



Material Characterization

Date: 2011/11/01

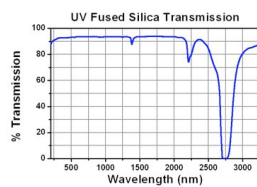
Dr. Yi-Chung Tung



Material Characterization

Characterization

Mechanic characterization
Thermo characterization (melting point)
Optical characterization (diffraction angle)
NMR (and other resonances)
Spectroscopy
Electrical characterization





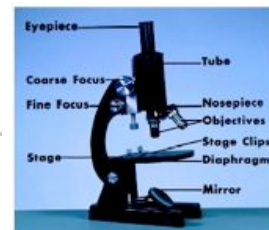
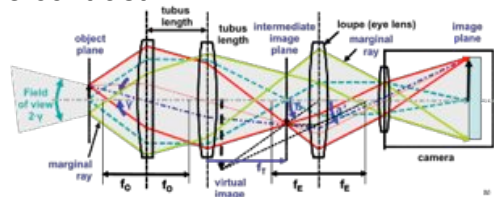
How to Observe Things Small?

- Optical Microscopy:
 - Optical Microscope.
 - Total Internal Reflection Fluorescence Microscope (TIRF).
 - Confocal Microscope.
 - Single Plane Illumination Microscope (SPIM).
- Non-Optical Microscopy:
 - Scanning Probe Microscope (SPM): Scanning Probe Microscope (STM), Atomic Force Microscope (AFM) etc.
 - Scanning Electron Microscope (SEM).
 - Transmission Electron Microscope (TEM).



Optical Microscope

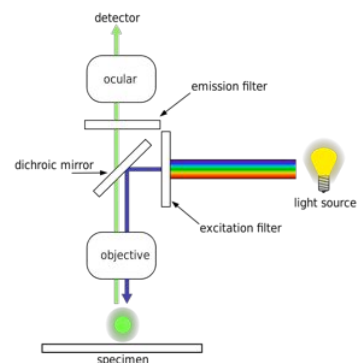
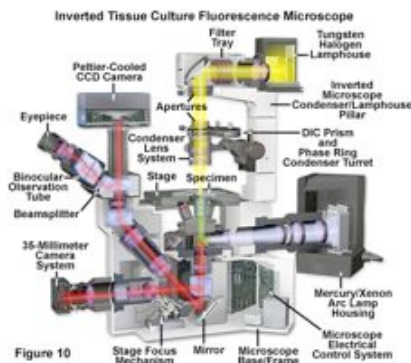
- The **optical microscope**, often referred to as the "**light microscope**", is a type of microscope which uses visible light and a system of lenses to magnify images of small samples. Optical microscopes are the oldest design of microscope and were possibly designed in their present compound form in the 17th century. Basic optical microscopes can be very simple, although there are many complex designs which aim to improve resolution and sample contrast.



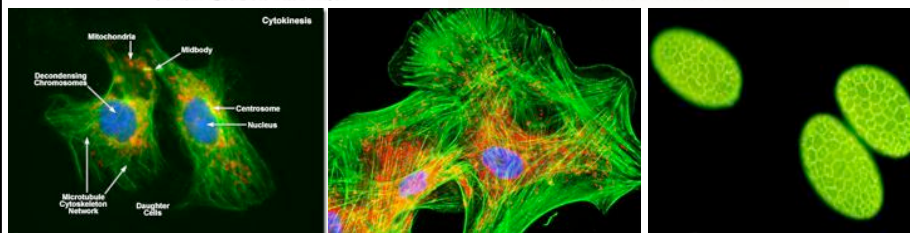
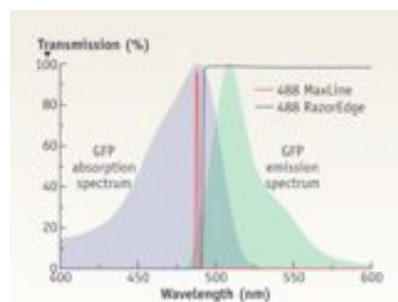
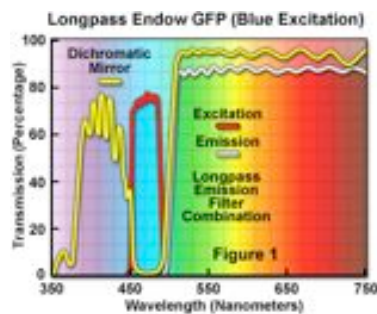


