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Characterization of nanomaterials SEM ,AFM

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Introduction to Nanometrology

- Nanometrology is a subfield of metrology, concerned with the science of measurement at thenanoscale level. Nanometrology has a crucial role in order to produce nanomaterials and devices with a high degree of accuracy and reliability innanomanufacturing.

- A challenge in this field is to develop or create new measurement techniques and standards to meet the needs of next-generation advanced manufacturing, which will rely on nanometer scale materials and technologies.

- The needs for measurement and characterization of new sample structures and characteristics far exceed the capabilities of current measurement science.

- Control of the critical dimensions are the most important factors in anotechnology. Nanometrology today, is to a large extent based on the development insemiconductortechnology. Nanometrology is the science ofmeasurementat the nanoscale level.

- In Nanotechnology accurate control of dimensions of objects is important. Typical dimensions of nanosystems vary from 10nm to a few hundred nm and while fabricating such systems measurement up to 0.1nm is required.

- In the last 70 years various techniques for measuring at nanoscale have been developed most of them based on some physical phenomena observed on particle interactions or forces at nanoscale. Some of the most commonly used techniques are

- Atomic Force Microscopy, X-Ray Diffraction, Scanning Electron Microscopy,

- Transmission Electron Microscopy, High Resolution Transmission Electron Microscopy, and Field Emission Scanning Electron Microscopy.

Analytical Imaging:

- The fundamental of nanotechnology lies in the fact that properties of materials change dramatically when their size is reduced to the nanometer range. But measuring this nano dimension is not a very easy task.

- Although research is going on to synthesise nanostructured and nanophasic materials, characterizing these nano sized materials is also an emerging field posing lot of challenges to scientists and technonologists.

- Thus, nanotechnology has motivated the upsurge in research activities on the discovery and invention of sophisticated nano characterization techniques to allow a better control of morphology, size and dimensions of materials in nano range.

- Nanomaterials to be thoroughly characterized as much as possible using a combination of experimental techniques due to the intrinsic complex nature of nanomaterials. For example, two of the most basic characteristics are size and shape of nanostructures.

- Another important property is the surface of nanomaterials due to their extremely large surface-to-volume ratio. Furthermore, it is often important to know the crystal structures of the nanomaterials. To study these different properties requires many other experimental methods, besides optical spectroscopy.

- Nanotechnology was mostly a dream until the invention of the Scanning Tunneling Microscope and the Atomic Force Microscope. In addition, all other existing facilities are used to characterize nanomaterials.

SCANNING ELECTRON MICROSCOPE (SEM)

Introduction :

Initially, the plan of SEM was offered by H. Stintzing in 1927 (a German patent application). His suggested procedure was unable to produce magnified image because the collimated beam with which sample was irradiated was light, X-rays and corpuscles. Then a German electrical engineer named M. Knoll contributed a paradigm of SEM in 1935 where specimen was scanned with electron beam to obtain image. In 1938, Von Ardenne developed SEM with slight modification by introducing DE magnifying lenses called scanning transmission electron microscope to scan thin samples. For scanning bulk samples Zworykin (in 1942) improved SEM with few other alterations. Eventually SEM was commercialized in 1965 with many alterations being done in the R & D of the Oatley Lab.

Definition:

The scanning electron microscope is an electron microscope technique capable of producing high resolution images of the surfaces of a sample.

Principle:

SEM (Figure 1) belongs to the family of electron microscopes which produce images of an object by scanning its surface with highly focused electron beam. The process involves the interaction of electrons with atoms of an object, creating signals containing information of object's composition and topography. Arrangement of constituent atoms is studied by 2D beam scanning upon the sample surface and image is produced from collected secondary electrons. Scan pattern is generated by the electron beam and the image is formed by merging beam's position and the detected signal.

