Atomic Force Microscopy (AFM)

Introduction

The atomic force microscope (AFM) or scanning force microscope (SFM) was invented in 1986 by Binnig, Quate and Gerber. Similar to other scanning probe microscopes, the AFM raster scans a sharp probe over the surface of a sample and measures the changes in force between the probe tip and the sample. Fig.1 illustrates the working concept for an atomic force microscope. A cantilever with a sharp tip is positioned above a surface. Depending on this separation distance, long range or short range forces will dominate the interaction. This force is measured by the bending of the cantilever by an optical lever technique: a laser beam is focused on the back of a cantilever and reflected into a photodetector. Small forces between the tip and sample will cause less deflection than large forces. By raster-scanning the tip across the surface and recording the change in force as a function of position, a map of surface topography and other properties can be generated.

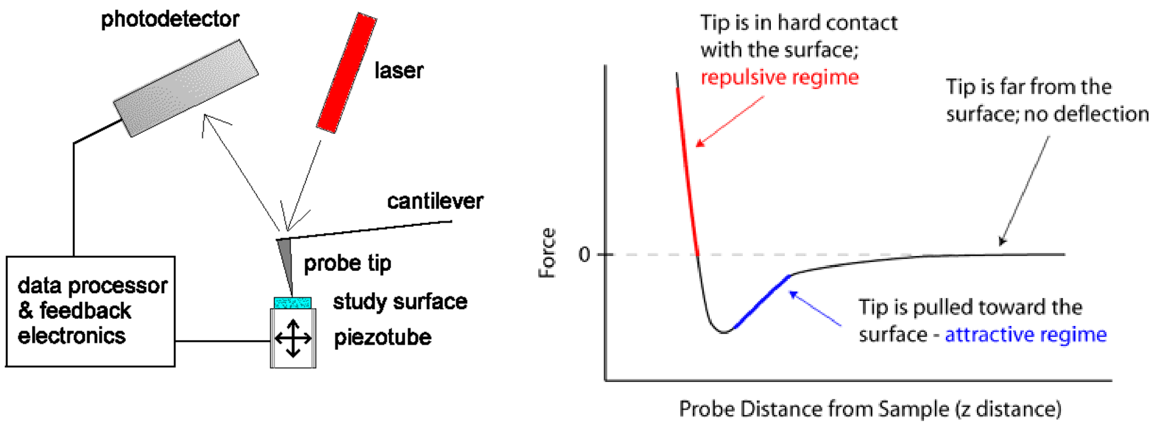

Figure 1. Scheme of an atomic force microscope and the force-distance curve characteristic of the interaction between the tip and sample.

The AFM is useful for obtaining three-dimensional topographic information of insulating and conducting structures with lateral resolution down to 1.5 nm and vertical resolution down to 0.05 nm. These samples include clusters of atoms and molecules, individual macromolecules, and biological species (cells, DNA, proteins). Unlike the preparation of samples for STM imaging, there is minimal sample preparation involved for AFM imaging. Similar to STM operation, the AFM can operate in gas, ambient, and fluid environments and can measure physical properties including elasticity, adhesion, hardness, friction and chemical functionality. A table of representative surfaces and structures is shown below.

Inorganic and Synthetic Materials	
Surfaces	***Nanostructures***
Surface topography	Carbon nanotubes
Surface Chemistry	Surfaces of polymers
Silicon wafers	Diffraction gratings
Data storage media	Integrated circuits
Ceramics	Nanowires
Biological Materials	
Polymers and Polymer Matrices	***Biological Structures***
Natural resins and gums	Bacterial flagellae
Muscle proteins	Amyloid-beta
DNA	Chromosomes
Plant cell walls	Cell and membrane surfaces

Basic set-up of an AFM

In principle the AFM resembles a record player and a stylus profilometer. The ability of an AFM to achieve near atomic scale resolution depends on the three essential components: (1) a cantilever with a sharp tip, (2) a scanner that controls the *x*-*y*-*z* position, and (3) the feedback control and loop.

1. *Cantiliever with a sharp tip*. The stiffness of the cantilever needs to be less the effective spring constant holding atoms together, which is on the order of 1 - 10 nN/nm. The tip should have a radius of curvature less than 20-50 nm (smaller is better) a cone angle between 10-20 degrees.

