Atomic Force Microscopy

Atomic force microscopy (AFM) is one of many techniques which fall under the Scanned Probe Microscopy (SPM) family of instruments.

The first of the SPM techniques was the Scanning Tunneling Microscope (STM), developed by Binnig & Rohrer, which got them the Nobel prize for Physics in 1986. STM is based on the tunneling of electrons from a sharp metal probe tip to a conductive sample.

AFM uses a fine tip to measure surface morphology and properties through an interaction between the tip and surface. It was invented in 1985 by G. Binnig, C.F. Quate and Ch. Gerber.

In an AFM a constant force is maintained between the probe and sample while the probe is raster scanned (parallel lines) across the surface. By monitoring the motion of the probe as it is scanned across the surface, a three dimensional image of the surface is constructed.

The constant force is maintained by measuring the force with the "light lever" sensor and using a feedback control electronic circuit (FCU) to control the position of the Z piezoelectric ceramic.

The motion of the probe over the surface is generated by piezoelectric ceramics that move the probe and force sensor across the surface in the X and Y directions.

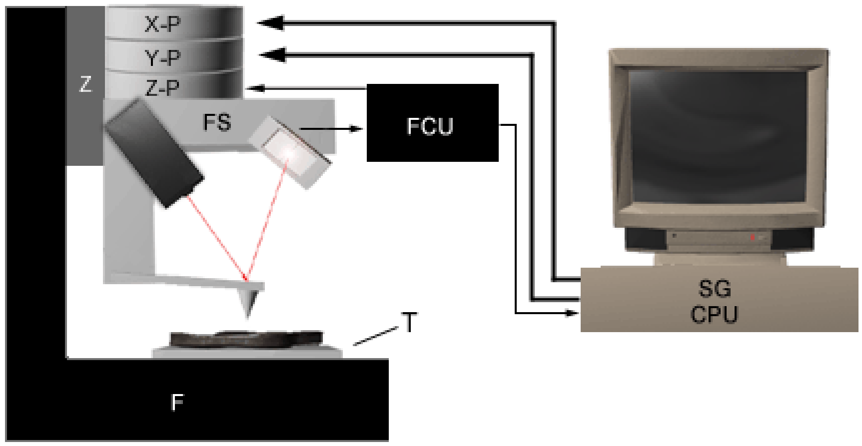
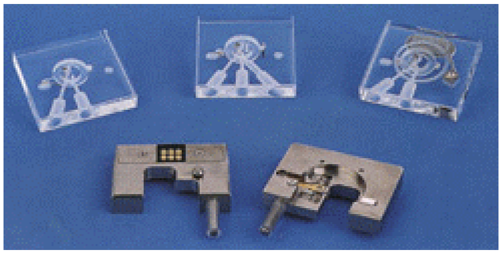


Figure 5: Shows all of the components and subsystems of an atomic force microscope system.

Instrumentation:

- (Z) Coarse Z motion translator- it moves the AFM head towards the surface so that the force sensor can measure the force between the probe and sample. The motion of the translator is usually about 10 mm.
- (T) Coarse X-Y translation stage - is used to place the section of the sample that is being imaged by the AFM directly under the probe.
- (X-P) X and Y piezoelectric transducer - With the X and Y piezoelectric transducer the (Y-P) probe is moved over the surface in a raster motion when an AFM image is measured.
- (FS) Force Sensor - The force sensor measures the force between the probe and the sample by monitoring the deflection of a cantilever.
- (ZP) Z piezoelectric Ceramic - Moves the force sensor in the vertical direction to the surface as the probe is scanned with the X and Y piezoelectric transducers.
- (FCU) Feedback control unit – FCU takes in the signal from the light lever force sensor and outputs the voltage that drives the Z piezoelectric ceramic. This voltage refers to the voltage that is required to maintain a constant deflection of the cantilever while scanning.
- (SG) X-Y signal generator - The motion of the probe in the X-Y plane is controlled by the X-Y signal generator. A raster motion is used when an image is measured.

