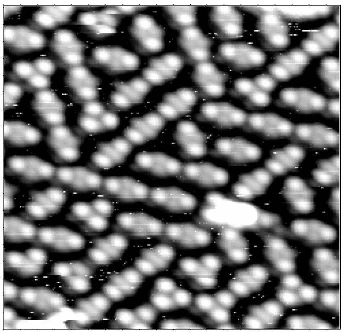
Scanning Probe Microscopy
2010



IF1602 Materialfysik för E

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2010

Scanning Probe Microscopy (SPM)

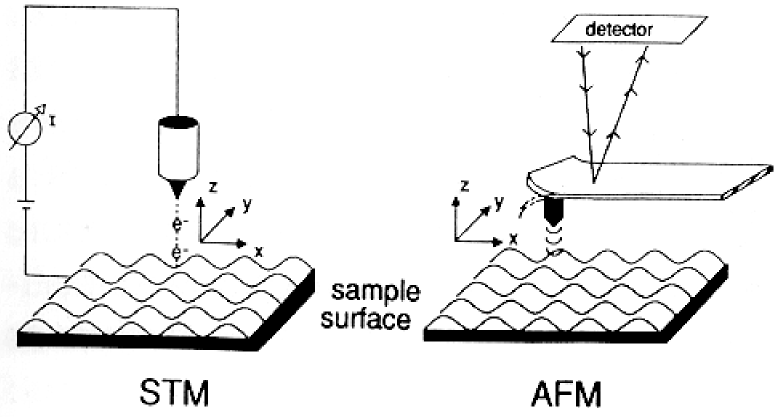
Scanning probe microscopy (SPM) is the name for a class of microscopy techniques that offers spatial resolution down to a few Ångströms. This extreme resolution has led to a new understanding of the structure of materials and forms of life. With the help of scanning probe microscopy it is possible to look into the fascinating world of the atoms.

SPM works without optical focusing elements, instead a sharp probe tip is scanned across a surface and probe-sample interactions are monitored to create an image of the surface. There are basically two types of microscopes, scanning tunnelling microscopy (STM) and atomic force microscopy (AFM).

Scanning Tunneling Microscopy (STM) was developed by Gerd Binnig and Heinrich Rohrer in the early 80's at the IBM research laboratory in Rüschlikon, Switzerland. For this revolutionary innovation Binnig and Rohrer were awarded the Nobel Prize in Physics in 1986. In STM, a small sharp conducting tip is scanned across the sample's surface, so close that a tunnel current can flow. With the help of that current the tip-surface distance can be controlled with such precision that the atomic arrangement of metallic or semiconducting surfaces can be determined. STM is restricted to electrically conducting surfaces.

A further development of STM called Atomic Force Microscopy (AFM) was developed by Gerd Binnig, Calvin Quate and Christoph Gerber. AFM extends the abilities of the STM to include electrically insulating materials.

Instead of measuring a tunnel current atomic-range forces between tip and sample surface are measured. The tip is attached to the end of a cantilever in order to measure these forces. The force acting on the tip can then be determined by detecting the deflection of this cantilever.