2. *Scanner*. The movement of the tip or sample in the *x*, *y*, and *z*-directions is controlled by a piezo-electric tube scanner, similar to those used in STM [see the STM lab manual for a detailed description]. For typical AFM scanners, the maximum ranges for are 80 μm x 80 μm in the *x*-*y* plane and 5 μm for the *z*-direction.

3. *Feedback control*. The forces that are exerted between the tip and the sample are measured by the amount of bending (or deflection) of the cantilever. By calculating the difference signal in the photodiode quadrants, the amount of deflection [(A+B)-(C+D)] can be correlated with a height (Figure 2). Because the cantilever obeys Hooke's Law for small displacements, the interaction force between the tip and the sample can be determined.

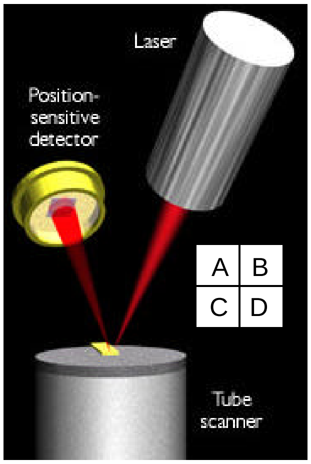

Figure 2. Schematic diagram of optical changes in the deflection of the cantilever and a quadrant photodiode.

The AFM can be operated with or without feedback control. If the electronic feedback is **on**, as the tip is raster-scanned across the surface, the piezo will adjust the tip-sample separation so that a constant deflection is maintained—or so the force is the same as its setpoint value. This operation is known as *constant force mode*, and usually results in a fairly faithful topographical (hence the alternative name, *height mode*).

If the feedback electronics are switched **off**, then the microscope is said to be operating in *constant height* or *deflection* mode. This is particularly useful for imaging very flat samples at high resolution. Often it is best to have a small amount of feedback-loop gain to avoid problems with thermal drift or the possibility of a slightly rough sample damaging the tip and/or cantilever. Strictly, this mode should then be called *error signal*. The error signal may also be displayed while the feedback is on; this image displays slow variations in topography and highlights the edges of features.

Modes of operation for the AFM

The three general types of AFM imaging are (1) *contact mode*, (2) *tapping mode* and (3) *non-contact mode*.

1. ***Contact mode*** is the most common method of operation of the AFM and is useful for obtaining 3D topographical information on nanostructures and surfaces. As the name suggests, the tip and sample remain in close contact as the scanning proceeds. "Contact" represents the repulsive regime of the inter-molecular force curve, the part of the curve above the x-axis (Figure 1, right image). Most cantilevers have spring constants < 1 Nm, which is less than effective spring constant holding atoms together. One of the drawbacks

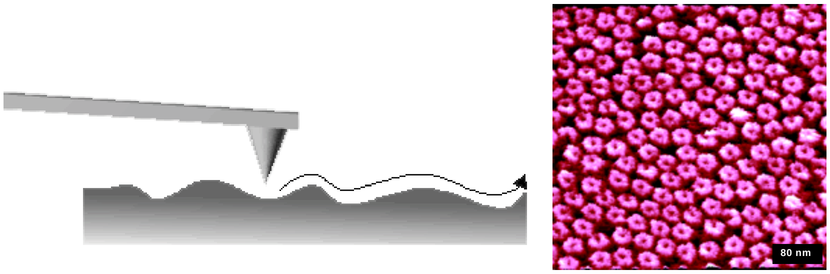

Figure 3. Scheme of contact mode imaging and an image of cholera oligomers by Shao (U. Virginia)

of the tip remaining in contact with the sample is that large lateral forces can be exerted on the sample as the tip is dragged over the specimen. These large forces can result in deformed images and damaged samples. Small lateral forces, however, can be used to provide information on the friction (drag resistance) between the tip and sample in a mode known as *lateral force microscopy* (LFM).

LFM measures the torsional deformation of the cantilever while the tip scans over the surface. While topographic images are recorded by the difference between the top and bottom quadrants of the photodiode, the frictional images are recorded by the difference between the left and right portions of the photodiode. Simultaneous measurement of the topographic and frictional images can be recorded. LFM is useful for obtaining chemical contrast in samples whose features are all of the same height.

