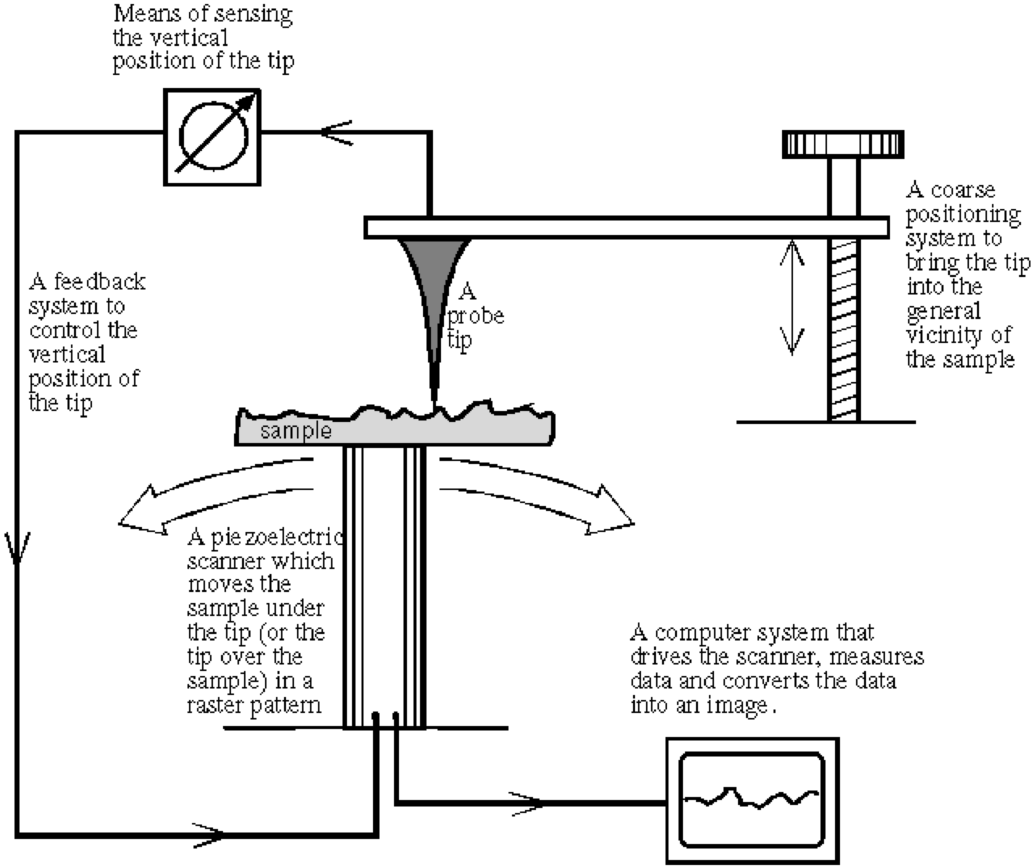
"SPM Techniques"

Scanning probe microscopes (SPMs) are a family of instruments used for studying surface properties of materials from the atomic to the micron level. All SPMs contain the components illustrated in Figure.



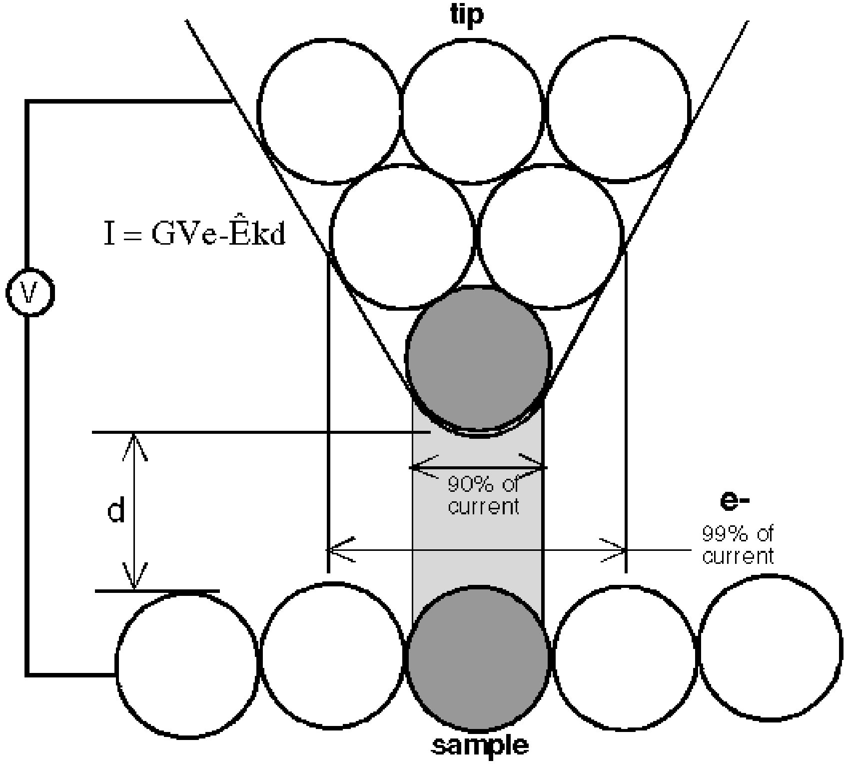
Schematic of a generalized SPM.

Scanning Tunneling Microscopy

The scanning tunneling microscope (STM) is the ancestor of all scanning probe microscopes. It was invented in 1981 by Gerd Binnig and Heinrich Rohrer at IBM Zurich. Five years later they were awarded the Nobel prize in physics for its invention. The STM was the first instrument to generate real-space images of surfaces with atomic resolution.

