

Atomic Force Microscopy

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Lecture Plan Introduction Atomic Force Microscopy Contact Non-contact Intermittent (Tapping Mode) **H**Tip Context SPM methods Applications



AFM vs Stylus Profilometry

In comparison with stylus profilometry (week 3 and 4).

#AFMs have a number of refinements

Sensitive detection

☐ flexible cantilevers

- △sharp tips
- high resolution tip-sample positioning
- force feedback





Force vs Distance : Contact mode

- The tip makes soft "physical contact"
- The tip is attached to the end of a cantilever with a low spring constant.
- During tracing, the contact force causes the cantilever to bend with changes in the topography.



Contact Mode Constant Height or Constant Force

Constant-force mode

The deflection of the cantilever can be used as input to a feedback circuit that moves the scanner up and down in z, responding to the topography by keeping the cantilever deflection constant.

Constant-height mode

A the spatial variation of the cantilever deflection can be used directly to generate the topographic data set because the height of the scanner is fixed as it scans.

Contact Mode Constant Height or Constant Force

Constant-force mode

The speed of scanning is limited by the response time of the feedback circuit, but the total force exerted on the sample by the tip is well controlled. Constant-force mode is generally preferred for most applications.

Constant-height mode

➢ is often used for taking atomic-scale images of atomically flat surfaces, where the cantilever deflections and thus variations in applied force are small. Constant-height mode is also essential for recording real-time images of changing surfaces, where high scan speed is essential.

Force vs Distance : Non Contact

The total force between the tip and the sample in the non-contact regime is low, generally about 10⁻¹²N. This low force is advantageous for studying soft or elastic samples. An additional advantage is that samples like silicon wafers are not contaminated through contact with the tip



Force vs Distance : Non Contact

- In non-contact mode, the system vibrates a stiff cantilever near its resonant frequency (typically 100 to 400 kHz) with an amplitude of a few tens to hundreds of angstroms. It then detects the changes in resonant frequency or vibration amplitude as the tip comes near the surface. The sensitivity of this detection system allows sub-angstrom vertical resolution.
- In non-contact mode, the system monitors the resonant frequency or vibrational amplitude of the cantilever and keeps it constant with the aid of the feedback system that moves the scanner up and down.
- Non-contact measurement does not suffer from the tip or sample degradation effects that are sometimes present after taking numerous scans with contact AFMs.

Conventional contact mode probe is dragged across surface **Drawbacks**



Can cause damage to both sample and probe Itrapped electrostatic charge can contribute to

additional adhesive forces



Non-contact mode

probe is held a small distance above surface

Brawbacks Brawbacks

van de Waals forces are weak so that the tip needs to be oscillated to detect small forces between tip and surface.

Thus, non-contact provides lower resolution

CappingMode imaging overcomes the limitations of the conventional scanning modes by alternately placing the tip in contact with the surface to provide high resolution and then lifting the tip off the surface to avoid dragging the tip across the surface.



During scanning, the vertically oscillating tip alternately contacts the surface and lifts off, generally at a frequency of 50,000 to 500,000 cycles per second. As the oscillating cantilever begins to intermittently contact the surface, the cantilever oscillation is necessarily reduced due to energy loss caused by the tip contacting the surface. The reduction in oscillation amplitude is used to identify and measure surface features



Tapping Mode inherently prevents the tip from sticking to the surface and causing damage during scanning. Unlike contact and non-contact modes, when the tip contacts the surface, it has sufficient oscillation amplitude to overcome the tip-sample adhesion forces. Also, the surface material is not pulled sideways by shear forces since the applied force is always vertical



Measurement of liquids



Cantilevers

Highly flexible stylus exerts a down force on the sample
Spring constant is 0.1 N/m
AFM has a high resonant frequency

resonant frequency=

 $\frac{1}{\tau}\sqrt{\frac{\text{spring constant}}{\text{mass}}}$

cantilever can have both a low spring constant and high resonant frequency if it has a small mass



Cantilevers

%Typically measure 100 microns in length
%Silicon oxynitride
%thin film of gold for reflectivity



Tips Types

Common types of AFM Tips
Inormal tip (3 microns tall)
for contact mode



Super tip for tapping mode

Ultralever (tapping mode)



Ultralevers[™] MAXIMUM RESOLUTION CANTILEVERS

FEATURES:

Tips

- Silicon cantilever with silicon conical tip.
- Tip height nominally 5 7 µm.
- Sharp, high aspect ratio tip.
- Typical radius of curvature 10 nm (60 nm for MFM Ultralevers).
- Recommended for non-contact mode.
- + Gold coated for high reflectivity.
- Boron doped silicon, 0.001Ω/cm.
- + Wide range of spring constants.
- Four different cantilevers on every chip.
- Compatible with any major brand AFM.
- Also available are MFM Ultralevers coated with cobalt for Magnetic Force Microscopy, and Tipless Ultralevers for special applications.



ThermoMicroscopes Ultralevers are high quality cantilevers ideal for most topographic applications including, metrology, high resolution, non-contact or intermittant contact imaging, and IC measurements. MFM Ultralevers are ideal for high resolution MFM imaging on "hard" magnetic surfaces. Tipless cantilevers are a perfect platform for the reasercher who wants to make a tip out of a custom material or other chemical applications.





MFM Ultralever



Typical mechanical characteristics

Cantilever type	A	В	С	D	A	В	С	D
Standard mode of operation	Contact				Intermittent and non-contact			
Cantilever length	180 µm	180 µm	85 µm	85 µm	180 µm	180 µm	85 µm	85 µm
Cantilever width	25 µm	38 µm	18 µm	28 µm	25 µm	38 µm	18 µm	28 µm
Cantilever thickness	1 µm	1 µm	1 µm	1 µm	2 µm	2 µm	2 µm	2 µm
Force Constant	0.26 N/m	0.40N/m	1.6 N/m	2.1 N/m	2.1 N/m	3.2 N/m	13 N/m	17 N/m
Resonant Frequency	40 kHz	45 kHz	140 kHz	160 kHz	80 kHz	90 kHz	280 kHz	320 kHz

Tips Resolution

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- Step size of the image
- Minimum radius of the tip.

Manufacture : Silicon

- Silicon conical tips are made by etching into the silicon around a silicon dioxide cap.
- * The high aspect ratio of conical tips makes them suitable for imaging deep, narrow features such as trenches, but they may break more easily than the pyramidal or tetrahedral geometries, as described above.
- Silicon also has the advantage that it can be doped, so that tips can be made electrically conducting. Conducting tips are useful for controlling the bias between the tip and the sample, or for preventing unwanted charge buildup on the tip

Manufacture : Silicon Nitride

Ips

- Silicon nitride tips are fabricated by depositing a layer of silicon nitride over an etched pit in a crystalline silicon surface.
- His method produces the pyramidal or tetrahedral tip geometry.
- Here aspect ratio of a silicon nitride tip is thus limited by the crystallographic structure of the etch pit material, silicon.



Resolution

The sharpest tips available commercially can have a radius as small as 50 angstroms. Because the interaction area between tip and sample is a fraction of the tip radius, these tips typically provide a resolution of 10 to 20 angstroms. Thus, the resolution of AFM images larger than 1 micron by 1 micron is usually determined not by the tip but by the image's step size.







SPM Types Lateral Force Microscopy Measures lateral deflections (twisting) Useful for variations in surface friction



SPM Types Lateral Force Microscopy

- Changes in slope
- In order to determine between surface friction and slope both LFM and AFM images need to be collected simultaneously.





SPM Types Lateral Force Microscopy

- Useful for identifying surface compositional differences where the materials have differing frictional characteristics and the topography is relatively the same.
- Constraints of the Hamiltonian Hamilton Hamilton
 - rough topography



Polished polycrystalline silicon carbide

SPM Types Force Modulation Microscopy

Maps differences in surface stiffness or elasticity





SPM Types Force Modulation Microscopy

Drawback

image

 high frequency modulation can excite a number of the scanner's mechanical resonances
 can reduce quality of





SPM Types Phase Detection Microscopy

Phase imaging is useful for differentiating between component phases of composite materials.





Similarly, the twophase structure of the polymer blend in this figure is shown clearly by the high resolution of phase imaging.



SPM Types Electrostatic Force Microscopy

- EFM plots the locally charged domains of the sample surface, similar to the MFM plots.
- **EFM** is used to study the spatial variations of the surface charge carrier density, for example, EFM can map the electrostatic fields of electronic circuit as the device is turned on and off.

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Single-stranded DNA seen as the 'ghostly' strand surrounded by duplex DNA. Sample pRH3 (6.16kb) plasmid DNA, heated to melt duples then adsorbed to poly-l-lysine mice Scan size 900 x 900 nm



Contact mode height and friction image of oil droplets (fingerprint residue) on glass. Contact mode height and friction image of oil droplets (fingerprint residue) on glass.



AFM height (left) and phase (right) images of poly(methylmethacrylate)



http://www.di.com/NanoTheatre/theater.html

Track edge on a commercial hard disk. The surface topography (left) shows texture and 80nm-scale film morphology. The magnetic force gradient image (right),. 5µm scan.



http://www.di.com/NanoTheatre/theater.html

Phase and TappingMode images of wood pulp fibre



http://www.di.com/NanoTheatre/theater.html

Silicon wafer after an RCA-type clean. This particular clean increased the surface roughness of the silicon from its as received value, typically 0.1nm RMS or less, to 0.73nm RMS.



Summary

% Introduce AFMs
% Different Modes
% Construction
% Types
% Applications