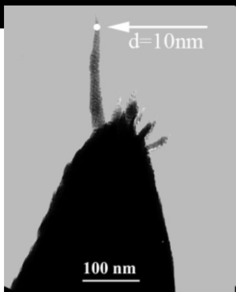
Tip properties

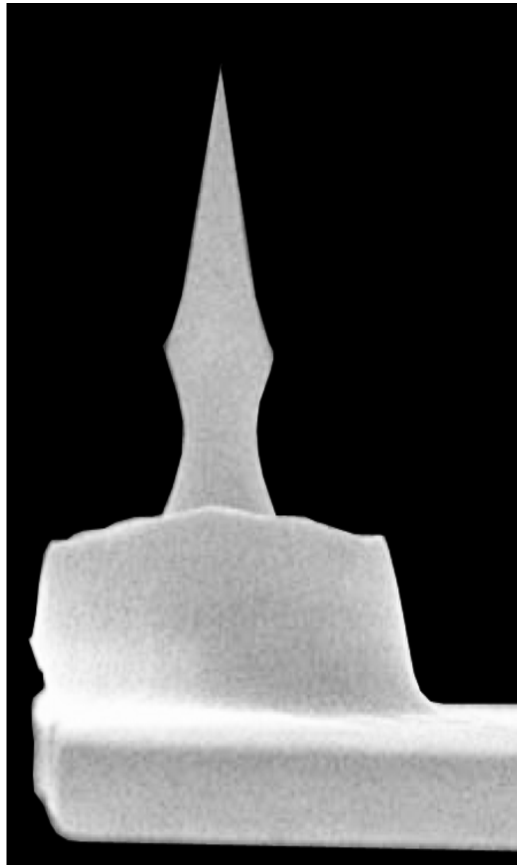
Shape – **Curvature Radius *R [nm]***

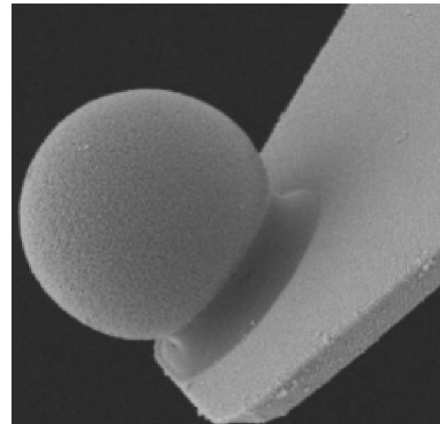


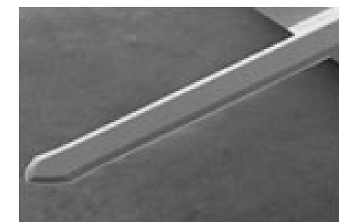
R 1 nm	10 nm	100 nm	NA
Supersharp	Standard	Special app.	Tip less



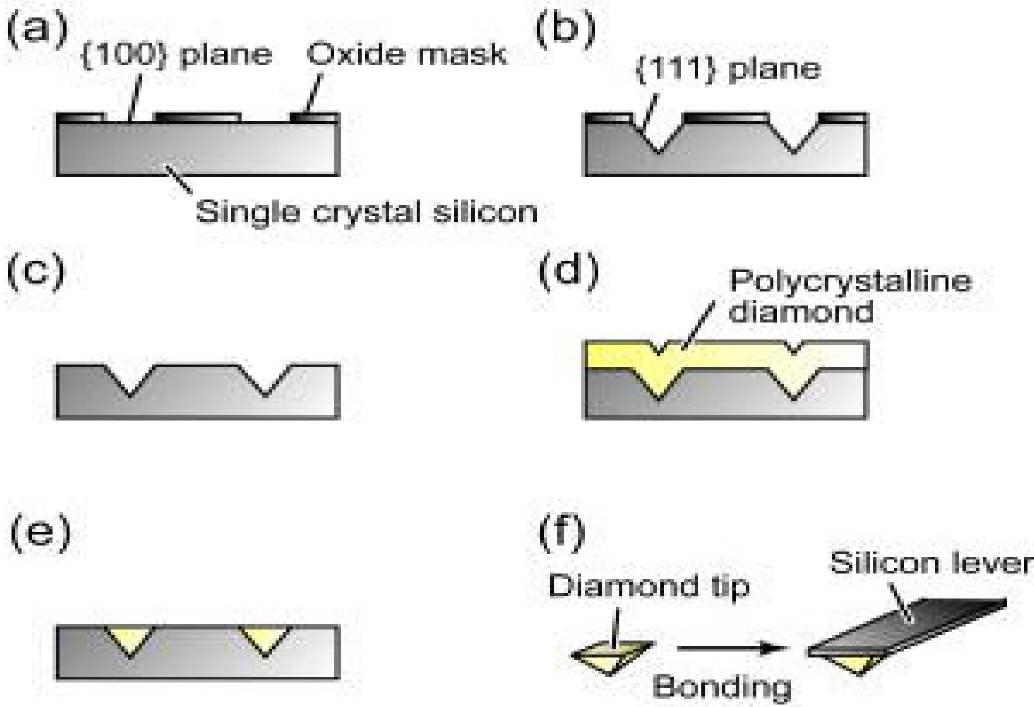




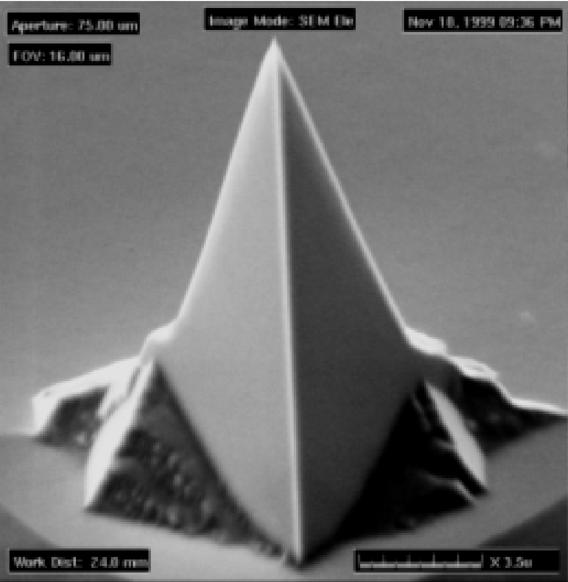


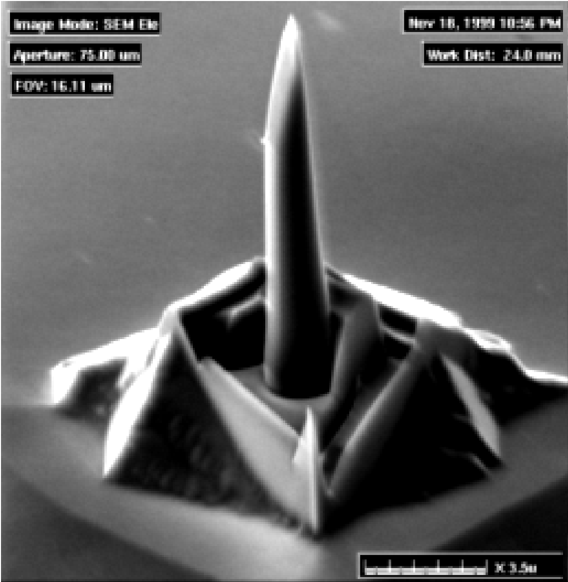


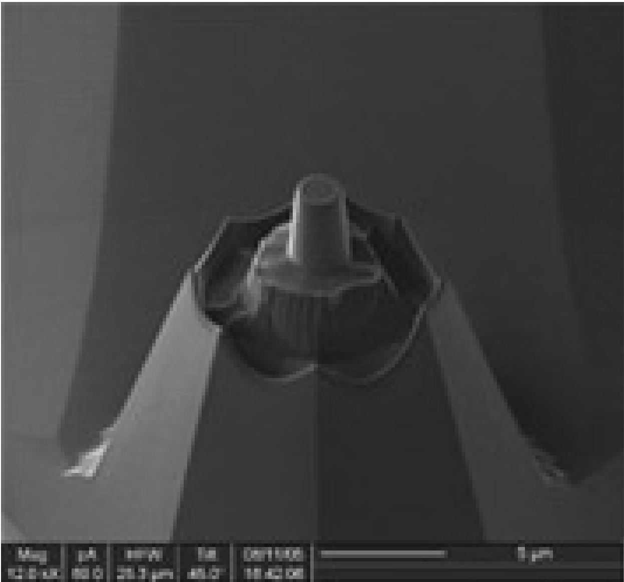
Cantilever **fabrication**



FIB (Focus Ion Beam) post-fabrication of AFM probes (tip)