Fluorescence Microscope - 1

- A **fluorescence microscope** is an optical microscope used to study properties of organic or inorganic substances using the phenomena of fluorescence and phosphorescence instead of, or in addition to, reflection and absorption.



Fluorescence Microscope - 2

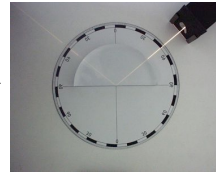
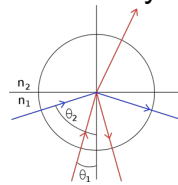




Total Internal Reflection Fluorescence Microscope (TIRF) - 1

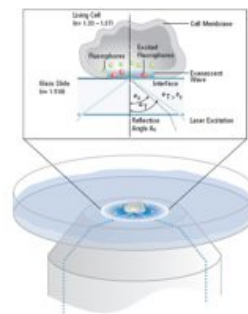
- A total internal reflection microscope (TIRF) is a type of microscope with which a thin region of a specimen, usually less than 200 nm, can be observed.
- A TIRFM uses an evanescent wave to selectively illuminate and excite fluorophores in a restricted region of the specimen immediately adjacent to the glass-water interface.
- The evanescent wave is generated only when the incident light is totally internally reflected at the glass-water interface. The evanescent electromagnetic field decays exponentially from the interface.

$$\sin \theta_i = \frac{n_2}{n_1} \sin \theta_t$$



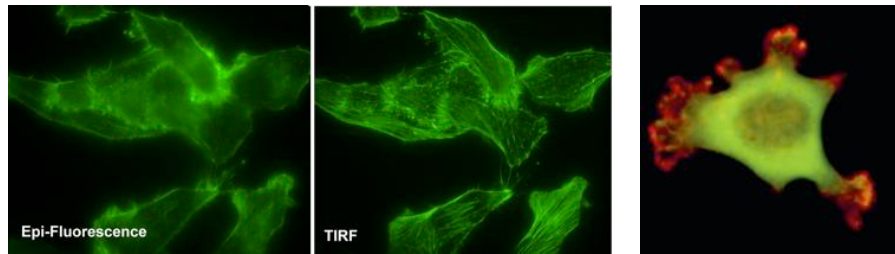
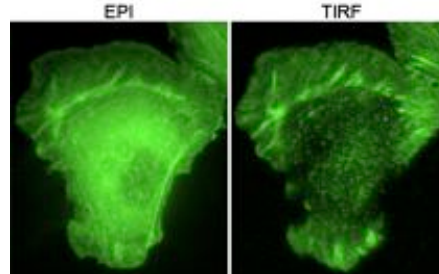
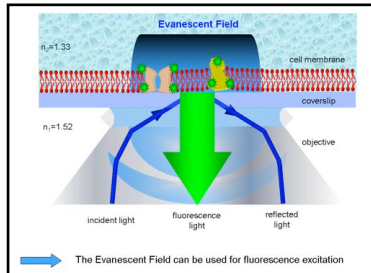
Total Internal Reflection Fluorescence Microscope (TIRF) - 2

- An **evanescent wave** is a near field standing wave with an intensity that exhibits exponential decay with distance from the boundary at which the wave was formed.