V = voltage applied

d ≤ 10 Å



For tunneling to take place, both the sample and the tip must be conductors or semiconductors

Figure 1-2. Schematic of tip and sample interaction

STMs can be designed to scan a sample in either of two modes:
constant-height or ***constant-current*** mode

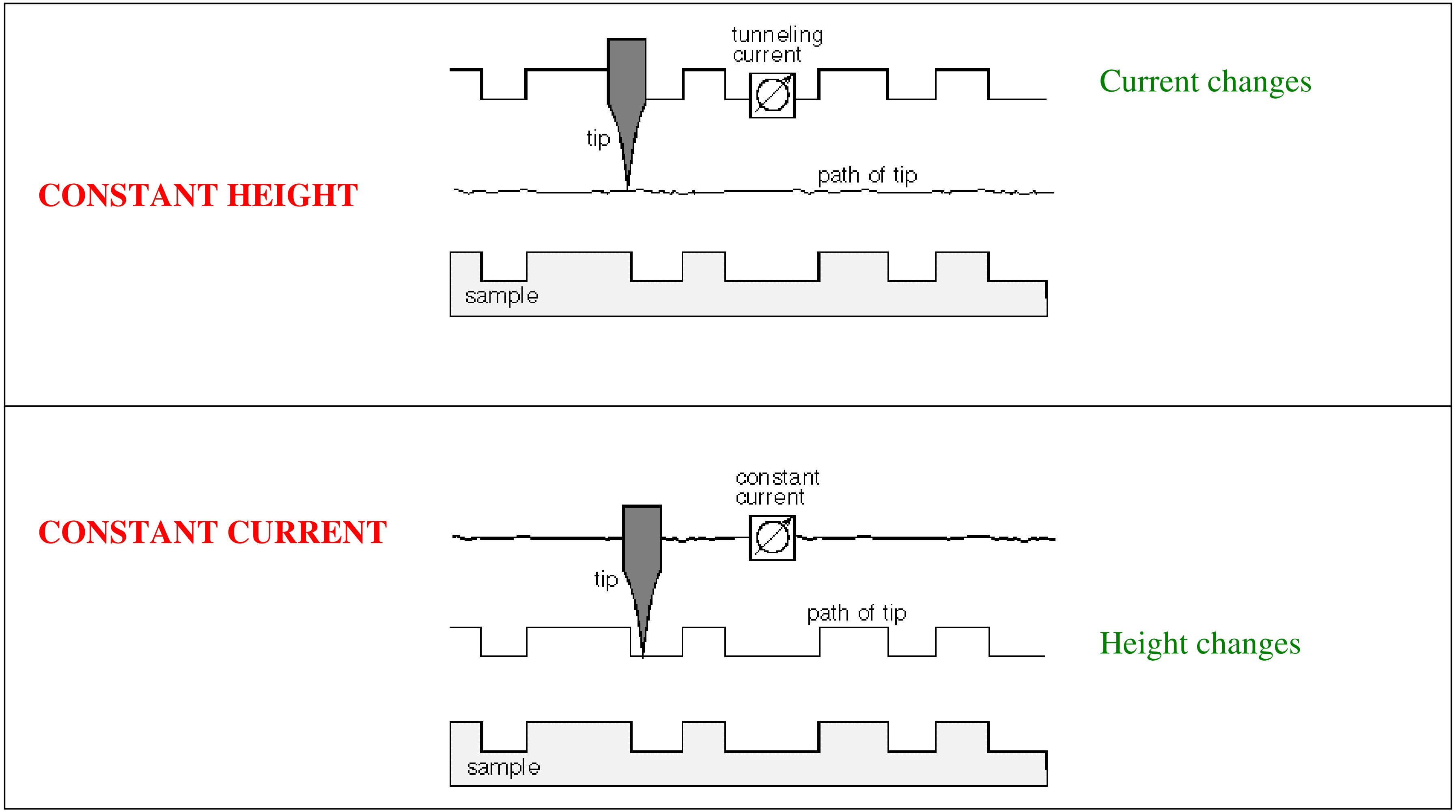
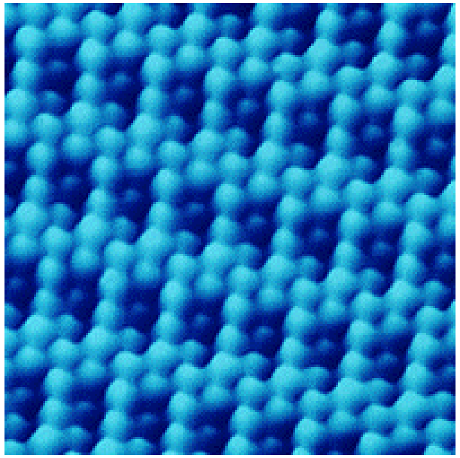
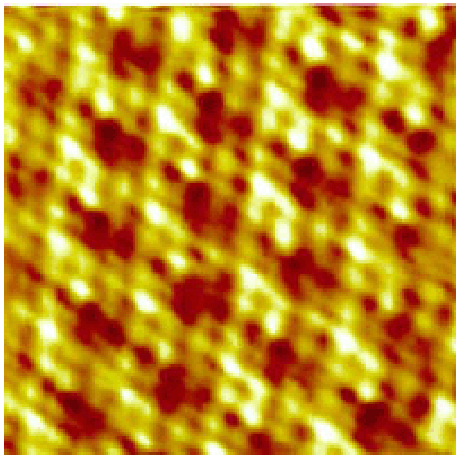


Figure: Comparision of constant-heightand constant-current mode for STM

O on Single Crystal



STM image of oxygen atom lattice on rhodium single crystal; part of study of electrocatalysis. 4nm scan courtesy Purdue University.