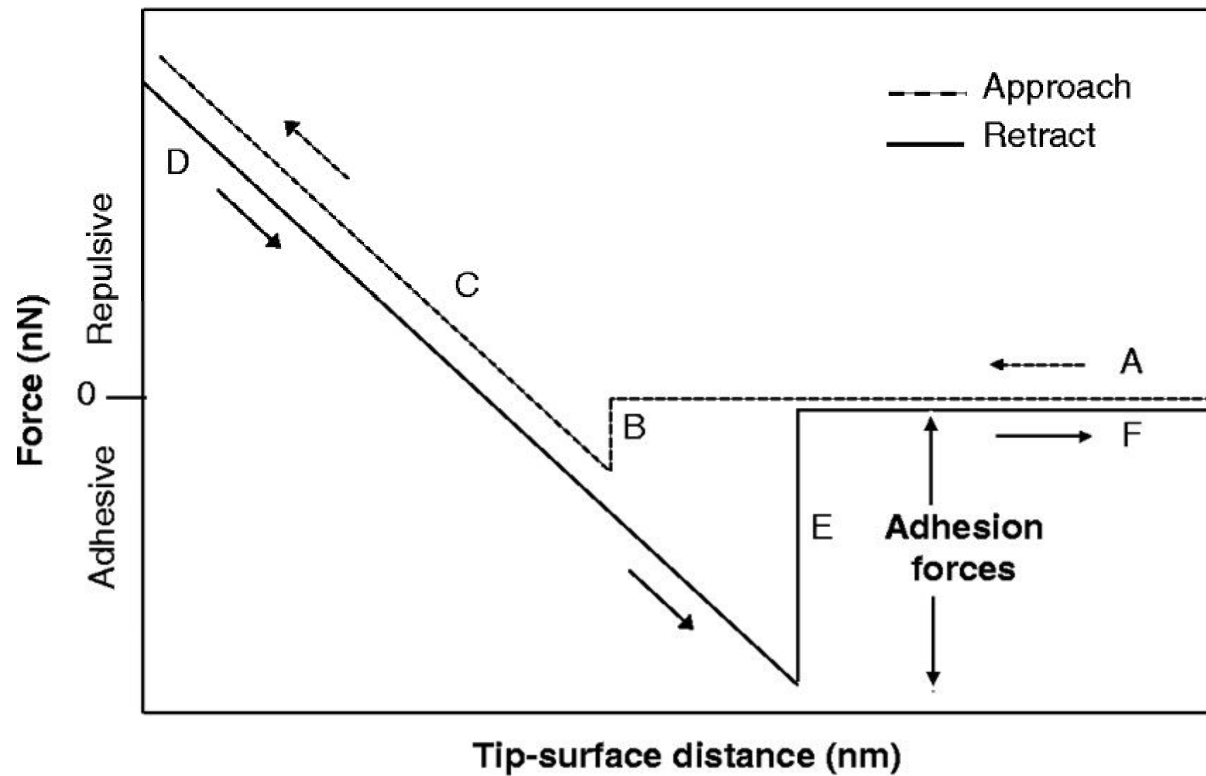


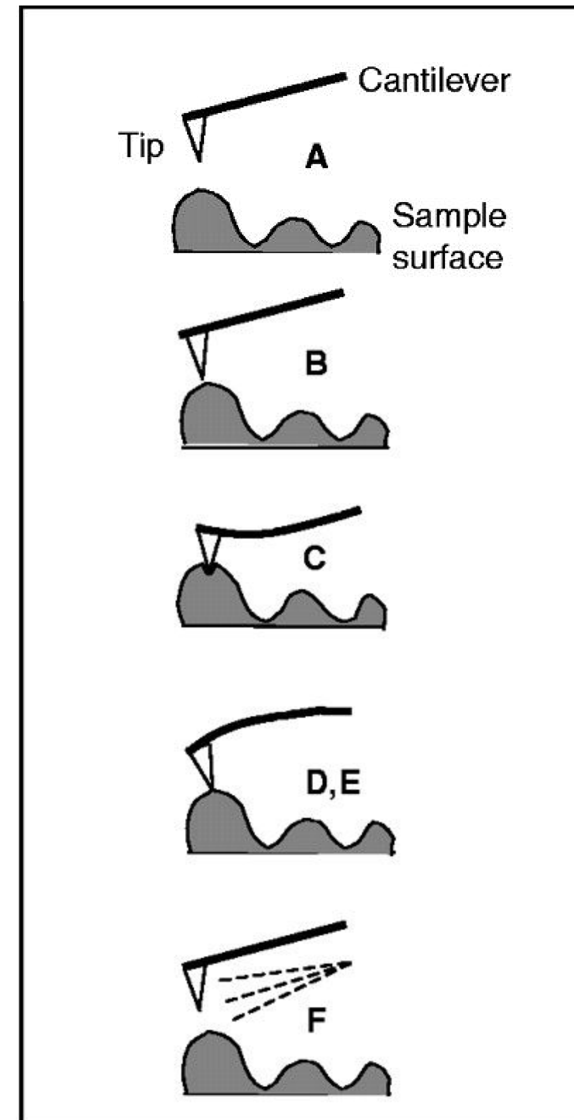




Plateau Tip

Idealized force-distance curve describing a single approach-retract cycle of the AFM tip, which is continuously repeated during surface scanning.





Victor Shahin et al. J Cell Sci 2005;118:2881-2889

Journal of Cell Science
jcs.biologists.org

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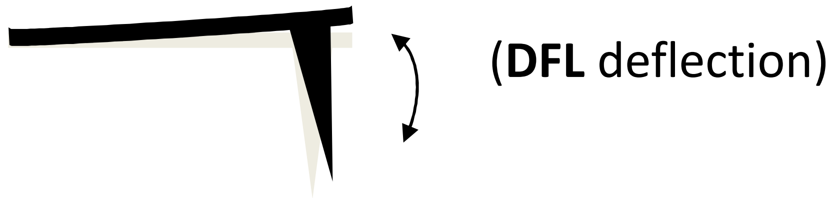
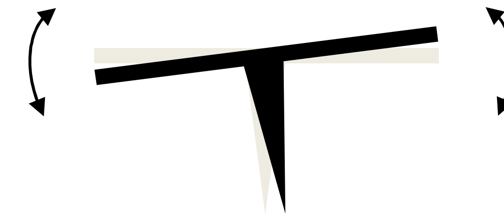
Cantilever bending – how to detect

Contact with surface

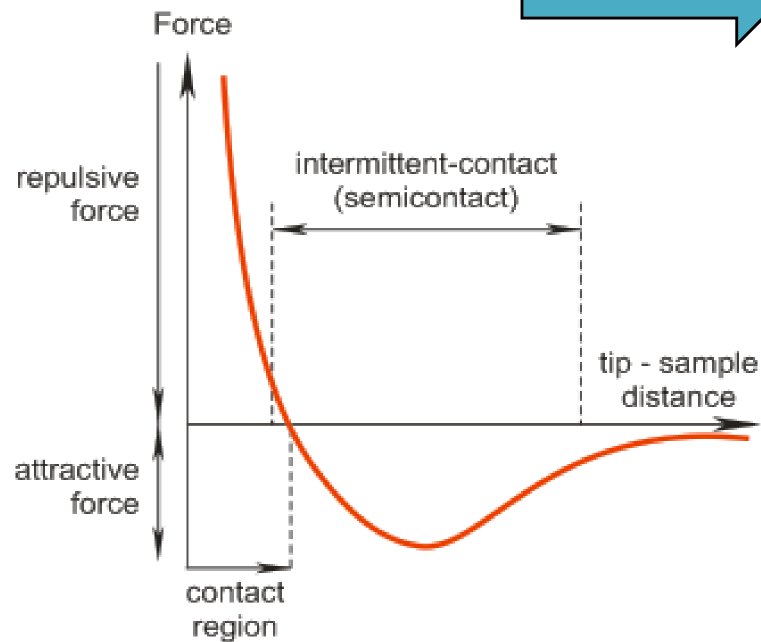
Torsion forces
(**LF** latheral forces)

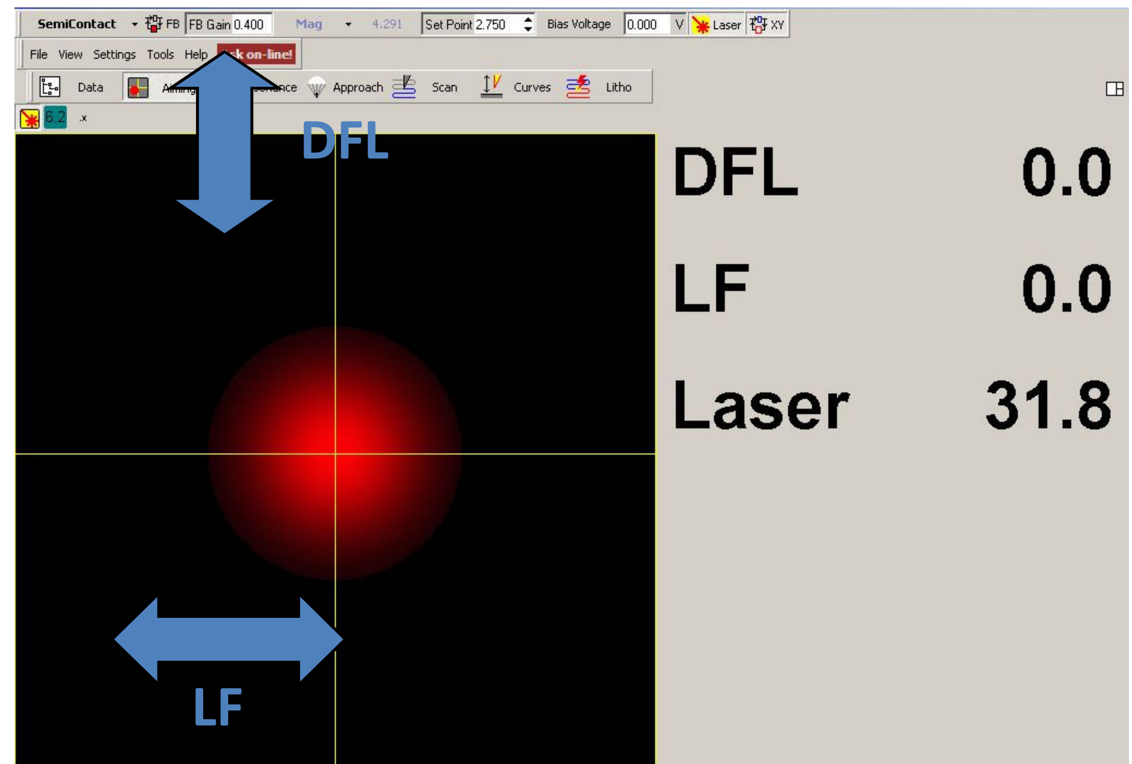


(**DFL** deflection)



