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Course: M.Sc.
Semester: IV
Branch: Inorganic Chemistry
Paper: III
Unit: 2
Topic: Atomic Force Microscopy
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Atomic Force Microscopy

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History of AFM

- 1^{st} AFM was made by **Gerd Binnig** and **Cristopher Gerber** in **1985**.
- It was constructed by sticking tiny pieces of diamond onto one end of tiny strip of gold foil.
- Small hook at the end of the tip gets pressed against the sample surface.
- Sample scanned by tracking deflection of cantilever by monitoring tunneling current.

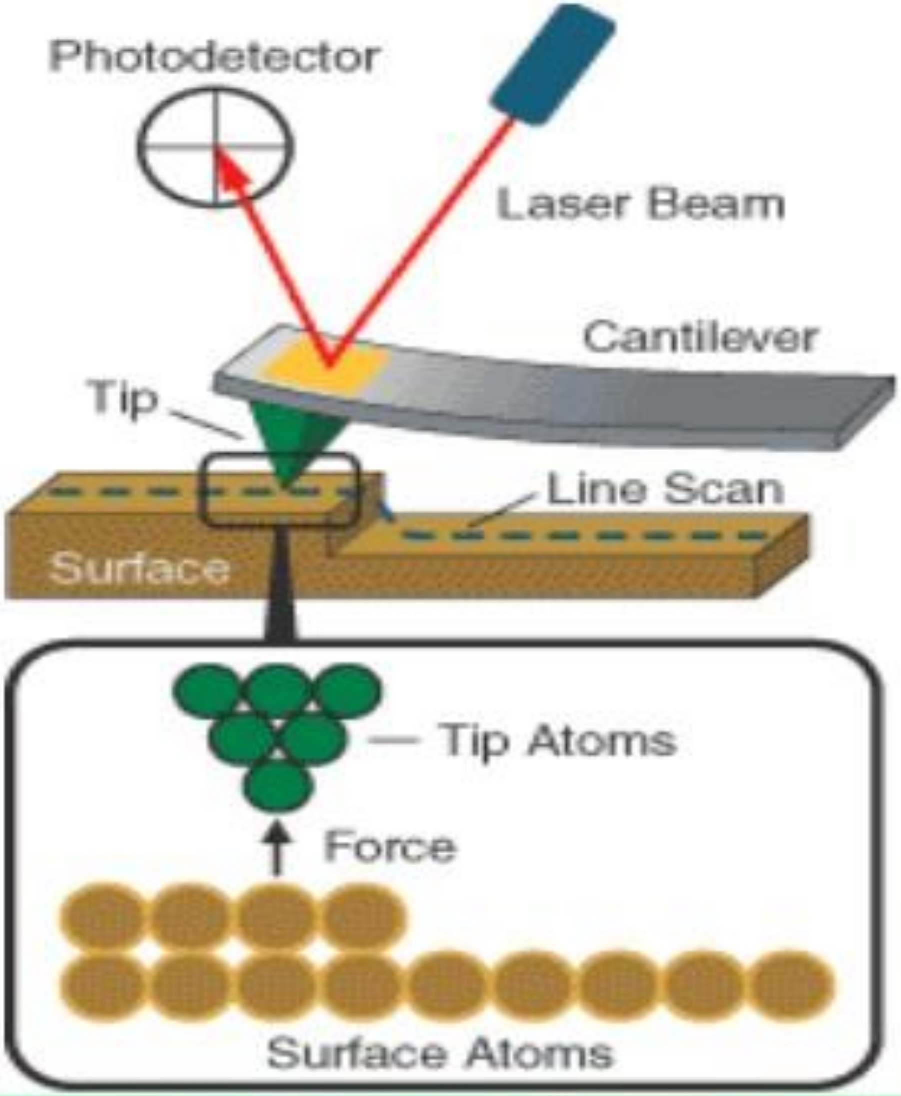
How does AFM work

- It belongs to the family of the **Scanning Probe Microscopy (SPM)**.
- In the SPM a sharp probe is scanned across a surface and some ***probe-sample*** interaction or interactions are monitored.
- The AFM senses inter-atomic forces that occur between a probe tip and a sample surface.
- So there is a certain kind of force existing between the atoms of probe and sample surface, hence the name AFM.