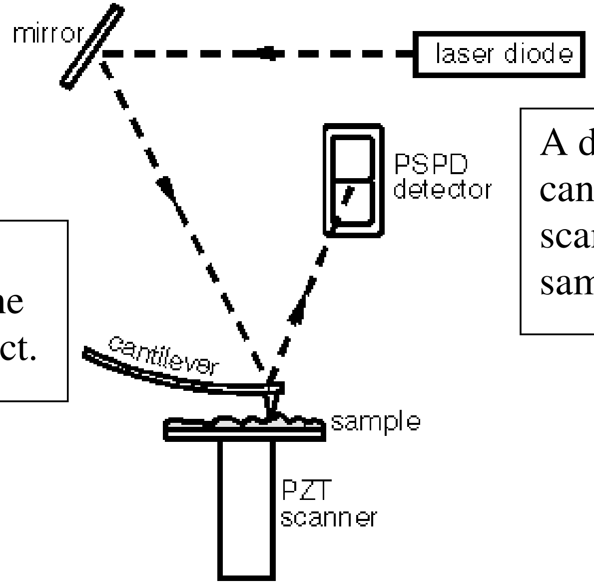


ECSTM image of Ag under potential deposition on Au (111) surface. The solution is 0.1M H2SO4 +0.001M AgClO4 and the potential is 302mV. The image was obtained from the first digital instruments ECSTM established in U.K. 6 nm scan courtesy , North Dakota State University, USA, and G. Attard, University of Wales, UK.

Atomic Force Microscopy

The atomic force microscope (AFM) probes the surface of a sample with a sharp tip, a couple of microns long and often less than 100Å in diameter.
The tip is located at the free end of a cantilever that is 100 to 200µm long.

Forces between the tip and the sample surface cause the cantilever to bend, or deflect.

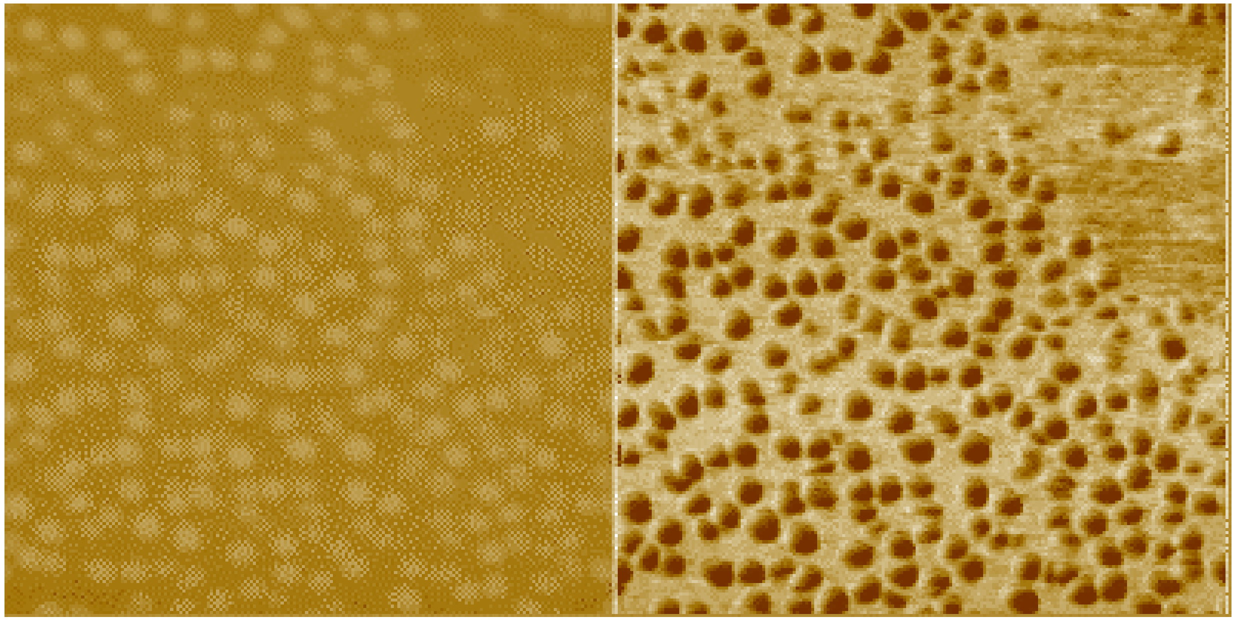


A detector measures the cantilever deflection as the tip is scanned over the sample, or the sample is scanned under the tip.

The beam-bounce detection scheme.

The measured cantilever deflections allow a computer to generate a map of surface topography.
Once the AFM has detected the cantilever deflection, it can generate the topographic data set by operating in one of two modes - ***constant-height*** or ***constant-force*** mode.
AFMs can be used to study insulators and semiconductors as well as electrical conductors.

Topographical and force modulation images of a silicon wafer pulled from a fab line after the lithography process

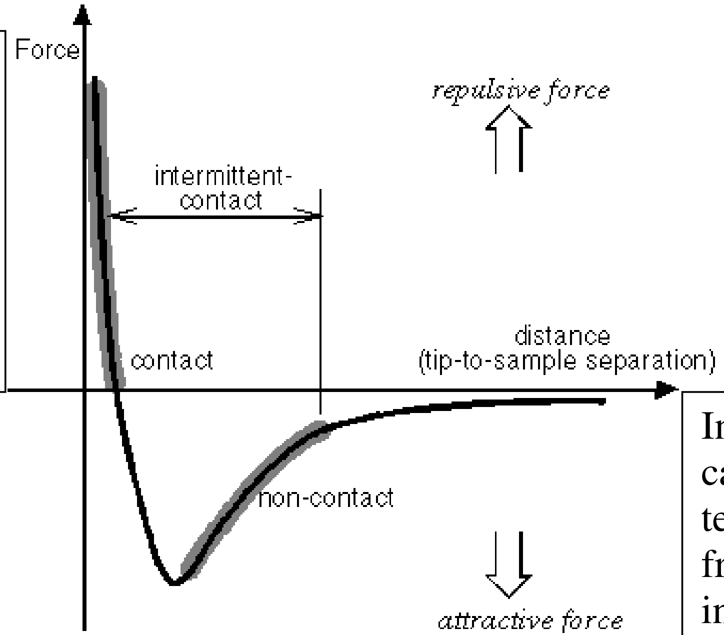


Contact and Non-contact AFM

Several forces typically contribute to the deflection of an AFM cantilever. The force most commonly associated with atomic force microscopy is an interatomic force called the van der Waals force. The dependence of the van der Waals force upon the distance between the tip and the sample is shown in Figure

Two distance regimes are labeled on Figure **1) the contact regime**; and **2) the non-contact regime** (largely a result of the long-range **van der Waals** interactions).

