4.9 Scanning Probe Microscopy

Scanning probe microscopy (SPM) is a new branch of microscopy that forms images of surfaces using a physical probe that scans the specimen. An image of the surface is obtained by mechanically moving the probe in a raster scan of the specimen, line by line, and recording the probe-surface interaction as a function of position. SPM was founded with the invention of the scanning tunneling microscope in 1981.

Some important types of scanning probe microscopy

- AFM, atomic force microscopy
- EFM, electrostatic force microscope
- FMM, force modulation microscopy
- MFM, magnetic force microscopy
- STM, scanning tunneling microscopy
- SVM, scanning voltage microscopy
- SHPM, scanning Hall probe microscopy

4.9.1 Atomic Force Microscope (AFM)

Introduction

The atomic force microscope (AFM) or scanning force microscope (SFM) was invented in1986 by Binnig, Quate and Gerber. Similar to other scanning probe microscopes, the AFM raster scans a sharp probe over the surface of a sample and measures the changes in force between the probe tip and the sample.

Working Concept

The physical parameter probed is a force resulting from different interactions. The origin of these interactions can be ionic repulsion, van der Waals, capillary, electrostatic and magnetic forces, or elastic and plastic deformations. Thus, an AFM image is generated by recording the force changes as the probe (or sample) is scanned in the x and y directions.

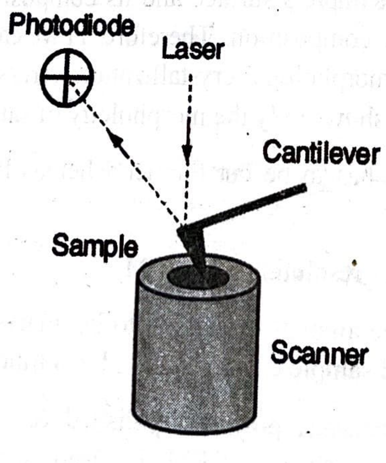

Fig.4.13 Working concept of AFM

The sample is mounted on a piezoelectric scanner, which ensures three-dimensional positioning with high resolution. The force is monitored by attaching the probe to a pliable cantilever, which acts as a spring, and measuring the bending or "deflection" of the cantilever. The larger the cantilever deflection, the higher the force that will be experienced by the probe. Most instruments today use an optical method to measure the cantilever deflection with high resolution; a laser beam is focused on the free end of the cantilever, and the position of the reflected beam is detected by a position-sensitive detector (photodiode). AFM cantilevers and probes are typically made of silicon or silicon nitride by micro fabrication techniques.

Basic set-up of an AFM

In principle the AFM resembles a record player and a stylus profilometer. The ability of an AFM to achieve near atomic scale resolution depends on the three essential components: (1) a cantilever with a sharp tip, (2) a scanner that controls the *x-y-z* position, and (3) the feedback control and loop.

1. *Cantiliever with a sharp tip.* The stiffness of the cantilever needs to be less the effective spring constant holding atoms together, which is on the order of 1 - 10 nN/nm. The tip should have a radius of curvature less than 20-50 nm (smaller is better) a cone angle between 10-20 degrees.

2. *Scanner.* The movement of the tip or sample in the *x*, *y*, and *z*-directions is controlled by a piezo-electric tube scanner, similar to those used in STM. For typical AFM scanners, the maximum ranges for are 80 mm x 80 mm in the *x-y* plane and 5 mm for the *z*-direction.

3. *Feedback control.* The forces that are exerted between the tip and the sample are measured by the amount of bending (or deflection) of the cantilever. By calculating the difference signal in the photodiode quadrants, the amount of deflection can be correlated with a height. Because the cantilever obeys Hooke's Law for small displacements, the interaction force between the tip and the sample can be determined.

A summary of the different modes of operation is found in Table 4.6

Table 4.6 AFM Modes of operation

Mode of Operation	Force of Interaction
Contact mode	strong (repulsive) - constant force or constant distance
Non-contact mode	weak (attractive) - vibrating probe
Tapping mode	strong (repulsive) - vibrating probe
Lateral force mode	frictional forces exert a torque on the scanning cantilever

Applications

The AFM is useful for obtaining three-dimensional topographic information of insulating and conducting structures with lateral resolution down to 1.5 nm and vertical resolution down to 0.05 nm. These samples include clusters of atoms and molecules, individual macromolecules, and biologic al species (cells, DNA, proteins). Unlike the preparation of samples for STM imaging, there is minimal sample preparation involved for AFM imaging. Similar to STM operation, the AFM can operate in gas, ambient, and fluid environments and can measure physical properties including elasticity, adhesion, hardness, friction and chemical functionality. A concise applications listing is given below.

- Metals: tooling studies, roughness measurements, corrosion studies...
- Solid powder catalysts: aggregate structural determination,

- Polymers: determination of morphology and surface properties, kinetic studies, aging phenomena, surface treatment modifications, adhesion force measurement and indentation,
- Biological samples, biomaterials: macromolecules association and conformation studies, adsorption kinetic of molecules on polymer surfaces,
- Nano- and microparticle structures, Langmuir-Blodgett. Film studies...

Advantages

The 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 true three-dimensional surface profile.
- Additionally, samples viewed by AFM do not require any special treatments (such as metal/carbon coatings) that would irreversibly change or damage the sample.
- 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 ultra-high vacuum (UHV).

Disadvantages

- A disadvantage of AFM compared with the scanning electron microscope (SEM) is the image size. The SEM can image an area on the order of millimetres by millimetres with a depth of field on the order of millimetres. The AFM can only image a maximum height on the order of micrometres and a maximum scanning area of around 150 by 150 micrometres.
- Another inconvenience is that at high resolution, the quality of an image is limited by the radius of curvature of the probe tip, and an incorrect choice of tip for the required resolution can lead to image artifacts.
- Traditionally the AFM could not scan images as fast as an SEM, requiring several minutes for a typical scan, while an SEM is capable of scanning at near real-time (although at relatively low quality) after the chamber is evacuated.
- AFM images can be affected by hysteresis of the piezoelectric material.

4.9.2 Scanning Near-Field Optical Microscopy

Scanning Near Field Optical Microscopy (SNOM/NSOM) is a form of scanning probe microscopy that allows optical imaging with spatial resolution beyond the diffraction limit. This is done by placing the detector very close to the specimen surface in the region called "optical near-field".

As a result, near field microscopy remains primarily a surface inspection technique

The interaction of light with an object, such as a microscope specimen, results in the generation of both near-field and far-field light components. The far-field light propagates through