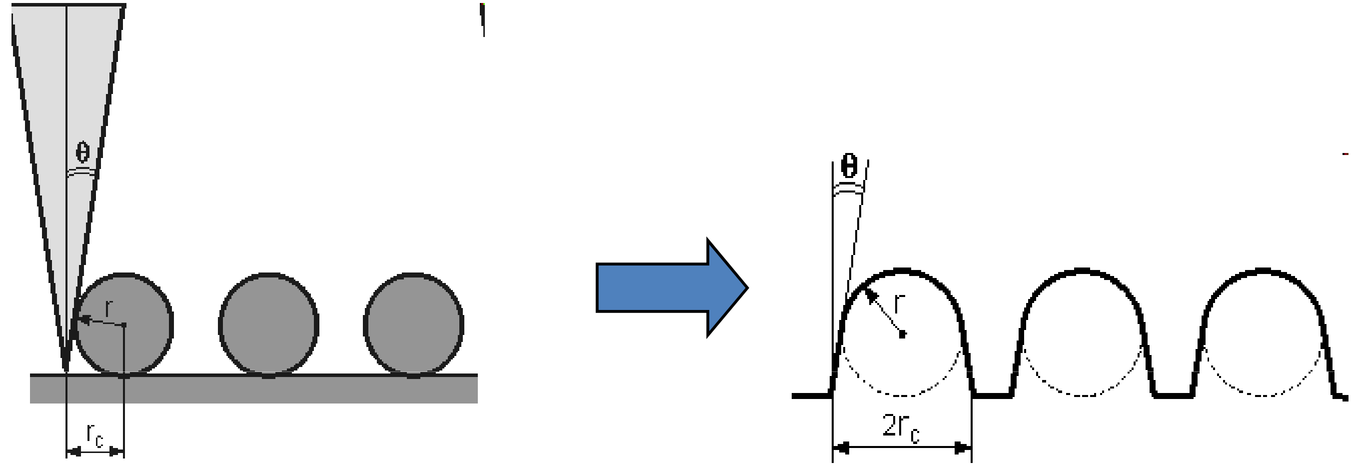
Change of cantilever properties
(DFL/LF) is detected by laser beam



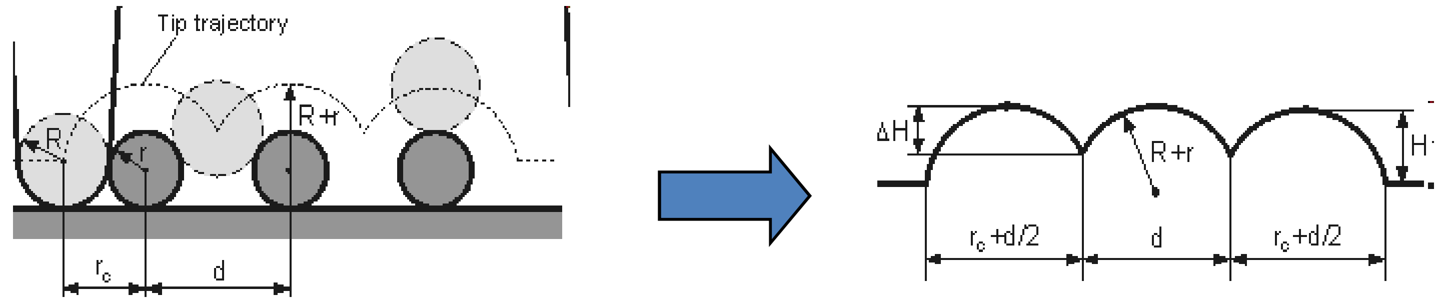


Curvature radius (R) effect

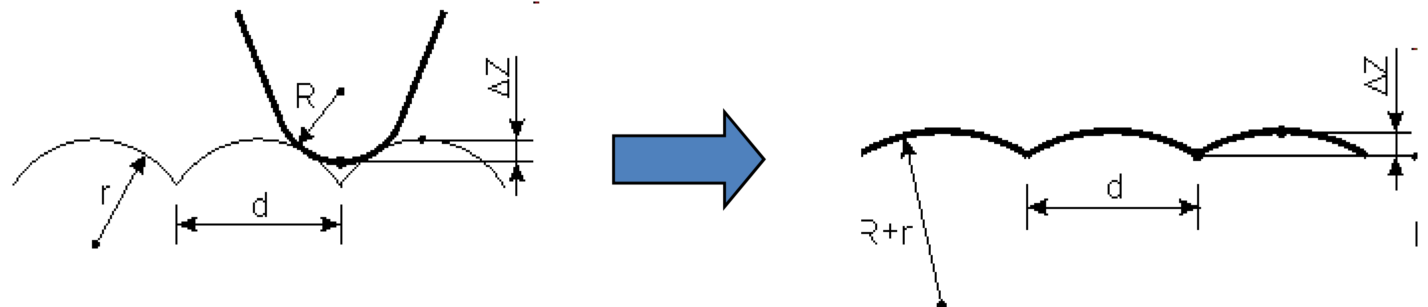
SuperSharp tip
= real image



Standard tip
= R ~ 5-10nm

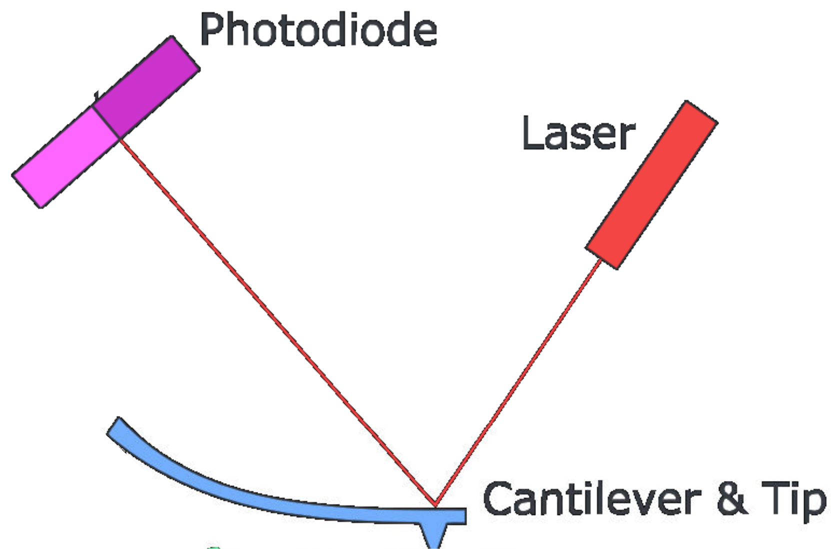


Blunt tip
= affecting real shape and size



Laser, photodiode a cantilever

Laser + photodiode
→ Detection of
cantilever bending



Upper side of cantilever - **reflective**

Laser beam reflects to detector

Detector = photodiode

Laser beam movement – bending detection

Aiming – 1st step of microscope setting

Change of laser beam position – **during scanning** over the sample

→ **Automatic transformation** to the 2D **image** (software)

SemiContact FB FB Gain 0.400 Mag 4.291 Set Point 2.750 Bias Voltage 0.000 V Laser XY

File View Settings Tools Help Ask on-line!

Data Aiming Resonance Approach Scan Curves Litho

DFL

LF

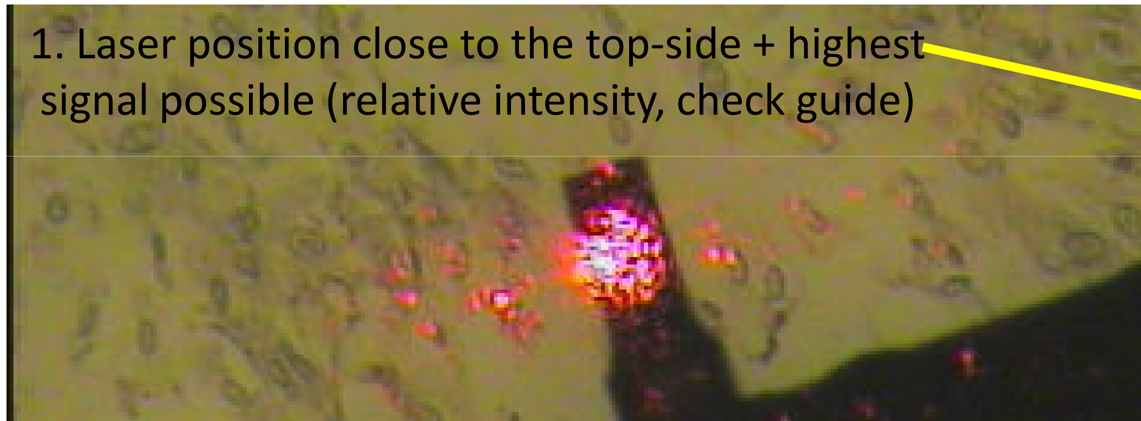
DFL	0.0
LF	0.0
Laser	31.8

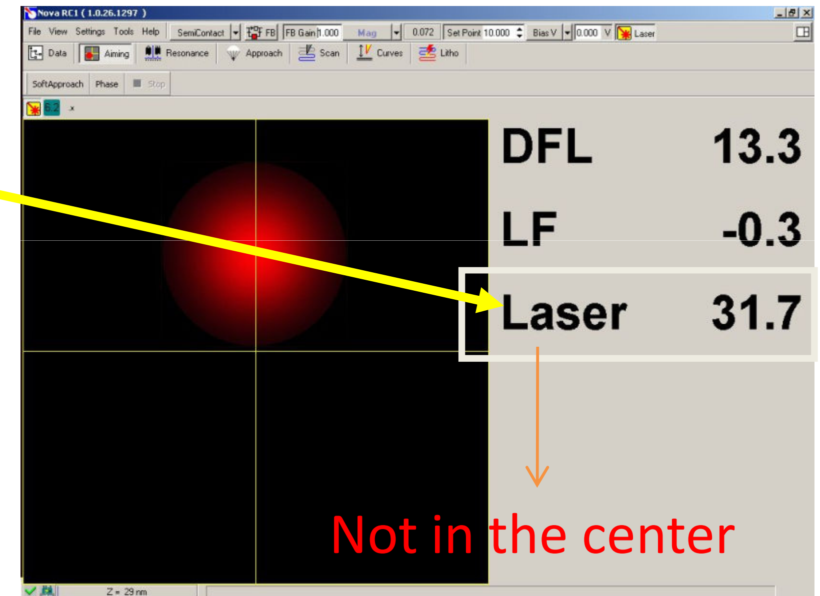
Aiming: initial setting of AFM microscope

Aim of aiming (2 steps):