A Digital Instruments Nanoscope IIIa Scanning Probe Microscope



Fluid cell attachment for Nanoscope IIIa enabling in situ measurement.

What AFM can do?

- ✓ Operates both in air or water,
- ✓ The topographic images at a resolution on the nanometer-scale
- ✓ Can measure surfaces that are not a good conductor.
- ✓ Can also measure surface/interface properties (mechanical, magnetic and electric, optical, thermal, chemical properties).
- ✓ AFM has been developed as an independent technique and has more applications than SPM/STM.

AFM operation does not require a current between the sample surface and the tip, thus can move into potential regions. Can image insulators, organic materials, biological macromolecules, polymers, ceramics and glasses in different environments, such as liquids, vacuum, and low temperatures.

To obtain similar resolution as in STM for insulating surfaces, AFM
A sharp tip (apex radius ~20 nm or 10-100 nm radius of curvature) formed on a soft cantilever is used to probe the interaction between the tip and sample surface.

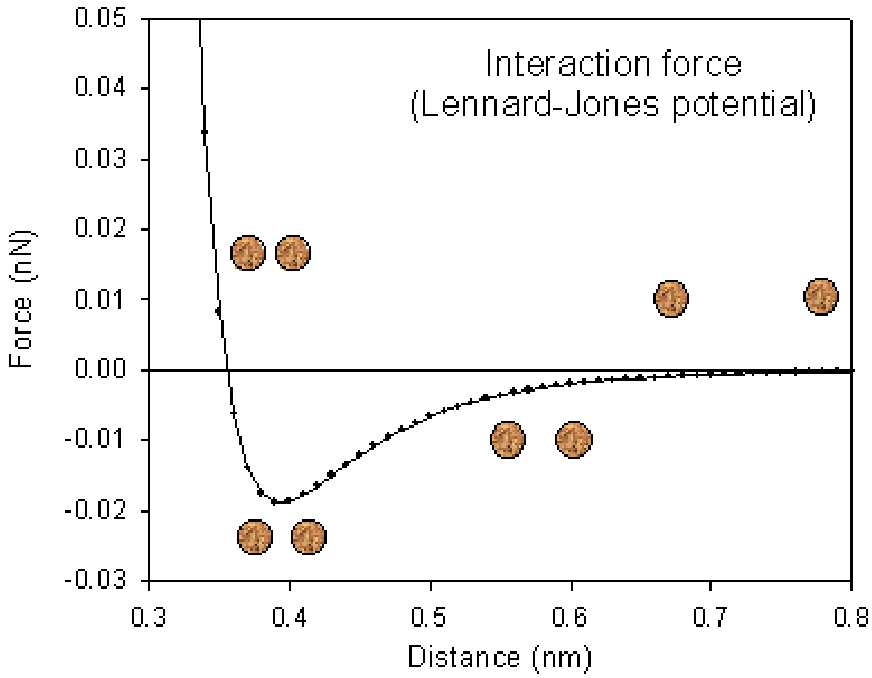
The phenomena could be understood through the Lennard-Jones interaction between two atoms:

$$E_r = -\frac{\alpha}{r^6} + \frac{\beta}{r^{12}}$$

where r is the separation of the two bodies, α and β are interaction constants. Then the interaction force,

$$F_r = -\frac{dE_r}{dr} = -\frac{6\alpha}{r^7} + \frac{12\beta}{r^{13}}$$

α and β are known to be 10^{-77} Jm^6 and 10^{-134} Jm^{12}, respectively.

