4.3: Scanning Tunneling Spectroscopy

In addition to scanning across the sample, information on the electronic structure at a given location in the sample can be obtained by sweeping voltage and measuring current at a specific location. This type of measurement is called scanning tunneling spectroscopy (STS) and typically results in a plot of the local density of states as a function of energy within the sample. The advantage of STM over other measurements of the density of states lies in its ability to make extremely local measurements: for example, the density of states at an impurity site can be compared to the density of states far from impurities.

4.4: Atomic Force Microscopy (AFM)

Introduction

Atomic force microscopy (AFM) or scanning force microscopy (SFM) is a very high resolution type of scanning probe microscopy (SPM), with demonstrated resolution of the order of fractions of a nanometer (atomic resolution), more than 1000 times better than the optical diffraction limit.

Using an atomic force microscope (AFM), it is possible to measure a roughness of a sample surface at a high resolution, to distinguish a sample based on its mechanical properties (for example, hardness and roughness) and, in addition, to perform a microfabrication of a sample (for example, an atomic manipulation).

In a field of semiconductor physics, for example, (a) an identification of atoms at a surface, (b) evaluation of an interaction between a specific atom and its neighboring atoms and (c) a change in physical properties arisen from a change in an atomic arrangement thorough the atomic manipulation have been studied.

In a field of a cellular biology, for example, (a) an attempt to distinguish cancer cells and normal cells based on a hardness of cells and (b) an attempt to evaluate of an interaction between a specific cell and its neighboring cells in a competitive culture system have been made.

In some variations, electric potentials can also be scanned using conducting cantilevers. In more advanced versions, currents can be passed through the tip to probe the electrical conductivity or transport of the underlying surface.

Construction

The AFM consists of a flexible cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface.The dimensions of the cantilever are in the scale of micrometers. The radius of the tip is usually on the scale of a few nanometers to a few tens of nanometers. The cantilever holder, also called holder chip, often 1.6 mm by 3.4 mm in size allows the operator to hold the AFM cantilever/probe assembly with tweezers and fit it into the corresponding holder clips on the scanning head of the atomic force microscope. This device is most commonly called an "AFM probe", but other names include "AFM tip" and "cantilever" (employing the name of a single part as the name of the whole device).

An AFM probe is a particular type of SPM (scanning probe microscopy) probe. Most AFM probes used are made from silicon (Si), but borosilicate glass and silicon nitride are also in use. AFM probes are considered consumables as they are often replaced when the tip apex becomes dull or contaminated or when the cantilever is broken. They can cost from a couple of tens of dollars up to hundreds of dollars per cantilever for the most specialized cantilever/probe combinations.

In most cases a feedback mechanism is employed to adjust the tip to sample distance to maintain a constant force between the tip and the sample. Traditionally the tip or sample is mounted on a 'tripod' of three piezo crystals, with each responsible for scanning in the *x*, *y* and *z* directions. Piezoelectric elements that facilitate tiny but accurate and precise movements on (electronic) command enable the very precise scanning.

Working

When the tip is brought into proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever, which is what the AFM is designed to measure. A spatial map of the force between tip and sample atoms as a function of tip position on sample can be made by measuring the deflection by scanning the tip at sample surface, giving us surface topography of sample.

The magnitude of force due to tip sample interaction can be measured from Hooke's law F=kx, where k is spring constant of cantilever, which depends on nature (stiffness) of the cantilever and x is the cantilever deflection. Typically, the deflection is measured using a laser spot reflected from the top surface of the cantilever onto a position sensitive detector. This detector consist of an array of position sensitive photodiodes which can measure very small deflection of the order of nanometer very precisly. Then force can be calculated from Hook’s law.

Several types of forces or interactions can be detected. Depending on the situation, forces that are measured in AFM include, mechanical contact force, interatomic Van der Waals or Coulomb repulsive forces, chemical bonding, , magnetic forces etc., giving us different information about different properties of a sample e.g, mechanical strength, surface topography, chemical composition and magnetic properties respectively.

Depending on the interaction under investigation, the surface of the tip of the AFM probe needs to be modified with a coating. Among the coatings used are, diamond for increased wear resistance and magnetic coatings for detecting the magnetic properties of the investigated surface etc. The surface of the cantilevers can also be modified to increase the reflectance of the cantilever and to improve the deflection signal.