How does AFM Work

- AFM provides a **3D profile** of the surface on a nanoscale, by measuring ***forces*** *between a* ***sharp probe (<10 nm) and surface*** *at very short distance* ***(0.2-10 nm probe-sample separation)****.*
- *The probe is supported on a* ***flexible cantilever****.* The AFM tip “gently” touches the surface and records the small force between the probe and the surface.

How It Works

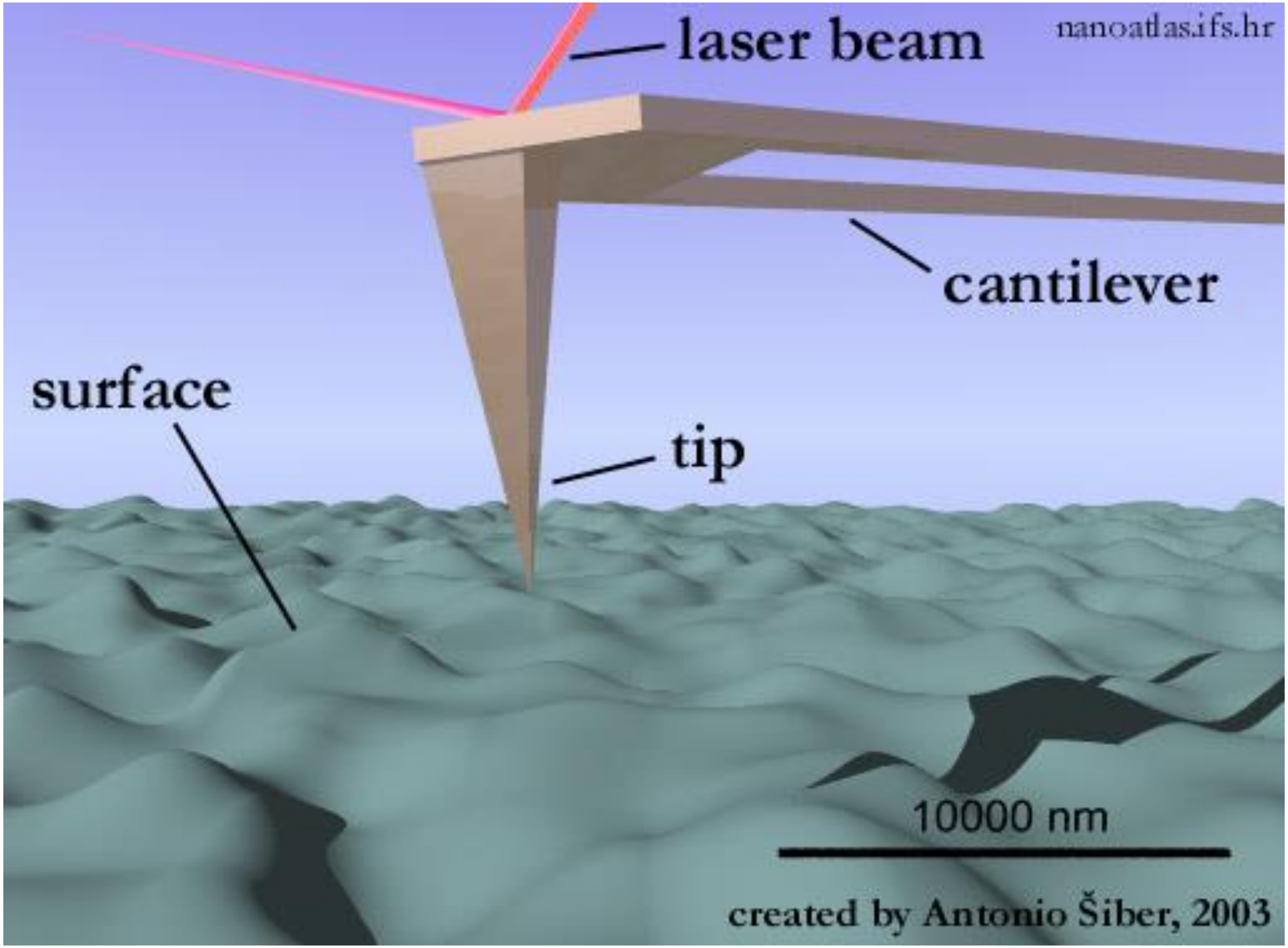