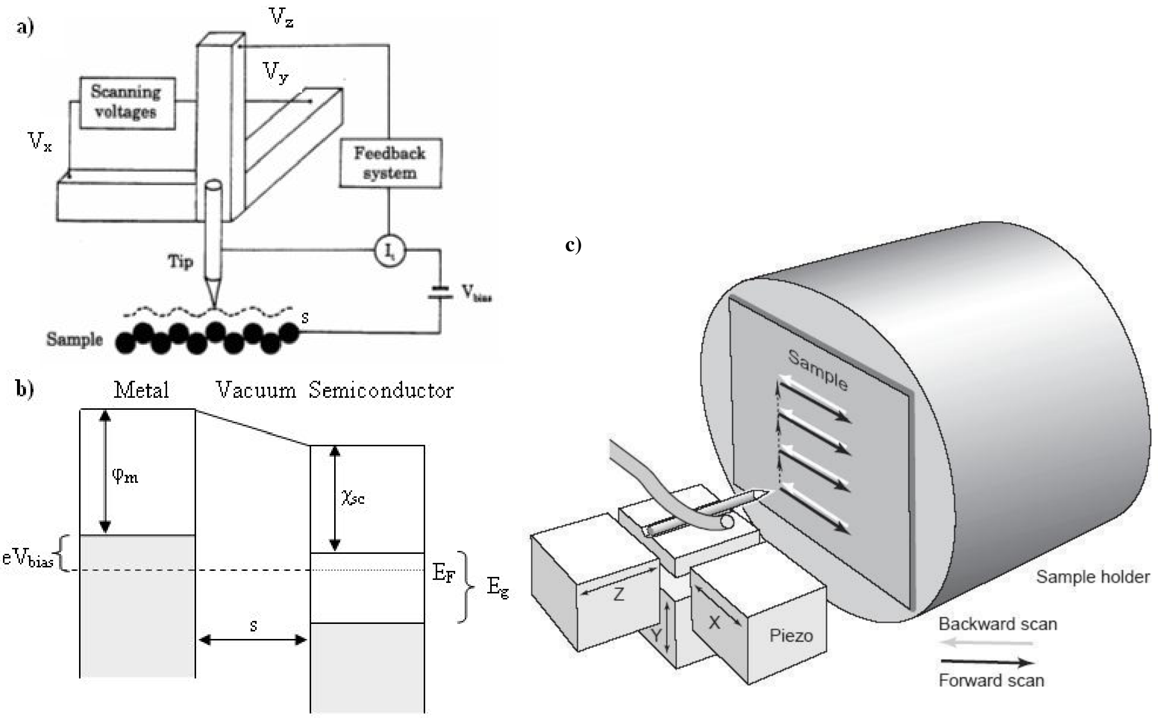


Principles of STM and AFM

The scanners are made from piezoelectric material, which changes its shape in response to an applied voltage. The magnitude of the change in shape (e.g. elongation or shearing) is roughly proportional to the applied voltage. Several independent piezoelectric elements are combined in order to enable motion in three dimensions. To achieve atomic resolution, ultra-clean and flat surfaces prepared in highly sophisticated vacuum systems are needed in most cases. Nevertheless, measurements in air can give useful results for many technically relevant surfaces. The instruments you will use are the Nanosurf® easyScan2 STM and AFM systems. You can find out more on their homepage www.nanosurf.com under *products*, *Nanosurf® easyScan2*.

Scanning Tunneling Microscopy (STM)

In the easyScan 2 STM, a platinum-iridium tip is moved in three dimensions using piezo-electric translators that are driven with sub-nanometer precision. The sample to be examined approaches the tip within a distance of 1 nanometer. Classical physics does not allow electrons in the small gap between a tip and a sample, but if a sharp tip and a conducting surface are put under a low voltage (V_{bias} on the order of a few volts) a very small tunneling current (I_t in the order of nA) may nevertheless flow between tip and sample. This tunneling current is an effect of quantum physics.

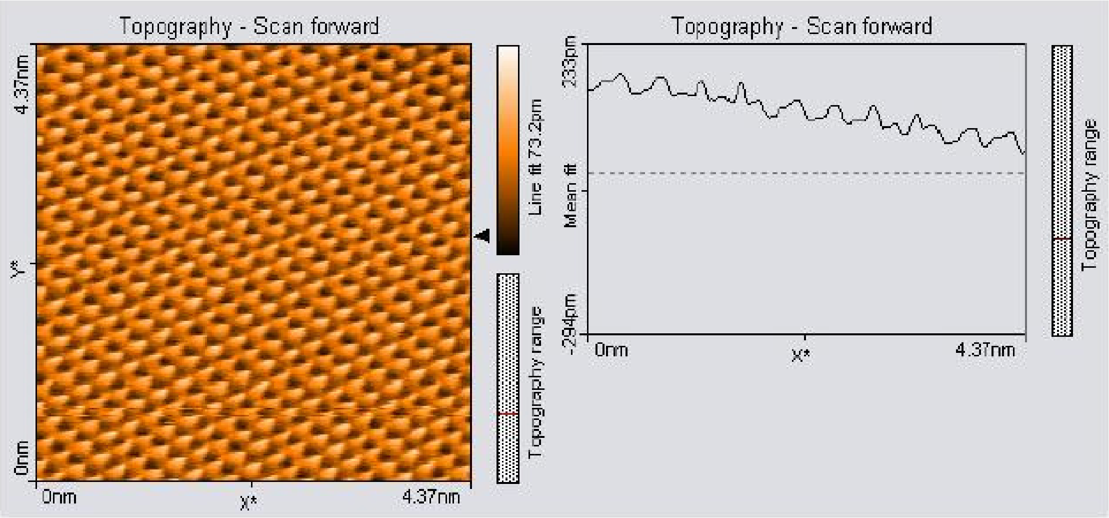


The schematics of the scanning tunneling microscope: a) fundamental layout, the tunnel current (I_t) is kept constant between the tip and the sample via a feedback loop. The.scanning motion of the tip is controlled by applying voltages (V_x and V_y) to the piezoelectric elements. A topographic image is created by recording V_z. b) energy level alignment in STM, in this case for a semiconducting sample. c) the layout of the microscope easyScan used in this lab.

