STRUCTURAL AND SURFACE ANALYSIS
ATOMIC FORCE MICROSCOPY

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ATOMIC FORCE MICROSCOPY

- Aim:

acquisition of three-dimensional, spatially localized images revealing structure and properties of investigated material

- belongs to the group of Scanning Probe Microscopy (SPM) techniques
- beginning dates back to the 80s of the XXth century
- 1982 - Gerd Binning, Heinrich Rohrer – invention of Scanning Tunnelling Microscopy (STM) – Nobel Prize in physics (1986)
- 1986 – Gerd Binning, Calvin Quate, Christopher Gerber – Atomic Force Microscopy (AFM)
- obtained image is not optical, but it results from interaction between the probe (tip) and the investigated surface
- the technique can be applied to conductive as well as non-conductive materials
- nanotechnological tool, which allows obtaining the images with atomic resolution (in advantageous conditions)

TYPES OF ATOMIC INTERACTIONS

- short-range interactions

- **van der Waals forces** responsible for formation of solids or the phenomenon of wetting the surface of solids by liquids

- within the distance of few nanometres these forces are capable of moving macroscopical objects, for instance probe of the AFM

- the main components of van der Waals forces:
- polarization connected with permanent dipoles,
- induced dipoles

TYPES OF ATOMIC INTERACTIONS

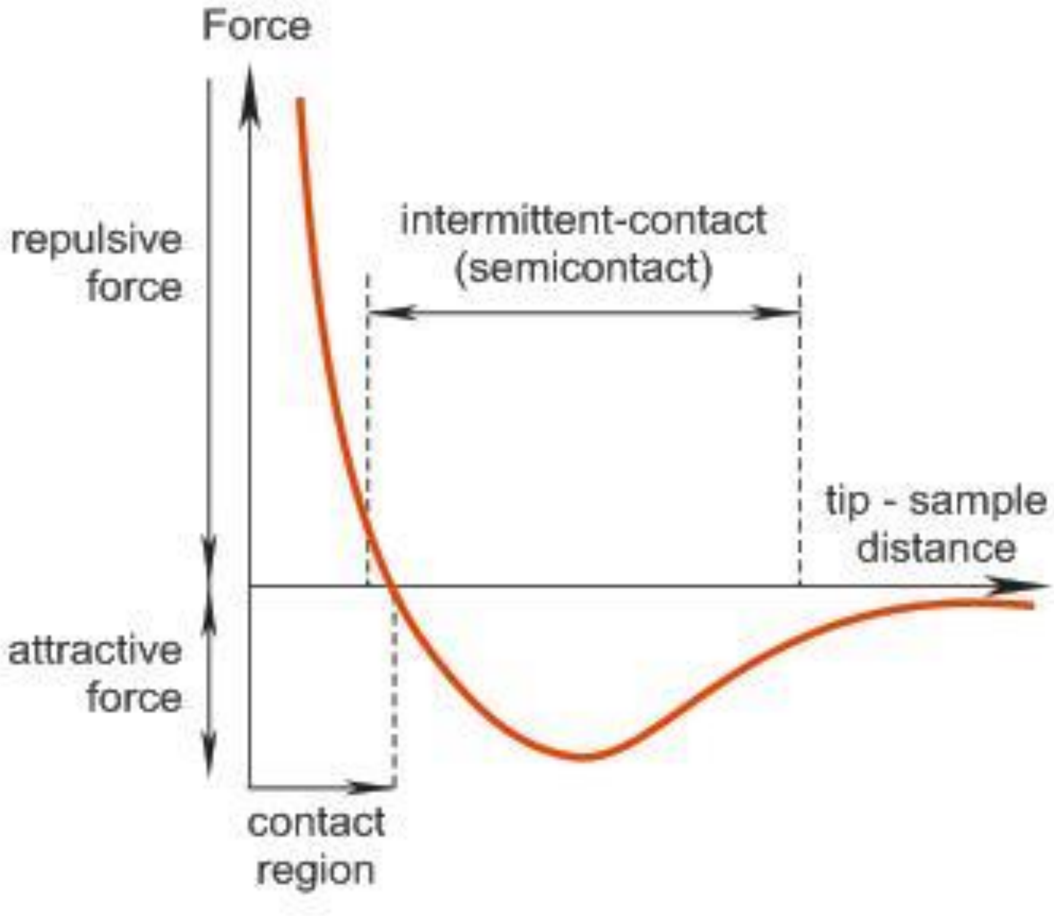
Distance [nm]	**1**	**10**	**100**
Force [N]	10^{-9}	10^{-11}	10^{-13}
detection threshold in the AFM technique: 10^{-18} N, easily measurable: 10^{-15} N			

Tab. 1 Van der Waals forces between two bodies versus distance between them

- long-range interactions

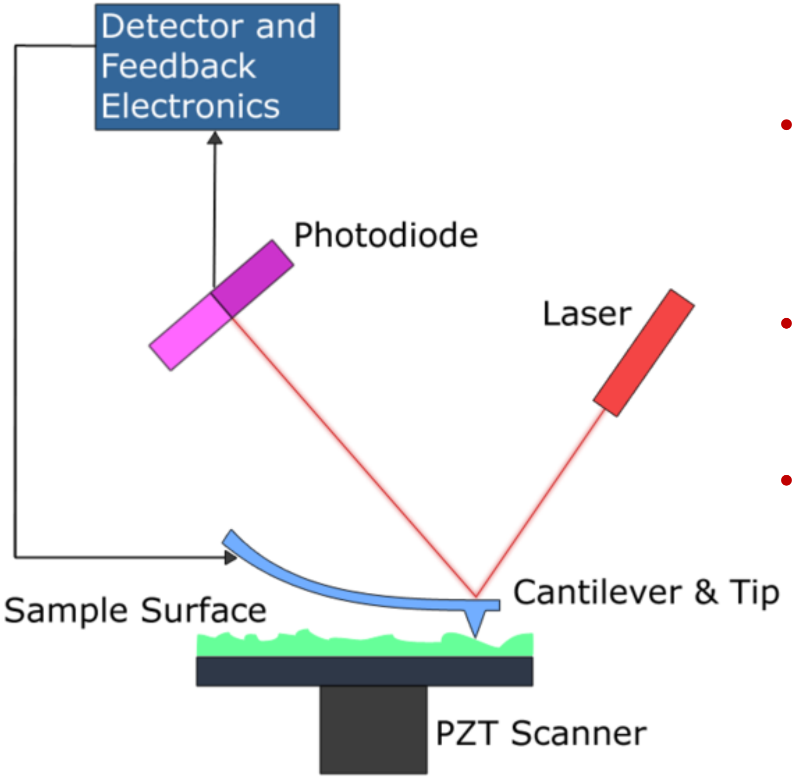
- attractive or repulsive electrostatic forces, magnetic forces,
- detection of particular types of forces requires application of suitable tip (for example coated with platinum or cobalt alloys)

TYPES OF ATOMIC INTERACTIONS



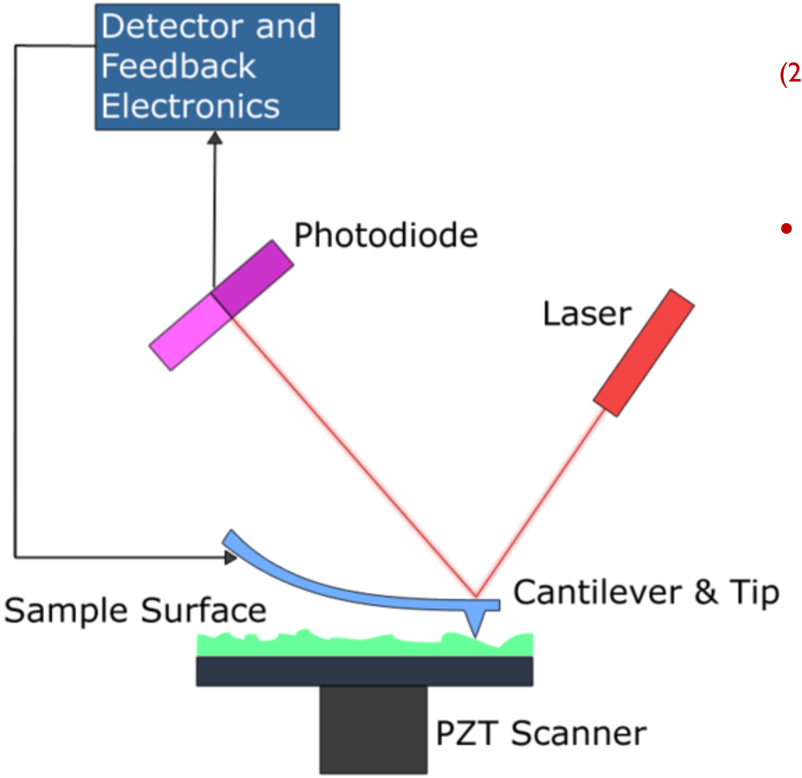
Interaction between two bodies versus distance between them