A Nanotechnology platform

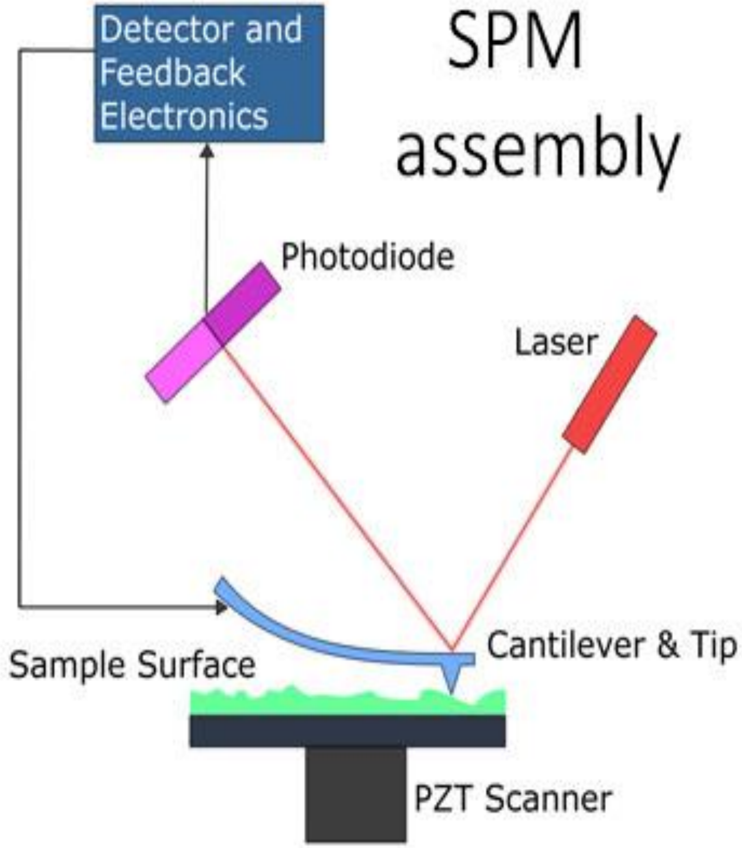
What are probes made of?

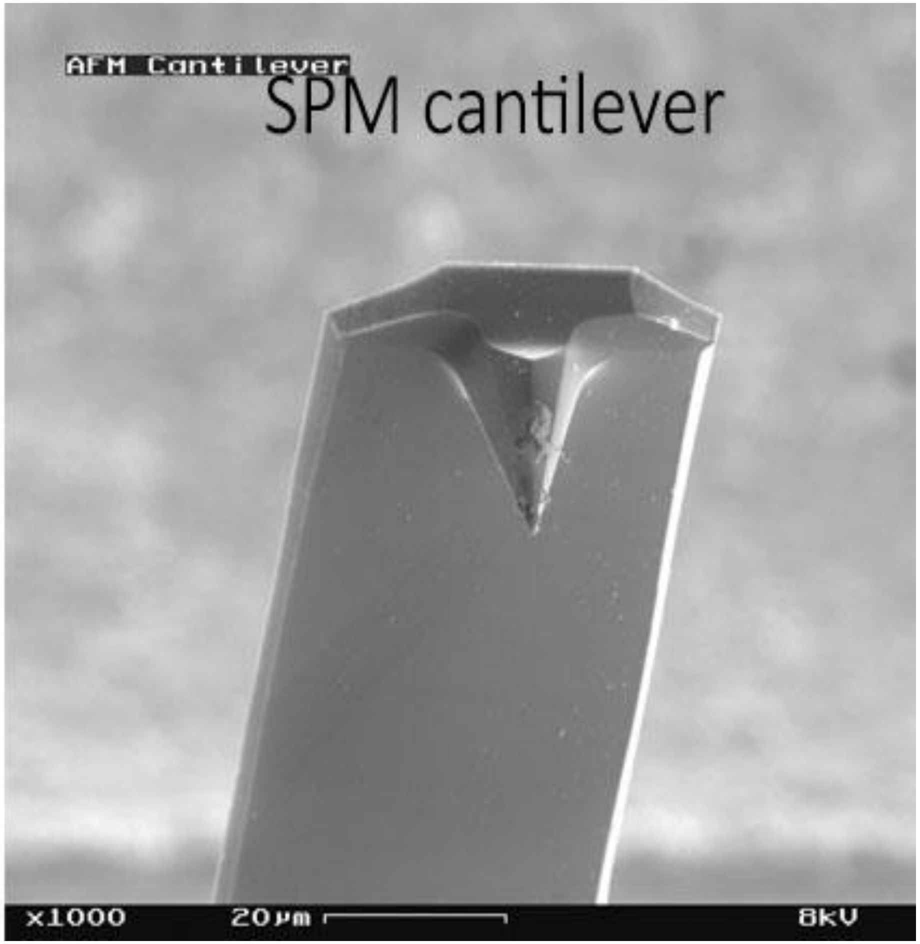
- Probes are typically made from **Si_3N_4 or Si**.
- Different cantilever lengths, materials, and shapes allow for varied spring constants and resonant frequencies.
- Probes may be coated with other materials for addition SPM applications such as **chemical force microscopy** (CFM) and **magnetic force microscopy** (MFM).