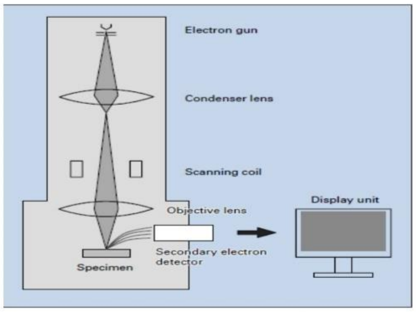
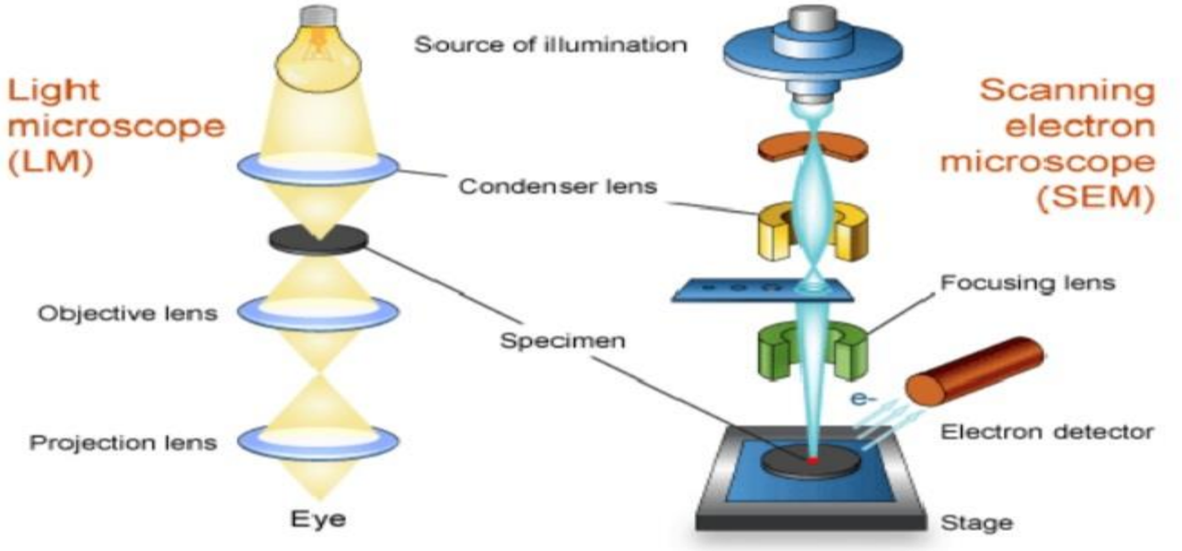


Figure 1 Basic Construction of SEM.



Instrumentation of SEM

The basic components used in electron optical system are:

- ✓ A source of electrons, called electron gun
- ✓ Lenses
- ✓ Scanning Coils
- ✓ Detectors to collect signals
- ✓ Sample Stage
- ✓ Display/Data output devices

Infrastructure Requirement

- Power supply
- Vacuum system
- Cooling system
- Vibration free floor
- Room free of ambient electric and magnetic fields

Description of Components

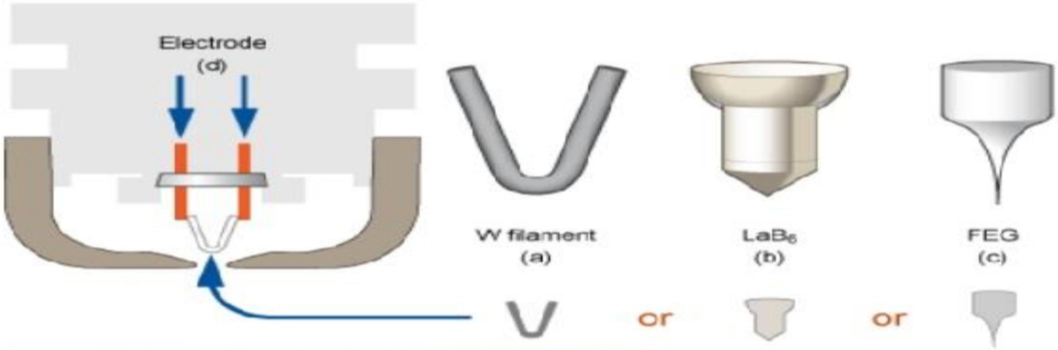
1) Electron Beam: It has two variables i.e. energy and current. The voltage is variable from about 1 - 60keV and the current from 1e-7 to 1e-12 A. These values are specific to the instrument model.

2) Electron Gun: It is used to produce fine electron beam (and is also called as electron probe). Several different types of electron guns used are:

a) TE (Thermionic- Emission) gun

b) FE (Field- Emission) gun

c) SE (Schottky- Emission) gun



Lenses

To produce finest beam of electron with desired crossoverdiameter, two- level lens system, i.e., condenser and objective lens, made of metal cylinders with cylindrical hole, operating in vacuumis used.These lenses are located beneath the electron gun. Magnetic field is generated in the inner part of the lenses to focus or de-focus the beam.