1. Highest possible reflection of laser beam from cantilever

2. Center beam position on the detector



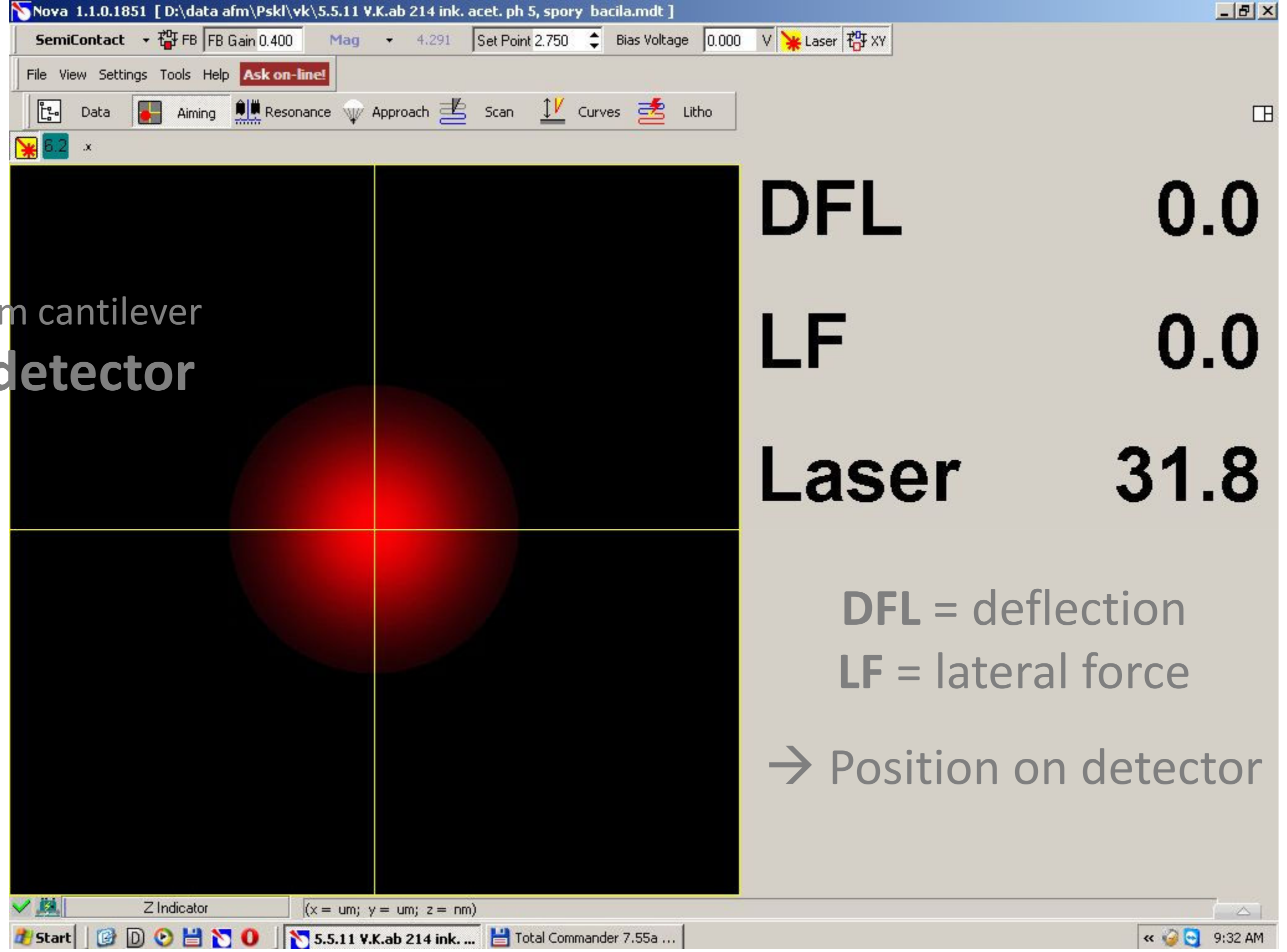


Screws to adjust
(labeled as **LASER**)

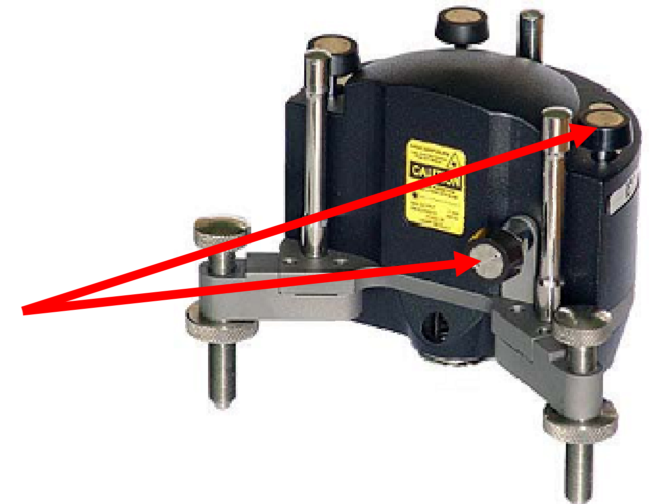
Aiming

Aim of aiming (2 steps):

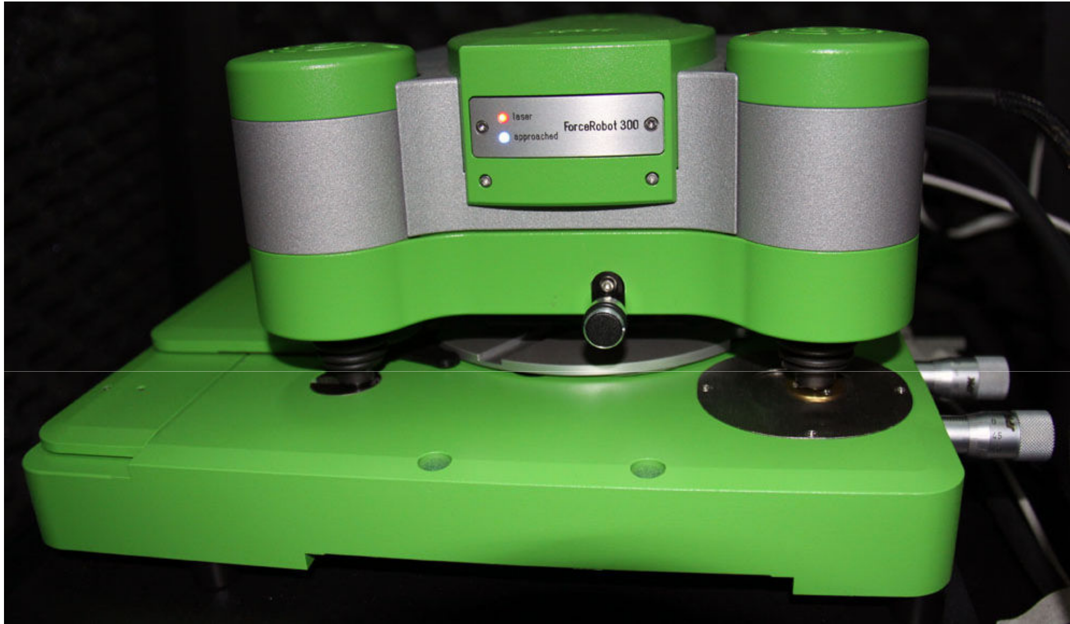
1. Highest possible reflection of laser beam from cantilever
2. **Center beam position on the detector**



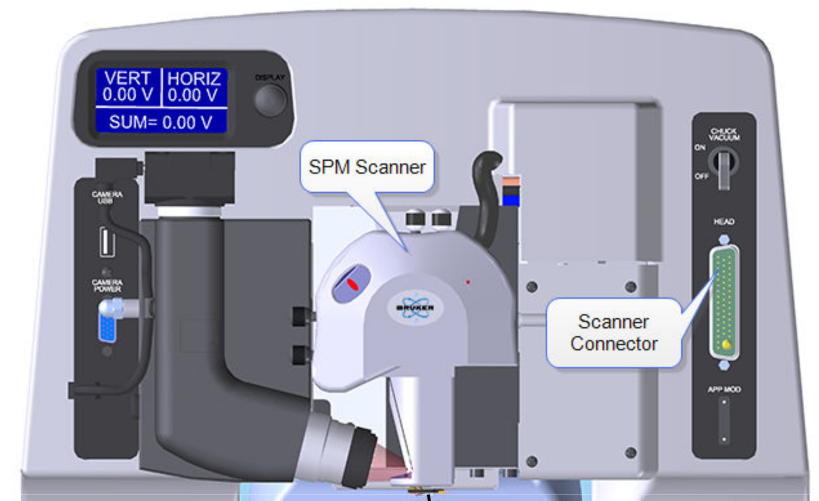
Screws to adjust
(labeled as **PHOTODIODE**)



Automatic adjustment available

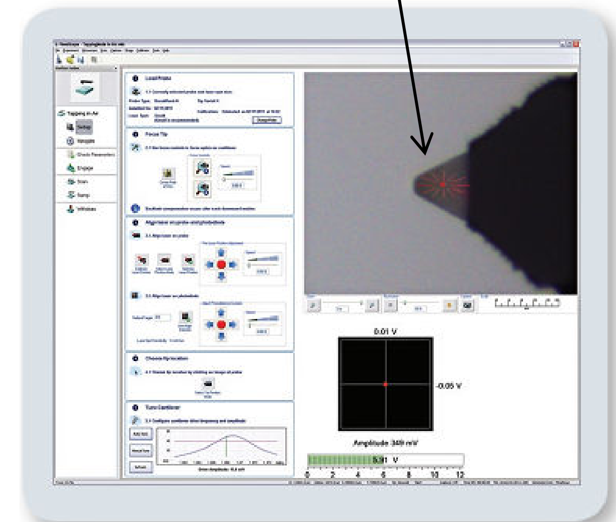


JPK Force Robot head



Bruker Icon/FastScan

NTMDT Solver Next



User interface with a sensible workflow and automatic setup.