AFM Probe


laser beam
nanoatlas.ifs.hr
cantilever
surface
tip
10000 nm
created by Antonio Šiber, 2003

How a AFM probe functions





How forces are measured?

- The probe is placed on the end of a cantilever *(which one can think of as a spring)*.
- The amount of force between the probe and sample is dependant on the ***spring constant*** *(stiffness)* of the cantilever and the distance between the probe and the sample surface. This force can be described using ***Hooke's Law:***

$$\boxed{F = k \, . \, X}$$

F = force *(b/w probe and sample surface)*

k = *Spring constant*

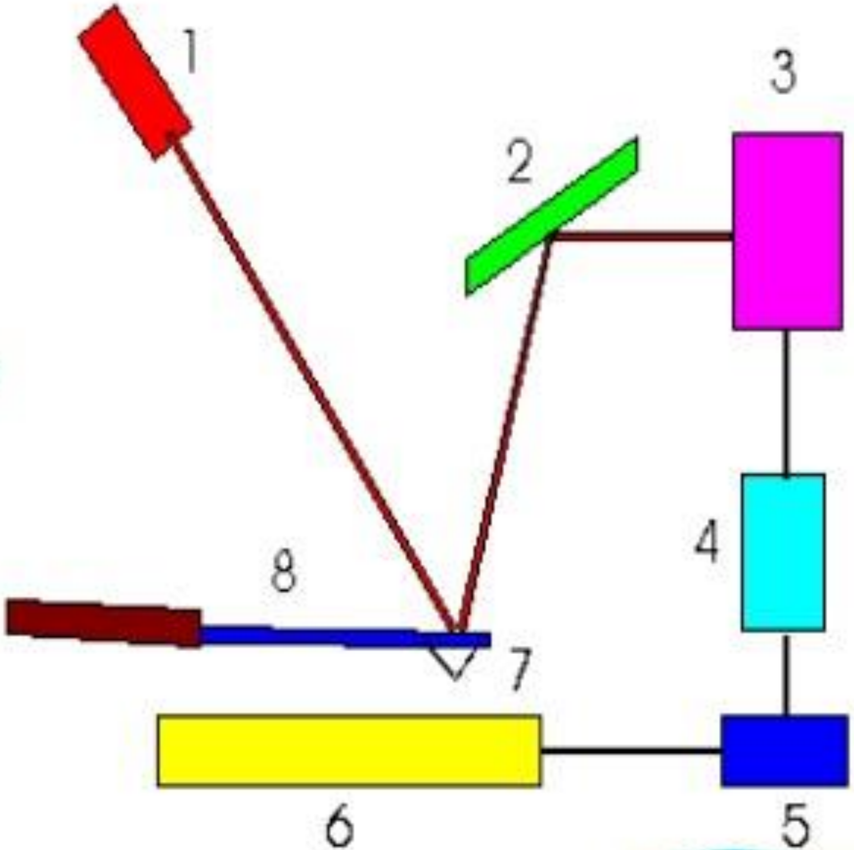
x = *cantilever deflection*

How forces are measured?

- If the spring constant of cantilever ***(typically ~0.1 – 1.0 N/m)*** is less than the surface, the cantilever bends and the deflection is monitored.
- This typically results in forces ranging from (10^{-9}) N/m to (10^{-6}) N/m in the open air.