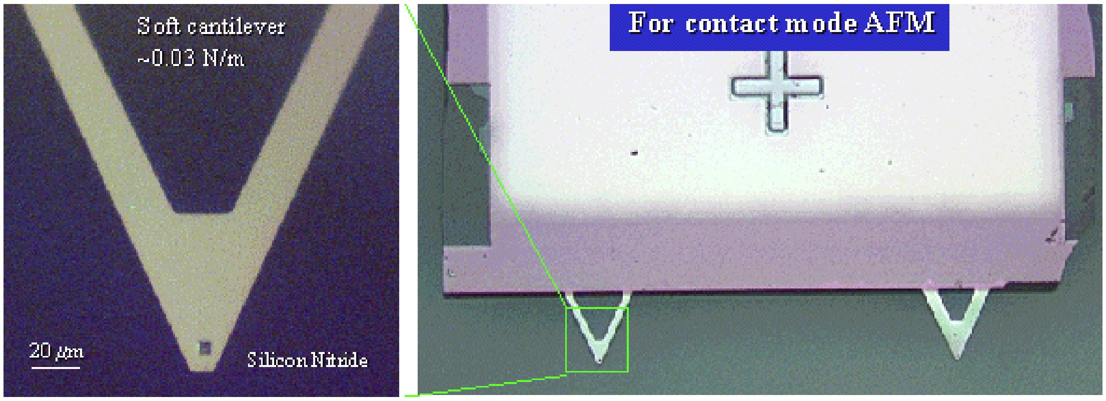


The attractive force between AFM probe tip and the sample surface is much larger than a two-atoms system. This is because the size of the tip is much larger than an atom. Typical radius of a commercial tip is ~ 20 nm.

The force range is also much longer.

The interaction between the tip and sample surface is measured through the deflection of the cantilever by means of a laser beam and photodetector.

The AFM uses the attractive or repulsive forces encountered by a probe tip when it is in close proximity to a sample surface (<200 nm).

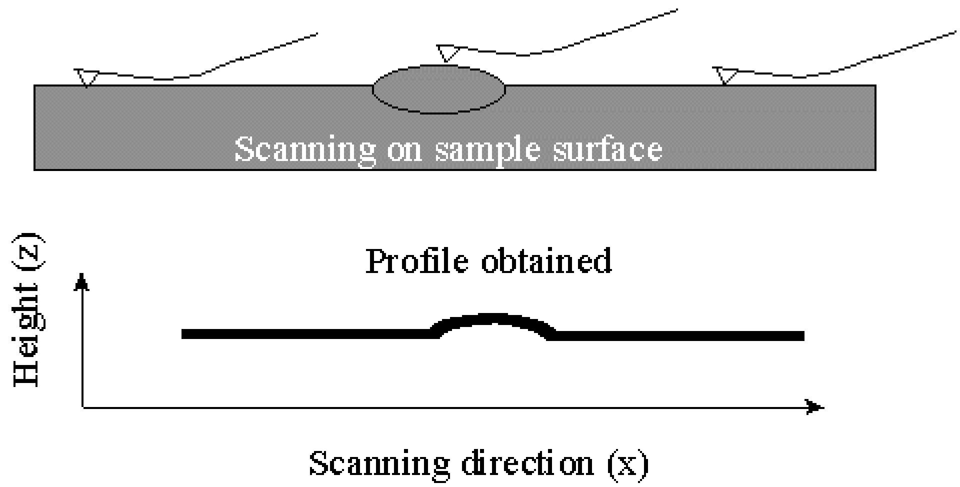


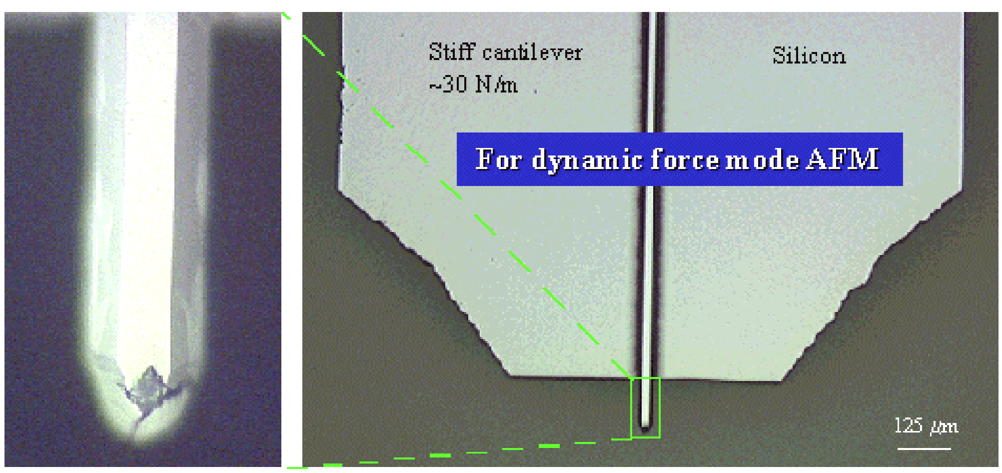
There are two types of AFM:

1. **contact mode** - Contact AFM is done by bringing the tip to a distance at which repulsive forces dominate the tip-sample interaction. A very soft cantilever ((0.01 ~ 1 N/m)tip is mechanically contacted with sample surface at an applied force. The AFM measures hard-sphere repulsion forces between the tip and sample.
   The tip and sample remain in close contact as the scanning proceeds.
   By "contact" mean in the repulsive regime of the inter-molecular force curve.
   Ionic repulsion forces take the leading role.
   The repulsive region of the curve lies above the x-axis.
   One of the drawbacks of remaining in contact with the sample is that there exist large lateral forces on the sample as the drip is "dragged" over the specimen.

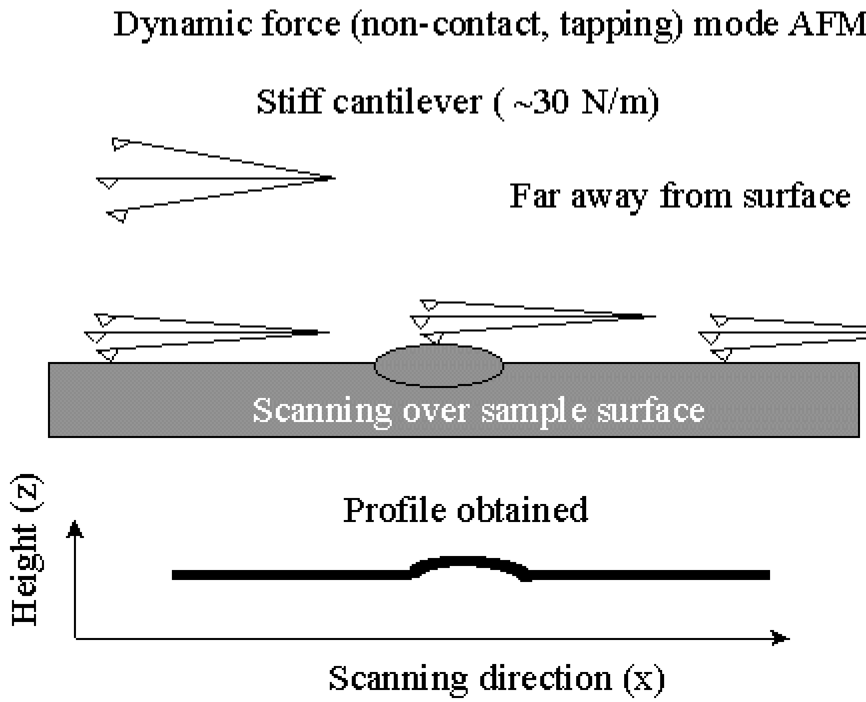
Contact mode AFM

Soft cantilever (~0.03 N/m)



