Introduction to Scanning Probe Microscopy (SPM)

Basic Theory

Atomic Force Microscopy (AFM)

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Outline

- How does the AFM work?
  - How are forces measured?
    - - Spring Constants
    - - Probes
    - - Instrumentation
  - Imaging Methods
    - What types of forces are measured?
    - Modes of Operation
      - -Contact
      - -Non-contact
      - -Tapping
  - What are force curves?
- What are the limitations of AFM?

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Atomic Force Microscopy

HOW DOES THE 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 are Forces 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:

$$F=-k \cdot x$$

F = Force
k = spring constant
x = cantilever deflection

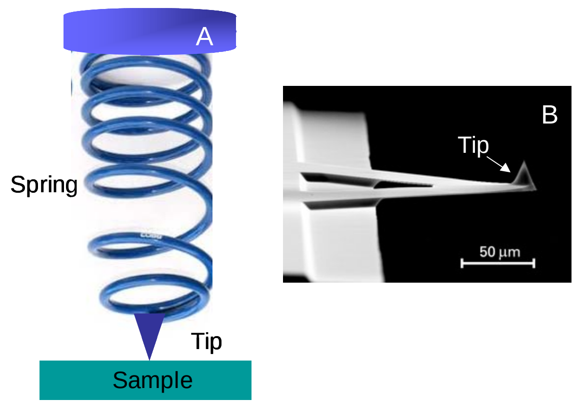


Figure 1. a) Spring depiction of cantilever b) SEM image of triangular SPM cantilever with probe (tip). (Image from MikroMasch)[1]

If the spring constant of cantilever (typically ~ 0.1-1 N/m) is less than surface, the cantilever bends and the deflection is monitored.

This typically results in forces ranging from nN (10^{-9}) to µN (10^{-6}) in the open air.

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. A description of the variety of different probes can be found at various vendor sites.[2] Probes may be coated with other materials for additional SPM applications such as chemical force microscopy (CFM) and magnetic force microscopy (MFM).

Instrumentation

The motion of the probe across the surface is controlled similarly to the STM using *feedback loop* and *piezoelectronic scanners.* **(See STM basic theory)** 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 measure 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 during the tip is scanned over the sample. The measured cantilever deflections are used to generate a map of the surface topography.

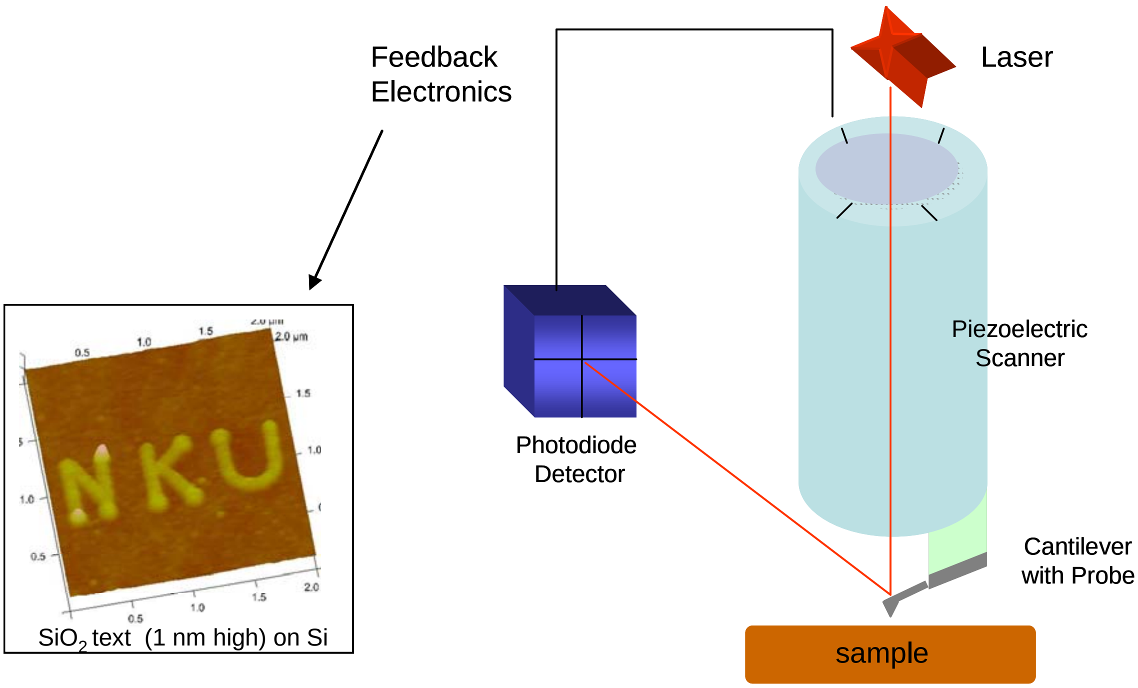


Figure 2. Schematic of AFM instrument showing "beam bounce" method of detection using a laser and position sensitive photodiode detector.