Parts of AFM

- 1. Laser – deflected off cantilever
- 2. Mirror –reflects laser beam to photodetector
- 3. Photodetector –dual element photodiode that measures differences in light intensity and converts to voltage
- 4. Amplifier
- 5. Register
- 6. Sample
- 7. Probe –tip that scans sample made of Si
- 8. Cantilever –moves as scanned over sample and deflects laser beam



Instrumentation

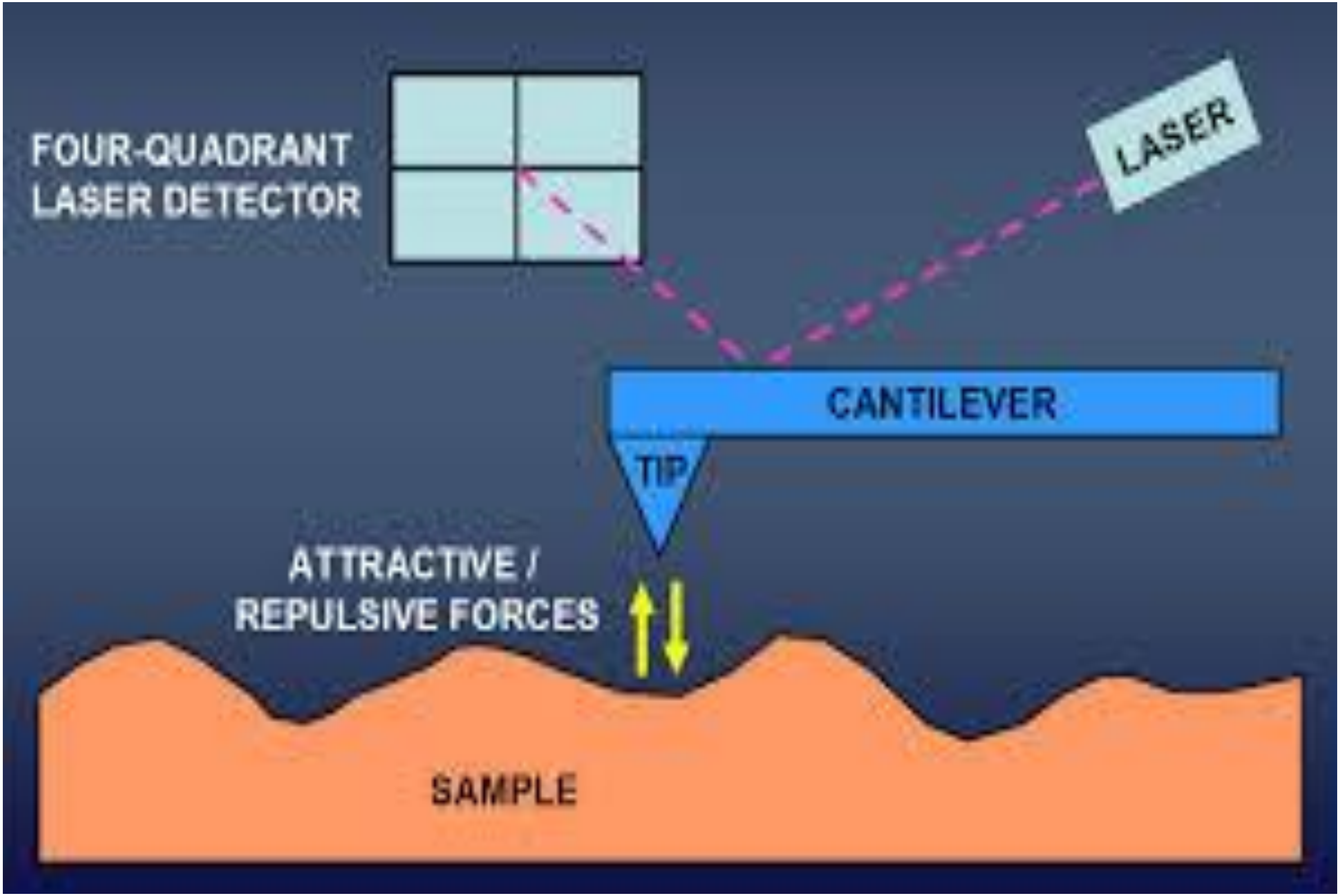
- The motion of the probe across the surface is controlled using ***feedback loop*** and ***piezoelectronic scanners****.*
- The primary difference in instrumentation design is how the forces between the probe and sample surface are monitored.
- The deflection of the probe is typically measured by a **"beam bounce"** method. A semiconductor diode laser is bounced off the back of the cantilever onto a position sensitive photodiode detector.
- This detector measures the bending of cantilever when the tip is scans over the sample surface. The measured cantilever deflections are used to generate a map of the surface topography.