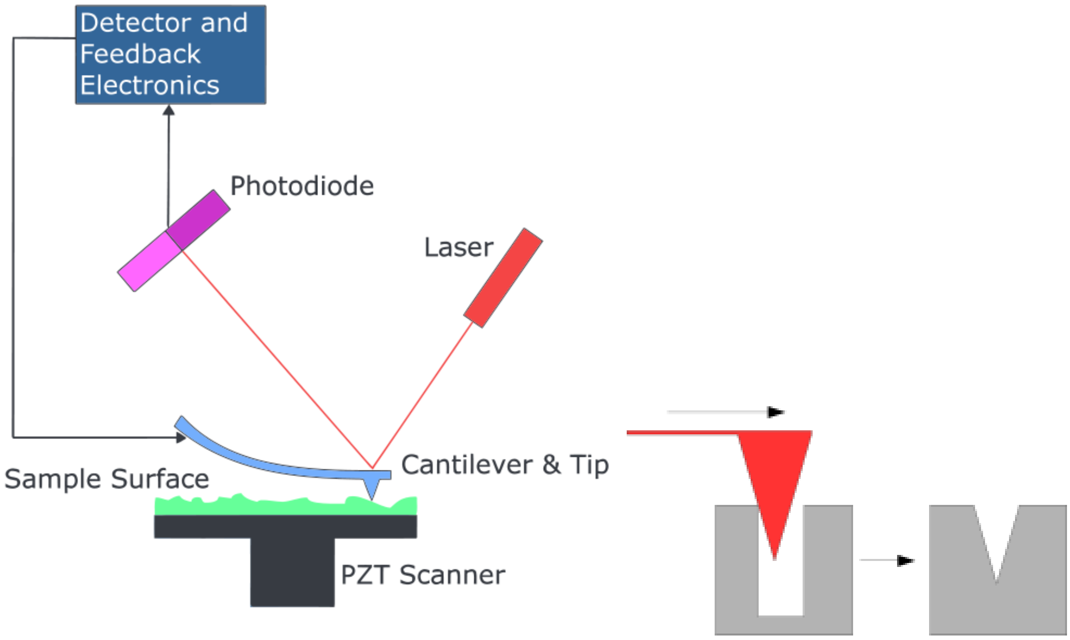

Fig. 4.4 (a): Schematic diagram showing working of Atomic force microscopy

Beam deflection measurement

The most common method for cantilever deflection measurements is the beam deflection method. In this method, laser light from a solid state diode is reflected off the back of the cantilever and collected by a position sensitive detector (PSD) consisting of two closely spaced photodiodes whose output signal is collected by a differential amplifier. Angular displacement of the cantilever results in one photodiode collecting more light than the other photodiode, producing an output signal (the difference between the photodiode signals normalized by their sum), which is proportional to the deflection of the cantilever. It detects cantilever deflections <10 nm (thermal noise limited). A long beam path (several centimeters) amplifies changes in beam angle.

Modes of operation of AFM for imaging

The AFM can be operated in a number of modes, depending on the application. AFM operation is usually performed by one of two important modes, according to the nature of the tip motion: **contact mode**, also called static mode and other is **non contact mode** or also called dynamic or AC mode, or oscillating mode (where the cantilever is vibrated or oscillated at resonance frequency)

Contact or Static Mode

In contact mode, the tip is "dragged" across the surface of the sample and the surface is measured either using the deflection of the cantilever directly or, more commonly, using the feedback signal required to keep the cantilever at a constant position. Because the measurement of a static signal is prone to noise and drift, low stiffness cantilevers (i.e. cantilevers with a low spring constant, k) are used to boost the deflection signal. Thus, contact mode AFM is always done at a distance where the overall force is repulsive (coulomb repulsive force between electron clouds of tip and sample atoms, when there are sufficiently close to each other), that is the case where tip almost touches or in contact with the sample solid surface below any water adsorbed layers.

Non-contact or dynamic mode

In noncontact atomic force microscopy mode, the tip of the cantilever does not contact the sample surface. The cantilever is instead oscillated at either its resonant frequency (frequency modulation) or just above (amplitude modulation) where the amplitude of oscillation is typically a few nanometers (<10 nm) down to a few picometers. The van der Waals forces, which are strongest from 1 nm to 10 nm above the surface, or any other long range force that extends above the surface acts to decrease the resonance frequency of the cantilever. This decrease in resonant frequency combined with the feedback loop system maintains a constant oscillation amplitude or frequency by adjusting the average tip to sample distance. Measuring the tip to sample distance at each (x,y) data point allows the scanning software to construct a topographic image of the sample surface.

In frequency modulation, changes in the oscillation frequency provide information about tip sample interactions. Frequency can be measured with very high sensitivity and thus the frequency modulation mode allows for the use of very stiff cantilevers. Stiff cantilevers provide stability very close to the surface and, as a result, this technique was the first AFM technique to provide true atomic resolution in ultrahigh vacuum conditions.

In amplitude modulation, changes in the oscillation amplitude or phase provide the feedback signal for imaging. In amplitude modulation, changes in the phase of oscillation can be used to discriminate between different types of materials on the surface.