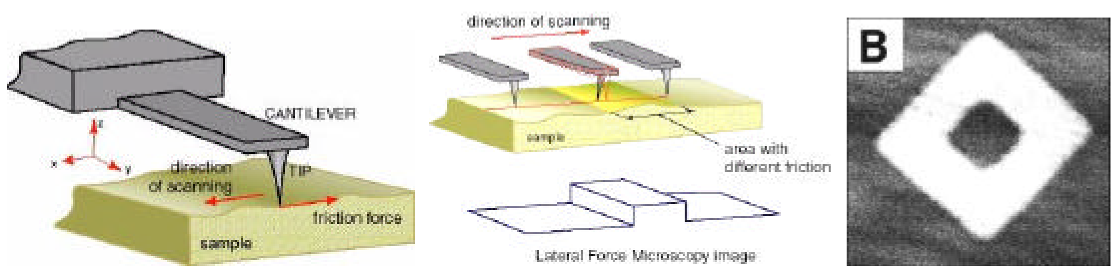

Figure 4. Scheme of lateral force imaging and a friction image of self-assembled monolayers on gold. The white regions terminate in –COOH, and the gray regions terminate in –CH_3; the tip is also functionalized with –COOH. C.M. Lieber, *Langmuir* 14, 1508 (1998).

2. ***Tapping mode*** is another mode of operation for AFM. Unlike the operation of contact mode, where the tip is in constant contact with the surface, in tapping mode the tip makes intermittent contact with the surface. As the tip is scanned over the surface, the cantilever is driven at its resonant frequency (hundreds of kHz). Because the contact time is a small fraction of its oscillation period, the lateral forces are reduced dramatically. Tapping mode is usually preferred to image samples with structures that

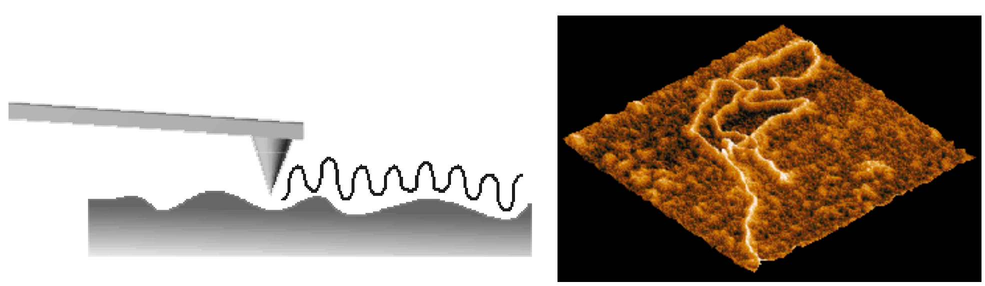

Figure 5. Scheme of tapping mode imaging and an image of DNA by B. Stine (Northwestern).

are weakly bound to the surface or samples that are soft (polymers, thin films). There are also two other types of image contrast mechanisms in tapping mode:

- *Amplitude imaging.* The feedback loop adjusts the *z*-piezo so that the amplitude of the cantilever oscillation remains (nearly) constant. The voltages needed to keep the amplitude constant can be compiled into an (error signal) image, and this imaging can often provide high contrast between features on the surface.

- *Phase imaging*. The phase difference between the driven oscillations of the cantilever and the measured oscillations can be attributed to different material properties. For example, the relative amount of phase lag between the freely oscillating cantilever and the detected signal can provide qualitative information about the differences in chemical composition, adhesion, and friction properties.

3. ***Non-contact*** mode is a method where the cantilever is oscillated above the surface of the sample at distance such that it is no longer in the repulsive regime but in the attractive regime of the inter-molecular force curve. The operation of non-contact imaging is quite difficult in ambient conditions because of the existing thin layer of water on the tip and the surface. As the tip is brought close to the surface, a small capillary bridge between the tip and the sample and cause the tip to "jump-to-contact."