Instrumentation

- **Piezoelectricity** is the electric charge that accumulates in certain solid materials (such as crystals, certain ceramics, and biological matter such as bone, DNA and various proteins called peizometarials) in response to applied mechanical stress. ***The word piezoelectricity means electricity resulting from pressure and latent heat.***

What type of forces are measured?

- The dominant interactions at short probe-sample distances in the AFM are Vander Waals (VdW) interactions.
- During contact with the sample, the probe predominately experiences ***repulsive Vander Waals forces*** *(contact mode). This leads to the tip deflection.*
- *As the tip moves further away from the surface* ***attractive Vander Waals forces*** *are dominant (non-contact mode).*



When the probe moves down (attractive), cantilever moves up and vice - versa

Modes of operation

- There are 3 primary imaging modes in AFM:

(1) Contact AFM

< 0.5 nm probe-surface separation

(2) Intermittent contact *(tapping mode AFM)*

0.5-2 nm probe-surface separation

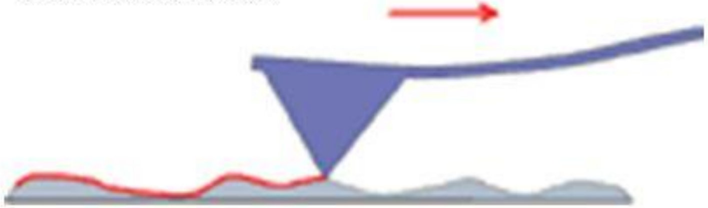
(3) Non-contact AFM

2-10 nm probe-surface separation

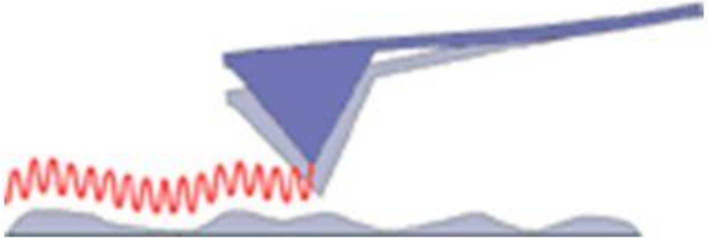
Modes of operation. There are 3 modes of AFM operation

1. Contact mode
2. Non-contact mode
3. Tapping mode

Contact mode



Non-contact mode



Tapping mode

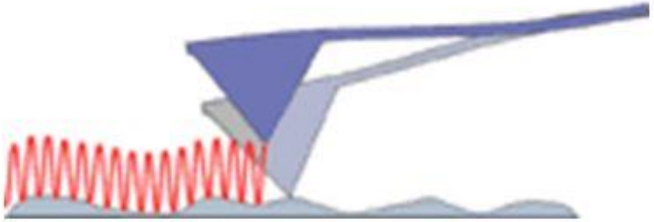


Fig. 7.3 Modes

Contact Mode AFM

- ***Repulsive VdW***- *When the spring constant of cantilever is less than surface, the cantilever bends. The force on the tip is repulsive.*
- *By maintaining a constant cantilever deflection (using the feedback loops) the force between the probe and the sample remains constant and an image of the surface is obtained.*
- ***Advantages***: *Fast scanning, good for rough samples, used in friction analysis*
- ***Disadvantages:*** *At times, such forces can damage/deform soft samples (however imaging in liquids often resolves this issue)*