For a visual depiction of the "beam bounce" method of detection in AFM and you can refer to the following web resource which utilizes Legos ®, magnetics, and a laser pointer to demonstrate this concept.

Imaging Methods

What types of forces are measured?

The dominant interactions at short probe-sample distances in the AFM are Van der Waals (VdW) interactions. However long-range interactions (i.e. capillary, electrostatic, magnetic) are significant further away from the surface. These are important in other SPM methods of analysis.

During contact with the sample, the probe predominately experiences *repulsive Van der Waals forces* (contact mode). This leads to the tip deflection described previously. As the tip moves further away from the surface *attractive Van der Waals forces* are dominant (non-contact mode).

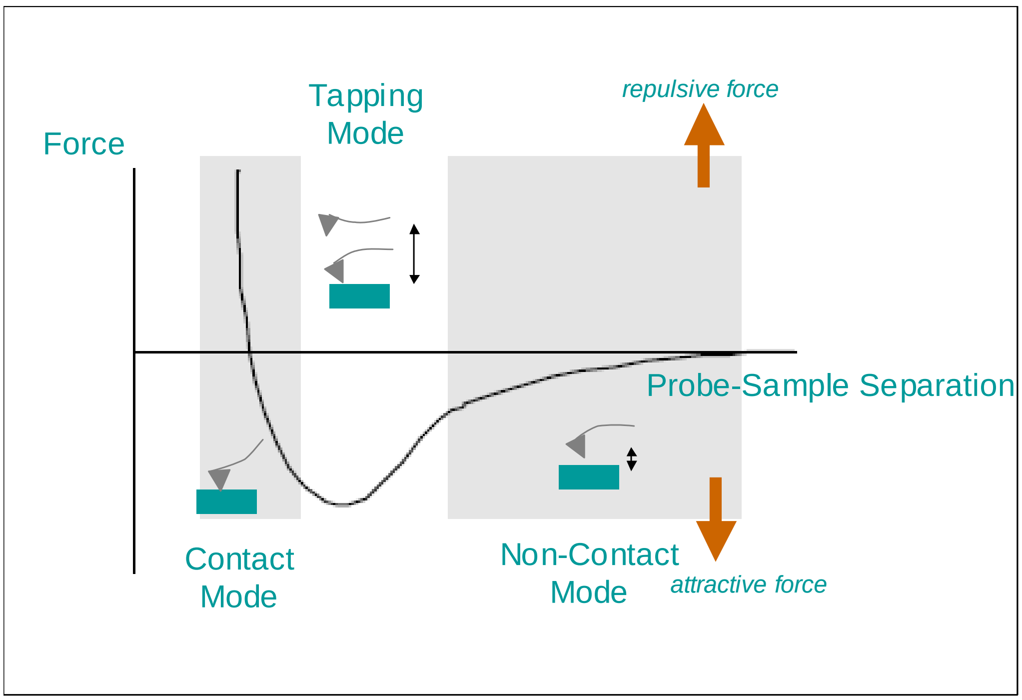


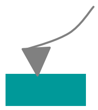
Figure 3. Plot of force as a function of probe-sample separation.

Modes of Operation

There are 3 primary imaging modes in AFM:

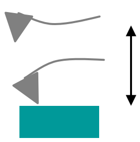
(1) Contact AFM
< 0.5 nm probe-surface separation
(2) Intermittent contact (tapping mode AFM)
0.5-2 nm probe-surface separation
(3) Non-contact AFM
0.1-10 nm probe-surface separation

Primary Modes of Imaging:



1. 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 time forces can damage/deform soft samples (however imaging in liquids often resolves this issue)

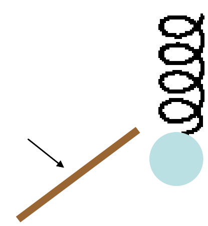


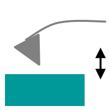
Oscillation Amplitude: 20-100 nm

2. Intermittent Mode (Tapping): The imaging is similar to contact. However, in this mode the cantilever is oscillated at its *resonant frequency*, Figure 4. 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

Figure 4. Resonant frequency is a natural frequency of vibration determined by the physical parameters of the vibrating object. For example if you hit a spring with a mass at the end (probe) the main response will be a bob up and down at its natural frequency.





3. 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. *(Note: all samples unless in a controlled UHV or environmental chamber have some liquid adsorbed on the surface).* 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

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, Figure 5. Force curve analyses can be used to determine chemical and mechanical properties such as adhesion, elasticity, hardness and rupture bond lengths.