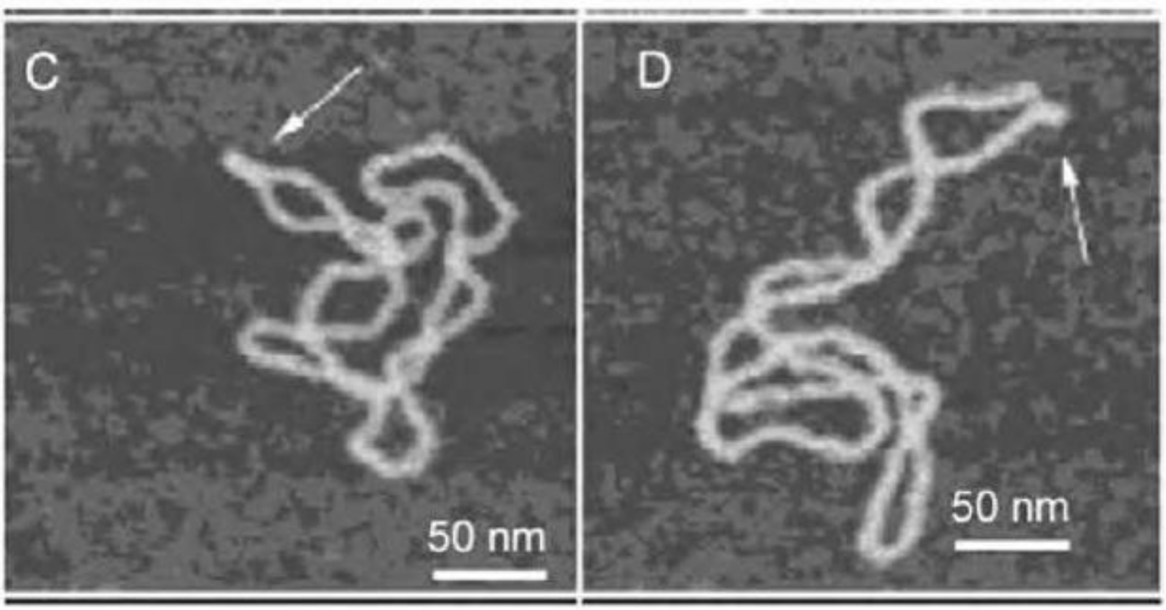

Fig. Atomic force microscope image of DNA structure

Advantages of non-contact mode

Noncontact mode AFM does not suffer from tip or sample degradation effects that are sometimes observed after taking numerous scans with contact AFM. This makes noncontact AFM preferable to contact AFM for measuring soft samples, e.g. biological samples and organic thin film. In the case of rigid samples, contact and noncontact images may look the same.

However, if a few monolayers of adsorbed fluid are lying on the surface of a rigid sample, the images may look quite different. An AFM operating in contact mode will penetrate the liquid layer to image the underlying surface, whereas in noncontact mode an AFM will oscillate above the adsorbed fluid layer to image both the liquid and surface.

Advantages of AFM over SEM

AFM has several advantages over the scanning electron microscope (SEM). Unlike the electron microscope, which provides a two dimensional projection or a two dimensional image of a sample, the AFM provides a three dimensional surface profile.

In addition, samples viewed by AFM do not require any special treatments (such as metal/carbon coatings) that would irreversibly change or damage the sample, and does not typically suffer from charging artifacts in the final image. While an electron microscope needs an expensive vacuum environment for proper operation, most AFM modes can work perfectly well in ambient air or even a liquid environment. This makes it possible to study biological macromolecules and even living organisms.

In principle, AFM can provide higher resolution than SEM. It has been shown to give true atomic resolution in ultrahigh vacuum (UHV) and, more recently, in liquid environments. High resolution AFM is comparable in resolution to scanning tunneling microscopy and transmission electron microscopy.

Disadvantages of AFM over SEM

- A disadvantage of AFM compared with the scanning electron microscope (SEM) is the single scan image size. In one pass, the SEM can image an area on the order of square millimeters with a depth of field on the order of millimeters, whereas the AFM can only image a maximum height on the order of 10 to 20 micrometers and a maximum scanning area of about 150×150 micrometers. One method of improving the scanned area size for AFM is by using parallel probes.

- The scanning speed of an AFM is also a limitation. Traditionally, an AFM cannot scan images as fast as a SEM, requiring several minutes for a typical scan, while a SEM is capable of scanning at near real time, although at relatively low quality.

- The relatively slow rate of scanning during AFM imaging often leads to thermal drift in the image making the AFM less suited for measuring accurate distances between topographical features on the image. However, several fast acting designs were suggested to increase microscope scanning productivity including what is being termed video AFM (reasonable quality images are being obtained with video AFM at video rate: faster than the average SEM). To eliminate image distortions induced by thermal drift, several methods have been introduced.

- As with any other imaging technique, there is the possibility of image artifacts, which could be induced by an unsuitable tip, a poor operating environment, or even by the sample itself, as depicted on the right. These image artifacts are unavoidable; however, their occurrence and effect on results can be reduced through various methods. Artifacts resulting from a toocoarse tip can be caused for example by inappropriate handling or de facto collisions with the sample by either scanning too fast or having an unreasonably rough surface, causing actual wearing of the tip.

- Due to the nature of AFM probes, they cannot normally measure steep walls or overhangs. Specially made cantilevers and AFMs can be used to modulate the probe sideways as well as up and down (as with dynamic contact and noncontact modes) to measure sidewalls, at the cost of more expensive cantilevers, lower lateral resolution and additional artifacts.