Tapping (Intermittent) mode

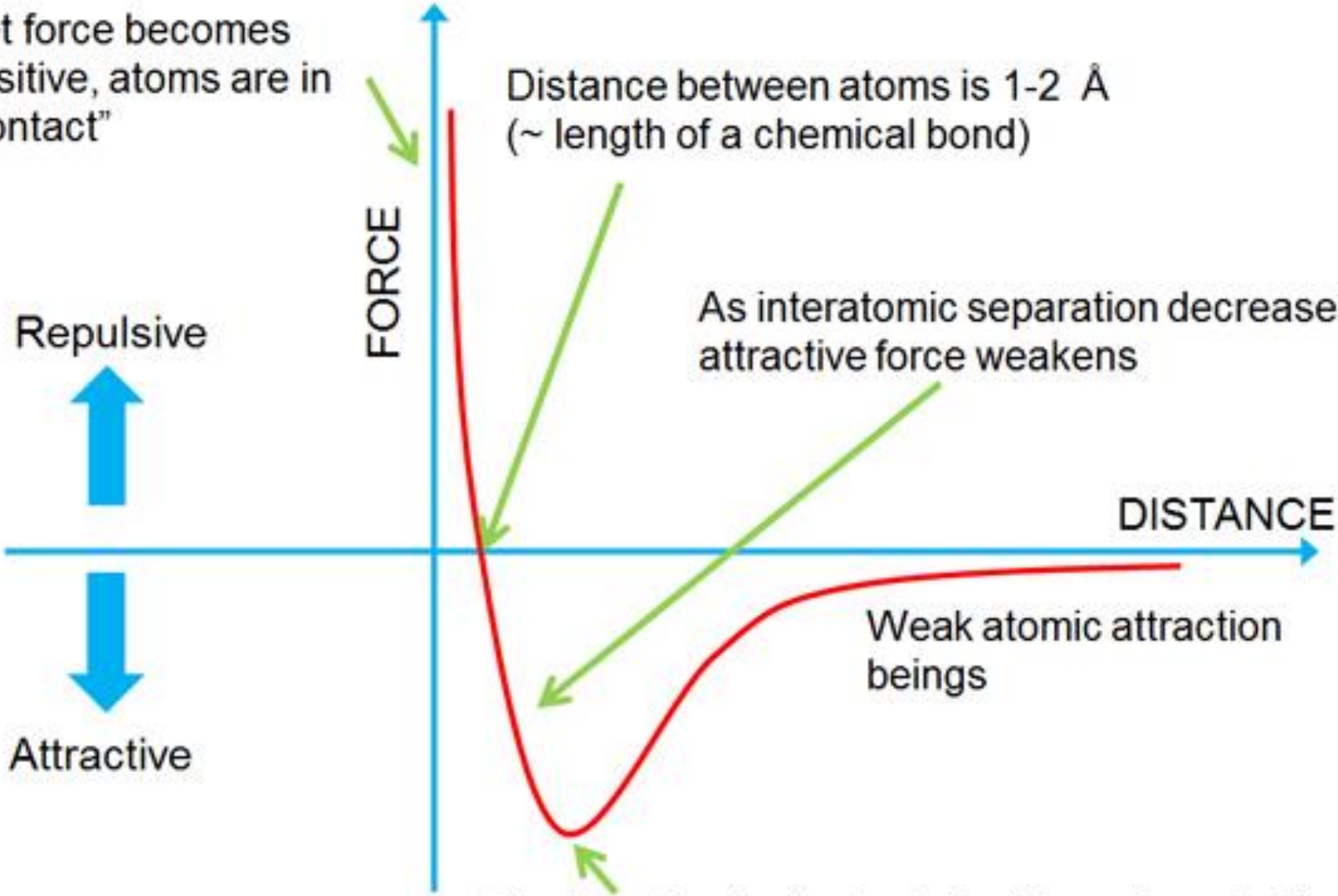
- The imaging is similar to contact. However, in this mode the cantilever is oscillated at its *resonant frequency.*
- *The probe lightly "taps" on the sample surface during scanning, contacting the surface at the bottom of its swing.*
- *By maintaining a constant oscillation amplitude a constant tip-sample interaction is* maintained and an image of the surface is obtained.
- ***Advantages:*** *allows high resolution of samples that are easily damaged and/or loosely held to a surface; Good for biological samples*
- ***Disadvantages:*** *more challenging to image in liquids, slower scan speeds needed*

Non-contact mode

- ***Attractive VdW-*** *The probe does not contact the sample surface, but oscillates above the adsorbed fluid layer on the surface during scanning.*
- *Using a feedback loop to monitor changes in the amplitude due to attractive VdW forces, the surface topography can be measured.*
- ***Advantages:*** *Very low force exerted on the sample(*10^{-12} *N), extended probe lifetime*
- ***Disadvantages:*** *Generally lower resolution; contaminant layer on surface can interfere with oscillation; usually need ultra high vacuum (UHV) to have best imaging*

Force Curve Analysis
What are Force Curves?

- Force curves measure the amount of force felt by the cantilever as the probe tip is brought close to - and even indented into - a sample surface and then pulled away.
- In a force curve analysis, the probe is repeatedly brought towards the surface and then retracted.
- Force curve analysis can be used to determine chemical and mechanical properties such as adhesion, elasticity, hardness and rupture bond lengths.