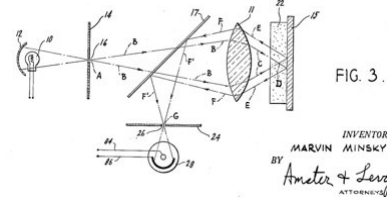


Total Internal Reflection Fluorescence Microscope (TIRF) - 3



Confocal Microscope - 1

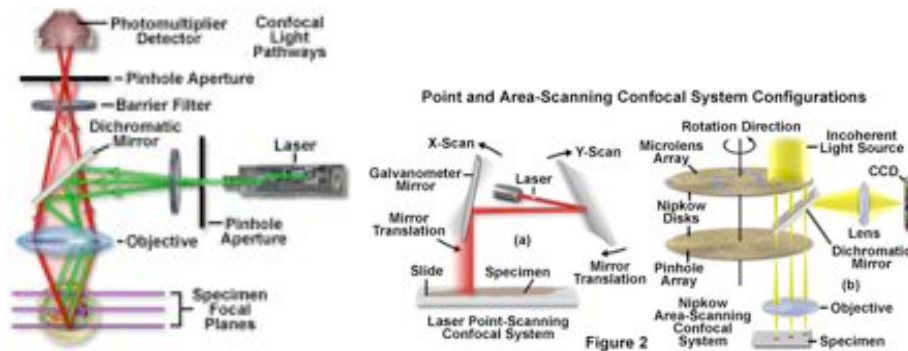
- The principle of confocal imaging was patented in 1957 by Marvon Minsky and aims to overcome some limitations of traditional wide-field fluorescence microscopes.
- It is an optical imaging technique used to increase optical resolution and contrast of a micrograph by using point illumination and a spatial pinhole to eliminate out-of-focus light in specimens that are thicker than the focal plane.
- It enables the reconstruction of three-dimensional structures from the obtained images.





Confocal Microscope - 2

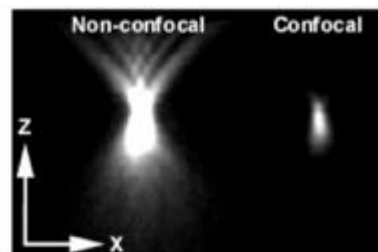
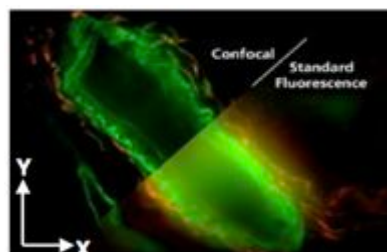
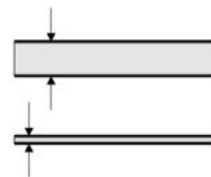
Confocal imaging relies upon the sequential collection of light from spatially filtered individual specimen points, followed by electronic signal processing and ultimately, the visual display as corresponding image points. The point-by-point signal collection process requires a mechanism for scanning the focused illuminating beam through the specimen volume under observation.