The strength of the tunneling current depends exponentially on the distance between the tip and the sample (usually referred to as z-distance). This extreme dependence on the z-distance makes it possible to measure the tip-sample movement very precisely. One of the three piezo crystals, the z-piezo, can now be used in a feedback loop that keeps the tunneling current constant by appropriately changing the z-distance. Applying a positive bias causes electrons to tunnel from the tip to unoccupied states and applying a negative bias causes tunneling to occur from occupied states.

To obtain an image of the sample, the tip is scanned using the x- and y-piezo crystals. The feedback loop will now let the tip follow the structure of the sample's surface. A height image can now be made by recording the position of the z-feedback loop as a function of the x- and y-piezo position. This ‘landscape’ (or topography) of the atomic surface is then drawn line by line on the computer screen.

The sample can also be scanned in a second mode: when the feedback loop is inhibited, the tip scans at a fixed distance from the sample (constant height mode). This time the variations in the tunneling current are measured and drawn line by line on the computer screen. However, this mode only works when the sample is atomically flat, because the tip would otherwise crash in to the sample.



STM image of graphite as seen in the easyScan software

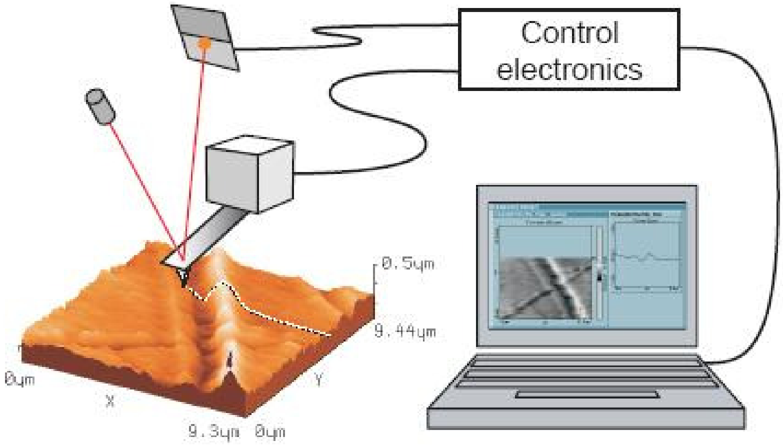
Atomic Force Microscopy (AFM)

The sensitive probe of an AFM is an elastic cantilever with a sharp tip placed at its free end. The tip is brought so close to the surface that the atoms of the tip and the surface are influenced by interatomic forces. As the tip is scanned across the surface, it moves up and down following the contours of the surface. By measuring the displacement of the tip, one can theoretically map out the surface topography with atomic resolution. The displacement of the tip is measured via a laser beam that is reflected from the cantilever, see the figure below. The cantilever must be soft enough to deflect a measurable amount without damaging the surface of the sample.

The easyScan 2 AFM is a microscope that can be used in both static and dynamic operating mode.

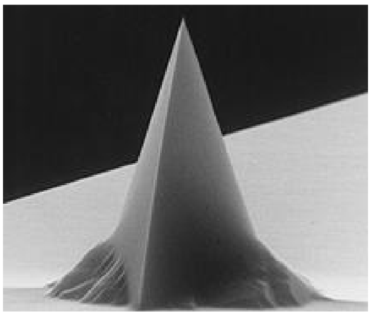
In the static force operating mode, a repulsive force that increases with decreasing tip-sample distance is acting on the tip. This causes the cantilever to bend and the motion of the cantilever is measured using a laser beam deflection system.

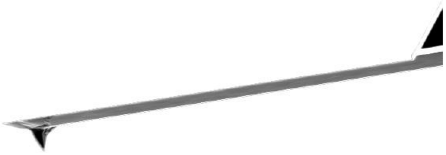
In dynamic operating modes, the cantilever is excited using a piezoelectric element. This element is oscillated with fixed amplitude at an operating frequency close to the resonance frequency of the cantilever. The repulsive force acting on the tip will increase the resonance frequency of the cantilever. This will cause the vibration amplitude of the cantilever to decrease. The vibration of the cantilever is also detected using the laser beam deflection system. The measured laser beam deflection or cantilever vibration amplitude can now be used as an input for a feedback loop that keeps the tip-sample interaction constant by changing the tip height. The output of this feedback loop thus corresponds to the local sample height.