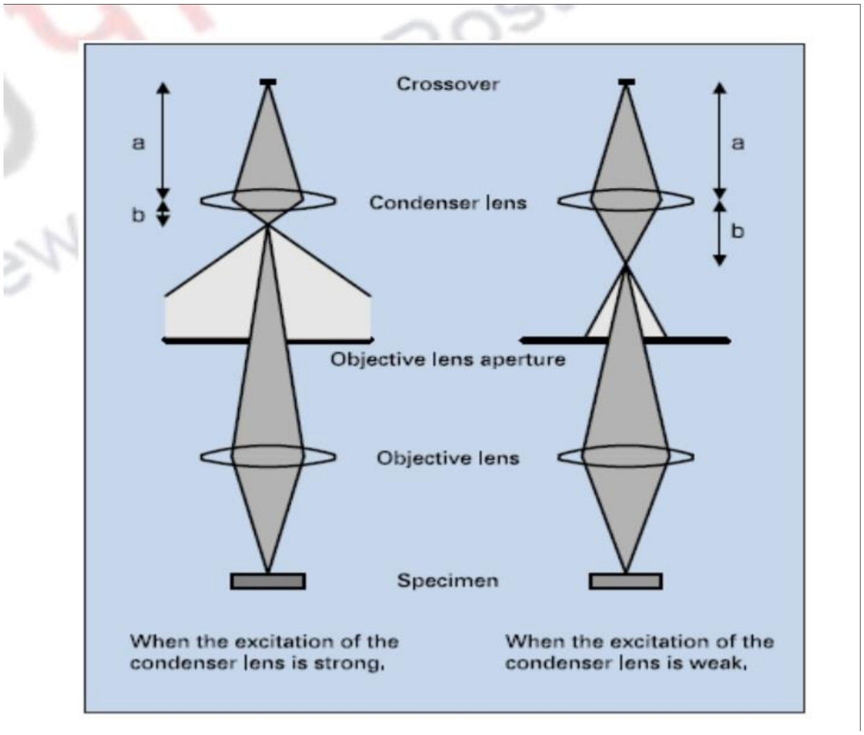


Fig 3: formation of electron probe by lenses.

Role of condenser lens:

Condenser lens affects the probe size. If it is strengthened then probe size is narrowed with a smaller ratio of b/a, whereas if it is weakened then probe size is broadened. C1 and C2 lenses control the beam current by varying size and intensity of beam spot. Aperture is formed by making a small hole in the metalplaced between the two condenser lenses and the objective lensto allow the beam to pass through it and reach the objective. Resolution is dependent upon aperture as it controls the spot size.

Role of objective lens:

It is used for focusing and determines the final diameter of probe.

Scanning Coils

These coils deflect the beam in X or Y directions in order to scan the sample surface in a raster pattern.

Principle of SEM image formation

When an electron beam is incident on the sample then many different types of signals are generated which are eventually used to observe or analyze morphology/ topology of the sample. SEM is also used for elemental and state analysis. These signals includes: Secondary electrons, Backscattered electrons, Auger electrons, Cathodoluminescence and X-rays