Confocal Microscope - 3

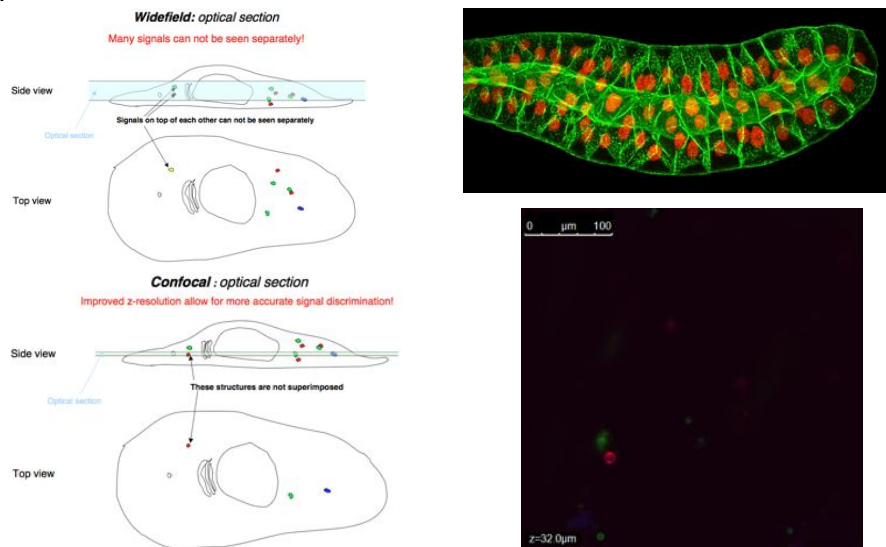
Widefield 2 - 3 μm

Confocal 0.5 μm





Confocal Microscope - 4

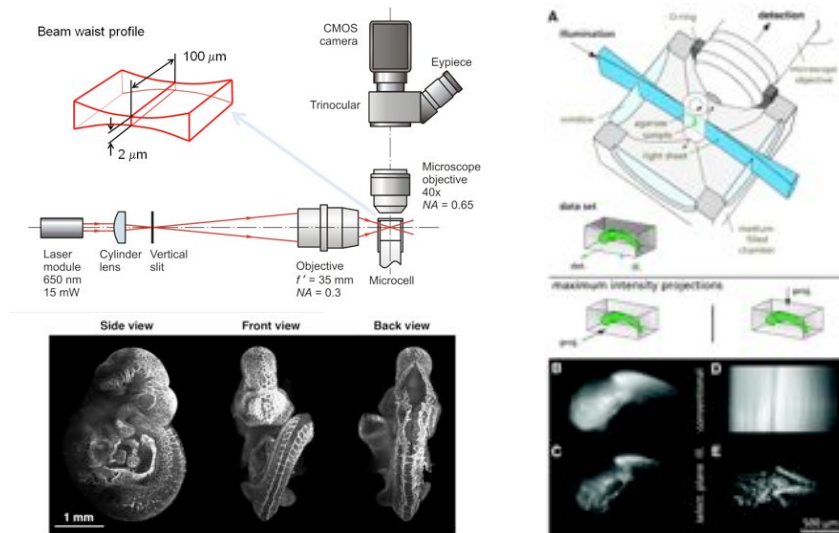


Single Plane Illumination Microscope (SPIM) - 1

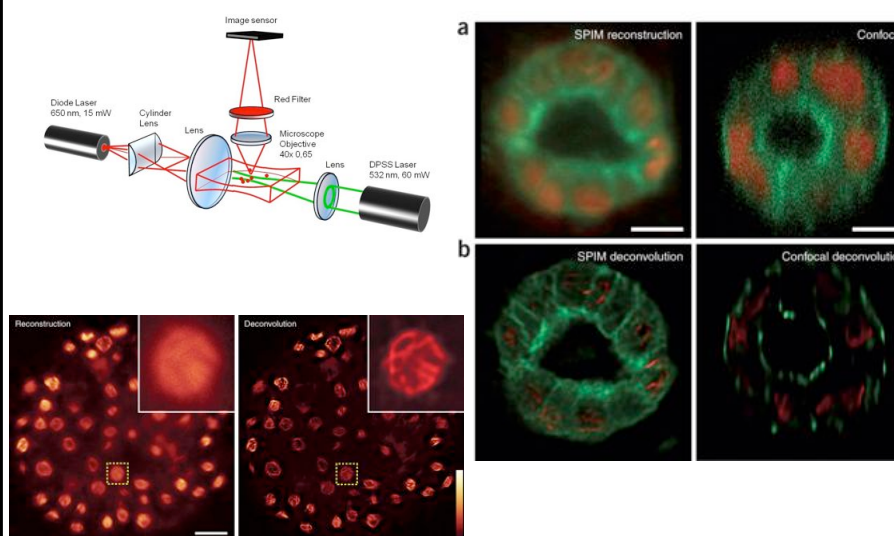
- Single Plane Illumination Microscopy (SPIM) is a fluorescence microscopy technique, where the illumination is done perpendicularly to the detection. The technique shapes the illumination laser beam into a rectangle and then focuses it down only in one direction, using a cylindrical lens.
- This forms a thin "sheet of light" right in the focal plane of the detection objective. As the lightsheet can be tailored to be thin ($< 2\mu\text{m}$ FWHM), we achieve good sectioning of the sample and out-of-focus light suppression. The lateral resolution is given by the detection objective only.