The basic parts of the easyScan 2 AFM system:
computer, scanner with cantilever and deflection measurement system.

The AFM sensor is a micro-fabricated cantilever with an integrated tip mounted on a cantilever holder chip. The cantilever is made from monolithic silicon which is highly doped to dissipate static charge. The tip is shaped like a polygon based pyramid with a height of 10-15 µm. The cantilever is coated with an approximately 30 nm thick aluminum film on the detector side of the cantilever which enhances the reflectivity of the laser beam. Furthermore, it prevents light from interfering within the cantilever.



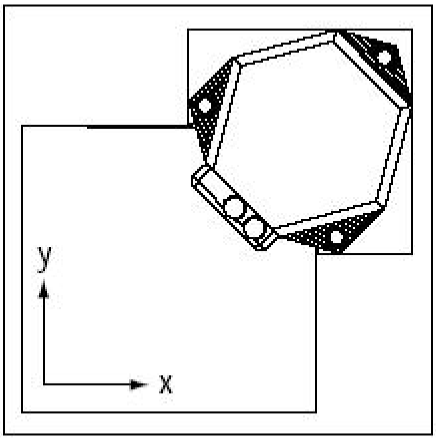


Right: cantilever, 228 µm long micro-fabricated silicon cantilever with integrated tip.

Left: AFM tip used in our setup: thickness: 2 µm, length: 450 µm, width: 50 µm.

The interaction between tip and surface is very complicated. The tip atoms closest to the sample surface experience only short range forces while atoms further away experience long range forces. The short range forces include quantum mechanical exclusion forces (repulsive), bond formation and adhesion (attractive), and friction (tends to twist the cantilever). Short range forces can also lead to elastic and plastic deformations of both tip and sample surface. Long range forces can extend tens of nanometers and include van-der-Waals forces, capillary forces, magnetic, and electrostatic forces. Capillary forces arise because the sample surface is covered with a 5 and 50 nm thick water layer if the measurement is carried out in normal ambient conditions (outside a vacuum chamber). Usually, the long range interaction is dominated by attractive forces while short range forces are dominated by repulsive interactions.

An image of the surface is made by recording the sample height as the tip is scanned over the sample surface in the x and y direction. The direction of the x- and y-axes of the scanner is shown in the figure Scanner coordinate system. The scanner axes may not be the same as the measurement axes, when the measurement is rotated, or when the X or Y measurement plane is changed. Therefore, the image x- and y-axes are denoted by an asterisk to avoid confusion (i.e. X*, Y*). The sample structure image is now obtained by recording the output of the height control loop as a function of the tip position.



Scanner coordinate system.

Lab session

The lab session is 4 hours and you have 2 hours on each microscope. One group will start with the AFM and one group with the STM and then you will switch after 2 hours.

AFM lab

Microscope operation:

1. Switch on hardware and start software
2. Cantilever and sample mounting
3. Manual approach
4. Automatic approach
5. The system is ready for acquiring images

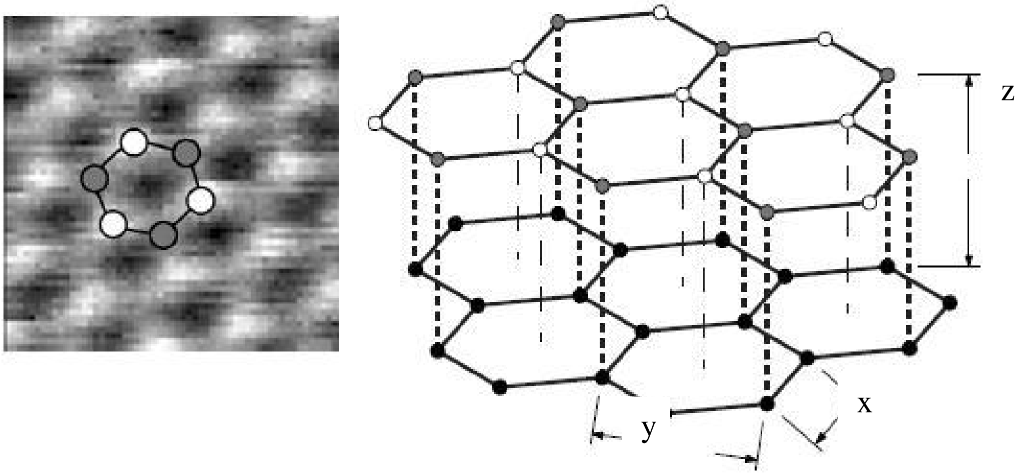
The samples investigated using the AFM equipment is a surface of silicon with some structures on it. We will measure the sample topography in the static mode and acquire images of the surface. Your task is to find out the dimensions of these structures. Also find out whether or not the edges of the structures are due to the slope of the tip or not.

