# Electron Microscopy

**Reading..............**

**Internet**

and

**Chapter-16**: Semiconductor Characterization Techniques

**Book:** Fundamentals of Solid State Engineering By: *Manijeh Razeghi*

## Microscope

- The main purpose of microscopy is to observe things that cannot be or are hardly observed by naked eye.
- To justify the purpose one can utilize everything that suits the purpose. For example, it can be different light paths coming from different parts of a sample or different reflection from different parts of a sample etc.
- Or in terms of AFM it can be different interaction forces between tip and different parts of a sample. So whatever physical quantity which varies depending on the position on a sample can be utilized to produce an image. And so can be utilized different spectroscopic features (intensity, wavelength, phase, FWHM of a peak etc.).

## Electron Microscope vs. Optical Microscope

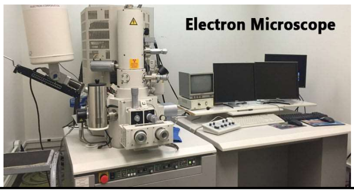
(first one built in 1931 by Ruska and Knoll) (Leeuwenhoek in 17th century)

Electron vs. Photon

Electron: charged, has rest mass, not visible

Photon: neutral, has no rest mass, visible at the wavelength ~ 400 nm-760 nm.

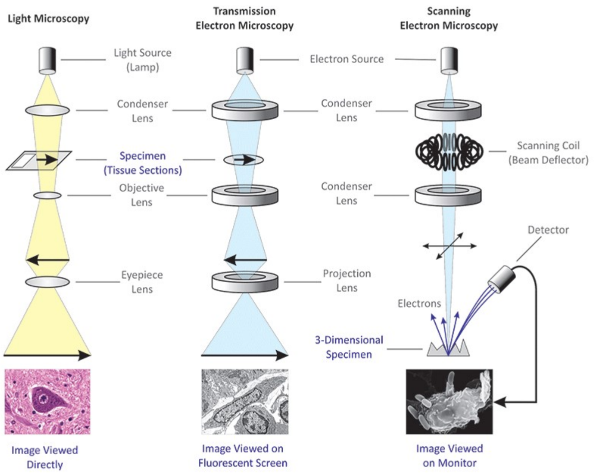
Because of these differences, the microscope construction will also be different



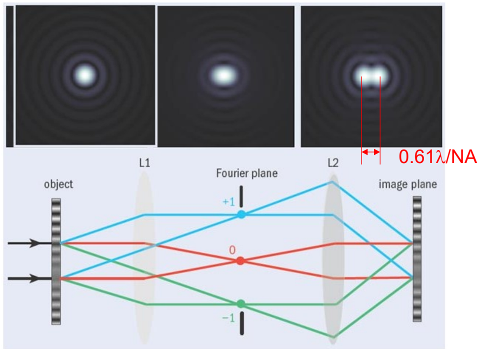

## Types of Electron Microscope

- **Transmission Electron Microscope** (TEM) uses a wide beam of electrons passing through a thin sliced specimen to form an image. This microscope is analogous to a standard upright or inverted light microscope
- **Scanning Electron Microscope** (SEM) uses focused beam of electrons scanning over the surface of thick or thin specimens.. Images are produced one spot at a time in a grid-like raster pattern. (will be discussed in a later lecture)
- **Scanning Transmission Electron Microscope (**STEM) uses a focused beam of electrons scanning through a thin sliced specimen to form an image**.** The STEM looks like a TEM but produces images as does an SEM (one spot at a time). It is most commonly used for elemental analysis of samples.
- **Scanning Auger Electron Microscopy** (SAM) The incident primary electrons cause ionization of atoms within the region illuminated by the focused beam. Subsequent relaxation of the ionized atoms leads to the emission of Auger electrons characteristic of the elements present in this part of the sample surface

# Light Microscope, TEM & SEM




# Resolution of a microscope



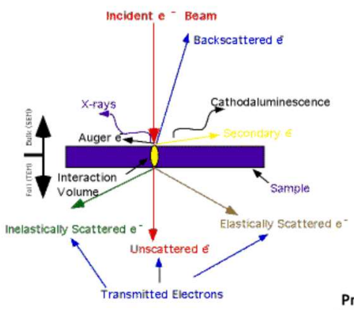

Where N.A. is the numerical aperture = n(sin$\alpha$)

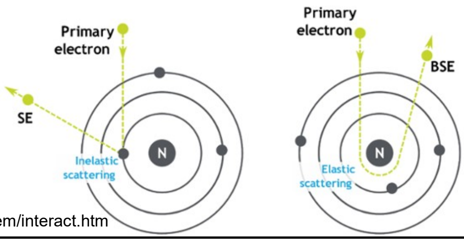
# The resolution is proportional to the wavelength!