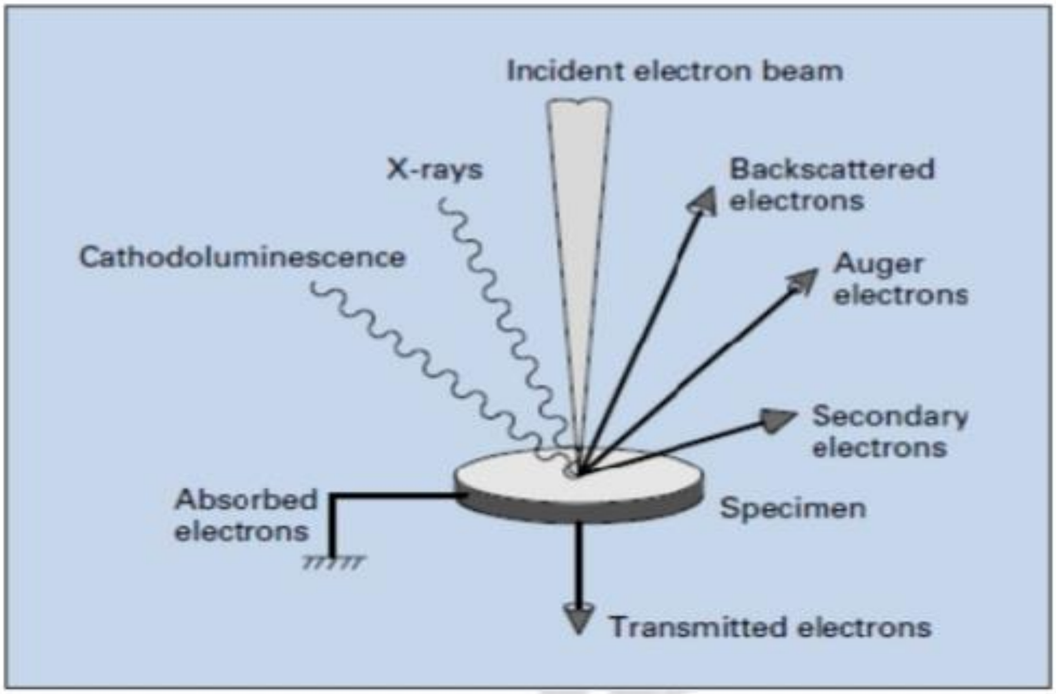
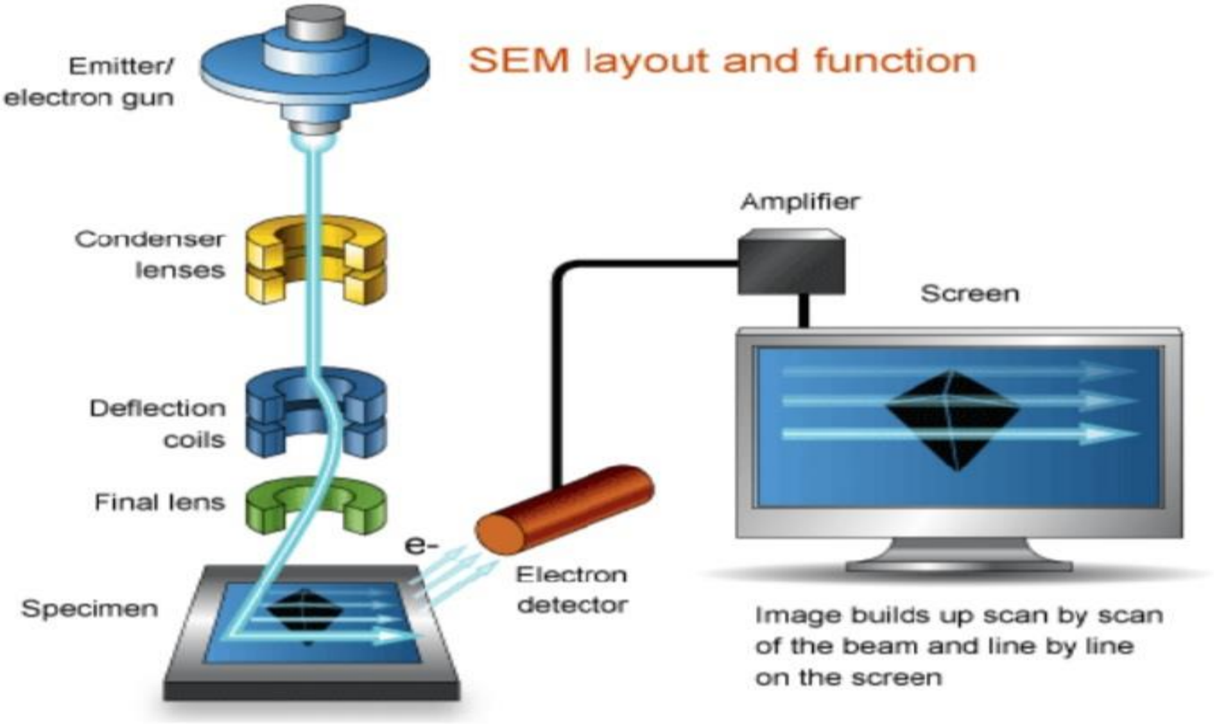


Fig4:Emission of various electrons and electromagnetic waves from the specimen.