ATOMIC FORCE MICROSCOPE



- is comprised of a cantilever made of silicon or silicon nitride terminated with a tip, which is used to scan the surface of investigated material
- motion of the tip versus the surface results in varying magnitude of atomic forces acting on the tip, which causes bending of the cantilever
- typically, bending of the cantilever is measured with a laser beam reflected from the top of it and directed to a photodiode detector

ATOMIC FORCE MICROSCOPE

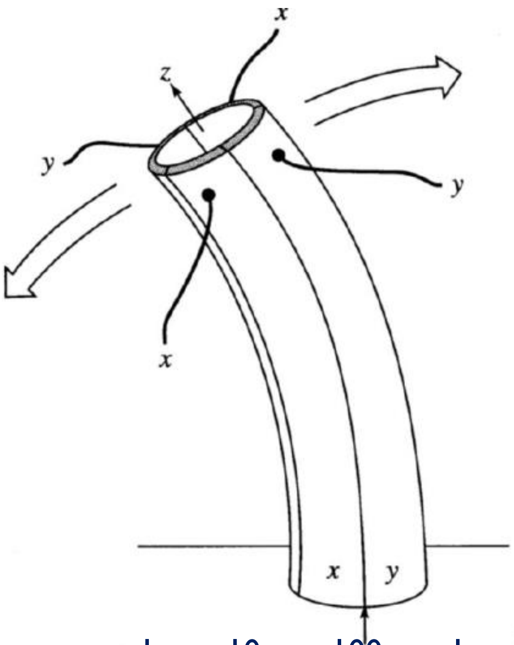


- SCANNING MODES:

(1) scanning by sample – sample moves in x and y directions with respect to the immobile tip

(2) scanning by tip – tip moves in x and y directions with respect to the immobile sample

- precise scanning motion is provided by piezoelectric element



- typical scanning ranges: 1 µm, 10 µm, 100µm along horizontal plane

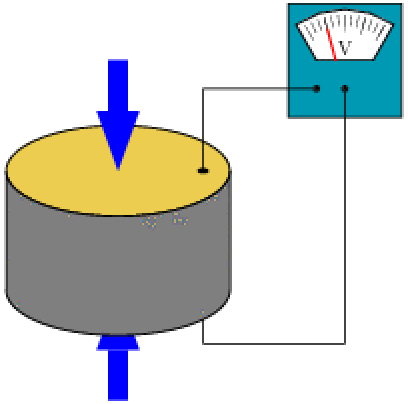
PIEZOELECTRICITY

- generation of voltage by the elements subjected to mechanical compression or tension

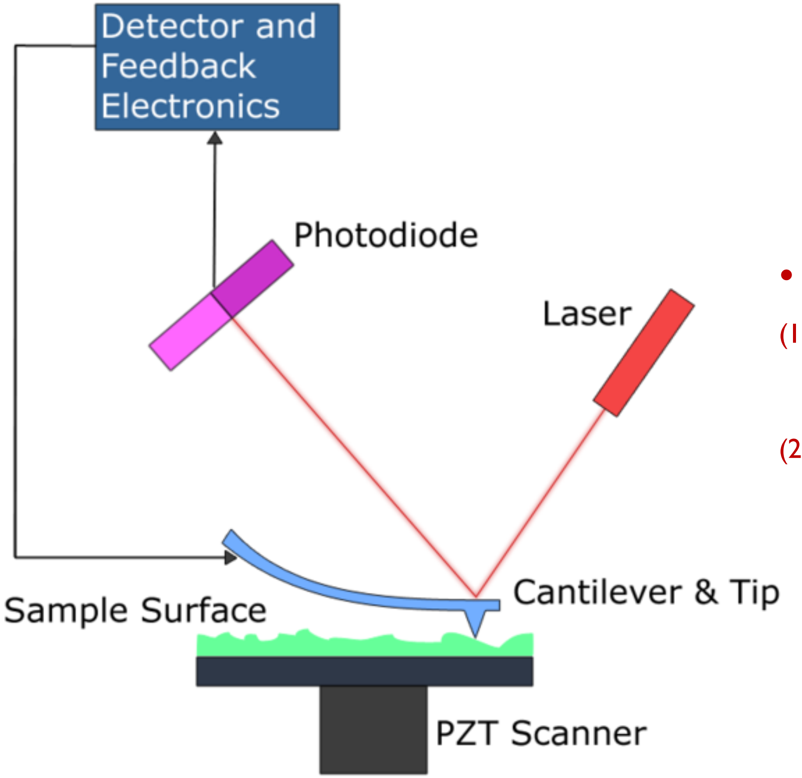
- dimensional changes of these elements due to applied voltage

- magnitude of generated voltage is proportional to the magnitude of force applied

- reversible phenomenon



ATOMIC FORCE MICROSCOPE

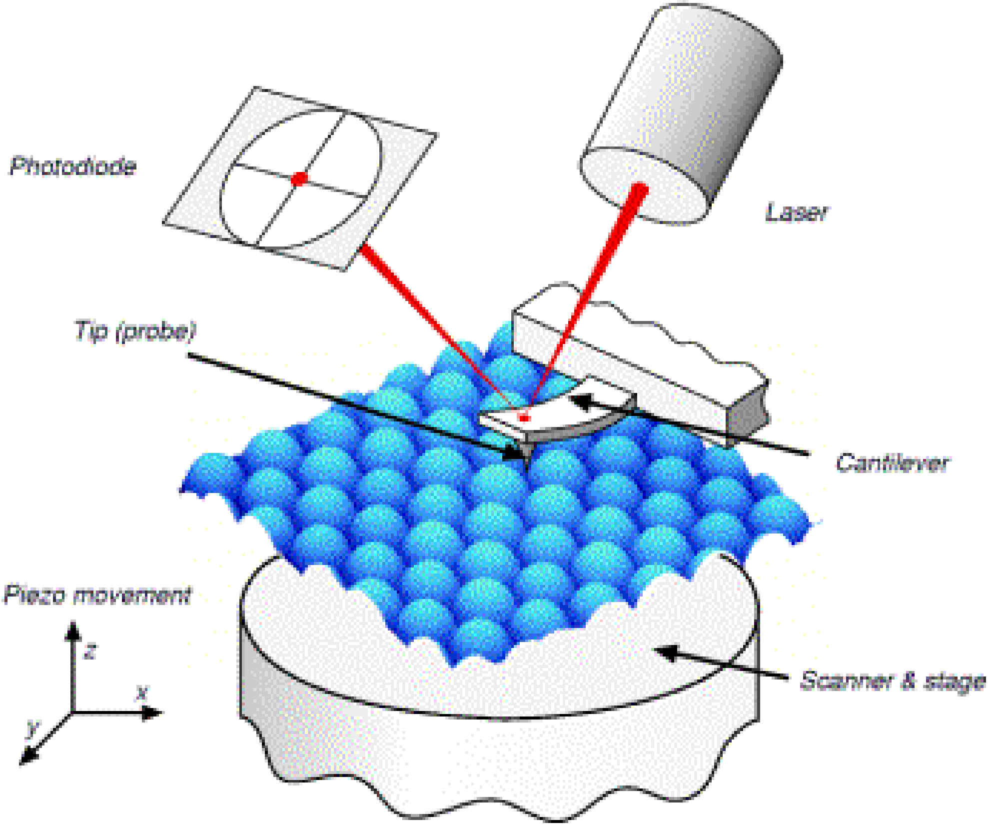


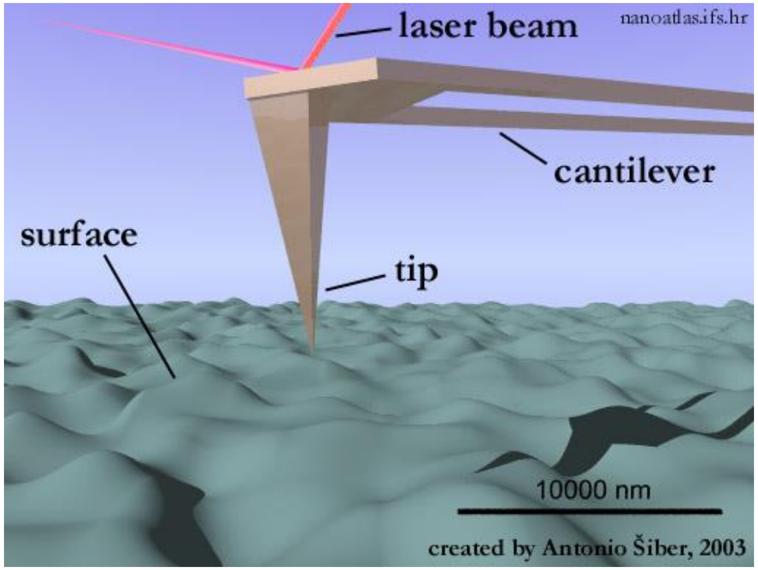
- MEASUREMENT MODE:

(1) contact – the tip remains in contact with the investigated surface, application: hard materials (for example metals, ceramics)

(2) non-contact – the tip moves within a small distance from the surface, application: soft materials (for example polymers, biological films)

ATOMIC FORCE MICROSCOPE

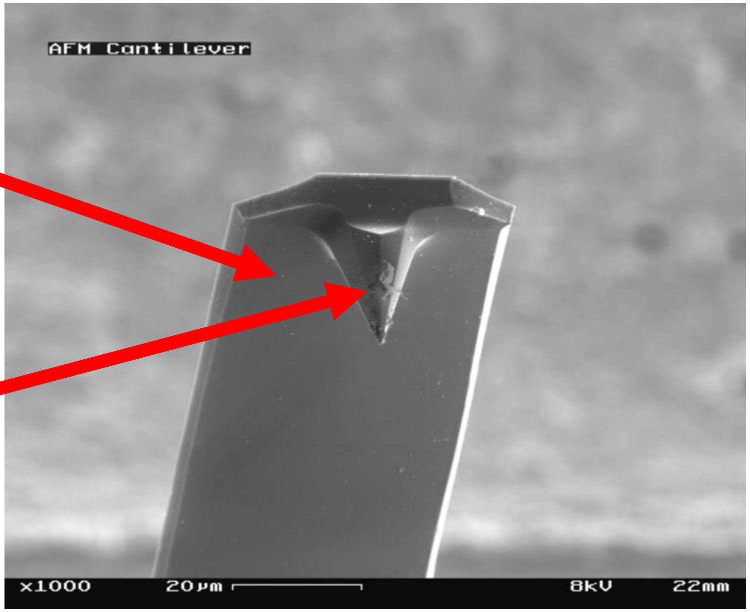


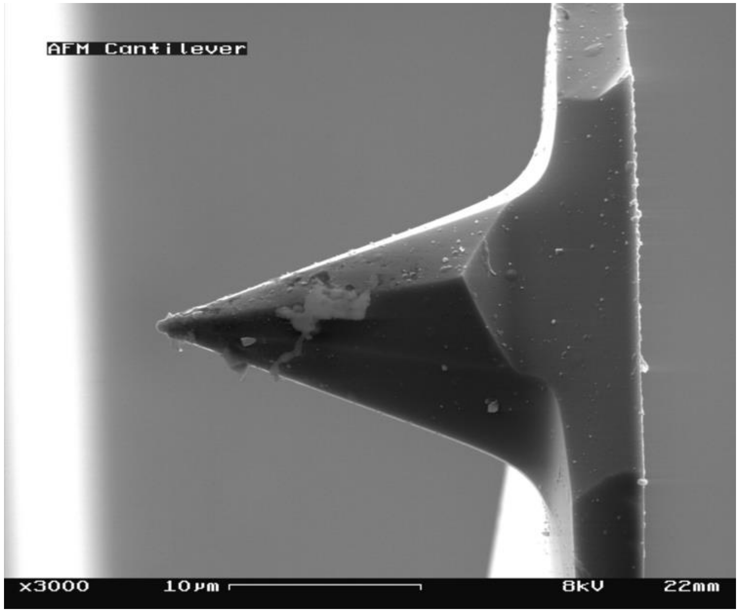


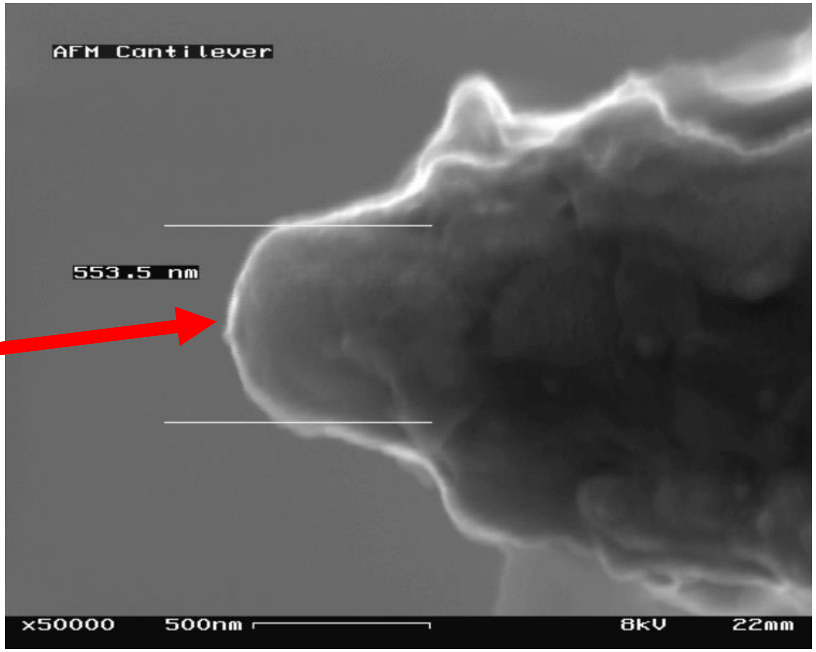
Surface imaging by the AFM technique via optical detection of cantilever bending

Cantilever
100-500µm

Tip

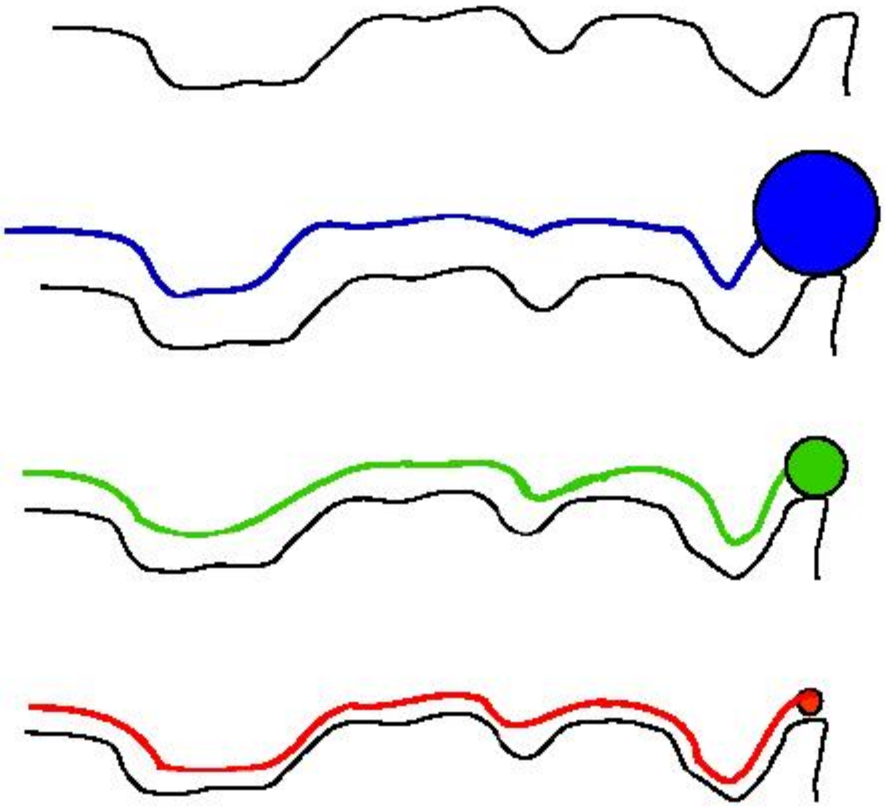






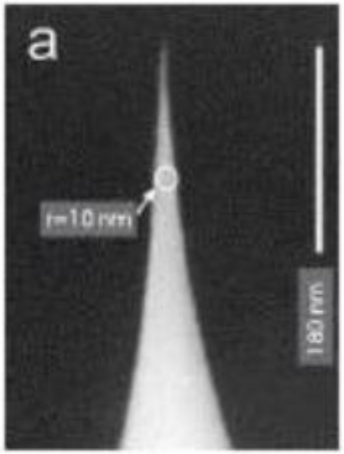
Tip curvature radius
(typically 10-20nm)