AFM modes of operation

Contact mode

- Measured parameter - cantilever **bending** (= **deflection**, **DFL**)
- Deflection ~ tip sample **force interaction**
- **Hook`s law:**

$$F = - k * \Delta h$$

F – force

k – force constant (stiffness)

Δh – change of height (=deflection)

NT-MDT

dc Contact techniques:

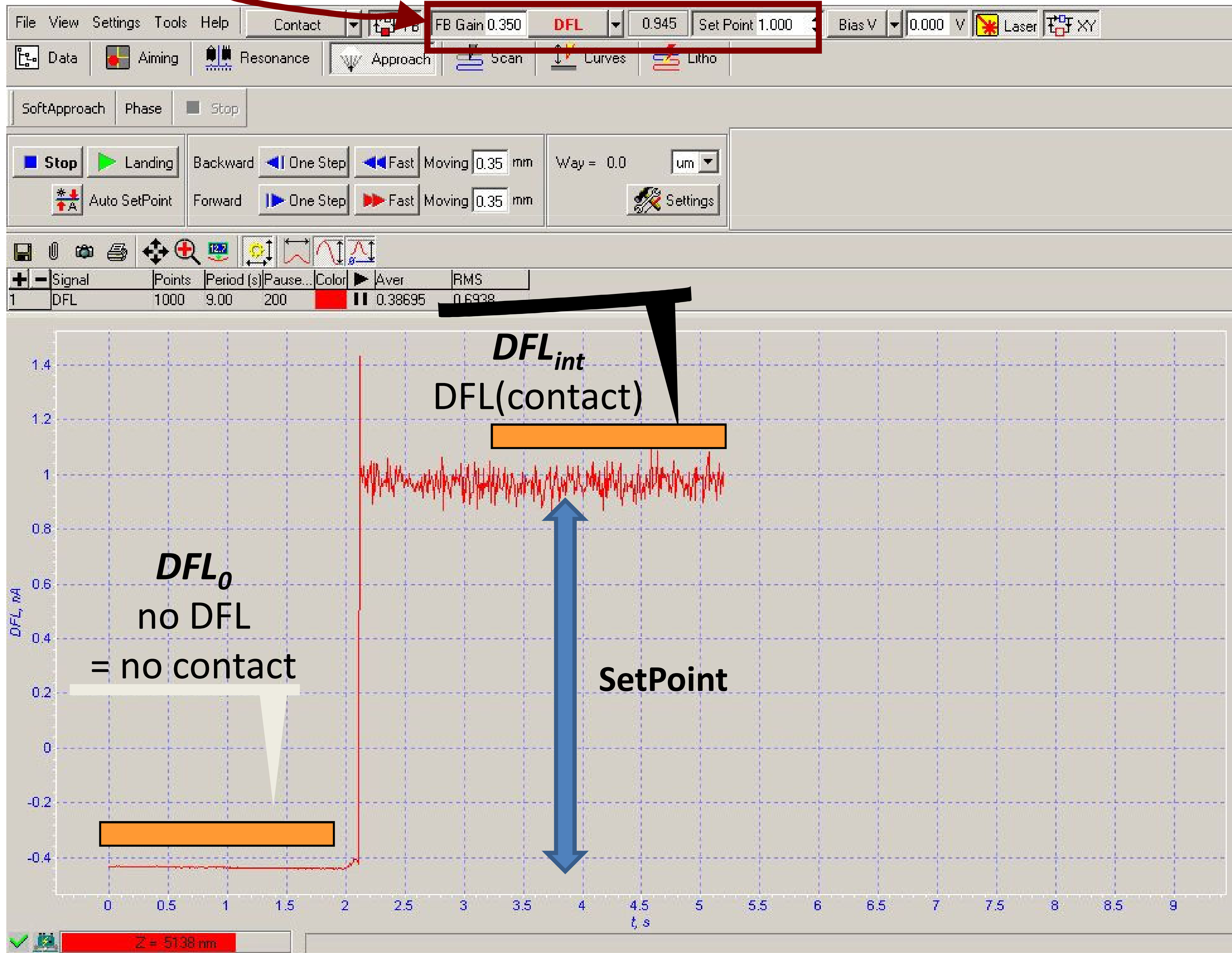
- Constant Height mode
- Constant Force mode
- Constant Error mode
- Lateral Force imaging
- Spreading Resistance imaging

Force
repulsive force
attractive force
tip - sample distance
contact region

Fig. 1. Idealized scetch of tip-sample forces.

In **Contact mode** of operation the cantilever deflection under scanning reflects repulsive force and is used as **such**, in **feedback circuitry** or in **their combination** to imagine the sample surface profile. Simultaneously with topography acquisition under scanning one can imagine some other characteristics of the investigated sample.

SetPoint = basic value of tip-sample interaction



Semicontact mode

(tapping mode, AC mode, oscillation mode, …)

- Measured parameter **amplitude of oscillation** (= **magnitude**, **MAG, …**)
- Measured as:
  - relative parameter, e.g. as **MAG [nA]**
  - absolute parameter – **A [nm]**

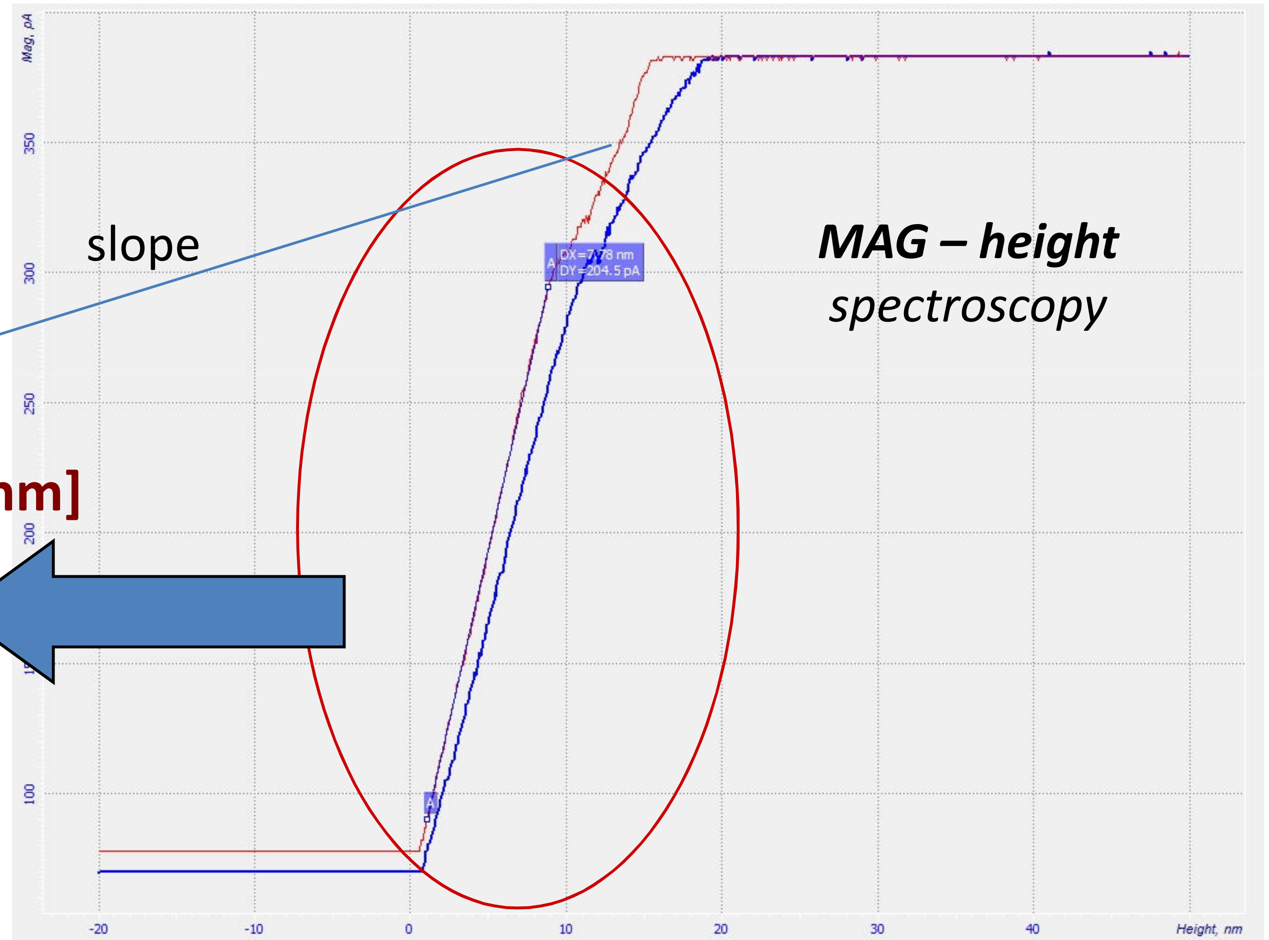
A_0
free
amplitude

A_{int}
damped
amplitude

Relative to absolute amplitude calibration

Amplitude is proportional:

- Voltage of an oscillator
- SetPoint



AMP_{real} = (dX/dY)*SetPoint [nm]

Semicontact mode:

Amplitude of oscillation ~ size of object

SetPoint = damping of free oscillation amplitude (relative/absolute)

File View Settings Tools Help SemiContact FB FB Gain 0.300 Mag 0.870 Set Point 1.000 Bias V 0.000 V Laser XY

Data Aiming Resonance Approach Scan Curves Litho

SoftApproach Phase Stop

Stop Landing Backward One Step Fast Moving 0.35 mm Way = 0.0 um

Auto SetPoint Forward One Step Fast Moving 0.35 mm Settings

	Signal	Points	Period (s)	Pause...	Color	▶	Aver	RMS
1	Mag	1000	9.00	200		❚❚	1.8412	1.2257

A_0

free amplitude

A_{int}

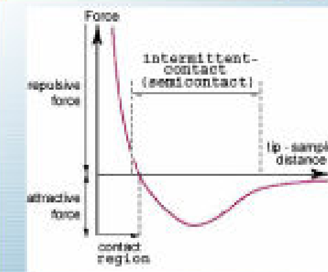
damped amplitude

Mag, nA: 0, 0.5, 1, 1.5, 2, 2.5, 3, 3.5

t, s: 0, 0.5, 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5, 5.5, 6, 6.5, 7, 7.5, 8, 8.5, 9