Applications :

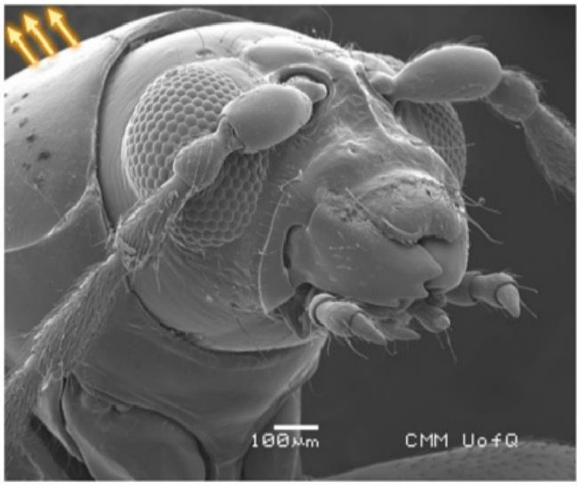
- The SEM shows very detailed three dimensional images at much high magnifications (up to ×300000) as compared to light microscope (up to × 10000). But as the images are created without light waves, they are black and white.
- The surface structure of polymer nanocomposites, fracture surfaces, nanofibres, nanoparticles and nanocoating can be imaged through SEM with great clarity. As very high resolution images of the dimension 1-5nm can be obtained, SEM is the most suitable process to study the surface of nanostrutures.
- Electrospun nanofibres are extensively studied in biomedical, environmental and other technical textile applications for their huge surface area. Electrospun nylon 6 nanofibres decorated with surface bound silver nanoparticles used for antibacterial air purifier can be characterized using SEM .
- In tissue engineering or cell culture applications, the SEM image is the prime characterization technique for scaffold construction, cell development and growth. SEM technique is used to observe the plied CNT yarns in 3D braided structures.
- The SEM technique can also be used to view dispersion of nanoparticles such as carbon nanotubes, nanoclays and nanofillers in the bulk and on the surface of nanocomposite samples

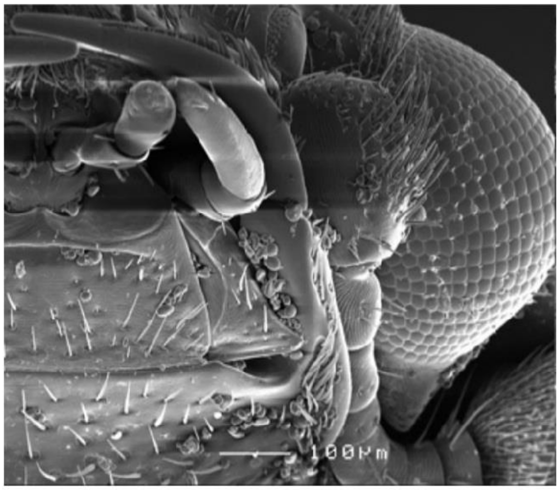
Types of SEM

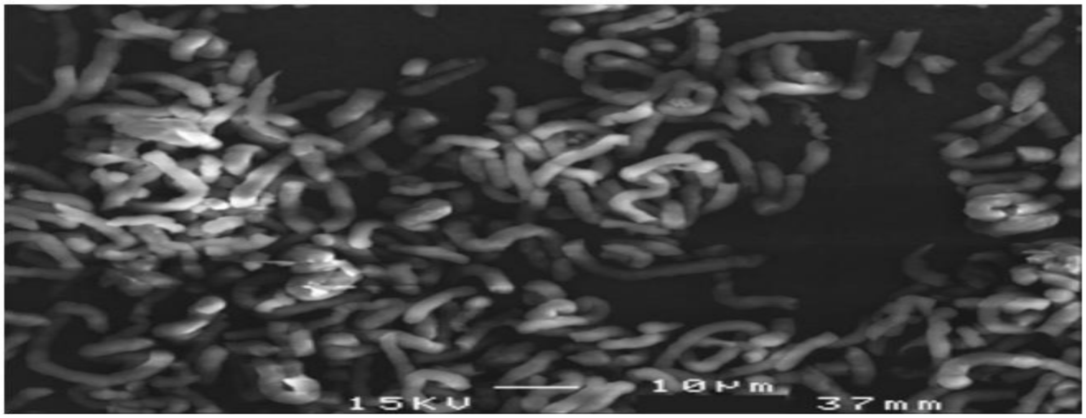
The different types of scanning electron microscopes in detail:

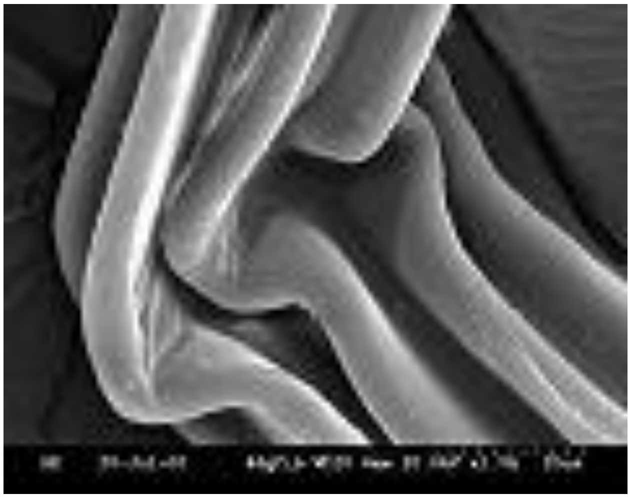
- **Conventional (high vacuum) SEM**
- **Variable Pressure or Low Vacuum SEM**
- **Cryo on an SEM (Cryo-SEM)**
- **Environmental SEM**
- **Focused ion beam (FIB)**
- **Electron-beam (E-beam) lithogr**

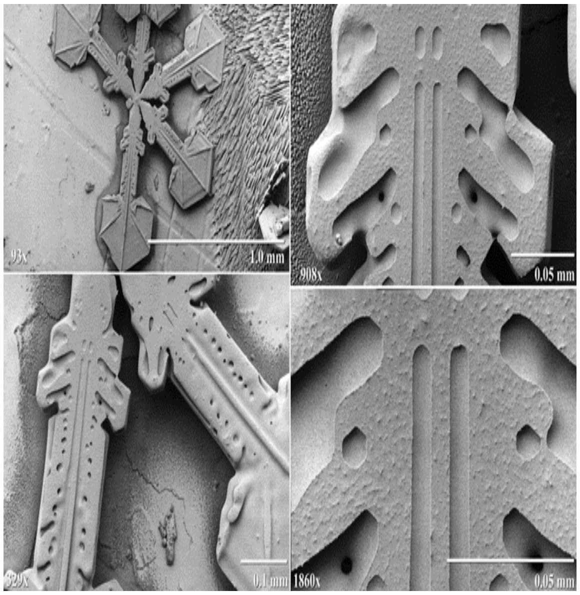
Images of SEM







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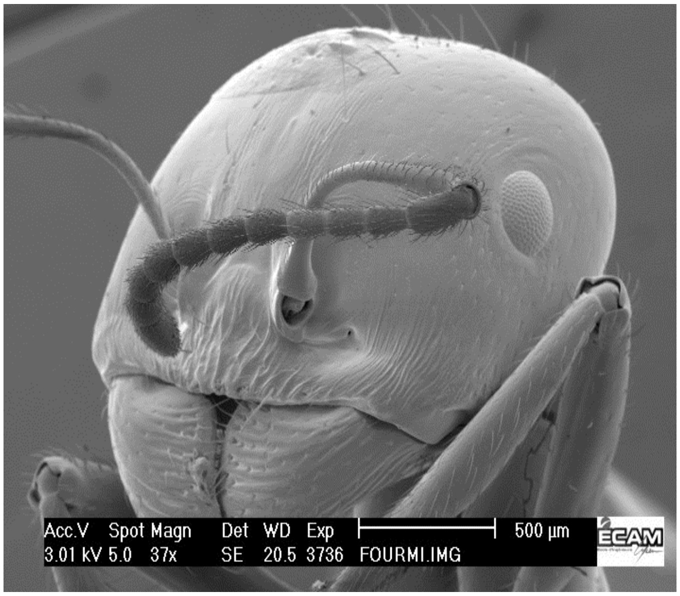
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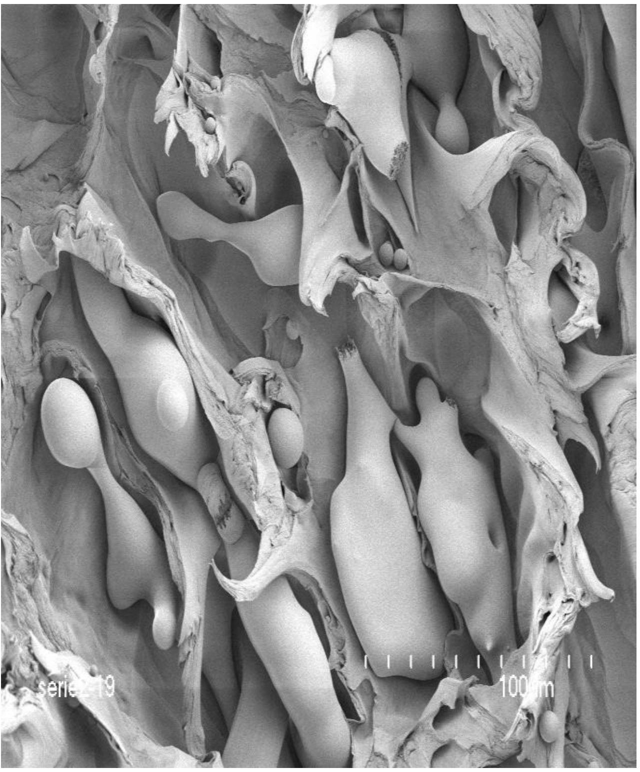
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Atomic Force Microscopy (AFM)