ATOMIC FORCE MICROSCOPE

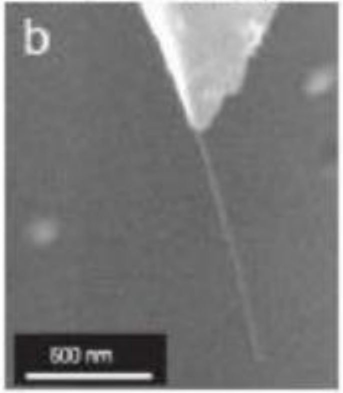


- suitable ratio of tip height to tip width (so-called aspect ratio) provides accuracy of surface imaging
- it is especially important in case of rough surfaces with significant differences in height profile

ultrasharp tip



nanotube tip



ATOMIC FORCE MICROSCOPE

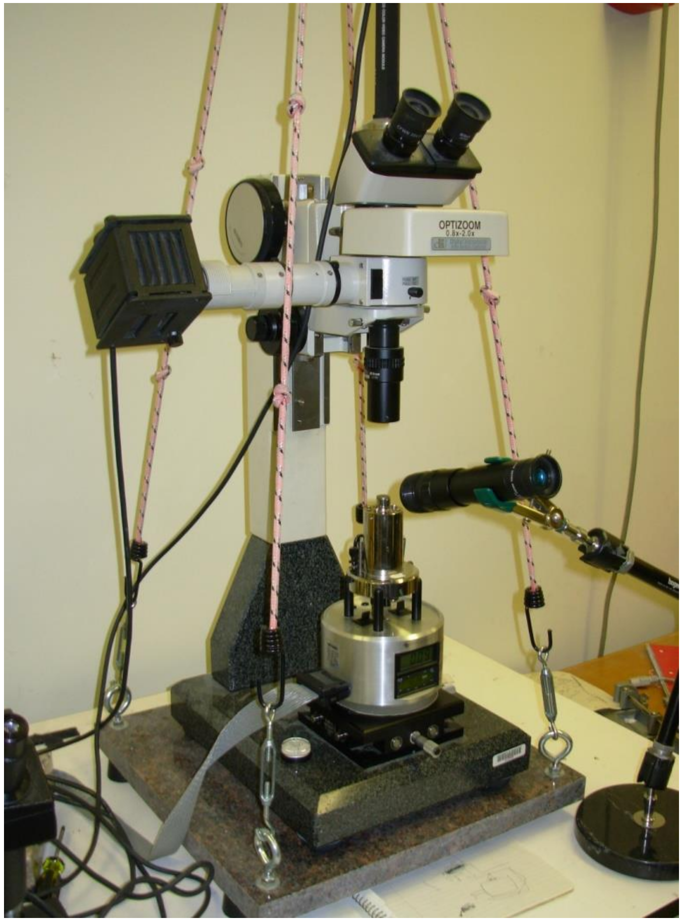
All systems of atomic force microscopy consist of five basic elements:

- tip,
- scanner,
- cantilever bending detection system,
- controller,
- anti-vibration system.

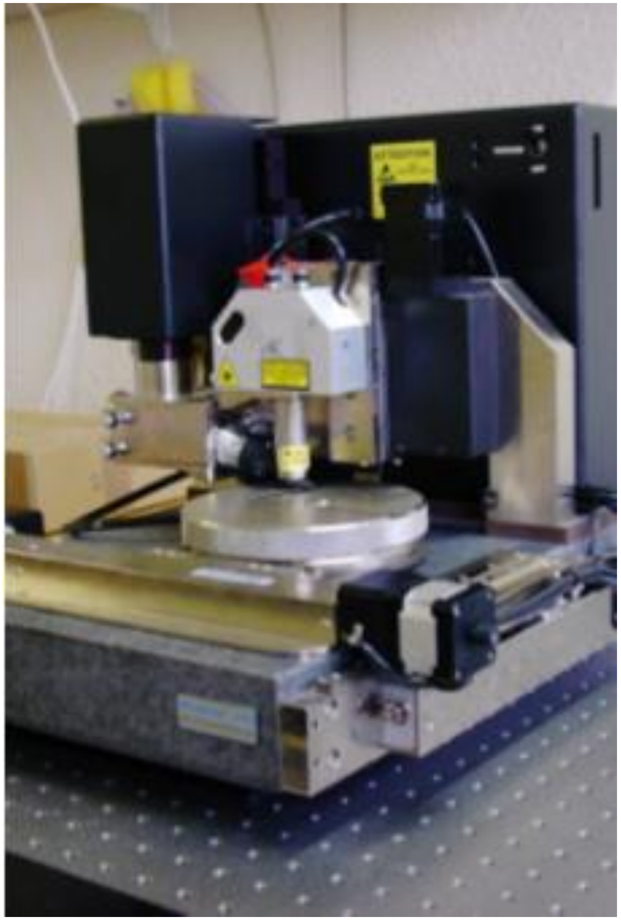
ATOMIC FORCE MICROSCOPE

Isolation from vibration

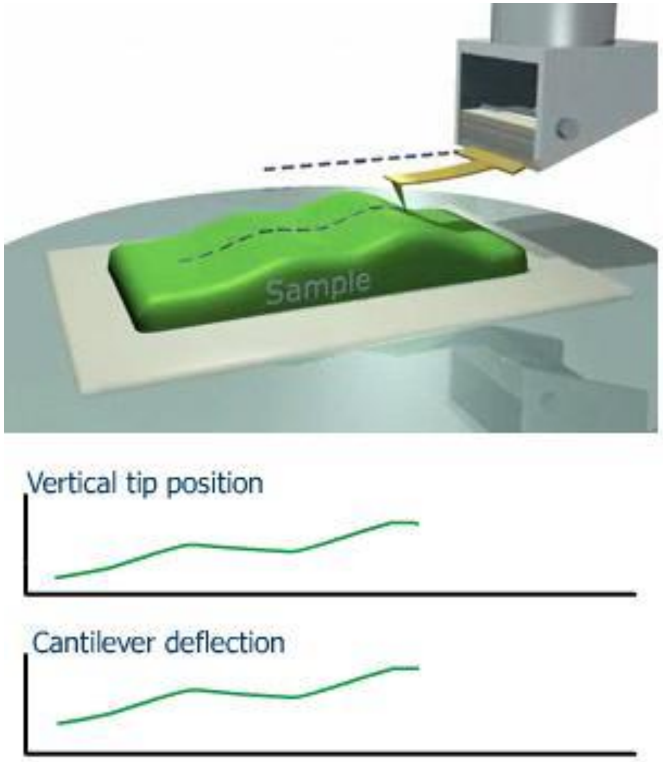
Suspended table



Anti-vibration table



MEASUREMENT MODES



- **CONSTANT HEIGHT MODE**:

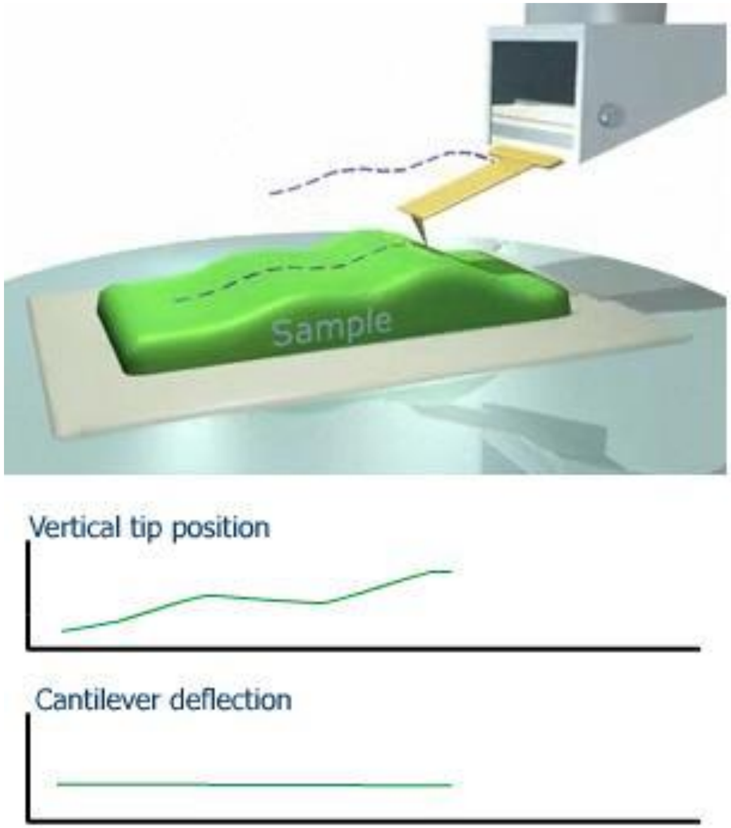
(1) a scanner is kept at constant height above the surface during measurement

(2) cantilever deflection during tip motion is used to reconstruct surface topography profile of the sample

(3) high scan rates possible

(4) problems with measurement on the surfaces with significant roughness

MEASUREMENT MODES



- **CONSTANT FORCE MODE**:

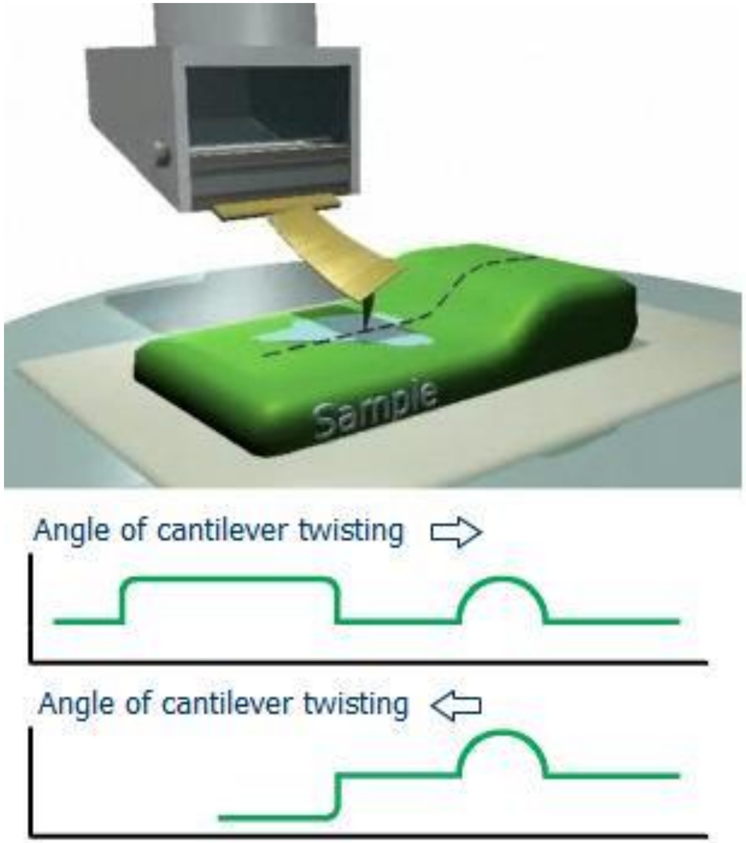
(1) constant cantilever deflection is maintained during measurement

(2) constant cantilever deflection is maintained by vertical motions of the scanner

(3) vertical positioning of the scanner is used to reconstruct surface topography profile of the sample

(4) limited scan rate

MEASUREMENT MODES



- **LATERAL FORCE IMAGING**:

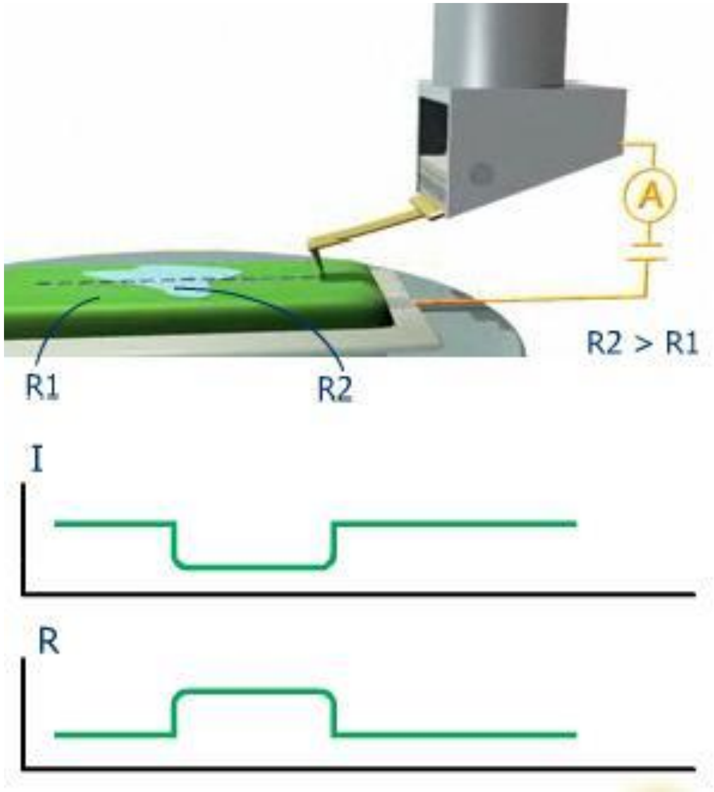
(1) due to friction forces the cantilever with tip undergoes twisting during measurement

(2) possibility of detection of the domains differing in coefficient of friction (not necessarily differing in topography)

(3) obtaining images with better resolved edges, corners and abrupt changes of profile

(4) application: polymers, semiconductors, data storage devices, deposits, identification of surface contaminants

MEASUREMENT MODES



- **SPREADING RESISTANCE IMAGING**

(1) application of bias voltage between the tip and the investigated sample; then measurement of flowing direct current

(2) possibility of detection of the domains differing in conductivity

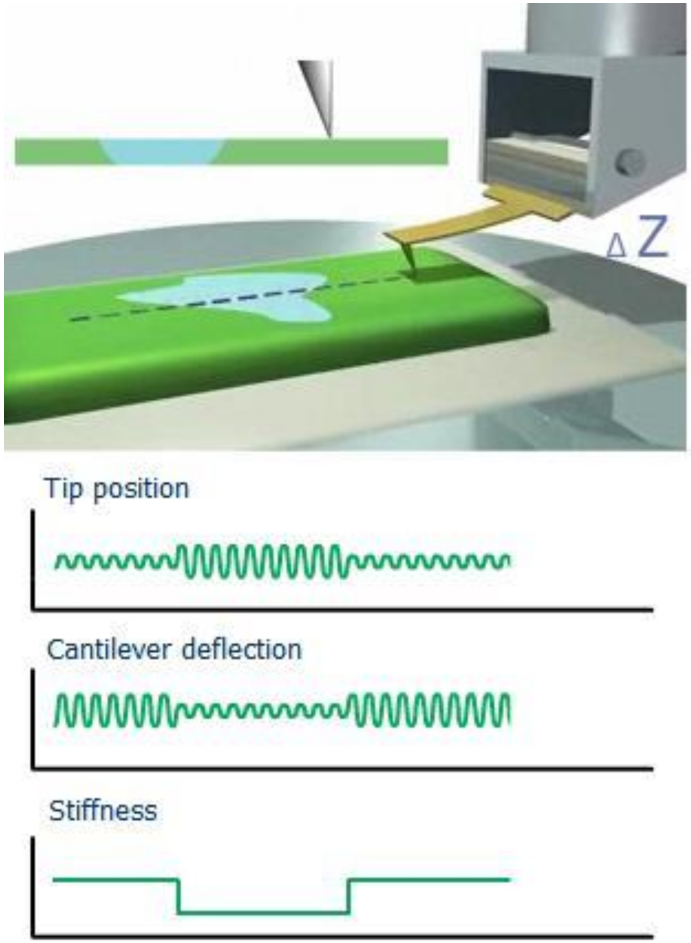
(3) the measurements must be carried out in contact mode

(4) the tip must be coated with platinum layer or doped with diamond to provide conductivity

(5) reliable results require appropriate contact force between the tip and the surface

(6) application: quality control of electronic printed boards

MEASUREMENT MODES



- **FORCE MODULATION MODE**:

(1) the cantilever with tip oscillates along vertical axis

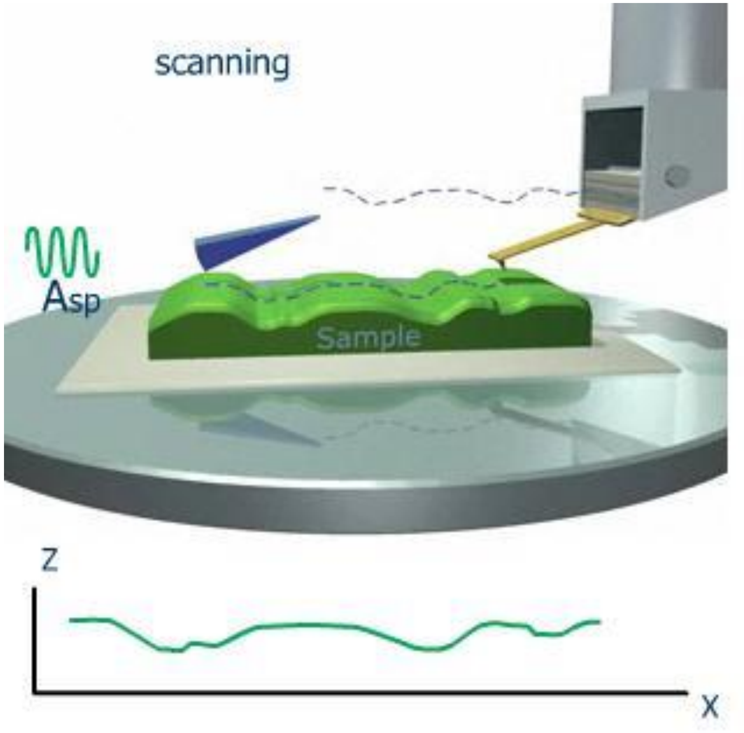
(2) during scanning the tip penetrates the surface to a depth depending on material's hardness

(3) oscillation amplitude and cantilever deflection change

(4) possibility of determination of local rigidity, elasticity and microhardness of particular domains on investigated surface

(5) application: polymers, semiconductors, biological materials, composite materials

MEASUREMENT MODES



- **NON-CONTACT MODE**:

(1) the cantilever with tip oscillates **above** investigated surface

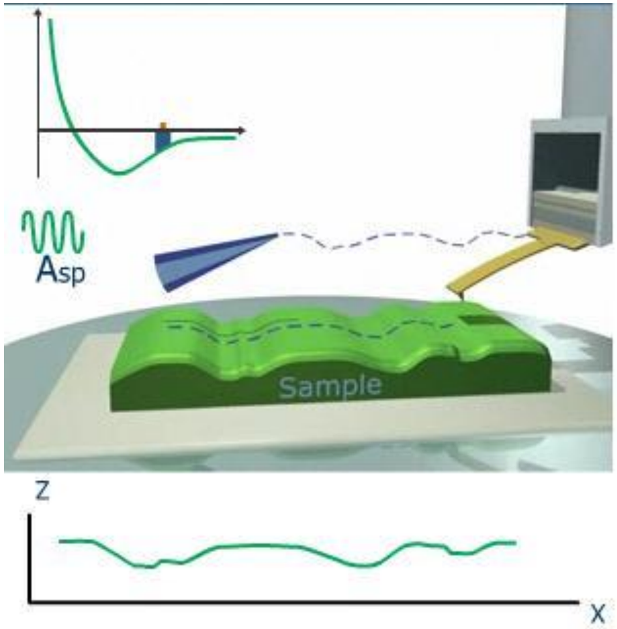
(2) the cantilever oscillates with resonant frequency (3-500Hz)

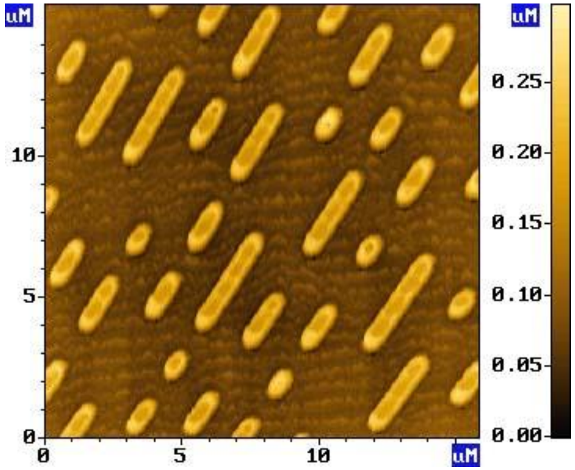
(3) when passing above particular regions the frequency of oscillation changes due to atomic forces impact

(4) change in the oscillation frequency is used to reconstruct topographic image

(5) application: biological materials, soft samples, which can be damaged while measured in contact mode

MEASUREMENT MODES





- **NON-CONTACT MODE – in long-range interaction distance**:

(1) the cantilever with tip oscillates **above** investigated surface in the **distance**, which is **far enough** to expose the tip to long-range forces

(2) the cantilever oscillates with resonant frequency

(3) when passing above particular regions the frequency of oscillation changes due to long-range atomic forces impact

(4) change in the oscillation frequency is used to reconstruct topographic image

(5) in practice the measurement involves two passes: **(1)** topographic image is acquired when passing in direct vicinity of the surface, **(2)** profile connected with long-range interaction is acquired when passing at a certain distance from the surface

(6) application: materials with magnetic domains, for example data storage devices

MEASUREMENT MODES

- **LITHOGRAPHY**:

(1) surface modification via interaction with the tip

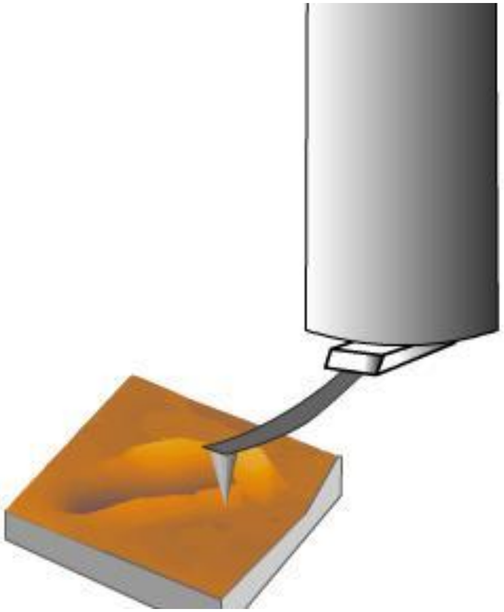
(2) allows creation of a desired pattern on the surface

(3) requires application of hard tips depending on the material to be grooved

TYPES OF LITHOGRAPHY:

- stylus
- pinhole
- oxidation

MEASUREMENT MODES

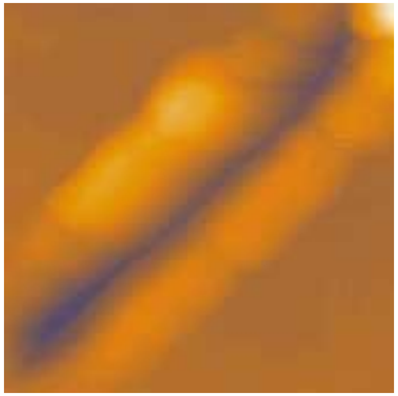


- **STYLUS LITHOGRAPHY**:

(1) scratching the material with hard tip

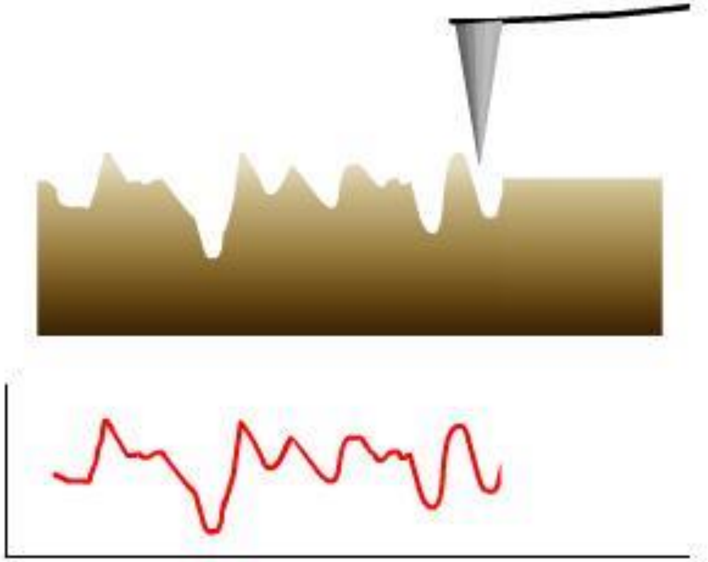
(2) usually performed on polymeric materials

(3) requires sufficient contact force to make the tip penetrate the material



1.6 x 1.6 μm

MEASUREMENT MODES



- **PINHOLE LITHOGRAPHY**:

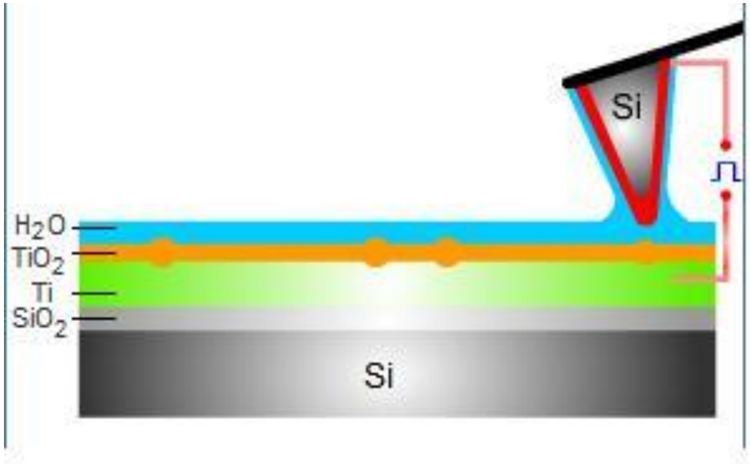
(1) indentation of the material with hard tip

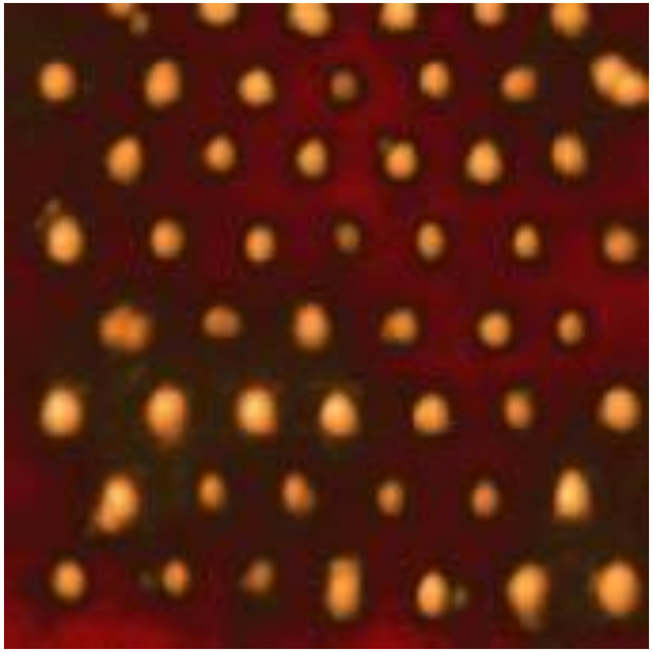
(2) typically performed on polymeric materials