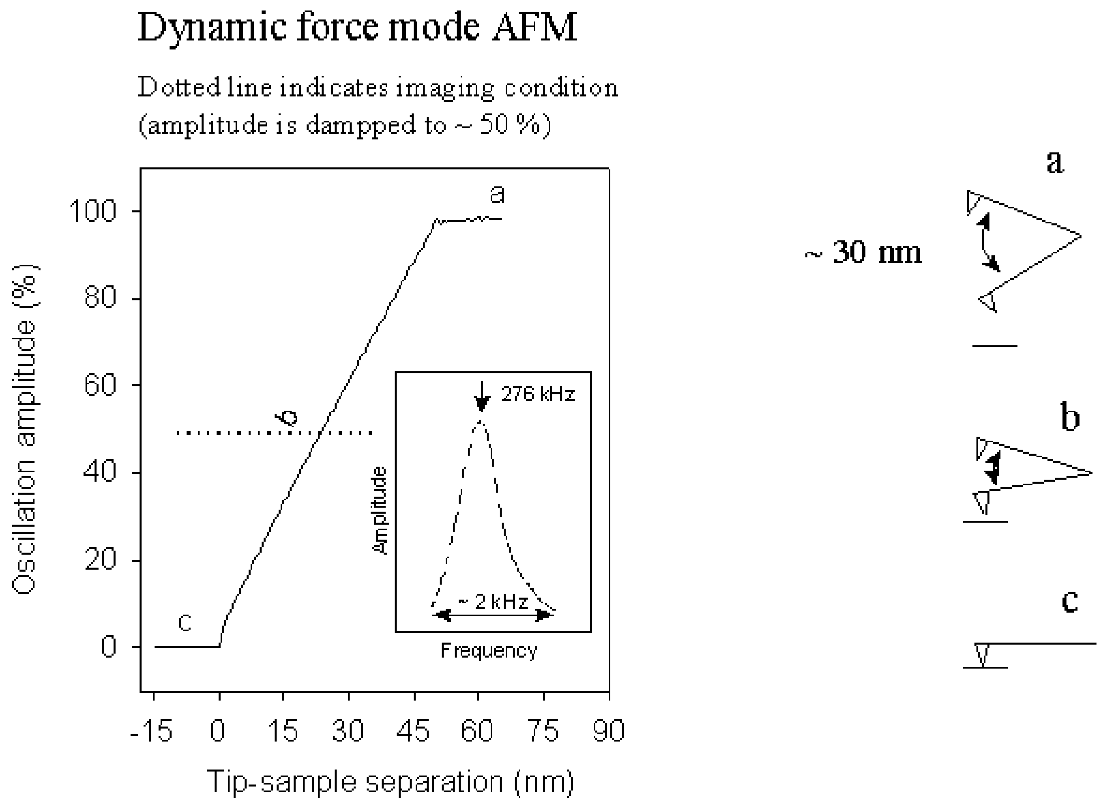


2. **dynamic force (tapping & non-contact) mode** - operated with a stiff cantilever (e.g., ~30 N/m) which is oscillated near its resonant frequency.



Non-Contact AFM -The cantilever must be oscillated above the surface of the sample at such a distance that we are no longer in the repulsive regime of the inter-molecular force curve such that the tip-sample interaction is in the attractive or van der Waals regime. In order to perform measurements in this attractive force region the cantilever is oscillated with a low amplitude (<5 nm), near its resonant frequency. For Non-Contact AFM the force is measured by comparing the frequency and/or amplitude of the cantilever oscillation relative to the driving signal.

The tip and the sample surface distance > 10Å Van der Waals, electrostatic, magnetic or capillary forces produce images of topography.



Tapping or Intermittant Contact mode is also done by oscillating the cantilever near its resonant frequency, but the amplitude is significantly higher (~10-50 nm). This Intermittant contact mode operates in the repulsive force region, but touches the surface only for short periods of time, in order to reduce damage to potentially fragile samples. The tip lightly taps the surface, touching the sample through the absorbed fluid layer during each oscillation. The technique is useful for “soft” surfaces. The feedback loop adjusts so that the amplitude of the cantilever oscillation remains (nearly) constant. An image can be formed from this amplitude signal, as there will be small variations in this oscillation amplitude due to the control electronics not responding instantaneously to changes on the specimen surface.

Resolution in AFM

Traditional microscopes have only one measure of resolution - in the focal plane of an image.

An atomic force microscope has two measures of resolution,

- In the plane of the measurement - The in-plane resolution depends on the geometry of the probe that is used for scanning. The sharper the probe the higher the resolution of the AFM image.