In the contact regime, the cantilever is held less than a few angstroms from the sample surface, and the interatomic force between the cantilever and the sample is repulsive.



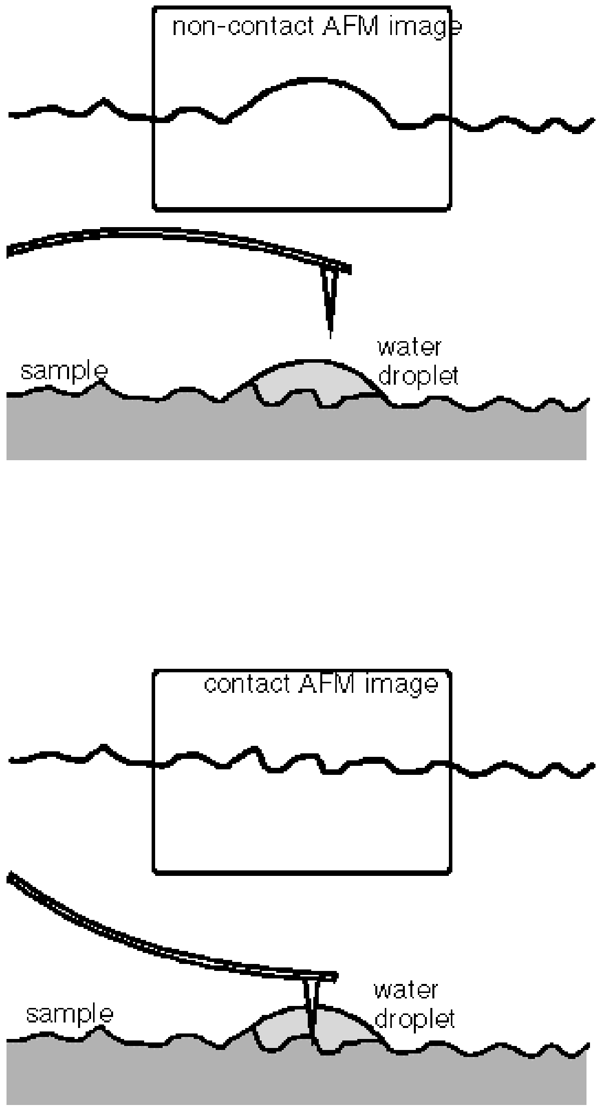
In the non-contact regime, the cantilever is held on the order of tens to hundreds of angstroms from the sample surface, and the interatomic force between the cantilever and sample is attractive

Interatomic force vs. distance curve.

In **contact AFM** mode, also known as repulsive mode, an AFM tip makes soft "physical contact" with the sample. The tip is attached to the end of a cantilever with a low spring constant, lower than the effective spring constant holding the atoms of the sample together.
The magnitude of the total force exerted on the sample varies from 10^{-8} to the more typical operating range of 10^{-7} to 10^{-6}N.

Non-contact AFM (NC-AFM) is one of several techniques in which the cantilever is vibrated near the surface of a sample. The spacing between the tip and the sample is on the order of tens to hundreds of angstroms.
NC-AFM is desirable because it provides a means for measuring sample topography with little or no contact between the tip and the sample.
The small force values in the non-contact regime (10^{-12} N) and the greater stiffness of the cantilevers used for NC-AFM are both factors that make the NC-AFM signal small, and therefore difficult to measure. Thus, a sensitive, AC detection scheme is used for NC-AFM operation.
In non-contact mode, the system vibrates a stiff cantilever near its resonant frequency (typically from 100 to 400 kHz) with an amplitude of a few tens of angstroms. Then it detects changes in the resonant frequency or vibration amplitude as the tip comes near the sample surface.

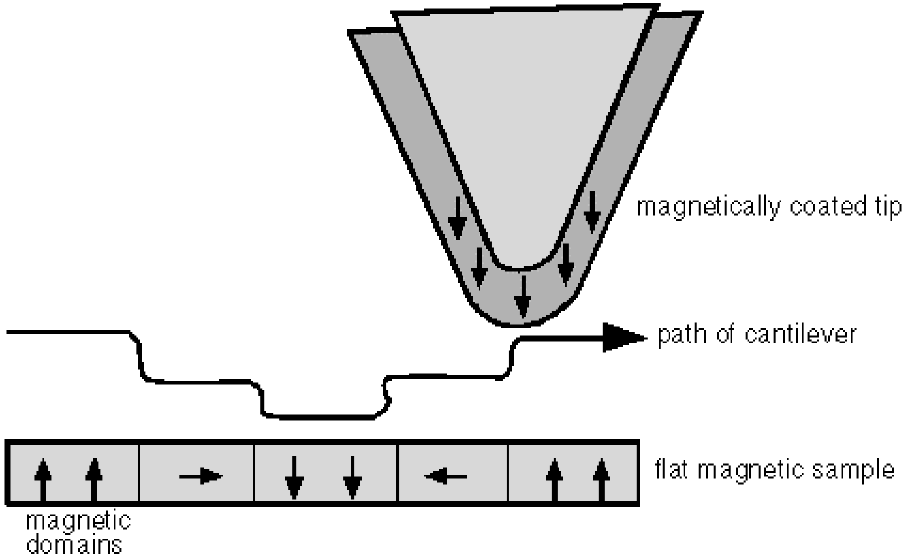
Intermittent-contact mode (IC-AFM) is similar to NC-AFM, except that for IC-AFM the vibrating cantilever tip is brought closer to the sample so that at the bottom of its travel it just barely hits, or "taps" the sample.