## Electron equivalent wavelength

The dualism wave/particle is quantified by the De Broglie equation: **λ = h/p = h/mv**
λ : wavelength; h: Planck constant; p: momentum

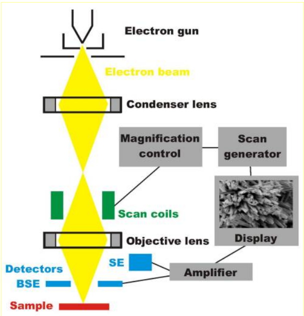
# Electron-Mater interaction

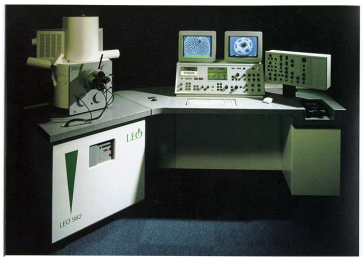



https://www.unl.edu/ncmn-cfem/xzli/em/interact.htm

# SEM



Leo 982 SEM

SEM: a fine electron beam spot is formed by condenser lens and its size determines the resolution (this differs from the TEM which is diffraction limited)

# Electron Gun

## Types of electron guns

**Heated tungsten**

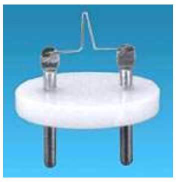
A heated filament made from the metal tungsten. Much in the way that an incandescent lightbulb works, the high voltage that is fed through the filament causes electrons to be kicked off the filament. The amount of energy required is known as the work function.

**Lanthanum hexaboride ($LaB_6$)**

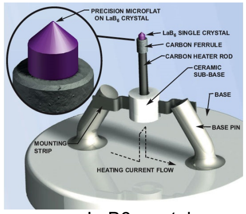
The $LaB_6$ filament is also a thermal filament. However, its work function is lower than for a tungsten filament, so it is more efficient.

**Tungsten field emission gun (FEG)**

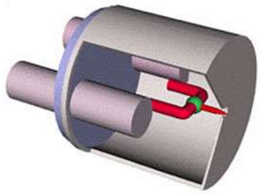
The FEG gun is not a thermal filament. Instead, electrons are expelled by applying a very powerful electric field very close to the filament tip. The size and proximity of the electric field to the electron reservoir in the filament causes the electrons to tunnel out of the reservoir.



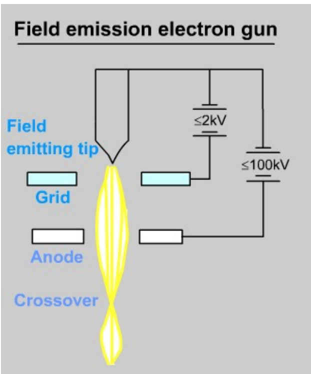
W hairpin




LaB6 crystal

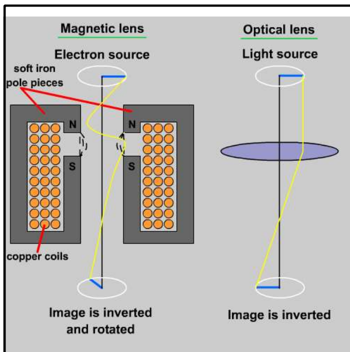


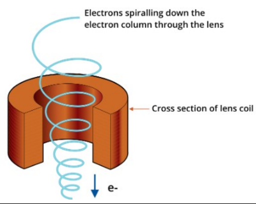
FEG




In the field emission gun, a very strong electric field ($10^9$ $Vm^{-1}$) is used to extract electrons from a metal filament. Temperatures are lower than that needed for thermionic emission.

This gives a much higher source **brightness** than in thermionic guns, but requires a very good vacuum.




# Electromagnetic lens

A lens that converges electron beams by a magnetic field. The magnetic field in the lens, which bends electron beams, is generated by a solenoid magnet.

By changing the electric current to the solenoid, the generated magnetic field is changed, leading to changes of the focal length and magnification.

## Backscattered Electrons:

*Formation*

Caused by an incident electron colliding with an atom in the specimen which is nearly normal to the incident's path. The incident electron is then scattered "backward" 180 degrees.