Single Plane Illumination Microscope (SPIM) - 2



Single Plane Illumination Microscope (SPIM) - 3

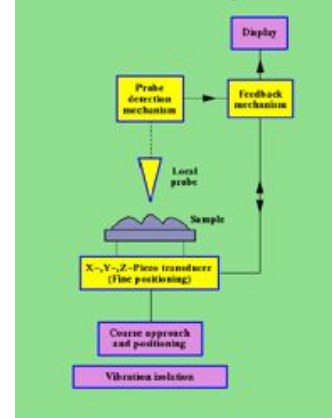




Scanning Probe Microscopy (SPM)

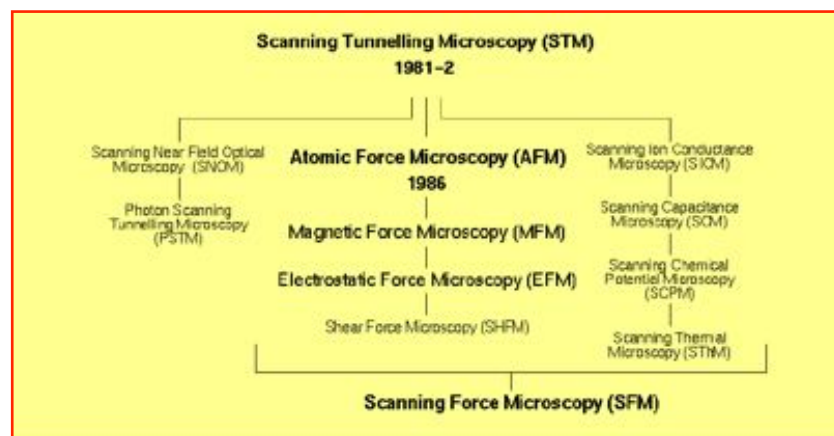
Scanning Probe Microscopy (SPM) is a branch of microscopy that forms images of surfaces using a physical probe that scans the specimen. An image of the surface is obtained by mechanically moving the probe in a raster scan of the specimen, line by line, and recording the probe-surface interaction as a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

Generalized Schematic of a Scanning Probe Microscope



Scanning Probe Microscopy (SPM)

SPM Family:





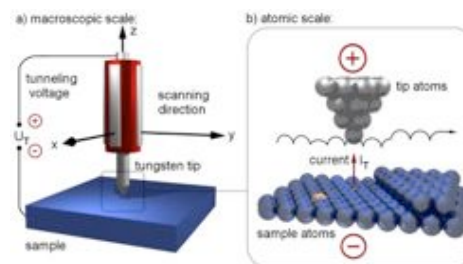
Scanning Tunneling Microscope

- A **scanning tunneling microscope** (STM) is an instrument for imaging surfaces at the atomic level. Its development in 1981 earned its inventors, Gerd Binnig and Heinrich Rohrer (at IBM Zürich), the Nobel Prize in Physics in 1986.^[1] For an STM, good resolution is considered to be 0.1 nm lateral resolution and 0.01 nm depth resolution.
- With this resolution, individual atoms within materials are routinely imaged and manipulated.