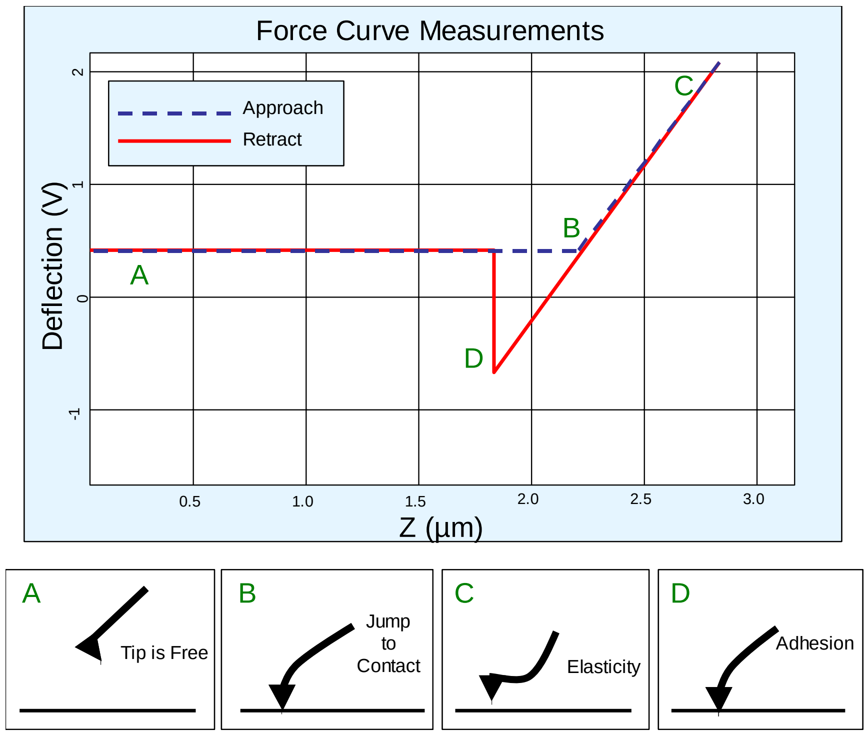


Figure 5. A typical force curve showing the various probe-sample interactions.

The slope of the deflection (C) provides information on the hardness of a sample. The adhesion (D) provides information on the interaction between the probe and sample surface as the probe is trying to break free. Direct measurements of the interactions between molecules and molecular assemblies can be achieved by functionlizing probes with molecules of interest (see Chemical Force Microscopy).

Interactive Tools:
An interactive force curve analysis can be found here: http://www.ntmdt.ru/SPM-Techniques/Principles/Spectroscopies/Force-distance_curves_mode20.html

WHAT ARE THE LIMITATIONS OF AFM?

The AFM can be used to study a wide variety of samples (i.e. plastic, metals, glasses, semiconductors, and biological samples such as the walls of cells and bacteria). *Unlike STM or scanning electron microscopy it does not require a conductive sample.* However there are limitations in achieving atomic resolution. The physical probe used in AFM imaging is not ideally sharp. As a consequence, an AFM image does not reflect the true sample topography, but rather represents the interaction of the probe with the sample surface. This is called tip convolution, Figure 6.

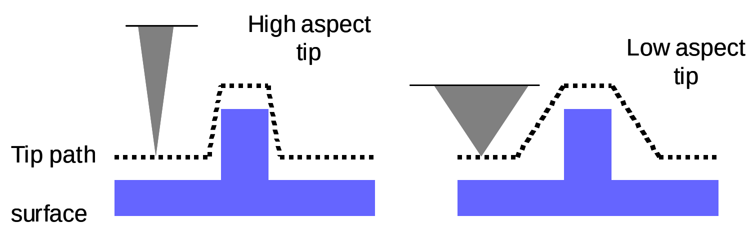


Figure 6. Ideally a probe (tip) with a high aspect ratio will give the best resolution. The radius of curvature of the probe leads to tip convolution. This does not often influence the height of a feature but the lateral resolution.

Commercially available probes are becoming more widely available that have very high aspect ratios. These are made with materials such as carbon nanotubes or tungsten spikes.[3] However these probes are still very expensive to use for every day image analysis.

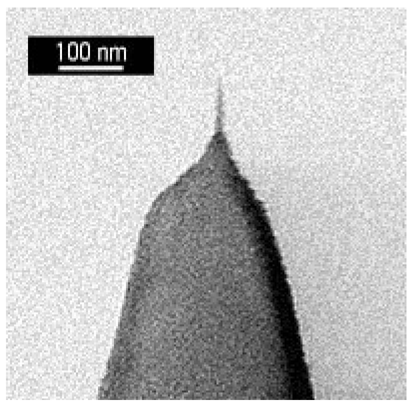


Figure 7. Example of High aspect probes from MikroMasch (1nm radius of curvature HI'RES-W probe).[3]

Online Resources

Another useful source on the principles of SPM:
http://www.physics.leidenuniv.nl/sections/cm/ip/group/Principle_of_SPM.htm

Another useful source on how AFM works
http://stm2.nrl.navy.mil/how-afm/how-afm.html#General%20concept

References:

1. MikroMasch http://www.spmtips.com/products/cantilevers/datasheets/sc11/ (Accessed 11/06/06).
2. Nanosensors Homepage http://www.nanosensors.com (Accessed 11/06/06).
3. MikroMasch http://www.spmtips.com/products/cantilevers/datasheets/hi-res-w/ (Accessed 1/26/07).

Acknowledgements:

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