*Utilization*

The production of backscattered electrons varies directly with the specimen's atomic number. This differing production rates causes higher atomic number elements to appear brighter than lower atomic number elements. This interaction is utilized to differentiate parts of the specimen that have different average atomic number.

## Secondary Electrons:

*Source*

Caused by an incident electron passing "near" an atom in the specimen, near enough to impart some of its energy to a lower energy electron (usually in the K-shell). This causes a slight energy loss and path change in the incident electron and the ionization of the electron in the specimen atom.

This ionized electron then leaves the atom with a very small kinetic energy (5eV) and is then termed a "secondary electron". Each incident electron can produce several secondary electrons.

## Secondary Electrons:

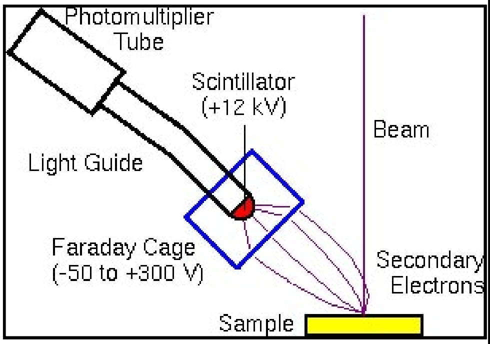
*Utilization*

Production of secondary electrons is dependent on the topography. Due to their low energy, 5eV, only secondaries that are very near the surface (< 10 nm) can exit the sample and be examined.

Any changes in topography in the sample that are larger than this sampling depth will change the yield of secondaries due to collection efficiencies.

The collector is a grid or mesh with a +100V potential applied to it which is placed in front of the detector, attracting the negatively charged secondary electrons to it which then pass through the grid-holes and into the detector to be counted.

## Sec. Elect. Detector




A conventional secondary electron detector is positioned off to the side of the specimen. A faraday cage (kept at a positive bias) draws in the low energy secondary electrons. The electrons are then accelerated towards a scintillator which is kept at a very high bias in order to accelerate them into the phosphor.

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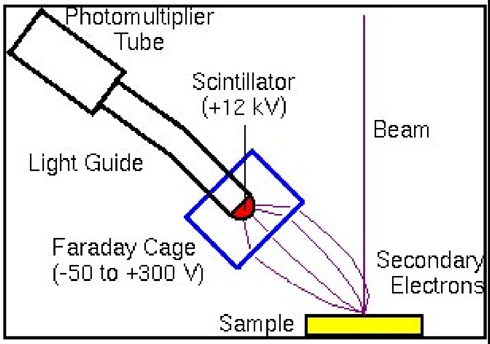


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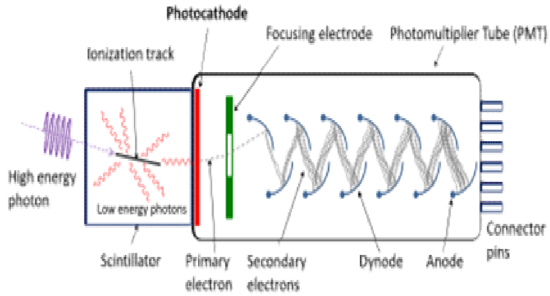
## Scintellator + Photomultiplier Tube (PMT)

Scintillator generates photons in response to incident radiation. PMT converts the light to an electrical signal and electronics to process this signal.




https://en.wikipedia.org/wiki/Scintillation_counter

## Wroking Principle: SEM

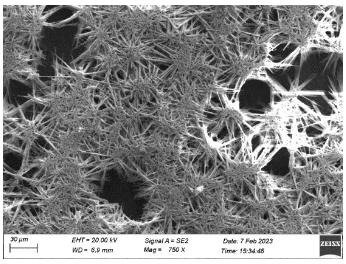
Electrons are emitted from a tungsten cathode either thermionically or via field emission and are focused by two successive condenser lenses into a very narrow beam.

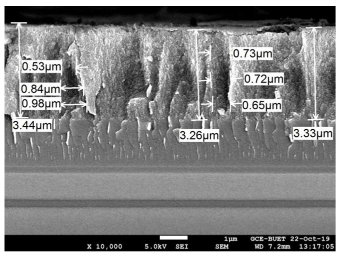
Two pairs of coils deflect the beam over a rectangular area of the specimen surface. Upon impinging on the specimen, the primary electrons transfer their energy inelastically to other atomic electrons and to the lattice.