Contact and non-contact AFM images of a surface with a droplet of water.

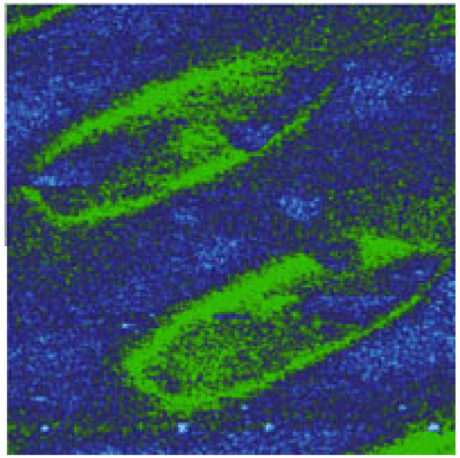
Other SPM Techniques

Magnetic Force Microscopy



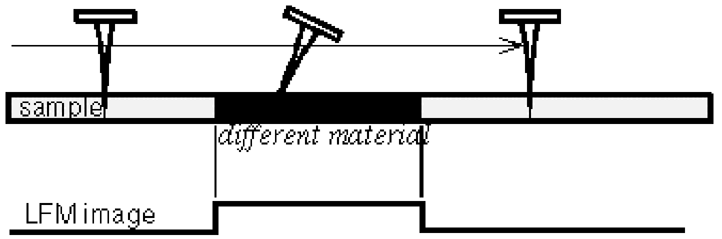
MFM maps the magnetic domains of the sample surface.

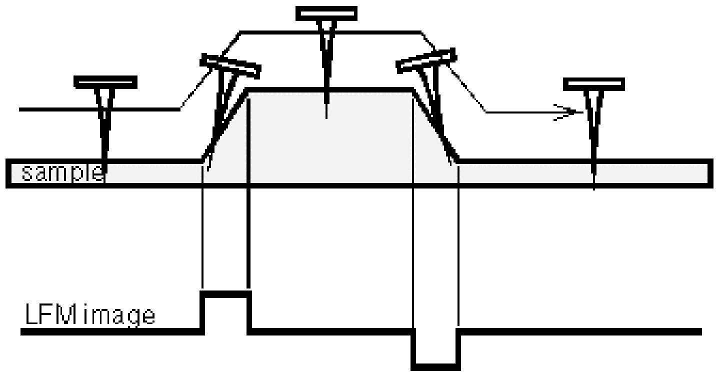
Magnetic memory bits



Phase image of magnetic memory bits showing locations where the total contribution from two magnetic layers has a net non-zero magnetization (green areas). 3.5 μm scan in lift mode, lift =150 nm, courtesy , M. Kowalewski, Oak Ridge National Laboratory, USA.

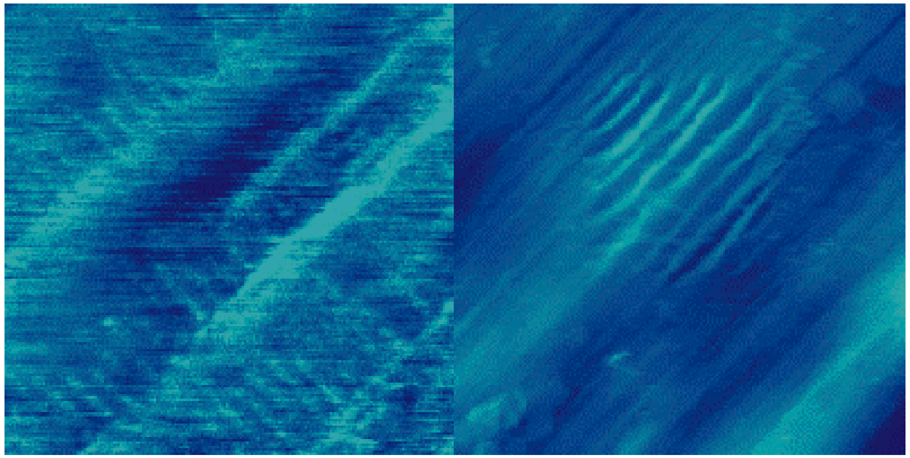
1.4 Lateral Force Microscopy

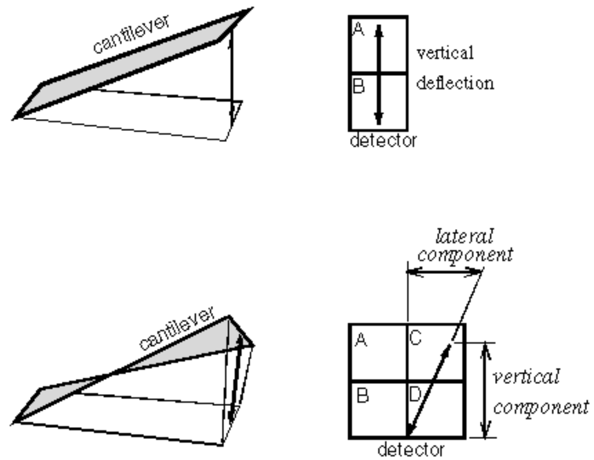




Lateral deflection of the cantileverfrom changes in surface friction (top) and from changes in slope (bottom).

Lateral force image of an oriented PE tape showing transverse nanofibrils. 125nm scan obtained under water





The PSPD for AFM (top) and LFM (bottom).