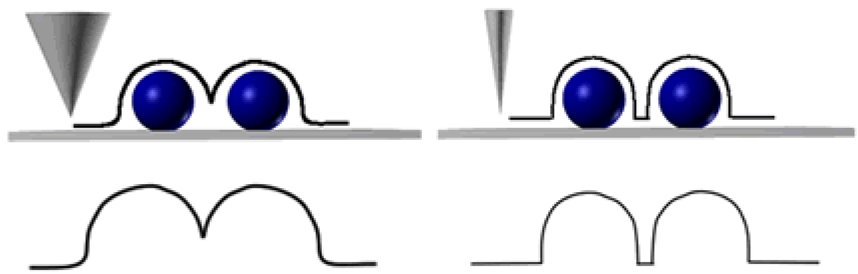


Figure : The image on the right will have a higher resolution because the probe used for the measurement is much sharper.

- in the direction perpendicular to the surface - The vertical resolution is established by relative vibrations of the probe above the surface. Can obtain maximum vertical resolution by minimizing the vibrations.

Probe Surface Interactions

The strongest forces between the probe and surface are mechanical, which are the forces that occur when the atoms on the probe physically interact with the atoms on a surface.

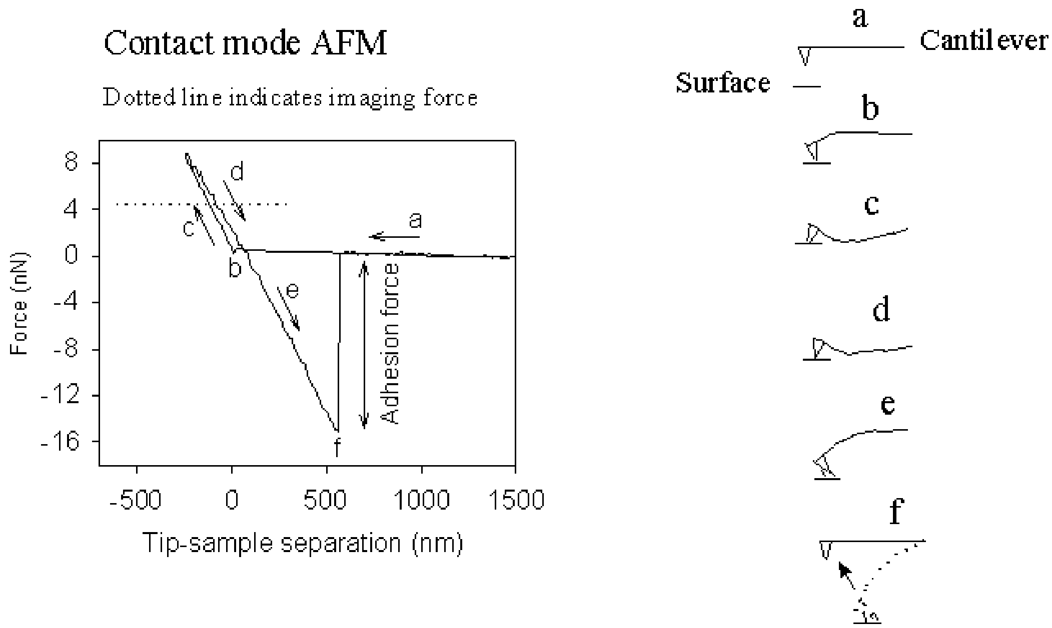
Other forces between the probe and surface can have an impact on an AFM image,

- surface contamination,
- electrostatic forces, and
- surface material properties.

The probe tip is mounted on the end of a triangular cantilever arm. A piezoelectric device raster scans the sample beneath the probe tip. As the probe tip undergoes attractive or repulsive forces, the cantilever will bend. This bending of the cantilever can be monitored by reflected laser beam (diode laser) off the cantilever onto a 2 element photodiode (Position Sensitive Photo Diode). In normal operation the tip-sample force is held constant by a computer controlled feedback loop that examines the force (bend of the cantilever) and tells the piezoelectric device whether to move the sample closer or farther away in order to maintain the set force value. The AFM topography image is produced by taking the feedback signal at each pixel of the raster scan.