Through many random scattering processes, some electrons manage to leave the surface to be collected by a detector facing the specimen. Usually these are the **secondary electrons**, originated from a depth of no larger than several angstroms, that are collected by the detector. A photomultiplier tube (PMT) amplifier is used to amplify the signal and the output serves to modulate the intensity of a cathode ray tube (CRT).

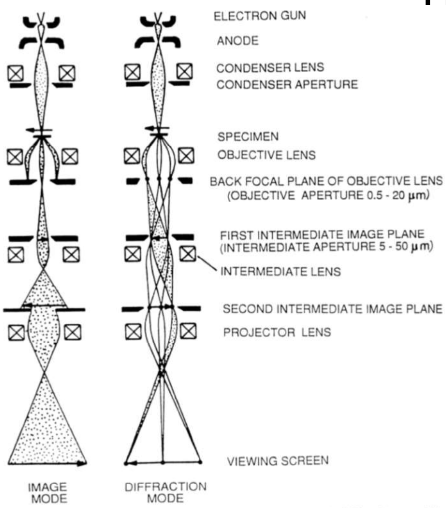
## SEM........

SEM not only can provide images of the surface but also by rotating the sample, one can obtain information about the thickness of various layers in the structure (cross-sectional SEM).





# TEM




Transmission electron microscopy (TEM) is a complex characterization technique that takes advantage of electron diffraction to give the user valuable information regarding the crystallography of the films and, in the image mode, provide high-resolution images of both plain-view and crosssectional view of the films.

# Working Principle of TEM

Electrons are thermionically emitted from the gun and are accelerated to high voltages (in excess of 100 keV). A condenser lens section projects the electron beam onto the specimen. Two types of scattering can occur when electrons hit the specimen: Elastic scattering results in no loss of energy while inelastic scattering involves some energy loss.

Diffraction patterns can be obtained from elastically scattered electrons while inelastically scattered electrons give rise to a spatial variation in the intensity of the transmitted beam. Interactions between the electron beam and the specimen at grain boundaries, dislocations, defect sites, density variations, etc. are the cause of inelastic scattering. Dislocations can be identified when any of the atomic planes terminates.

# Auger Electrons :

*Source*

Caused by the de-energization of the specimen atom after a secondary electron is produced. Since a lower (usually K-shell) electron was emitted from the atom during the secondary electron process an inner (lower energy) shell now has a vacancy. A higher energy electron from the same atom can "fall" to a lower energy, filling the vacancy. This creates and energy surplus in the atom which can be corrected by emitting an outer (lower energy) electron: an Auger Electron.

*Utilization*

Auger Electrons have a characteristic energy, unique to each element from which it was emitted from. These electrons are collected and sorted according to energy to give compositional information about the specimen. Since Auger Electrons have relatively low energy they are only emitted from the bulk specimen from a depth of < 3 nm

# X-rays

*Source*

Caused by the de-energization of the specimen atom after a secondary electron is produced. Since a lower (usually K-shell) electron was emitted from the atom during the secondary electron process an inner (lower energy) shell now has a vacancy. A higher energy electron can "fall" into the lower energy shell, filling the vacancy. As the electron "falls" it emits energy, usually X-rays to balance the total energy of the atom so it.

*Utilization*

X-rays or Light emitted from the atom will have a characteristic energy which is unique to the element from which it originated.

## Unscattered Electron

*Source*

Incident electrons which are transmitted through the thin specimen without any interaction occurring inside the specimen.

*Utilization*

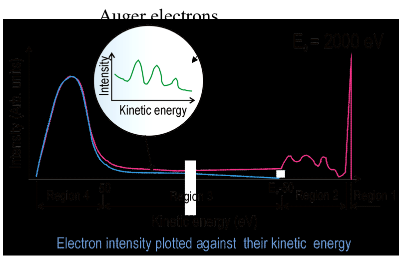
The transmission of unscattered electrons is inversely proportional to the specimen thickness. Areas of the specimen that are thicker will have fewer transmitted unscattered electrons and so will appear darker, conversely the thinner areas will have more transmitted and thus will appear lighter.

# Scanning Auger Microscopy & Spectroscopy

The **Auger Effect** is named after its discoverer, Pierre Auger, who observed a tertiary effect while studying photoemission processes in the 1920s. Auger electrons are emitted at discrete energies that allow the atom of origin to be identified. The idea of using electron-stimulated Auger signals for surface analysis was first suggested in 1953 by J. J. Lander. The technique became practical for surface analysis after Larry Harris in 1967 demonstrated the use of differentiation to enhance the Auger signals.