Z = 4919 nm

NT-MDT



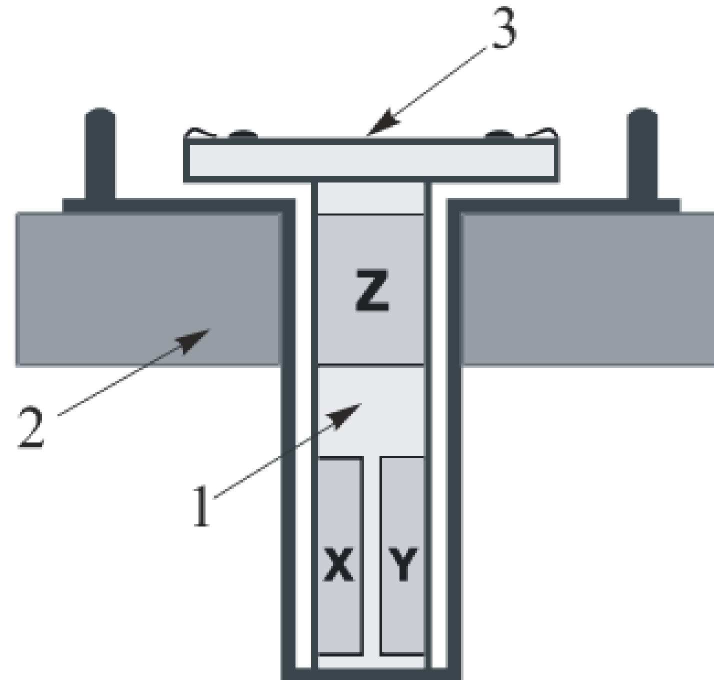
Semicontact techniques:

- Semicontact mode
- Phase Imaging mode
- Semicontact Error mode

Usage of **SFM** with oscillating cantilever was firstly anticipated by **Binnig**. Relatively small shift of cantilever oscillating frequency with sensing repulsive forces means that contact of cantilever tip with sample surface under oscillation is not constant. Only during small part of oscillating period the tip "feels" contact repulsive force. Scanning sample surface with cantilever oscillated in this manner is not non-contact, but intermittent contact (semicontact).

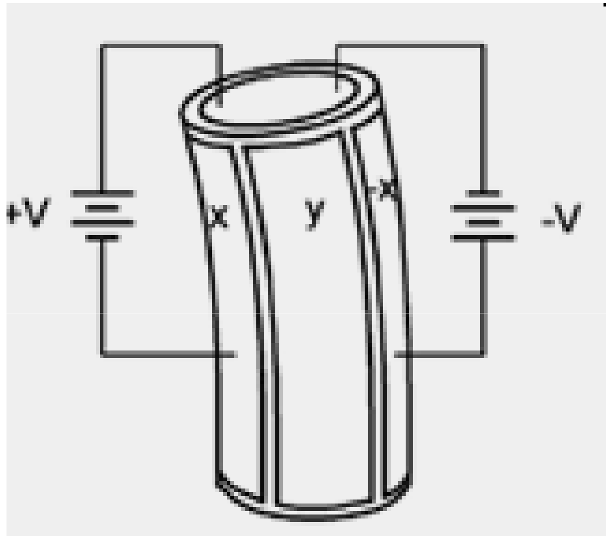
PZT

Piezoelectric tubes



Piezoelectric tubes PZT

Piezoelectrodes

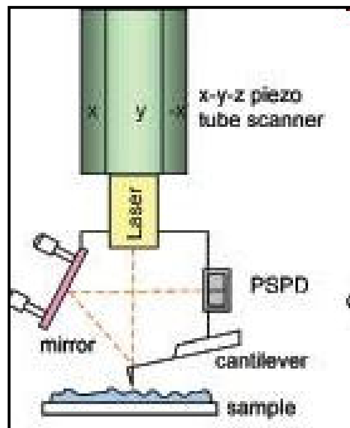


- **Hollow ceramic** tubes
- **Metal covered** in selected parts
- Voltage application → change of size

Notes + cautions

- *Fragile*
- *High voltage applied*

PZT – construction approaches of AFM

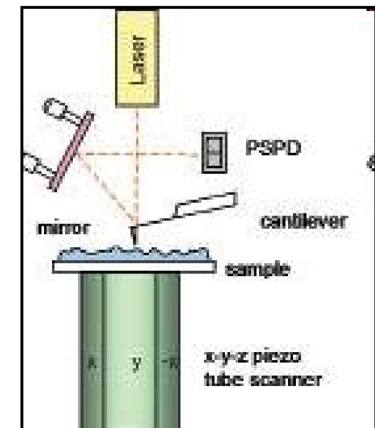


Scanning by probe construction

- *x,y,z* axes movement by head
- Oscillator in head
- Range x,y 100-150 um
- Range *z* 10-15 um

Scanning by sample construction

- *x,y,z* axes movement by sample
- Oscillator in head
- Range x,y 1-10 um
- Range *z* 1-3 um
- Low noise



Piezo-tubes PZT

in software

1. **Approaching to surface**

- first approach with step motors

- after wards Z-piezos

Jump to surface by Z-piezos

Z-piezo position

4.5
4
3.5
3
2.5
2
1.5
1
0.5
0

Mag, nA

0 0.5 1 2 2.5 3 3.5 4 4.5 5 5.5 6 6.5 7 7.5 8

t, s

Z Indicator

(x = um; y = um; z = nm)

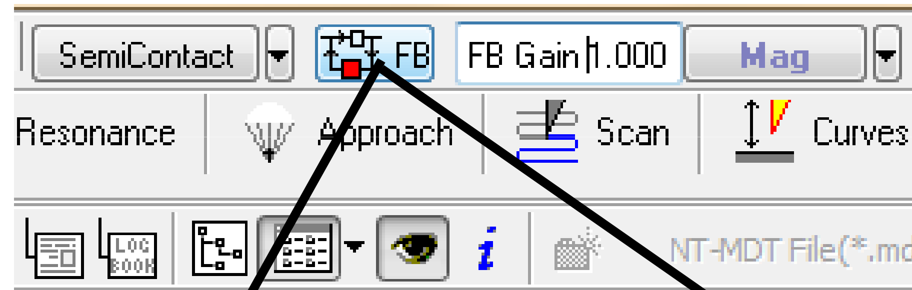
Piezo-tubes PZT

in software

2. **FBloop** (FeedBack Loop)

   - feed back driving of cantilever deflection (=constant) over the surface

   - **ON/OFF** of Fbloop leads to tip-sample interaction ON/OFF:



OFF

PZT

OFF

ZAP

ON

ZAPNUTO

vzorek

ON

ZAP

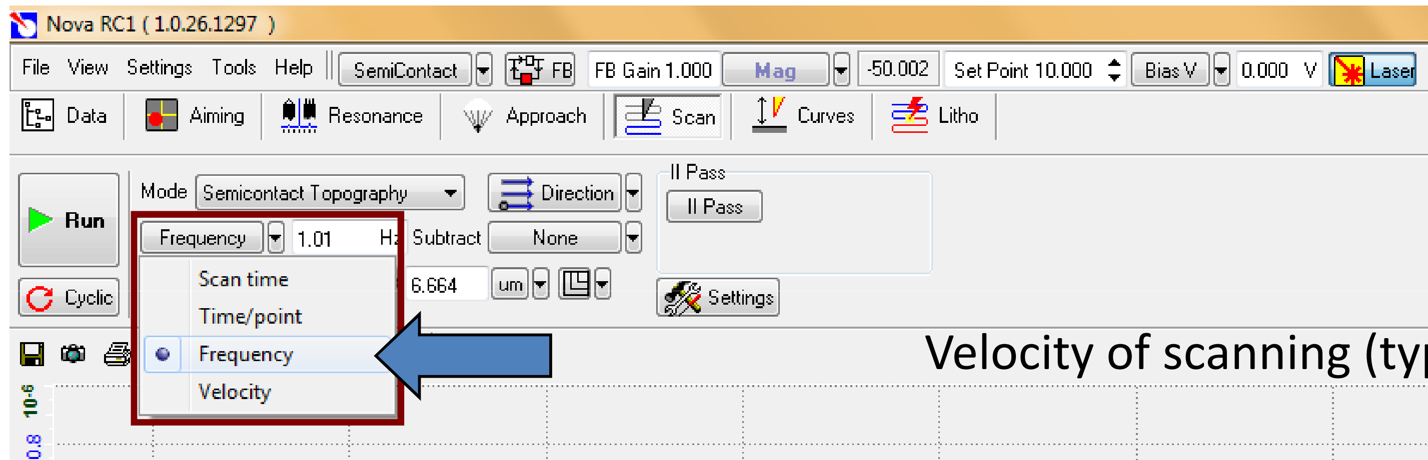
Z = 0 nm

Z Indicator

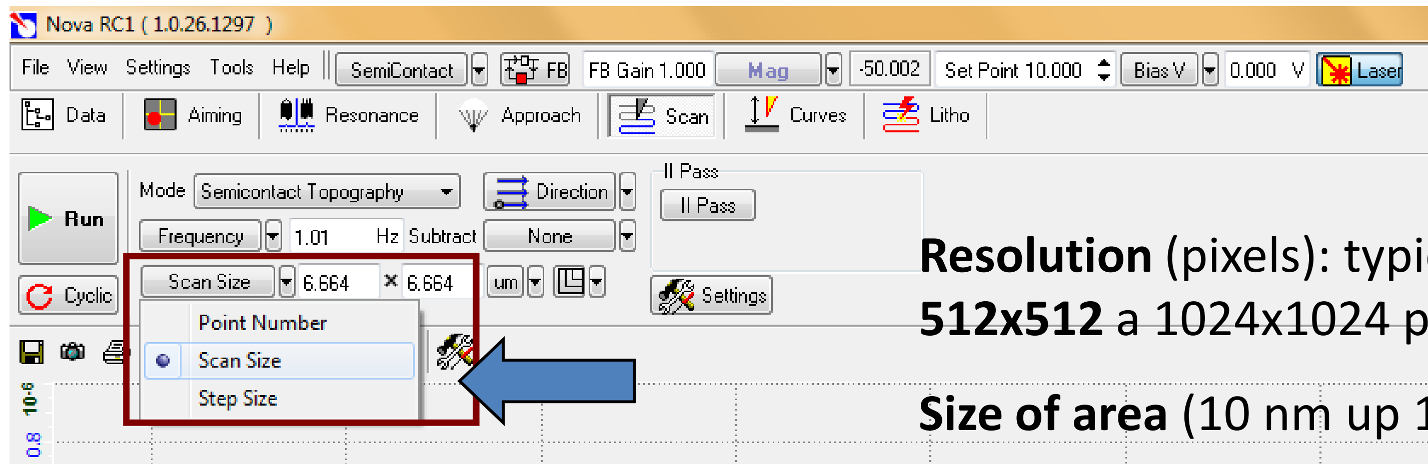
Mag, nA

t, s

3. **SCANNING OF SAMPLE:** parameters driven by **PZT**



Velocity of scanning (typically **0.35 – 0.7 Hz**)