TYPE OF IMAGING TECHNIQUES

1. Force distance curve



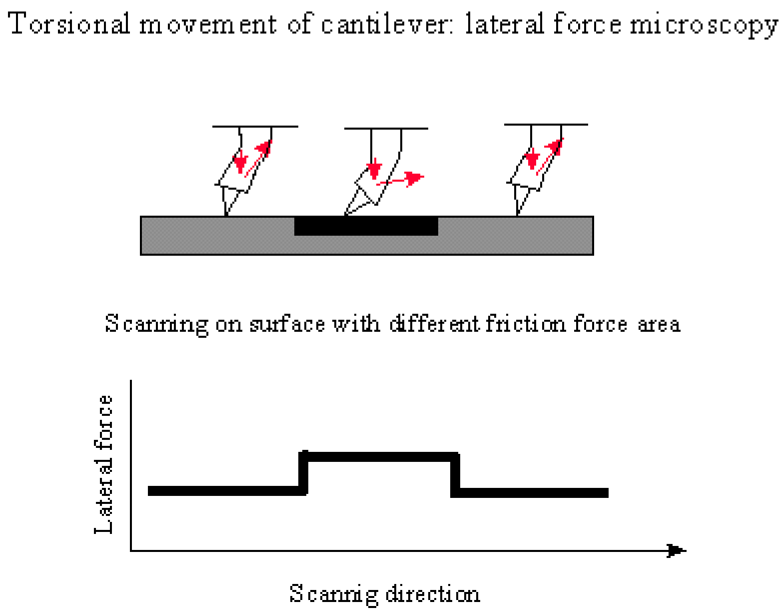
2. Force-distance curves

When the tip is brought to the surface followed by retracting the tip from the surface. The original point for the distance may be defined as the mechanical contact between the tip and surface in the extending cycle. Extending the tip beyond that point will result in load forces applied to the surface. The slope of this load force is a measure of the Young's modulus of the surface, possibly mixed with the spring constant of the cantilever. As a result, a cantilever whose spring constant is comparable with the surface stiffness should be used to measure the elasticity information.

In the retracting cycle, because of the adhesion properties between the tip and surface, the tip will not depart from the surface until the force used to pull the tip from the surface exceeds the adhesion force between them. This pull-off force can be considered as a measure of the adhesion force between the tip and surface.

3. Lateral Force Microscopy (LFM)

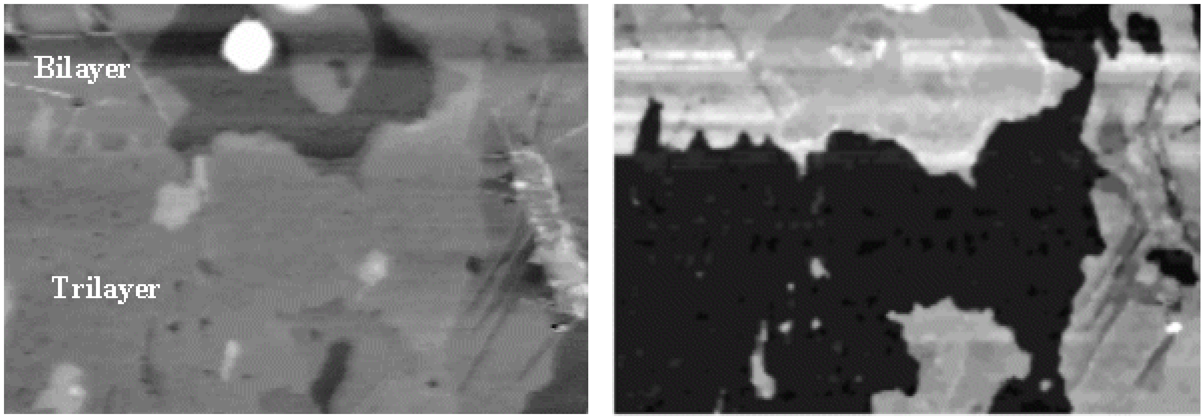
Lateral force microscopy (LFM) is based on measuring the torsional movement of the cantilever when the tip scans the surface.