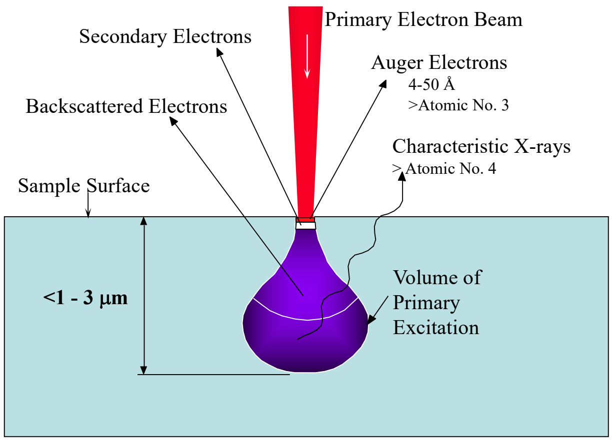
Pierre Auger

**Distribution of Energies of Emitted Electrons**

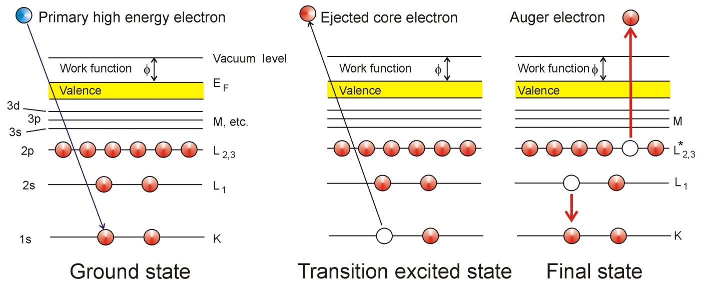



Today Auger electron spectroscopy is a powerful surface analytical tool to probe surfaces, thin films, and interfaces. This utility arises from the combination of surface specificity (0.5 to 10 nm), good spatial surface resolution (as good as 10 nm), periodic table coverage (except hydrogen and helium), and reasonable sensitivity (100 ppm for most elements).

## Electron Beam - Sample Interaction



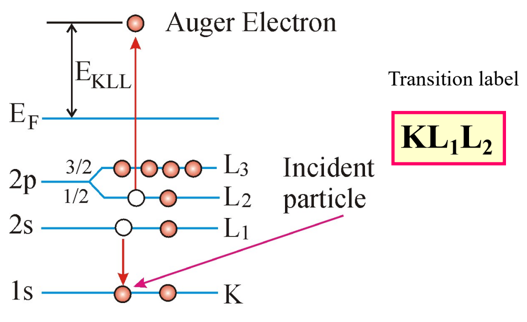
## Auger Process




The Auger process starts with the removal of an inner shell atomic electron to form a vacancy. Several processes are capable of producing the vacancy, but bombardment with an electron beam is the most common. The inner shell vacancy is filled by a second electron from an outer shell. The energy released kicks a third electron, the Auger electron, out of the atom.

Auger is a radiationless process. The process of an excited ion decaying into a doubly charged ion by ejection of an electron is called the Auger process.

## Nomenclature for Auger Transitions




The three symbols in the transition label correspond to the three energy levels involved in the transition.

| | |
|---|---|
| $N_{10}$ | $4f_{7/2}$ |
| $N_9$ | $4f_{5/2}$ |
| $N_8$ | $4f_{3/2}$ |
| $N_7$ | $4f_{1/2}$ |
| $N_6$ | $4d_{5/2}$ |
| $N_5$ | $4d_{3/2}$ |
| $N_4$ | $4d_{1/2}$ |
| $N_3$ | $4p_{3/2}$ |
| $N_2$ | $4p_{1/2}$ |
| $N_1$ | 4s |
| $M_6$ | $3d_{5/2}$ |
| $M_5$ | $3d_{3/2}$ |
| $M_4$ | $3d_{1/2}$ |
| $M_3$ | $3p_{3/2}$ |
| $M_2$ | $3p_{1/2}$ |
| $M_1$ | 3s |
| $L_3$ | $2p_{3/2}$ |
| $L_2$ | $2p_{1/2}$ |
| $L_1$ | 2s |
| K | 1s |

## Energy dispersive analysis using x-rays (EDX)

In EDX an electron from an outer shell of an atom (e.g. the 2s shell) lowers its energy to fill the hole in a lower shell (e.g. the 1s shell) which results in the emission of an x-ray. These emitted x-ray are characteristic of the particular atom undergoing emission. Therefore, by looking at the x-ray spectral lines of an atom one could identify that specific atom.