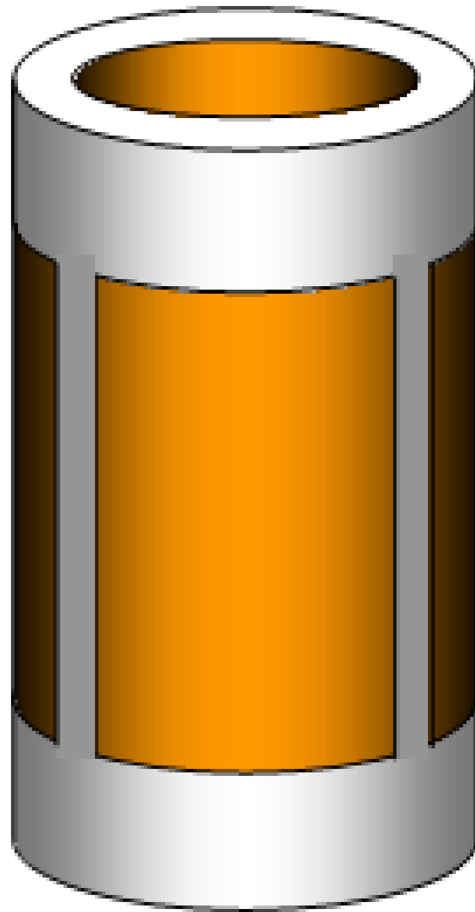
Resolution (pixels): typically 128x128; 256x256, **512x512** a 1024x1024 pix.

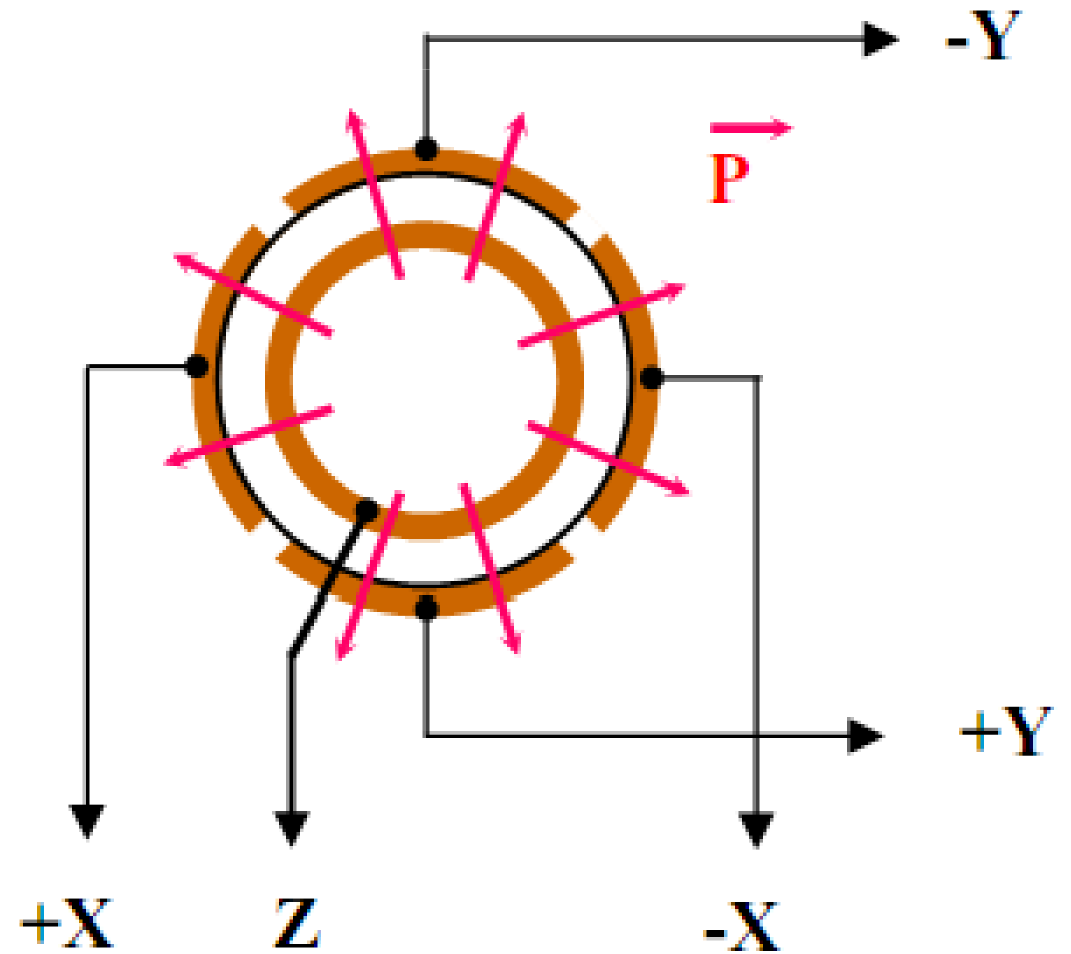
Size of area (10 nm up 150 um)