STM lab

Microscope operation:

1. Switch on hardware and start software
2. tip and sample mounting
3. Manual approach
4. Automatic approach through the software
5. Measuring and acquiring images of the surface

The sample investigated using the STM equipment is a graphite surface. Your task is here to get atomic resolution and find out the lattice constants of graphite. Find out the distances x (nearest neighbor distance), y (second nearest in-plane neighbor distance) and z (height difference between adjacent planes) shown in the graphite crystal lattice above.



Graphite surface; *left*: measured image *right*: lattice model.

Preparation before the lab

In order to be admitted to the lab, please read this “lab-pek” carefully in advance and be prepared to answer the questions below. Read relevant parts of chapter 41 in Serway.

Questions

1. Describe the basic principles of STM and AFM.

2. One important parameter to choose is the scanning speed of the AFM tip relative to the sample. In which cases do you expect that a low scanning speed is required in order to accurately measure the surface relief?

3. The AFM tip should be long and sharp. Why should it not be long and thin (even if you exclude the possibility that it breaks)?

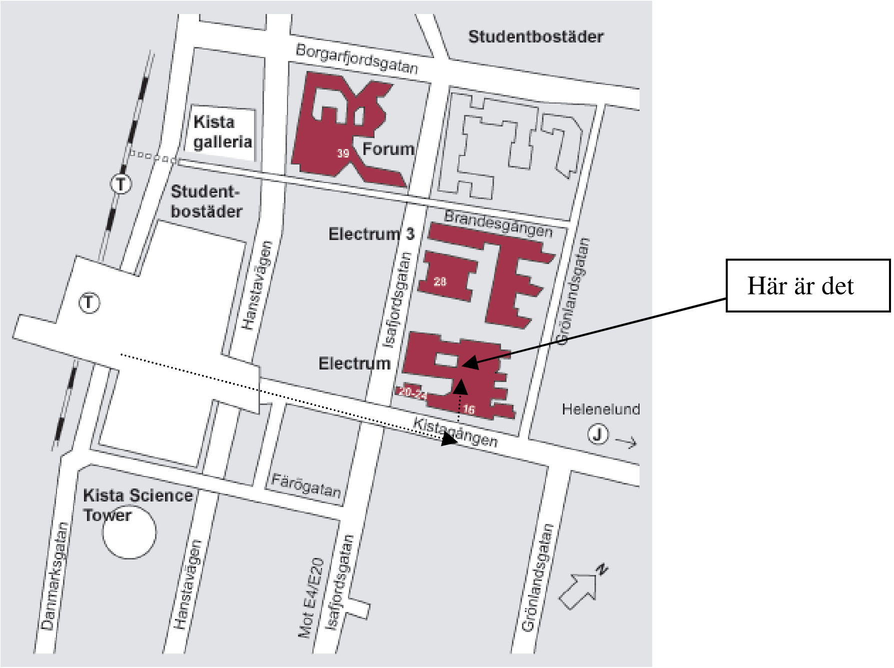
4. What could be advantages and drawbacks if you compare STM and AFM to an optical microscope?

5. What is limiting the resolution in these kinds of microscopes?

6. Which are the requirements on the sample in STM and in AFM respectively?

To get to us

The laboration takes place in Electrum close to Kista C (see map below). In Electrum, we will meet at the elevator B level 2..



Här är det

By subway
Number 11 Kungsträdgården - Akalla (takes about ca 20 minutes from the city center). Station Kista

Commuter trains
Södertälje - Märsta. Station Helenelund. Time about 15 minutes from the city center and 15 min walking to Kista.

By car
From Stockholm. E4 north to Uppsala. Take the Kistaexit (road 279). Take the first exit to Kista Centrum.

Parking
Parking is difficult in Kista, you can try Isafjordsgatan or the parking garage in i Kista Centrum.

Busses
155 Brommaplan - Kista - Akalla
178 Mörby station - Bergshamra -Helenelund - Kista
179 Vällingby - Spånga - Tensta - Kista
514 Vällingby - Spånga - Kista - Sollentuna
517 Spånga - Hjulsta - Kista
518 Vällingby - Backlura - Barkarby - Kista
537 Kista - Upplands Väsby
549 Brunna - Kista
554 Upplands Bro- Kista
627 Kista - Sollentuna - Täby - Arninge