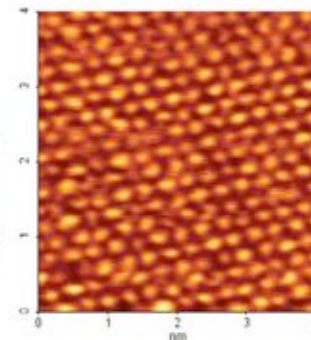
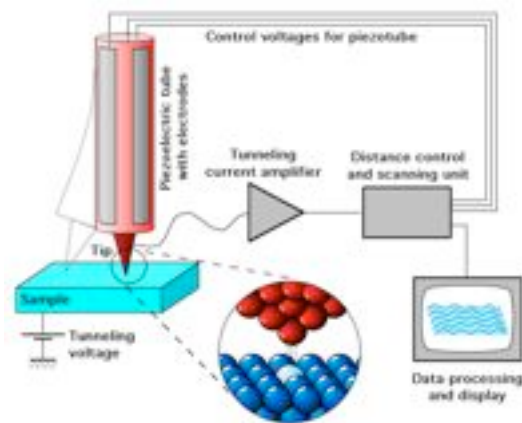
Scanning Tunneling Microscope (STM) - 1

- The STM is based on the concept of quantum tunneling. When a conducting tip is brought very near to the surface to be examined, a bias (voltage difference) applied between the two can allow electrons to tunnel through the vacuum between them. The resulting *tunneling current* is a function of tip position, applied voltage, and the local density of states (LDOS) of the sample.



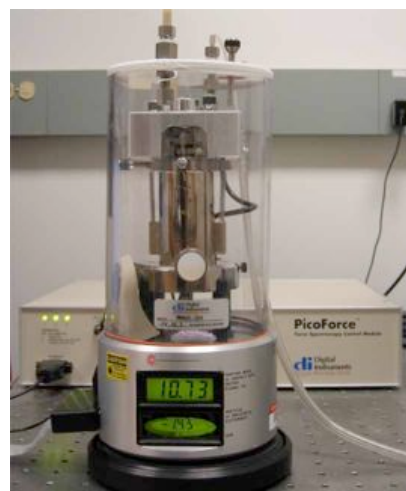


Scanning Tunneling Microscope (STM) - 2



Atomic Force Microscope (AFM) - 1

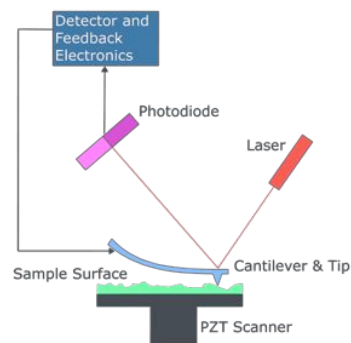
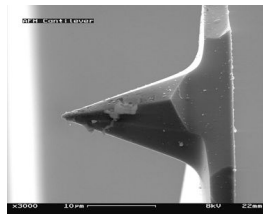
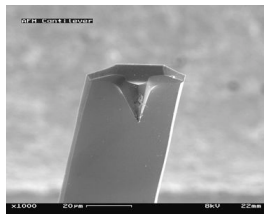
- Atomic force microscopy (AFM) or scanning force microscopy (SFM) is a very high-resolution type of scanning probe microscopy, with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction limit.



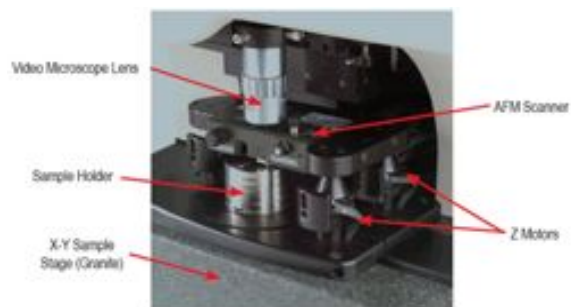
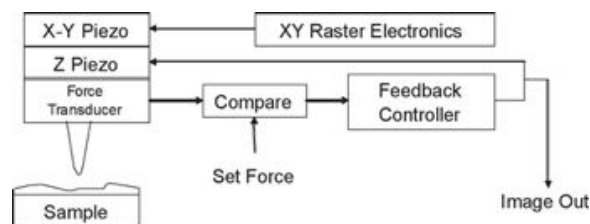


Atomic Force Microscope (AFM) - 2

The AFM consists of a cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface. The cantilever is typically silicon or silicon nitride with a tip radius of curvature on the order of nanometers. 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 according to Hooke's law.