(3) calls for sufficient amplitude of tip oscillation, which ensures tip penetration down the material

2.5 x 2.5 µm

MEASUREMENT MODES





200 x 200 nm

- **OXIDATION LITHOGRAPHY**:

(1) enforcing electrochemical reaction on the surface of the material, in the place of tip positioning

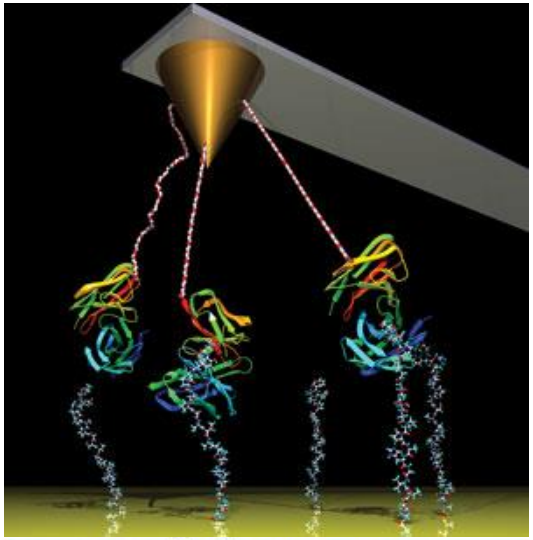
(2) the reaction is enforced by application of proper bias voltage between the tip and the scanned material

(3) presence of electrolyte is necessary

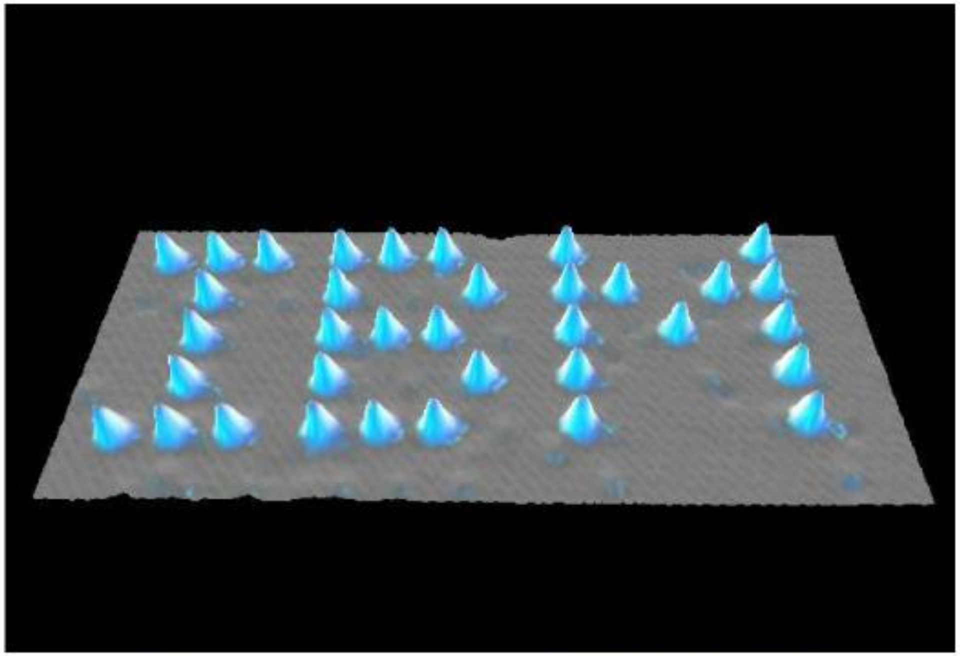
(4) applicable for the surfaces where electrochemical reaction can occur, for instance metals and alloys

Titanium oxide dots on the surface of metallic titanium

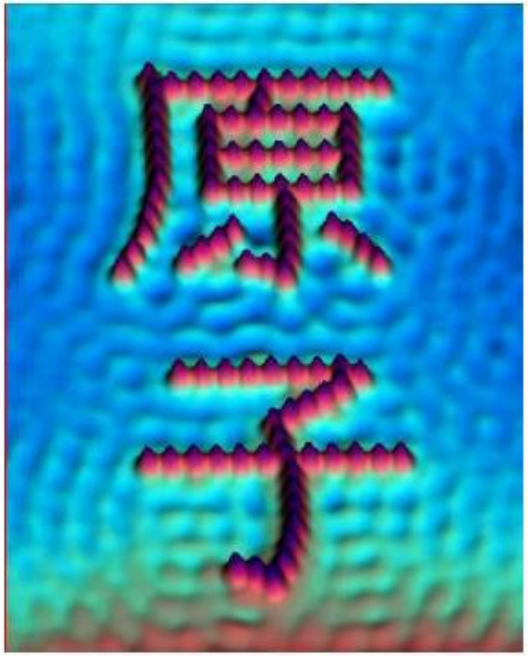
NANOGRAPHICS



- transfer („curling”) and positioning of individual atoms using the AFM tip



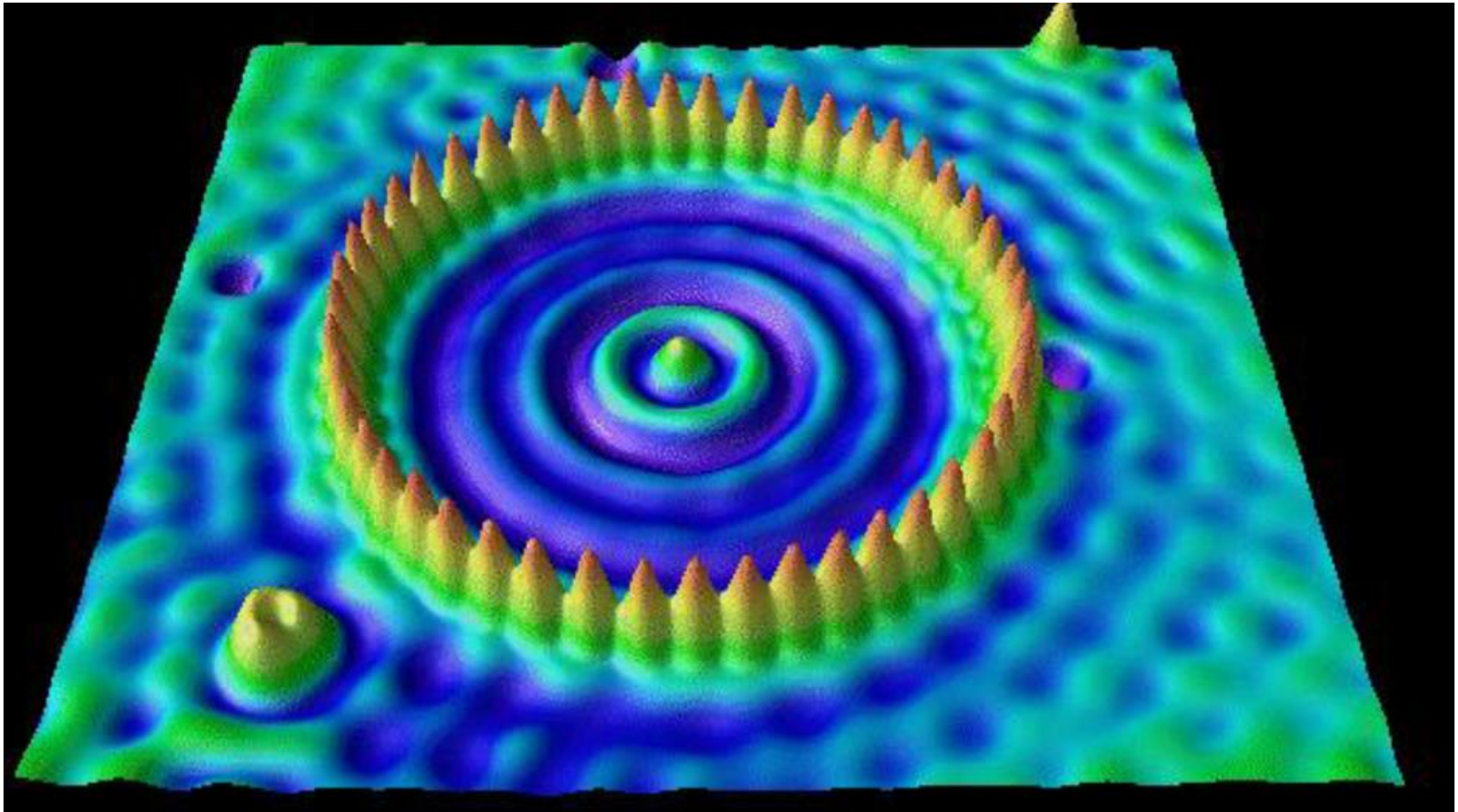
Xenon atoms on Nickel (110)