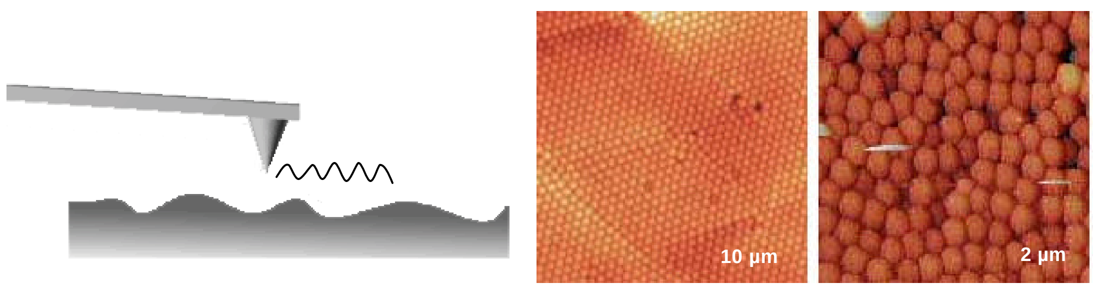

Figure 6. Scheme of non-contact mode imaging and an image of "raspberry" polymers, DI

The choice for which AFM mode to use is based on the surface characteristics of interest and on the hardness/stickiness of the sample. Contact mode is most useful for hard surfaces; a tip in contact with a surface, however, is subject to contamination from removable material on the surface. Excessive force in contact mode can also damage the surface or blunt the probe tip. Tapping mode is well-suited for imaging soft biological specimen and for samples with poor surface adhesion (DNA and carbon nanotubes). Non-contact mode is another useful mode for imaging soft surfaces, but its sensitivity to external vibrations and the inherent water layer on samples in ambient conditions often causes problems in the engagement and retraction of the tip. A summary of the different modes of operation is found in the table below.

Mode of Operation	**Force of Interaction**
Contact mode	strong (repulsive) - constant force or constant distance
Non-contact mode	weak (attractive) - vibrating probe
Tapping mode	strong (repulsive) - vibrating probe
Lateral force mode	frictional forces exert a torque on the scanning cantilever

Effects of the tip

Ideally the AFM tip would act simply as a probe, where it interacts with the surface only to characterize its properties but not influence the measured properties. There are several negative influences, however, that a tip can have on an image.

1. *Broadening of features.* Tip convolution occurs when the radius of curvature of the tip is comparable with, or greater than, the size of the feature that is imaged. Fig. x illustrates this problem; as the tip scans over the specimen, the sides of the tip make contact before the apex, and the feedback mechanism begins responding to the feature.
2. *Compression of features.* Compression occurs when the tip is situated over the feature that is imaged. It is difficult to determine in many cases how important this affect is, but studies on some soft biological polymers (such as DNA) have shown the apparent DNA width to be a function of imaging force. Although the force between the tip and sample may only be nN, the *pressure* may be in the range of MPa.
3. *Strong interaction with sample.* Interaction forces between the tip and sample are the reason for image contrast with the AFM. Some changes that may be perceived becaue of topography, however, may be due to a change in the force of interaction. For example, forces due to the chemical nature of the tip are important, and chemical mapping using specially treated or modified tips is an imaging mode called *chemical force microscopy*.

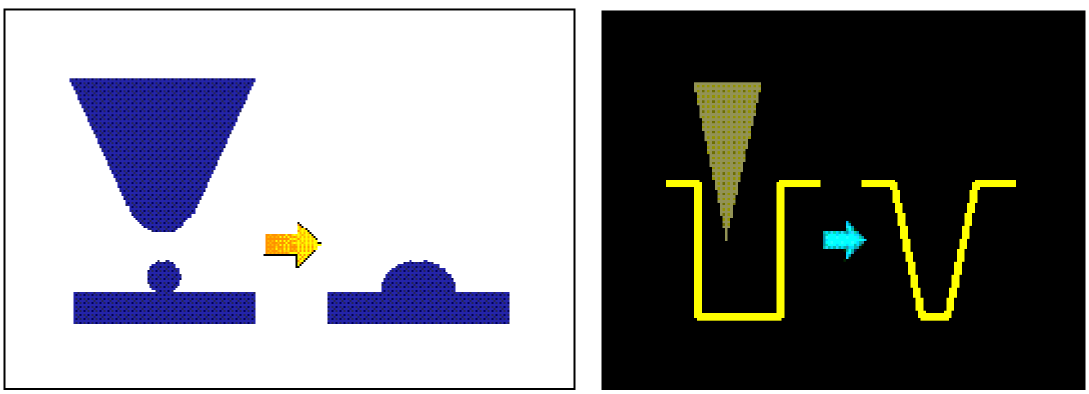

Figure 7. Tip convolution effects on sample features.

References

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3. Y. Xia and G.M. Whitesides, *Langmuir* **13,** 2059 (1997).