Force Modulation Microscopy

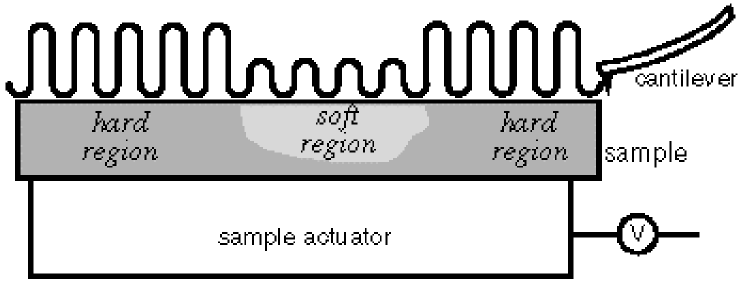


Figure 1-11. The amplitude of cantilever oscillation varies according to the mechanical properties of the sample surface. (bottom).

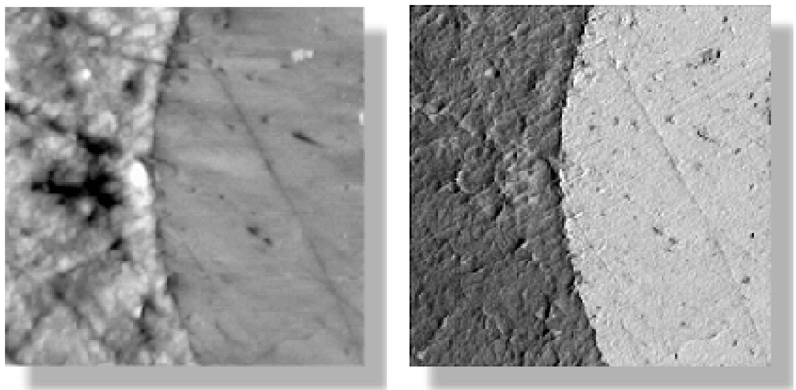


Figure 1-12. Contact-AFM (left) and FMM (right) images of a carbon fiber/polymer composite collected simultaneously Field of view 5μm

Phase Detection Microscopy

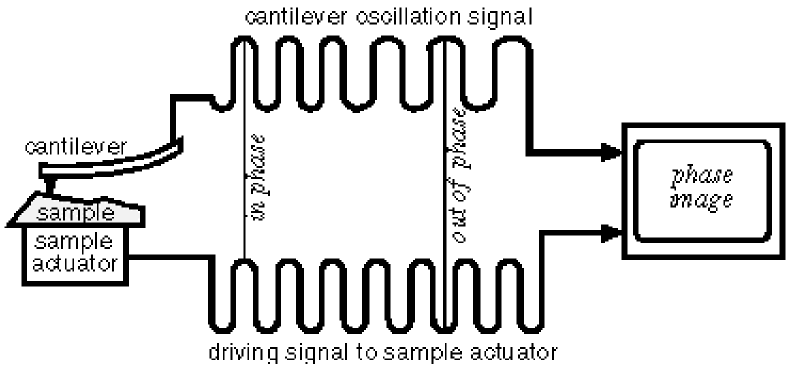


Figure 1-13. The phase lag varies in response to the mechanical properties of the sample surface

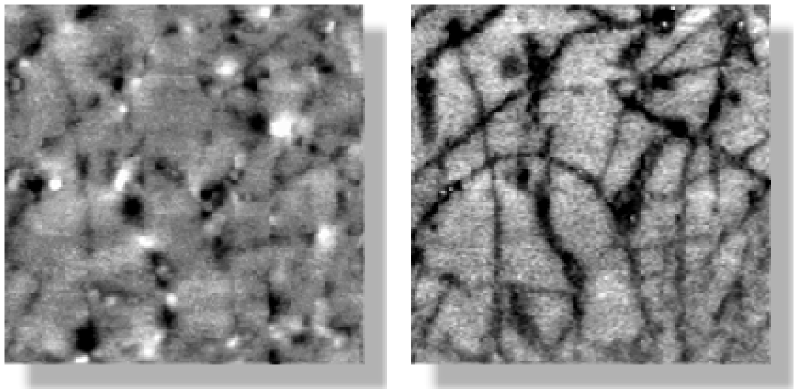
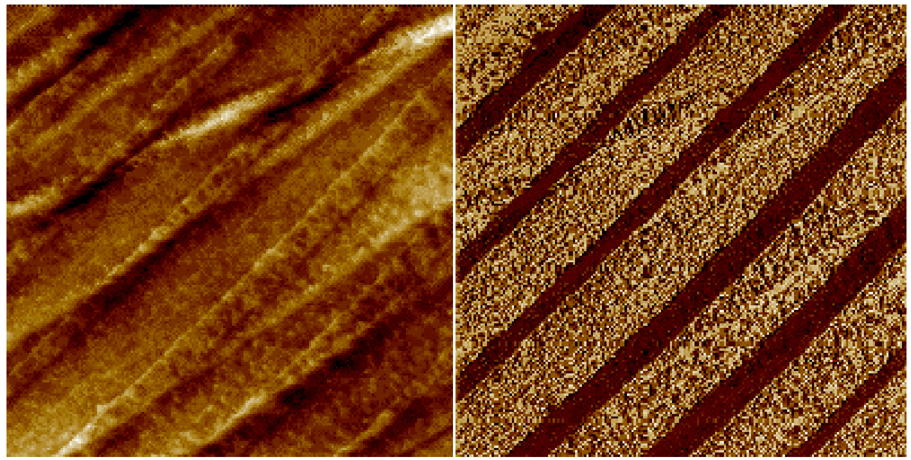


Figure 1-14. Non-contact AFM image (left) and PDM image (right) of an adhesive label, collected simultaneously. Field of view 3µm.