Lateral force detection in AFM is usually used to image different friction forces on a surface. The difference in the bi-directional lateral force images corresponds to the friction force image. Friction force imaging has the ability to identify such regions of higher hydrophilicity on the basis of increased interaction with the AFM tip. Combined with height measurement, it can clearly identify whether a molecular layer is a bilayer (terminated by the headgroup) or a trilayer (terminated by the tail).

On the other hand, there is another effect of lateral force imaging to reveal local topography change by an enhancement of the torsional movement of the cantilever when the tip crosses edges of the surface features. This technique has proven useful in detecting different phases on a surface whose height range is large, which is the case for some practical polymer samples.

Topography Friction force image



4. Force Modulation

This feature can probe local elastic properties of materials through a mechanical interaction between the surface and tip. This is done by oscillating the sample height while measuring the response of the cantilever with lock-in amplifier technique. Elasticity difference on a surface can be distinguished by using this technique. This technique actually measures the slope of the force-distance curves at the repulsive force region.

The oscillation of the sample height may be realized by applying sinusoidal voltage from a function generator to the Z-direction of the piezo (PZT) scanner on which the sample is fixed. A sinusoidal voltage is applied to the piezo scanner to oscillate the sample height with a peak-to-peak amplitude of about 1 nm. The response of the cantilever to this oscillation is detected with a lock-in amplifier and is used to obtain images relevant to local elasticity of sample surface. The oscillation of the sample height would not influence topographic images as far as its frequency is higher than the cutoff frequency of the feedback loop. Therefore, both topography and elasticity distribution images can be obtained simultaneously.

5. Phase Imaging

The phase shift in the oscillating cantilever is related to tip-surface interaction which is basically material specific.

Therefore, phase shift contrast in tapping mode AFM can be used to distinguish different surface compositions on a surface. There are many surface properties that may have an effect on the phase shift contrast. They could be difference in friction, viscoelasticity, adhesion, material, etc.

Phase imaging usually gives clear contrast on a surface if there are detectable differences in surface properties.

The phase shift image should be careful and usually depends on other observations and background knowledge on the sample.

6. Magnetic Force Microscopy (MFM)

Magnetic information on a sample can be measured with a magnetized tip.

A local topographic data in each scan line is first obtained by dynamic force mode AFM.

Then the tip is lifted up in a certain distance and repeats to scan the same line.

The magnetic interaction between the tip and surface will give a change in the magnitude (or phase) of the oscillating cantilever, which gives regional information of magnetic force distribution on a surface.

The difference of the magnetic properties can be measured in this method.

This technique is useful to image magnetic force distribution on the recorded magnetic media (data storage) and micromagnetic structure on some magnetic materials.

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7. Electric Force Microscopy (EFM)

Similar to MFM, EFM uses a conductive tip to probe the difference of electric filed gradient distribution on a surface. This technique can be used in the failure check on integrated circuit (IC).

8. Scanning Surface Potential Microscopy (SSPM)

A new technique which maps local surface potential distribution together with topography

keeping a certain separation between the sample surface and conductive tip to which a sinusoidal voltage is applied.

In case of that there is a difference in potentials between the tip and sample surfaces, an oscillating electromagnetic force appears between the tip and sample surface at the frequency of the applied sinusoidal voltage, which makes the cantilever oscillate.

This oscillation is used as the feedback parameter for the system which tries to stop this oscillation by applying a dc voltage to the tip so as to make the potential difference between the tip and sample surfaces vanish.

This applied dc voltage to the tip is thus equal to the surface potential of the sample, which makes the surface potential measurable together with the topography.