Atomic Force Microscope (AFM) - 3

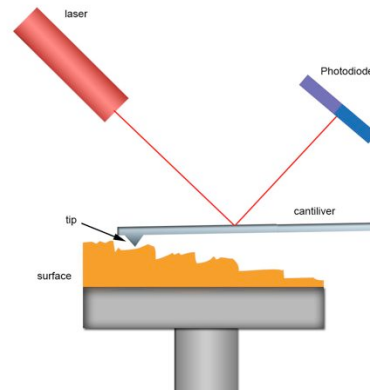




Atomic Force Microscope (AFM) - 4

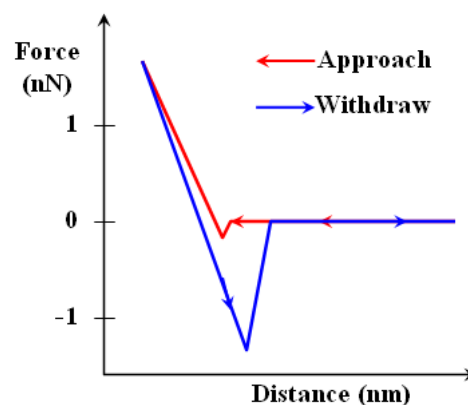
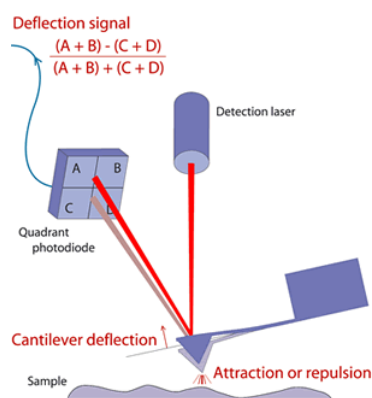
Contact mode

In the static mode operation, the static tip deflection is used as a feedback signal. Because the measurement of a static signal is prone to noise and drift, low stiffness cantilevers are used to boost the deflection signal. In contact mode, the force between the tip and the surface is kept constant during scanning by maintaining a constant deflection.



Atomic Force Microscope (AFM) - 5

Force-Displacement Curve:





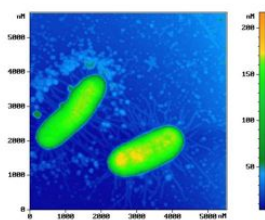
Atomic Force Microscope (AFM) - 6

Tapping mode

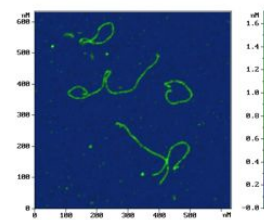
In *tapping mode*, the cantilever is driven to oscillate up and down at near its resonance frequency by a small piezoelectric element mounted in the AFM tip holder. Due to the interaction of forces acting on the cantilever when the tip comes close to the surface, cause the amplitude of this oscillation to decrease as the tip gets closer to the sample. An electronic servo uses the piezoelectric actuator to control the height of the cantilever above the sample. The servo adjusts the height to maintain a set cantilever oscillation amplitude as the cantilever is scanned over the sample.