TappingMode AFM height and phase images simultaneously recorded on a microtomed cross-section surface of a multilayered sample



Electrostatic Force Microscopy

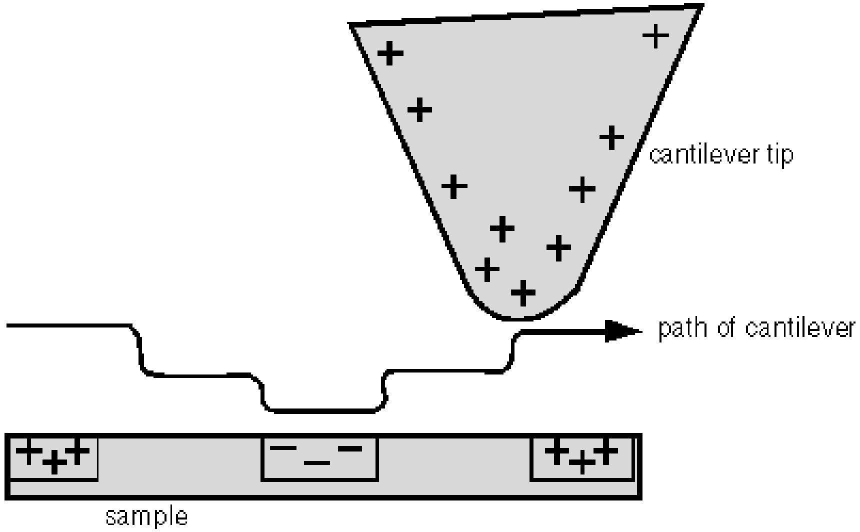
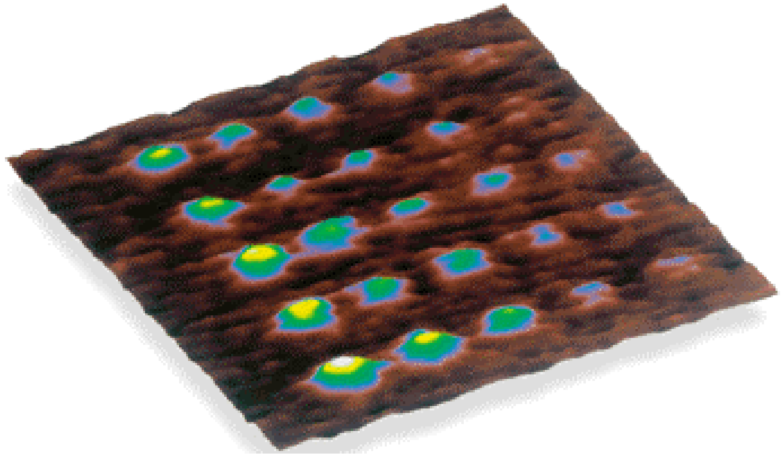


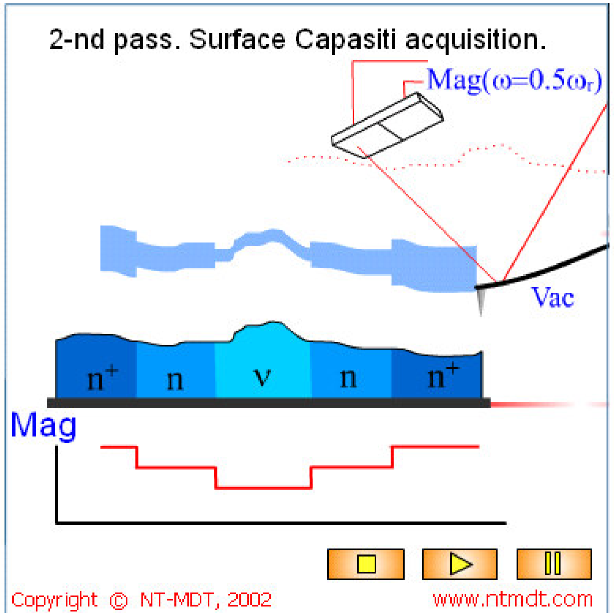
Figure 1-15. EFM maps locally charged domains on the sample surface.

Dots of charge



Dots of charge stored under various tip biases and imaged by EFM. Charge was stored in a double barrier Cerium Oxide/Silicon quantum well and remained up to 48 hours. the smallest dots in the array were measured to have the equivalent charge of 1 electron. 1.9µm scan courtesy of <u>P. Bridger</u> and J. Jones, California Institute of Technology, Pasadena.

Scanning Capacitance Microscopy



Scanning Capacitance Microscopy is kind of dynamic EFM. Generally [1] in EFM. the cantilever is biased directly by $V_{tip}=V_{dc} + V_{ac}\sin(wt)$, where V_{ac} is referred to as the driving voltage. Scanning is executed on some height h above the sample surface in according with the profile defined during the first scanning in Semicontact mode. The capacitive force $F_{cap}(z)$ between the tip and a sample surface at potential V_s is

$$F_{cap}(z) = (1/2)\,(V_{tip} - V_s)^2 (dC/dz)$$

where C(z) is the tip-surface capacitance dependent on tip geometry, surface topography and tip-surface separation z.

Second harmonic of the capacitive force depends only on (dC/dz) and V_{ac}

$$F_{cap2w}(z) = (1/2)(dC/dz)\,V_{ac}^2 \sin(2wt)$$

and can be used for acquisition additional information, e.g distribution of the surface capacity over the sample. For maximization of the second harmonic oscillations the ac frequency w is adjusted to be equal to half of cantilever resonance frequency w_r.