General Principle

The Atomic Force Microscope is a kind of scanning probe microscope in which a topographical image of the sample surface can be achieved based on the interactions between a tip and a sample surface. The atomic force microscope was invented by Gerd Binning et al. in 1986 at IBM Zurich based on the STM (Scanning Tunneling Microscope) already presented in 1981. While the latter depends on the conductive samples, the AFM allows also the use of non-conductive samples. In 1987, the inventors were awarded the Nobel Prize in Physics for the achievements. A typical AFM consists of a cantilever with a small tip (probe) at the free end, a laser, a 4-quadrant photodiode and a scanner. The surface characteristics can be explored with very accurate resolution in a range of 100 µm to less than 1 µm.

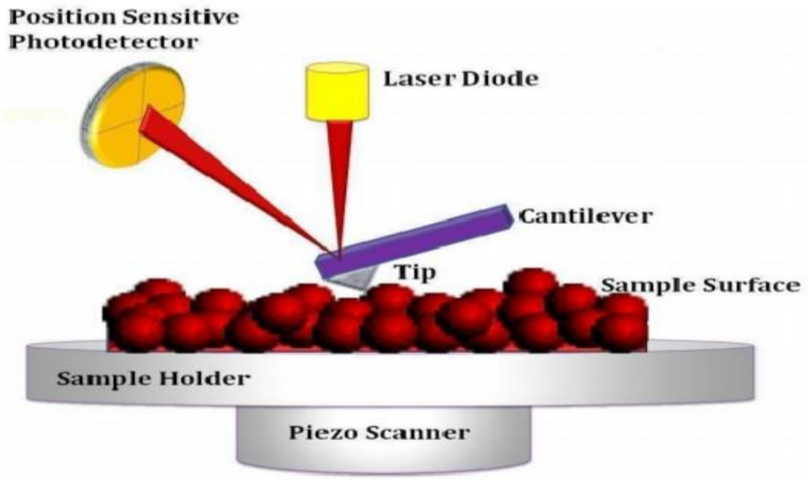
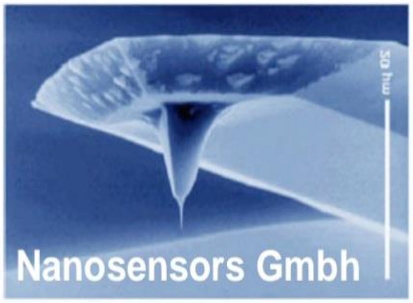
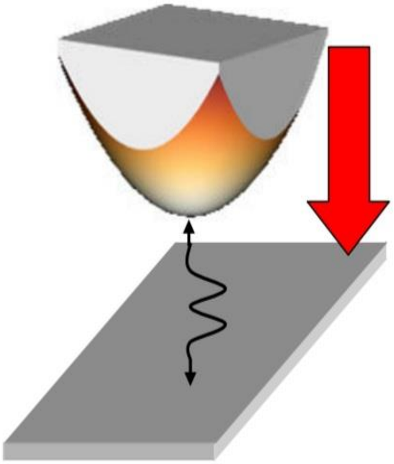


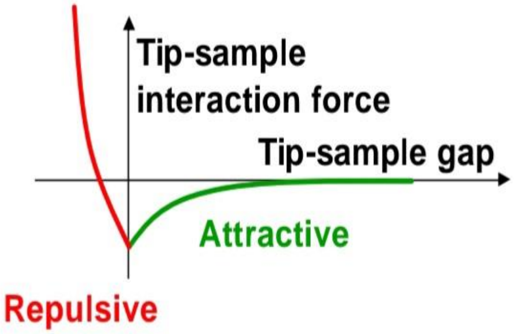
Figure 1. Principle of Atomic Force Microscopy

In AFM, a tip is used for imaging. It is generally made of silicon or silicon nitride (Si3N4). It approaches the sample in a range of interatomic distances (around 10 Å). The tip is commonly 3-15 microns in length. It is attached to the end of the spring cantilever. The cantilever is around 100-500 microns in length.