Fe atoms on Cu(111)

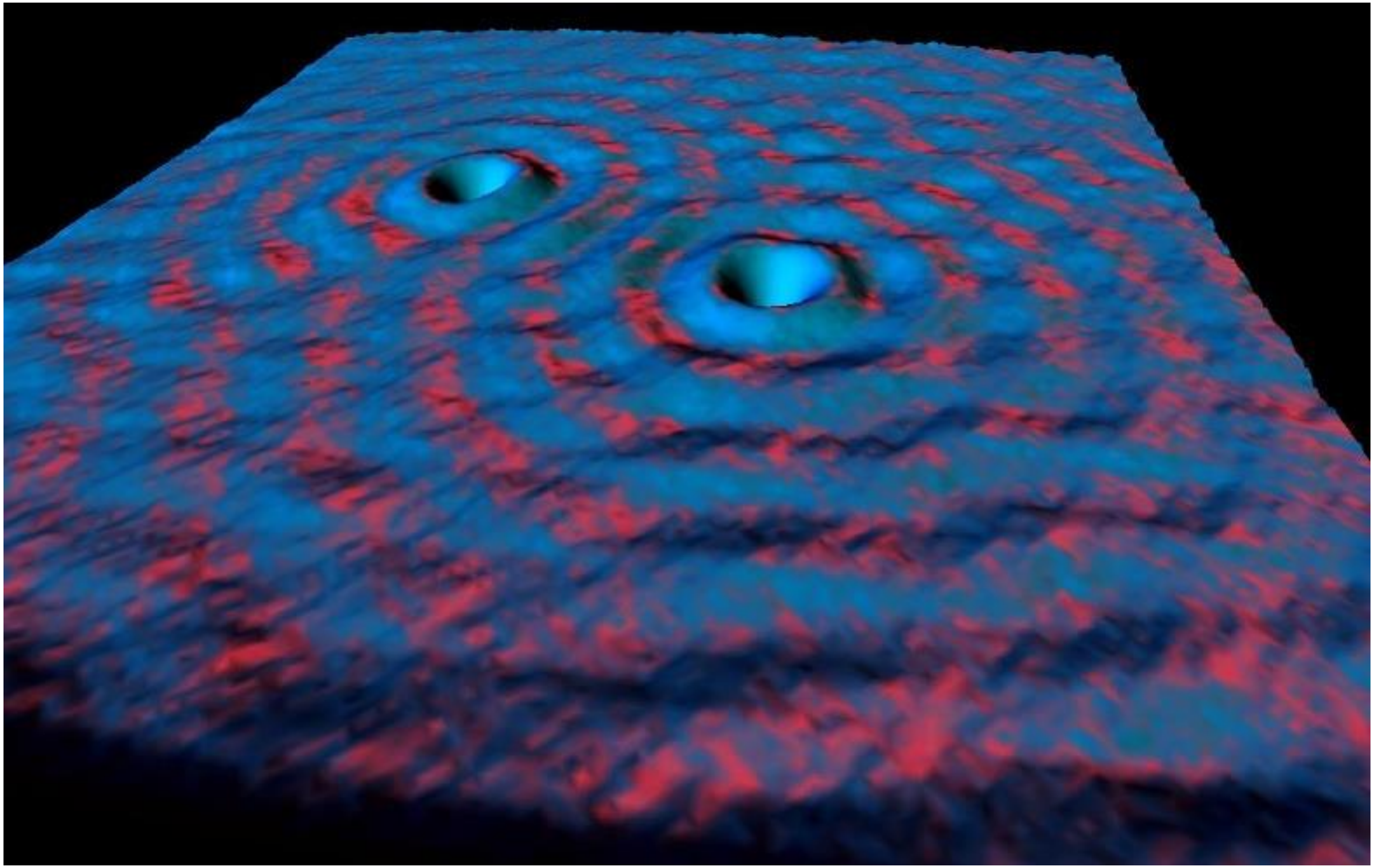
Don Eigler, IBM Alamden, http://www.almaden.ibm.com/vis/stm/atomo.html

NANOGRAPHICS



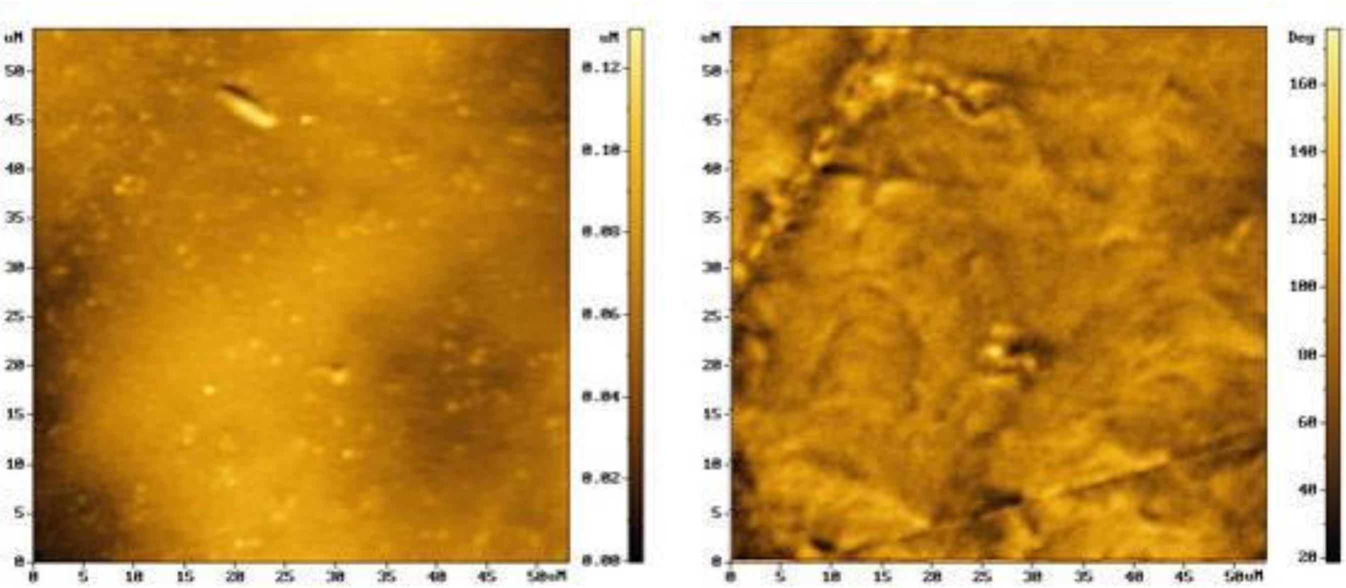
Iron atoms on the surface of Cu(111)

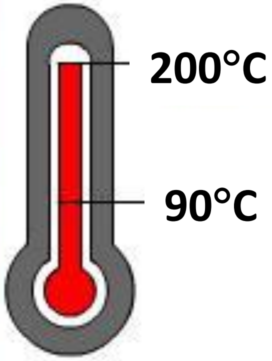
NANOGRAPHICS



MEASUREMENT AT VARIOUS TEMPERATURE

- requires application of dedicated heated stages and/or temperature chambers
- elements of the microscope must be resistant to elevated temperature
- investigation of the changes on oxide layers and alloys
- investigation of glass transition temperature of polymers
- observation of precipitation phenomena in solutions





Images of polymer surface below and above glass transition temperature

ADVANTAGES OF ATOMIC FORCE MICROSCOPY

- modern technique providing insight into surface properties of the material in micro- and nanoscale, inaccessible via the conventional methods
- a result of measurement is an image of high resolution – in advantageous conditions even of atomic resolution
- wide range of the measurement modes providing universal character of the method
- measurement of not only topography but several other physical and electrical quantities (hardness, coefficient of friction, conductivity, magnetic properties)
- measurements can be carried out in controlled atmosphere (vacuum, inert gas, solution) and temperature
- possibility of monitoring changes of the surface versus time
- very useful tool for analysis of new materials, at the design or pre-implementation stages – verification of effectiveness of introduced modifications
- indispensible tool in developing field of nanotechnology (biotechnology, electronics, precision mechanics, failure analysis)

DISADVANTAGES OF ATOMIC FORCE MICROSCOPY

- provides information only about surface properties of the material, not about bulk features
- high cost of equipment, execution of the measurement calls for qualified personnel
- sensitive to interference (vibrations, noise, air movement, electromagnetic noise)
- image interpretation can be troublesome – no information about chemical character of the investigated surface unless suitable adds-on are applied, for instance XPS
- necessity of application of the supplementary techniques to confirm the results obtained
- difficulties in landing the tip in exactly the sample place for the second time (need for precise sample markers)
- limited to laboratory applications