References

1. Nanotechnology 12, 485 (2001).
2. Appl. Phys. Lett. 52, 1103 (1988).
3. J. Appl. Phys. 61, 4723 (1987).

Near-field Scanning Optical Microscopy

Nanolithography

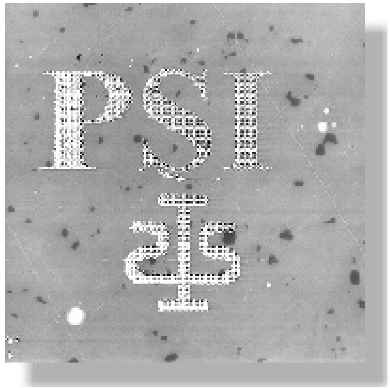


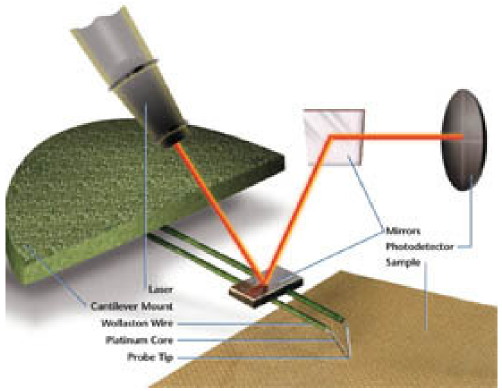
Image of a photoresistive surface that has been modified using nanolithography. Field of view 40µm.

Pulsed Force Mode

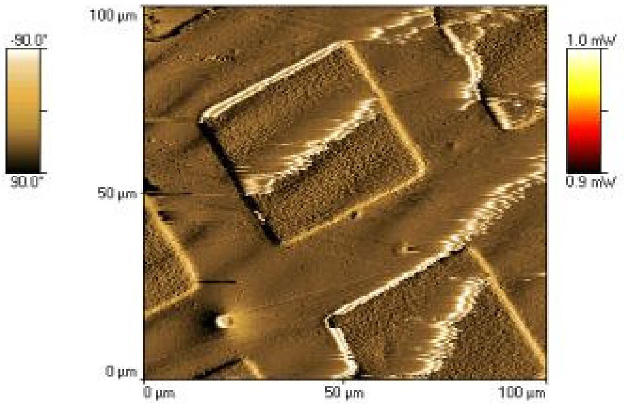
Scanning Thermal Microscopy

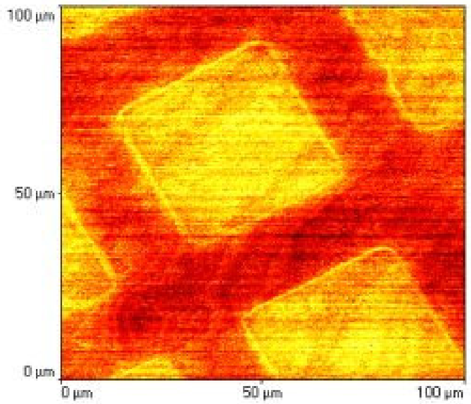
Micro-Thermal Analysis

Schematic representation of µ*TA* configuration



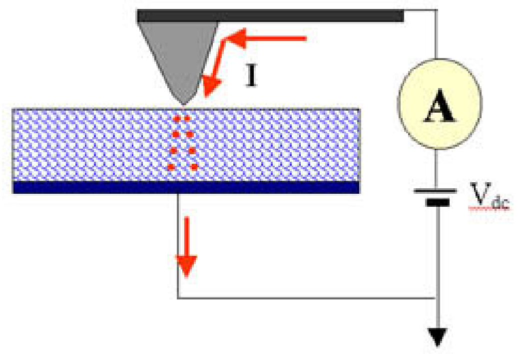
Characterisation of a Metal Coating on a Polymer Film by Micro-Thermal Analysis





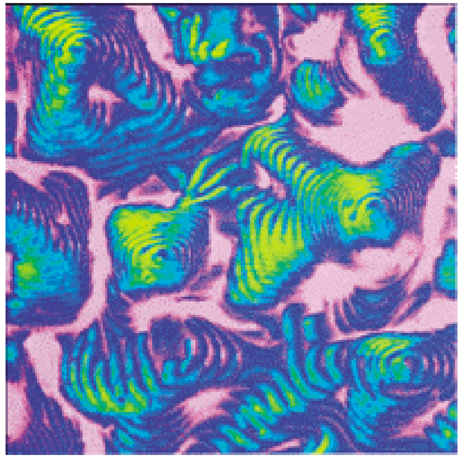
The image above shows the topography and thermal image of tin squares vacuum coated onto a polyester film. Localised thermal analysis carried out on the tin, shown below, measured the melting of the tin at 232°C followed by the melting of the polymer.

Conductive AFM



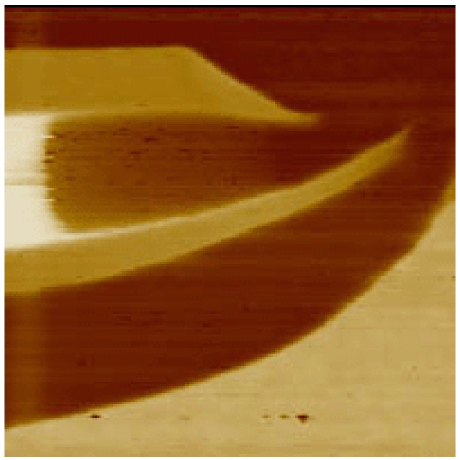
Current flow of conductive AFM Setup

Electrical Resistance between Thin Superconducting Film of YBaCuO and Conducting Probe



Cartography of the local electrical resistance between a thin superconducting film of YBaCuO (200nm) sputtered on a SrTi03 single-crystal substrate and a conducting probe made of n-doped silicon coated with p-doped diamond, obtained in contact AFM mode with a home-built extension of the NanoScope

Cross-sectioned InP-based laser



5x5 micron cross-sectioned InP-based laser structure imaged with Scanning Spreading resistance Microscopy (SSRM). The image displays the differently doped areas of the structure: n-type, p-type and isolaing areas. Scan courtesy , Digital Instruments, Veeco Metrology Group, Santa Barbara, CA.