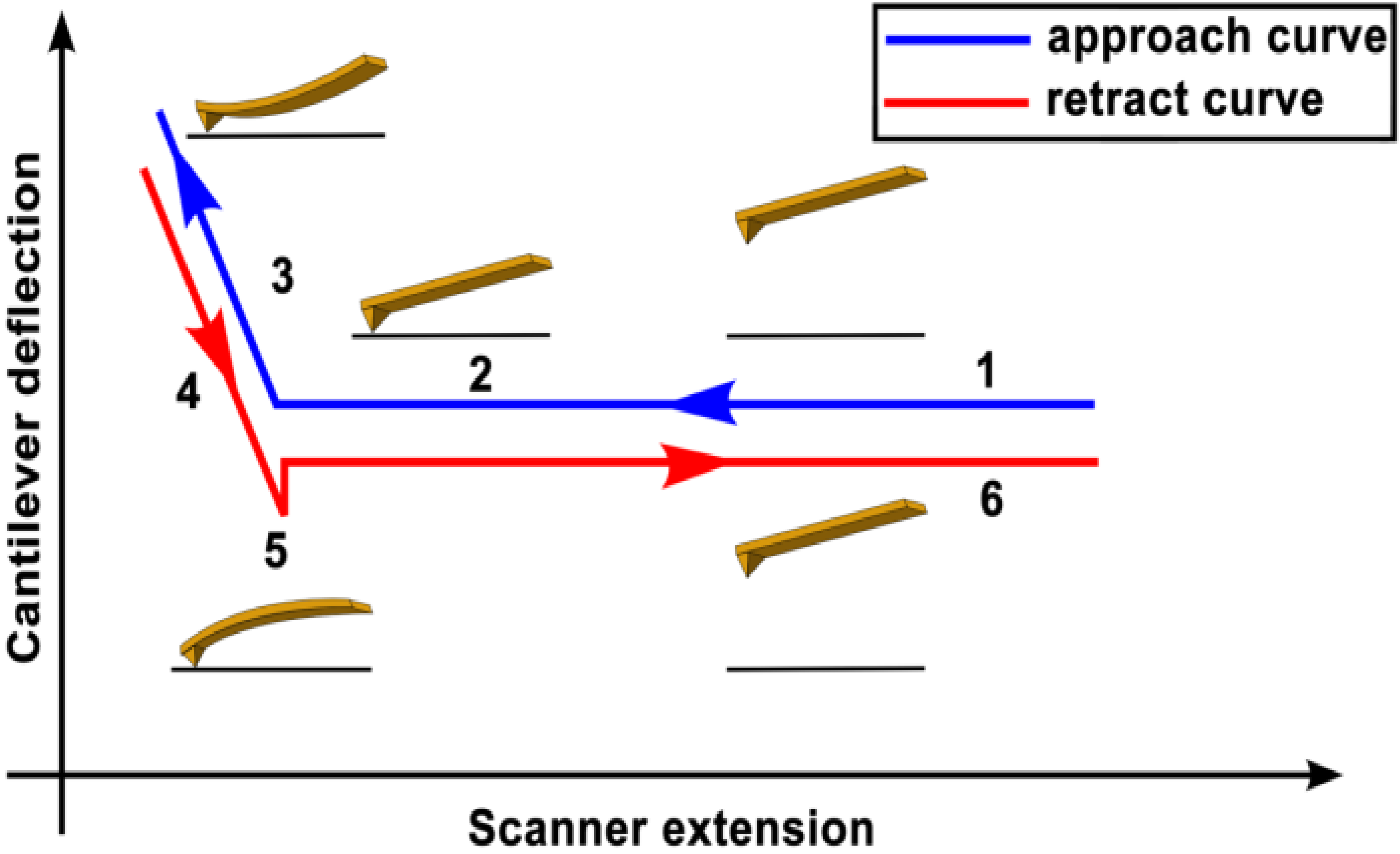
Net force becomes positive, atoms are in “contact”

Distance between atoms is 1-2 Å (~ length of a chemical bond)

As interatomic separation decreases, attractive force weakens

Weak atomic attraction beings

Electron clouds electrostatically repel each other


approach curve
retract curve
Cantilever deflection
Scanner extension
1
2
3
4
5
6

Advantages:

- Unlike electron microscope, samples do not need to be coated or stained- minimal damage and easy sample preparation.
- Unlike STM, samples do not need to be conductive.
- Sub-nm resolutions have been achieved on biological samples.
- also used for applications in which no scanning is required, where the tip is used as a sensitive force sensor