Step of scanning

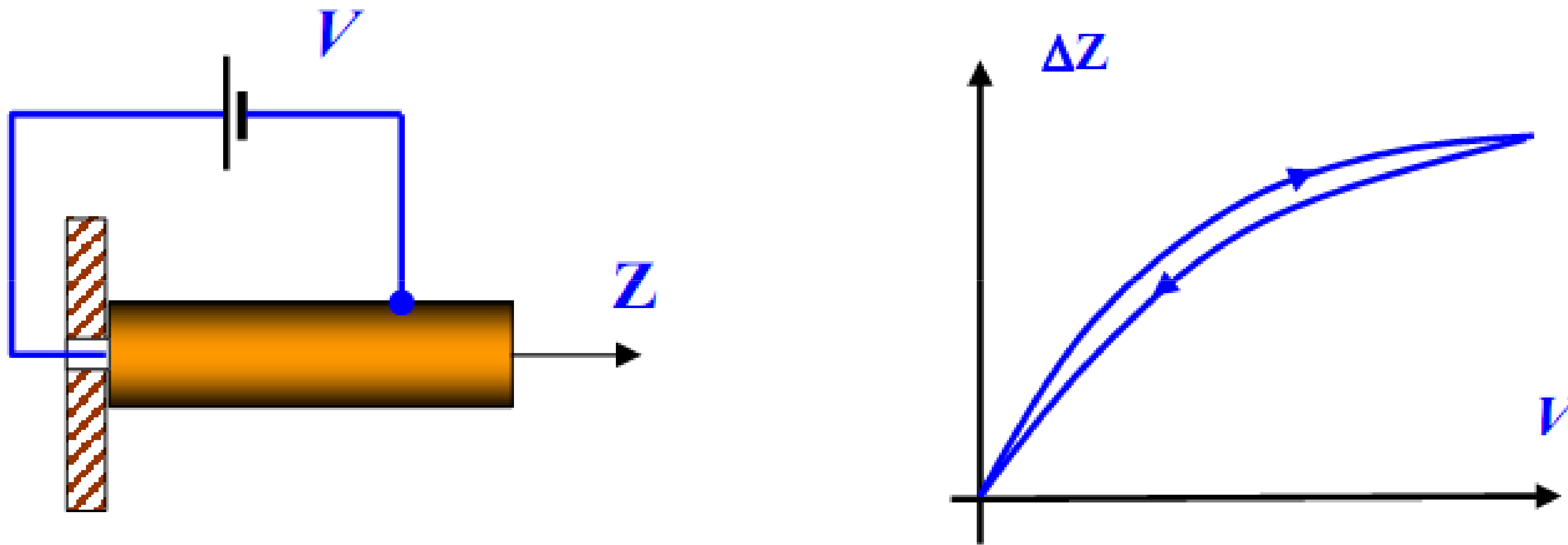
PZT electrodes

Detailed view

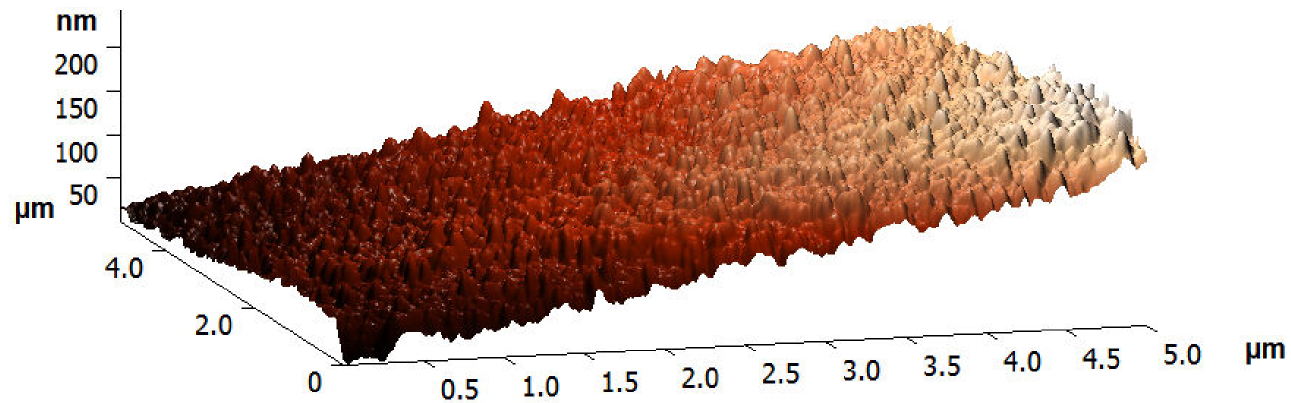




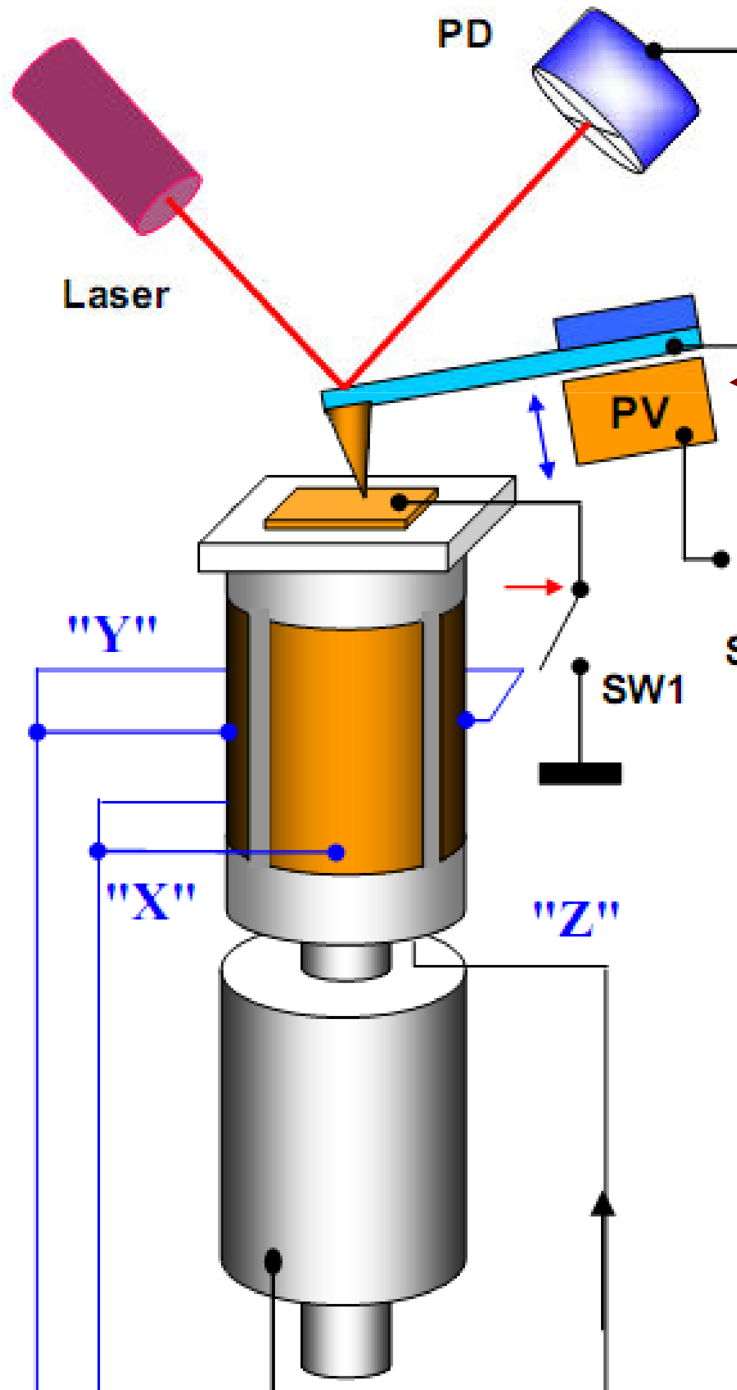
PZT: voltage-extension dependency



Native (raw) AFM **data** are **shifted**.
Removed e.g. by polynomal regression of data.

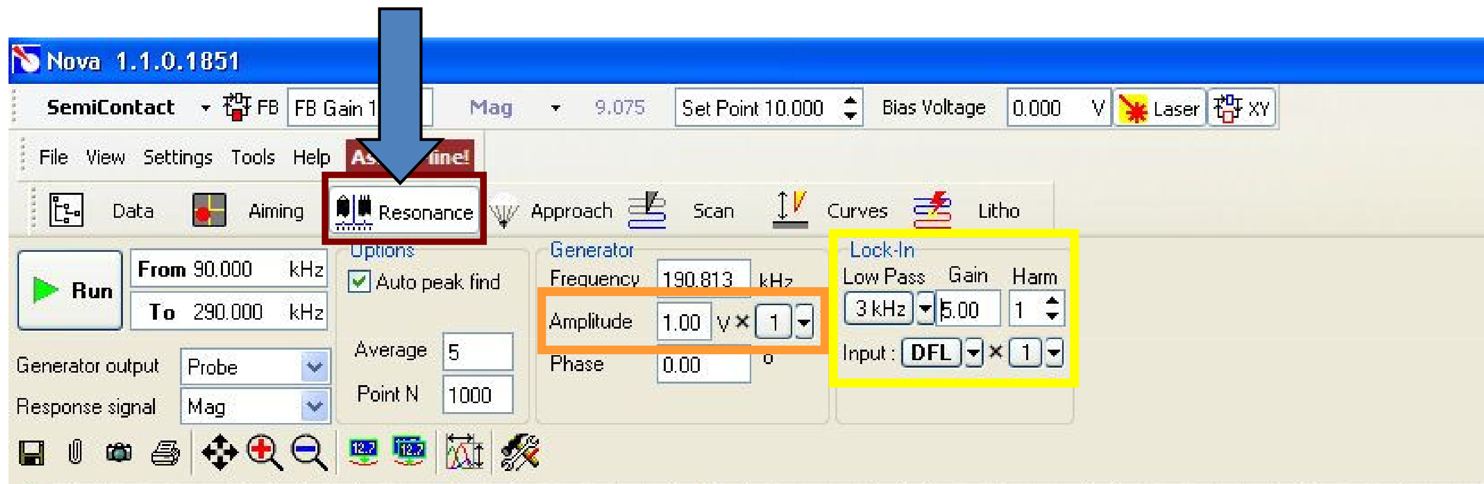


Oscillator



Oscillator(PV, PiezoVibrations) always located in the head

Oscillator setting



Amplitude = voltage [V]

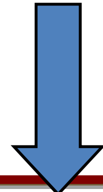
Gain = Lock-In amplifier

DFL x = numerical amplifier

Other components

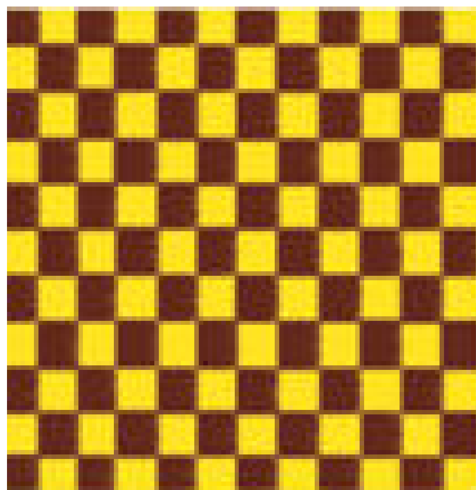
ClosedLoop *(X, Y - axis)*

ClosedLoop

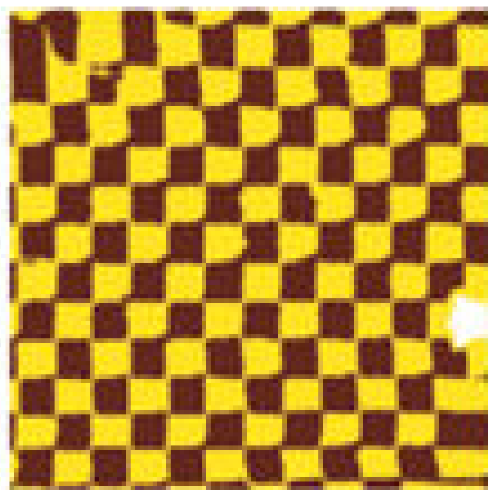


Capacitance sensors

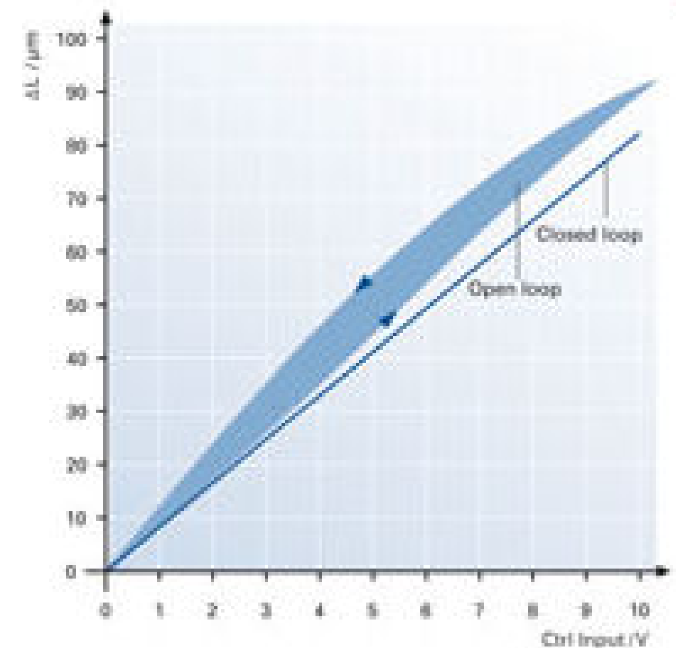
- Temperature drift correction
- PZT non-linearity correction
- Increasing noise



Closed Loop



Open Loop



Step motors

•Help to drive sample in the appropriate area of PZT action

•Driven automatically / manually