Tip-sample interaction forces in AFM



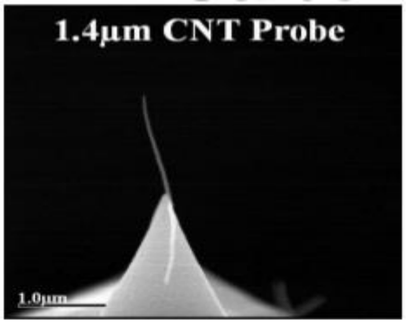


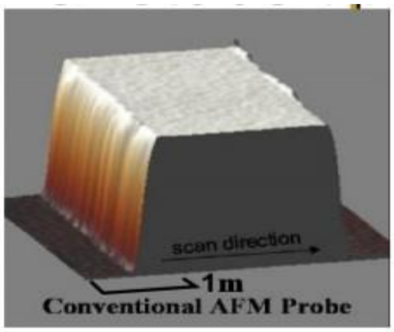


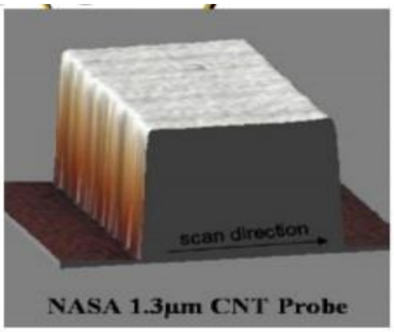
- Long-range electrostatic and magnetic forces (upto 100 nm)
- Capillary forces (few nm)
- Van der Waals forces (few nm) that are fundamentally quantum mechanical (electrodynamic) in nature
- Casimir forces
- Short-range chemical forces (fraction of nm)
- Contact forces
- Electrostastic double-layer forces
- Solvation forces
- Nonconservative forces (Dürig (2003))

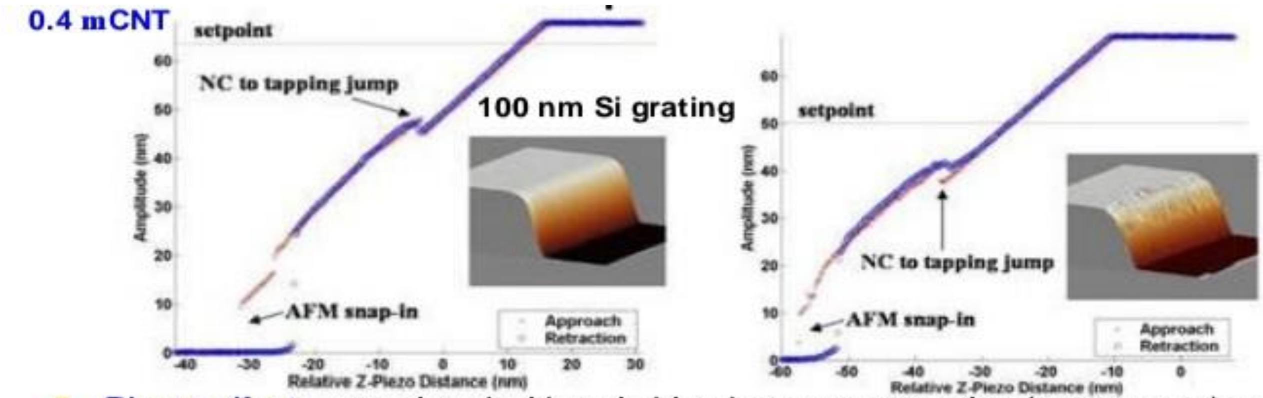
Applications and emerging areas

Carbon nanotube tips (CNT)









- Divot artifacts associated with switching between attractive (noncontact) and repulsive (tapping states)
- Ringing artifacts associated with CNT adhesion and stiction to sidewalls

300 nm Tungsten nanorods