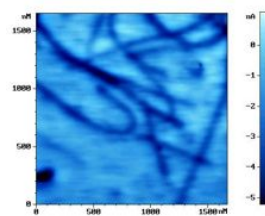
Atomic Force Microscope (AFM) - 7



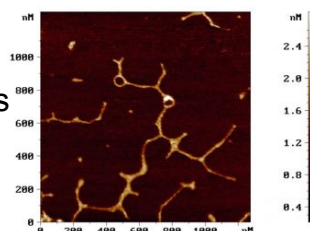
E Coli



Protein



Nanotubes



DNA

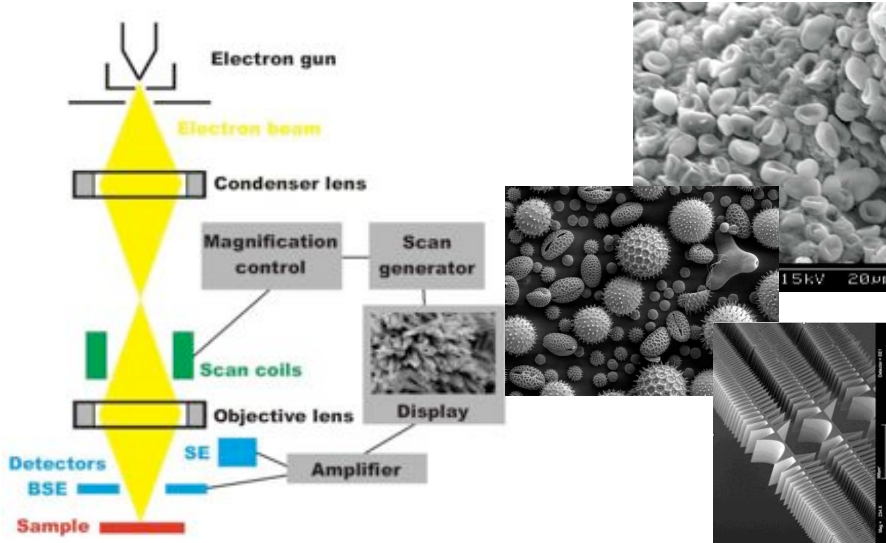


Scanning Electron Microscope (SEM) - 1

A **scanning electron microscope (SEM)** is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity.



Scanning Electron Microscope (SEM) - 2





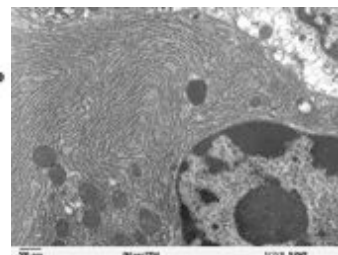
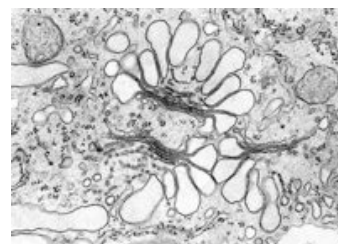
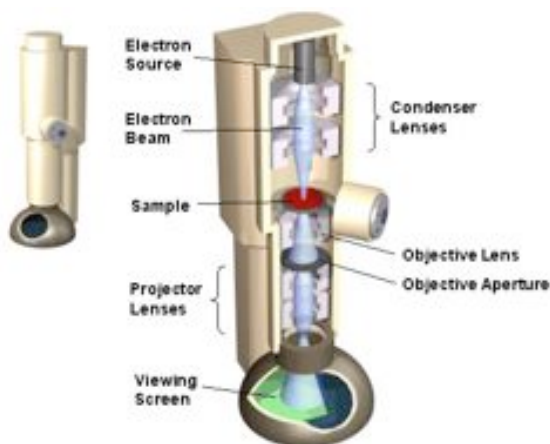
Transmission Electron Microscope (TEM) - 1

Transmission electron microscopy (TEM) is a microscopy technique whereby a beam of electrons is transmitted through an ultra thin specimen, interacting with the specimen as it passes through. An image is formed from the interaction of the electrons transmitted through the specimen; the image is magnified and focused onto an imaging device, such as a fluorescent screen, on a layer of photographic film, or to be detected by a sensor such as a CCD camera.

The first TEM was built by Max Knoll and Ernst Ruska in 1931, with this group developing the first TEM with resolving power greater than that of light in 1933 and the first commercial TEM in 1939.



Transmission Electron Microscope (TEM) - 2





Transmission Electron Microscope (TEM) - 3

SEM vs. TEM:

