NACK NETWORK

Building College-University Partnerships for Nanotechnology Workforce Development

Electron Microscopy

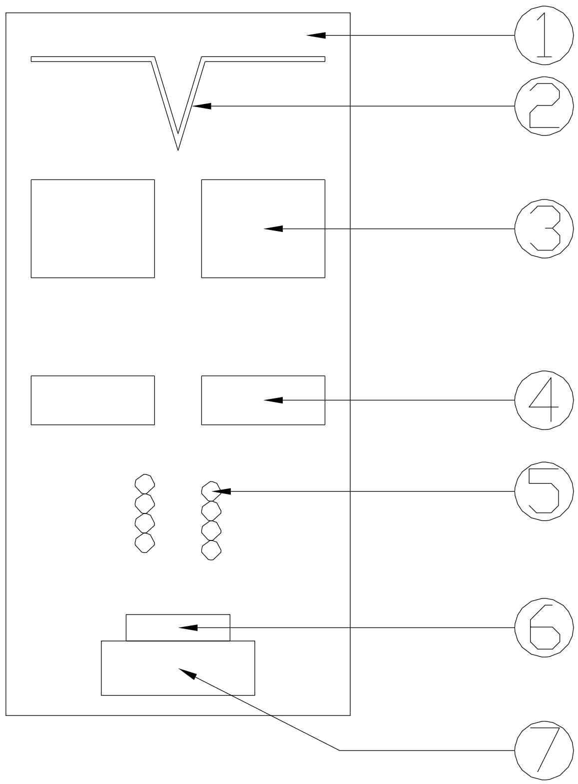
Outline

- Scanning Electron Microscopy
- Field Emission Scanning Electron Microscopy
- Transmission Electron Microscopy
- Energy Dispersive Spectroscopy

SEM

- A microscope that produces an image by using reflected electrons.
- Rudimentary SEM's use thermionic tungsten emitters.
- The electron beam scans the surface of an object and a computer constructs an image from the backscattered or emitted electrons.
- Capable of magnifications of up to 100,000 to 300,000X.
- Capable of resolutions of 4 to 5 nm.

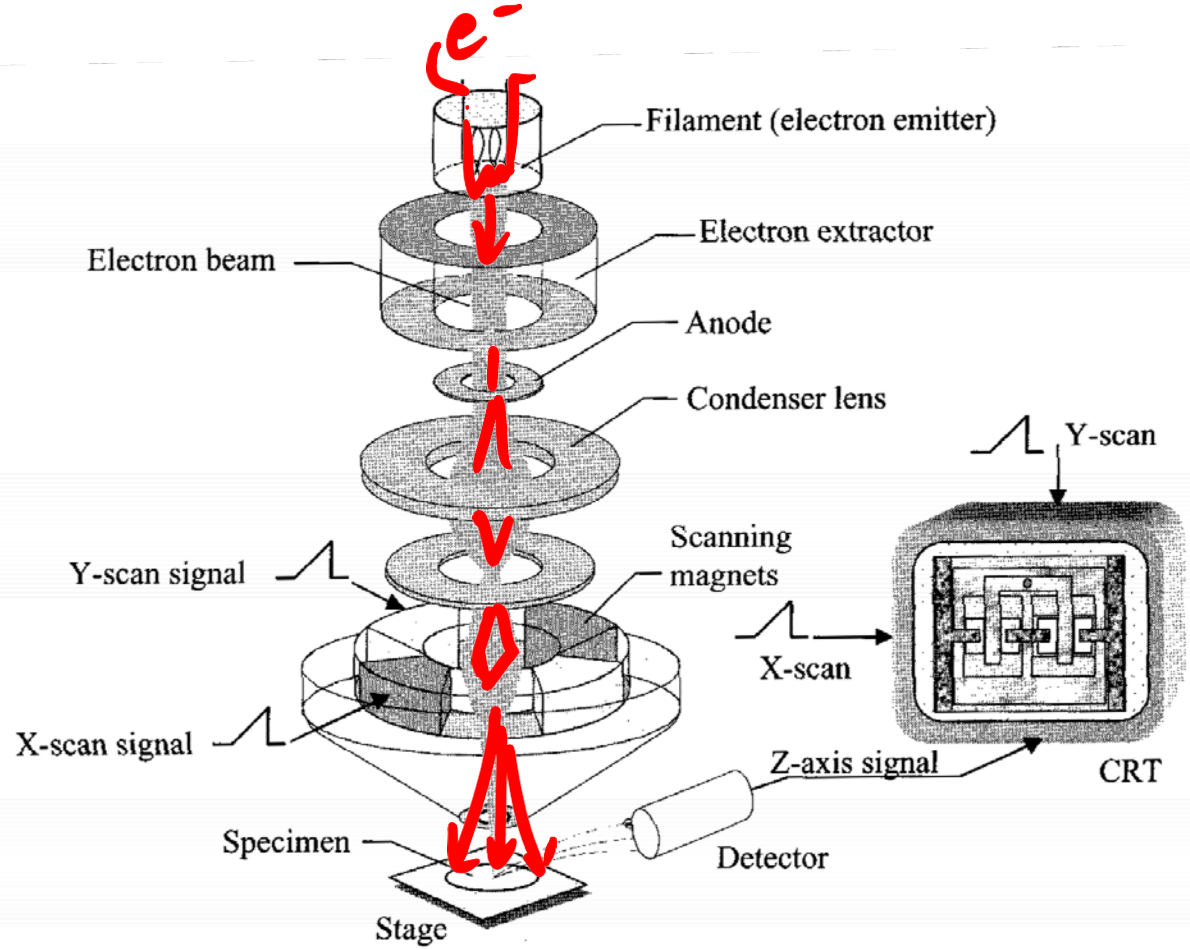
SEM



1. Vacuum Chamber
2. Filament
3. Columating Lens
4. Objective Lens
5. Scan Coils
6. Sample
7. Grounded Sample Stage

Penn Sate Center for Nanotechnology Education and Utilization

SEM



Simple Schematic of a SEM

Quirk, M., Serda, J. *Semiconductor Manufacturing Technology*. Prentice Hall, Upper Saddle River. 2001

SEM

- SEM utilizes an electron gun that produces electrons, focusing elements that shape the electrons into a beam, and a focusing system that makes the electrons strike the sample within a small 2 – 6 nm spot

Emission Sources

- Rudimentary SEMs utilize heated cathode emission sources that produce electrons by **thermionic emission.**
- The most common and least expensive is the sharpened tungsten tip.
- More specialized SEMs such as the FESEM use cold cathode emission sources that produce electrons by **field emission.**

Thermionic Emission

- A high electrical current is passed through a metal or ceramic filament.
- Electrons in the filament material gain thermal energy by joule heating.
- When the thermal energy is greater than the work function of the material, electrons are emitted from the filament and condensed into a beam.

Thermionic Emission

Filament Types:

- **Tungsten Hair-pin**
  - **Inexpensive**
  - **Short life (100s of hours)**
  - **Very resilient technology**
  - **Fast heating**
  - **Most common**
  - **Lowest resolution**
  - **Can be used in poor vacuum systems**

Tungsten hairpin

T_c = 2500 - 3000 K
ϕ = 4.5 eV
$j_c \simeq$ 1-3 A/cm^2
β=(0.3-2)×10^5 Acm^{-2}sr^{-1}
for E = 20 keV
ΔE = 1 - 2 eV
d_o = 20-50 μm

Courtesy "Image Formation in Low-Voltage Scanning Electron Microscopy".(16)

- **Lanthanum Hexaboride / Cerium Hexaboride**
  - **Relatively expensive**
  - **Longer life (1000s of hours)**
  - **Higher brightness than thermionic tungsten**
  - **Smaller Spot size → Higher resolution**
  - **Very sensitive to proper positioning in gun**
  - **Must be heated slowly to prevent damage**
  - **Requires high vacuum**

Pointed LaB_6 rod

T_c = 1400 - 2000 K
ϕ = 2.7 eV
$j_c \simeq$ 20 - 50 A/cm^2
β = (3-20)×10^{5}Acm^{-2}sr^{-1}

ΔE = 1 eV
d_o = 10-20 μm

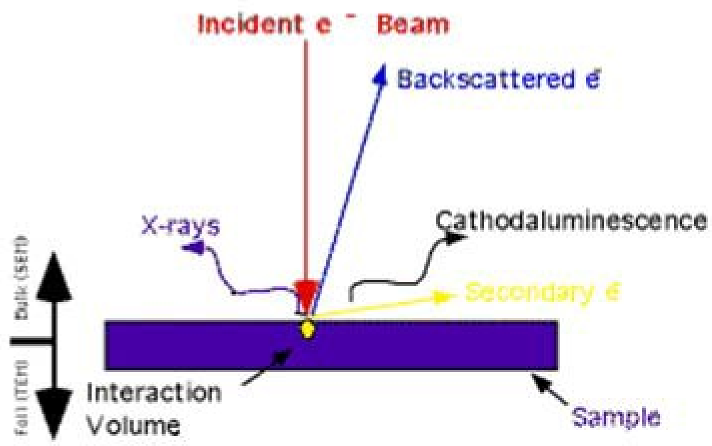
Courtesy "Image Formation in Low-Voltage Scanning Electron Microscopy".(16)

Field Emmision

- Utilizes a potential gradient to overcome the work function rather than thermal energy.
- A filament, usually LaB_6 wire sharpened to a fine point with a radius on the order of 100 nm, is placed in a large electric potential gradient (5-10kV).
- The large electric field gives the electrons enough energy to overcome the material's work function and escape from the filament surface.

SEM Generated Species Types

- These finely focused incident electrons strike the sample with a high voltage (120-200keV).
- The following types of species are created once the electron beam hits the sample:
  - Backscattered electrons
  - Secondary electrons
  - Auger electrons
  - X-Rays



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SEM Generated Species Types

Backscattered Electrons

- Formed when an incident electron strikes the sample and is bounced back from the sample
- Elements with higher atomic numbers absorb more incident electrons allowing fewer to be backscattered
- This gives contrast to different materials in the sample

SEM Generated Species Types

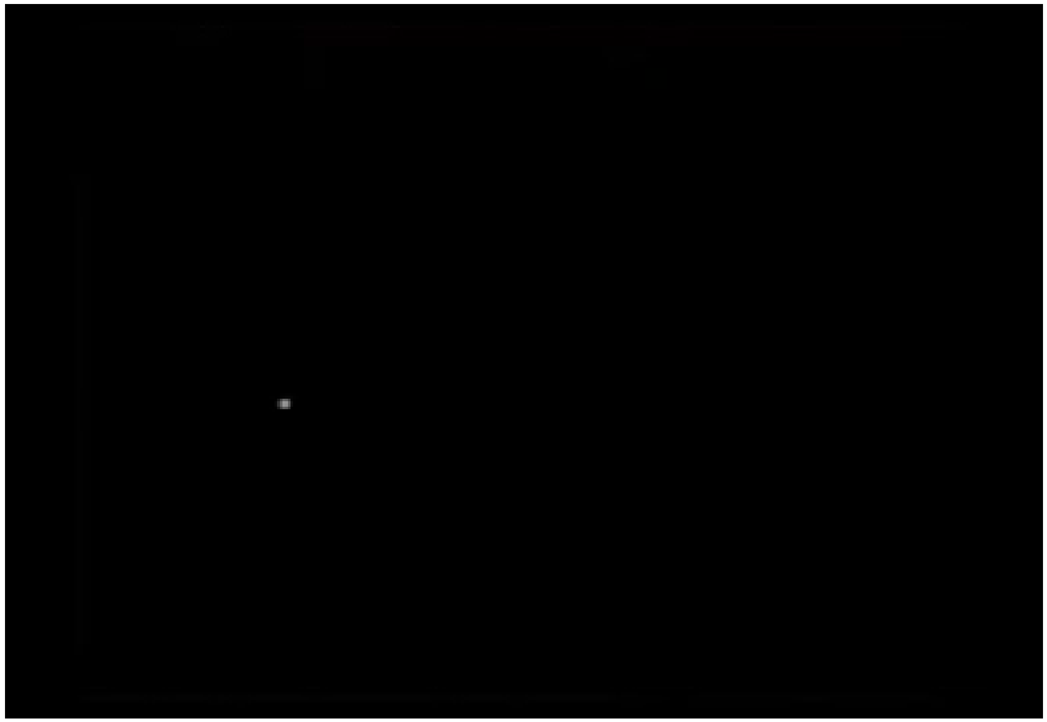
Secondary Electrons

- Formed when an incident electron strikes the sample ejecting an electron from the sample
- Several secondary electrons can be produced from one incident electron
- Secondary electrons are typically emitted by atoms close (10nm) to the surface of the sample and are highly influenced by the topography of the sample

SEM Generated Species Types

Secondary Electrons continued

- Secondary electrons have low energy ~5eV
- Since secondary electrons have such a low energy there is often a biased collecting cage (~ +100V) attached to the secondary electron detector so that the electrons are attracted to the detector



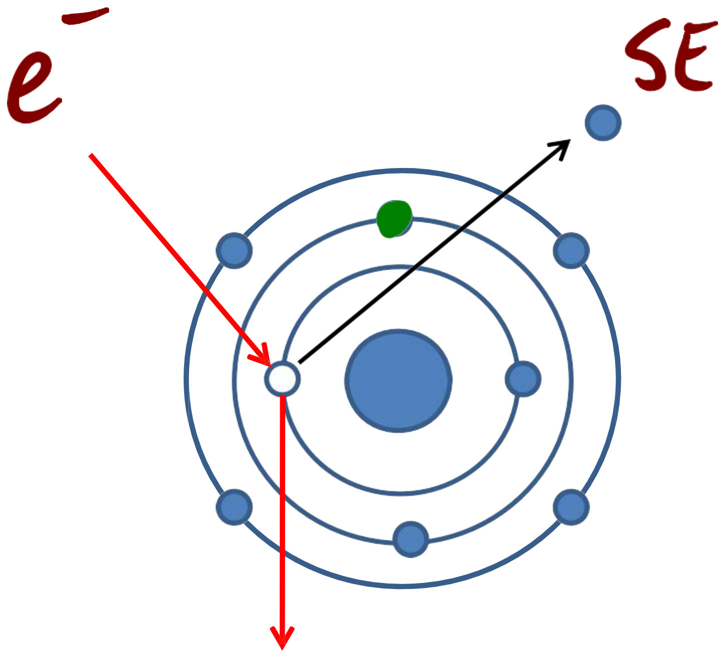
SEM Generated Species Types

Auger Electrons

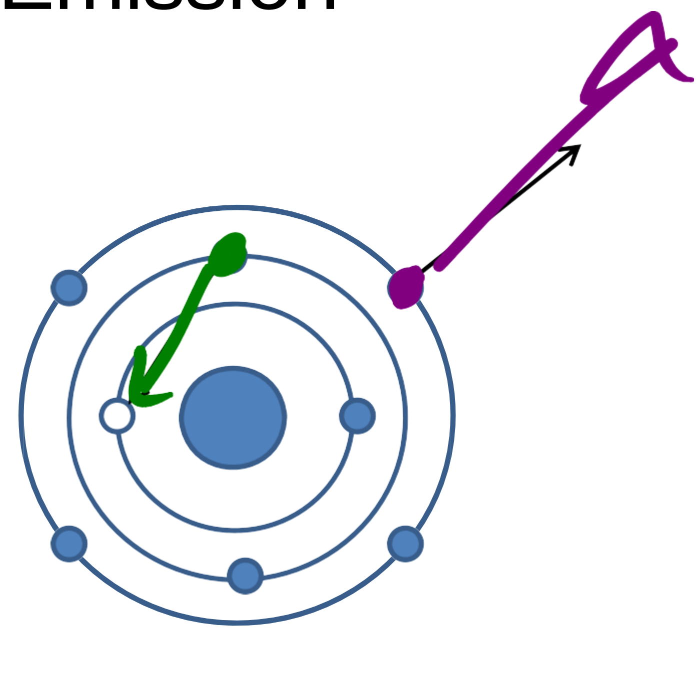
- These low energy electrons are emitted from the sample to achieve an energy balance after a secondary electron has been dislodged
- When an incident electron causes a secondary electron to leave an atom, a vacancy is produce in an orbital near the nucleus
- An electron from an outer shell of the atom will fall to fill the vacancy near the nucleus, creating a surplus of energy
- This surplus of energy causes an Auger electron to be released

SEM Generated Species Types

Auger Electron Emission



Electron Collision



Auger Emission

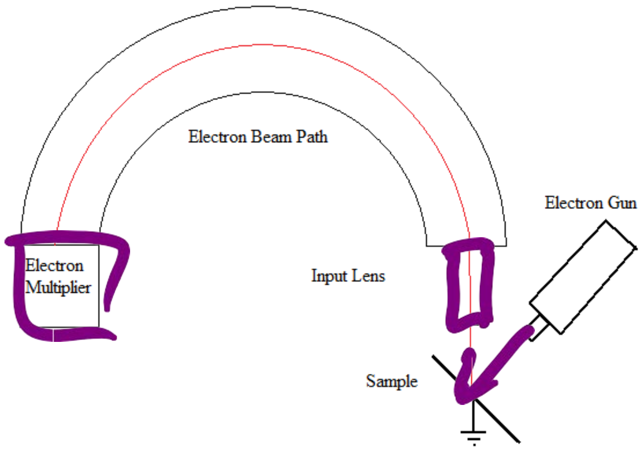
SEM Generated Species Types

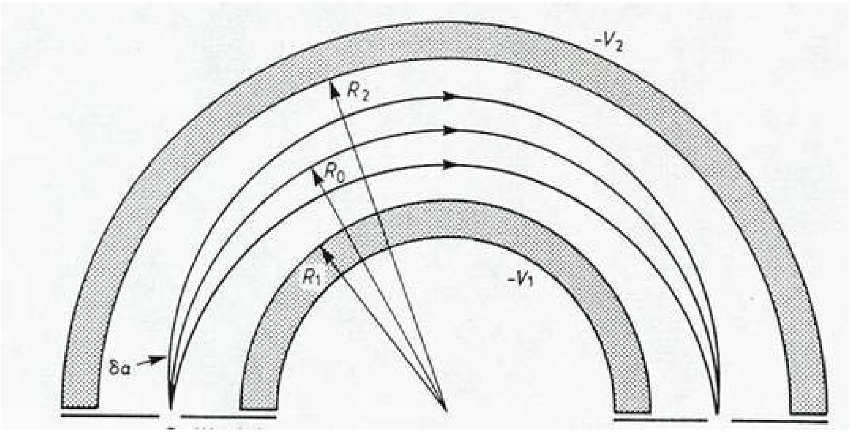
Auger Electrons continued

- Since each element has a different number of orbitals, electrons, and energies, Auger electrons are specific for each atom
- Auger electrons are used to determine the elemental composition of the sample
- Because Auger electrons have such low energy they cannot be emitted from a depth greater than 3 nm

Auger Electron Detectors: Concentric Hemispherical Analyzer

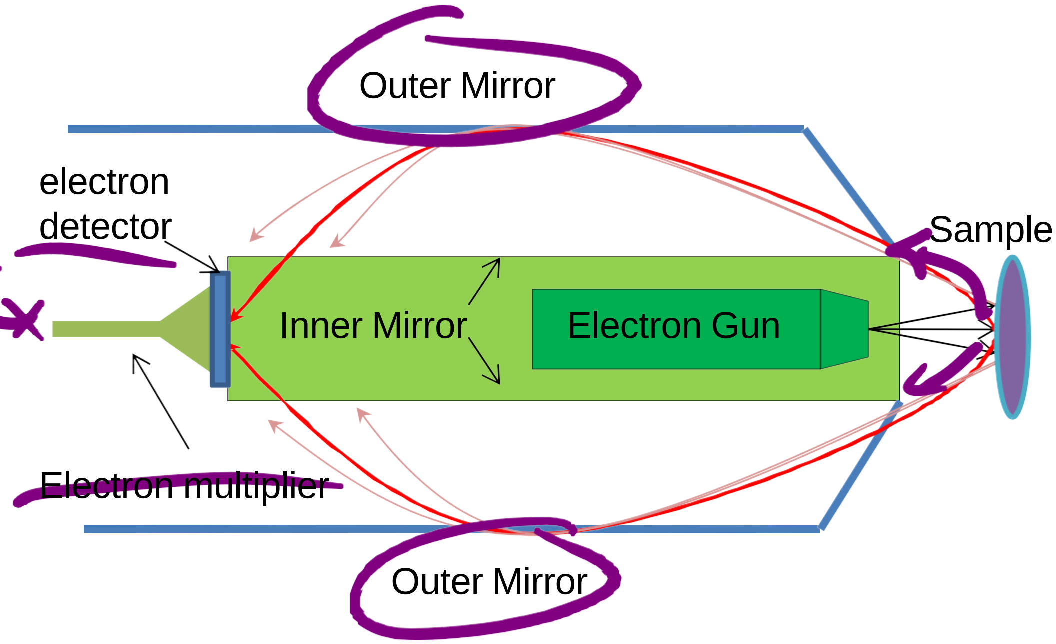
- Electron Gun (E-Beam) strikes sample
- Input lens retards the ejected electrons
- Electrons enter two precision-machined stainless steel concentric hemispheres
- Electrons exiting concentric path are then multiplied and detected for analysis





Auger Electron Detectors: Cylindrical Mirror Analyzer

- Electrons emitted from the sample are focused by two concentric cylindrical mirrors.
- Electrons with energies consistent with auger emission are captured by the detector. Other electrons are reflected by the inner mirror.
- The resulting signal is enhanced by the electron multiplier and sent through an amplifier circuit prior to data acquisition.



“Single pass” Cylindrical Mirror Analyzer

R.J Lee Personal SEM

Specifications	R.J. Lee Personal SEM
Filament	Tungsten Wire
Voltage Range	2kV - 15kV
Secondary Electron Detector	Everhart-Thornley
Backscattered Electron Detector	Solid State Annular
Magnification	10x-200,000x
X-Ray Detector	Elemental Analysis
Pressure	10^{-6} Torr

Outline

- Scanning Electron Microscopy
- Field Emission Scanning Electron Microscopy
- Transmission Electron Microscopy
- Energy Dispersive Spectroscopy

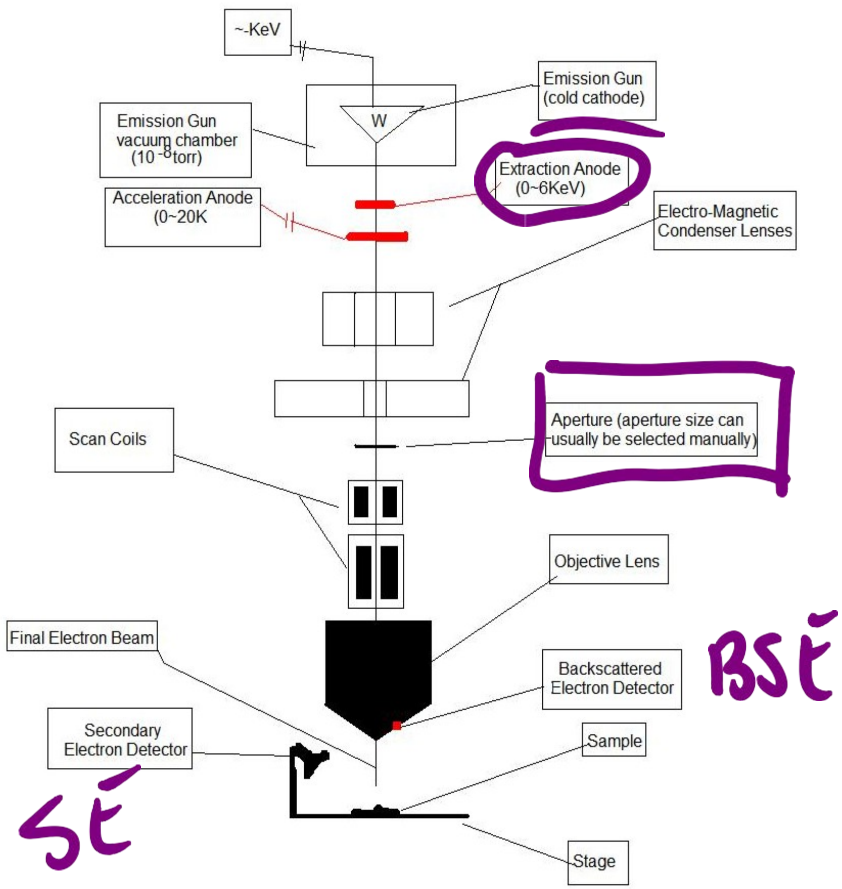
FESEM

- The FESEM is similar to the SEM,
  - The SEM uses a rather large diameter (60,000nm) thermionic tip
  - The FESEM utilizes smaller emission tip with a much smaller diameter (100 – 1000nm)
  - Intrinsically smaller beam gives better resolution

Emission

- A cold cathode is the emission source.
- The emitter has a negative bias of a few kV.
- A positive high voltage anode extracts electrons from the emitter, eliminating the need for thermionic emission.

FESEM



- Field electron emission source.
- Very fine image resolution (~1-2nm).
- Samples should be conductive.

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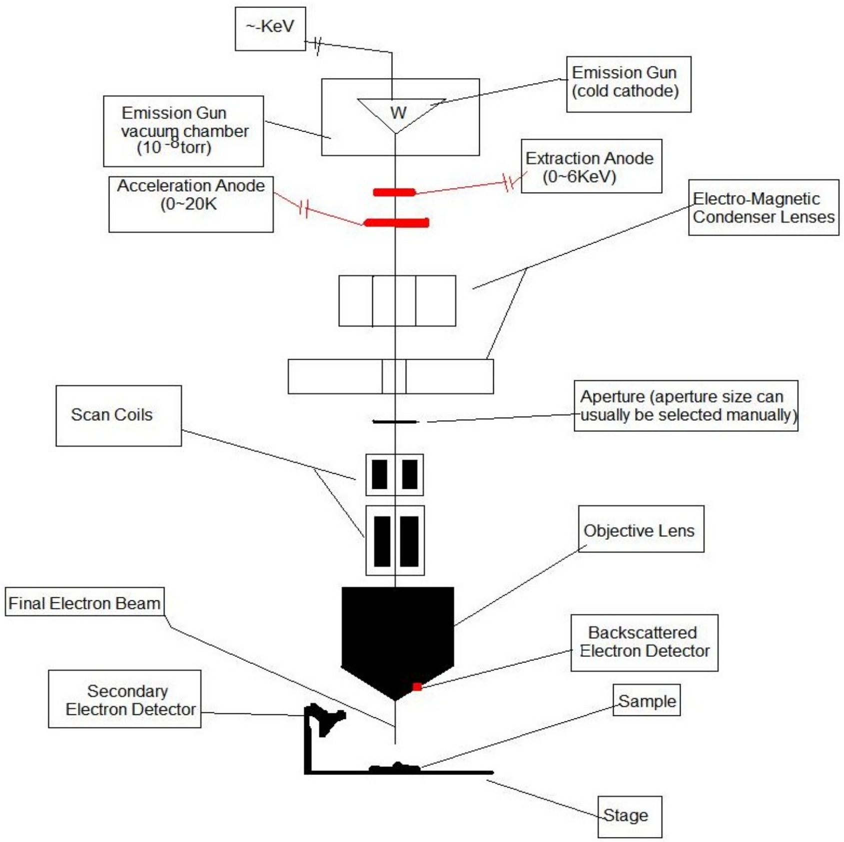
Field Emission Sources

2 types:

- Cold Cathode (W)
  - Very sharp point, tip diameter is ~100nm
  - ~KeV charge, not heated or cooled
  - Electrons “tunnel” out of the tip
  - Vacuum chamber under very low pressure of at least 10^{-8} torr, cryo pump is often used
- Schottky (W, ZrO2)
  - Sharp tip diameter (~1000nm)
  - Heated to at least 1500 C
  - Vacuum chamber at low pressure (~ 10^{-5} torr) achieved by roughing and turbo pumps

FESEM Anodes

- Extraction Anode:
  - 0-6KeV applied to extract or tunnel the electrons from the fine tip
  - Located just below the field emission source
- Acceleration Anode:
  - 0-20KeV applied to accelerate the electrons down through the tube
  - Located below the extraction anode



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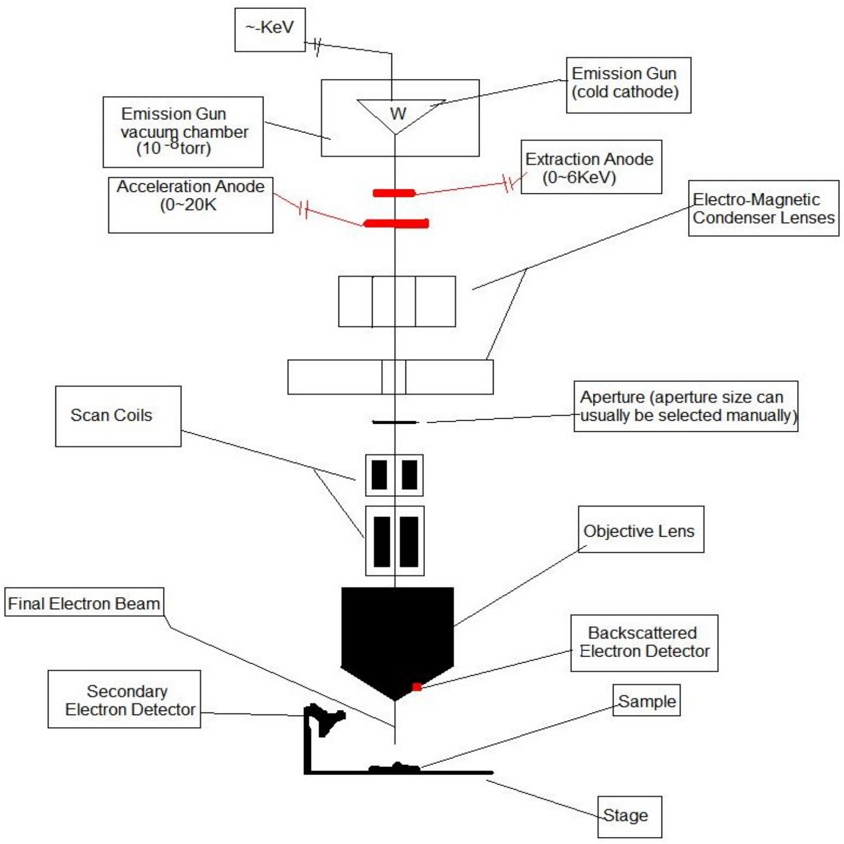
Electro-Magnetic Lenses



- A series of electromagnetic lenses condense the electron beam to its desired size.
- Beam must be smaller than the feature being examined.
- Beam must maintain a constant current density whilst being condensed.
- Avg. FESEM current density is ~17,000A/cm^2

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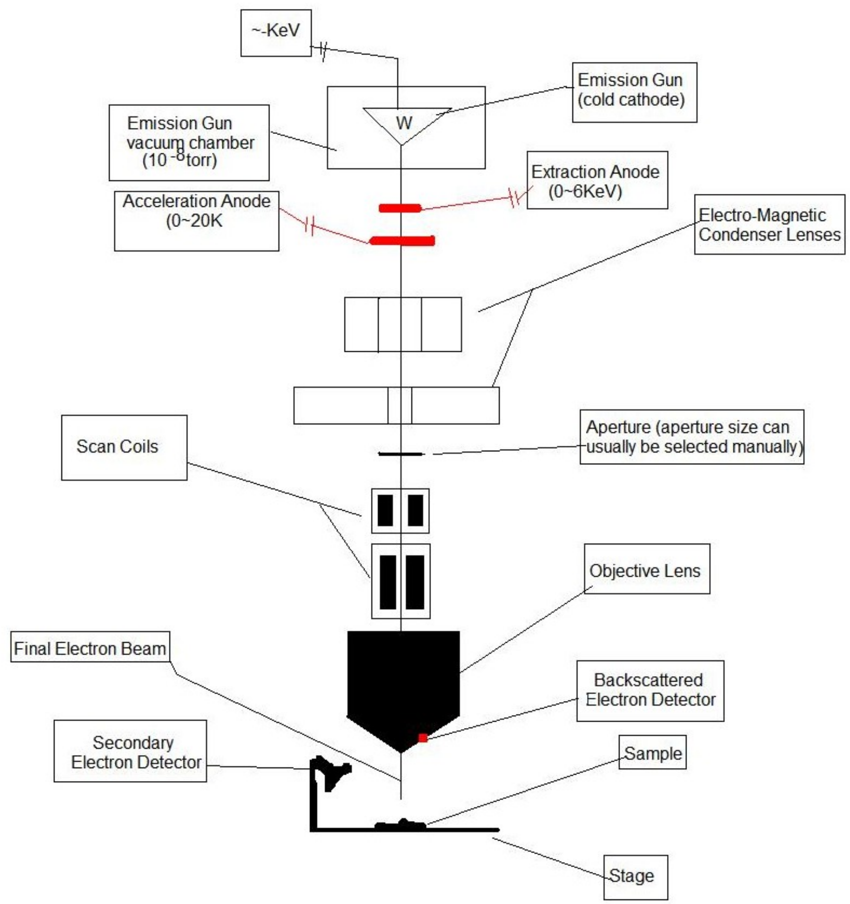
Aperture

- Plate with a small hole for the beam to pass through.
- Usually can be adjusted by the operator.
- Also helps to get the electron beam down to its final diameter and uniformity.



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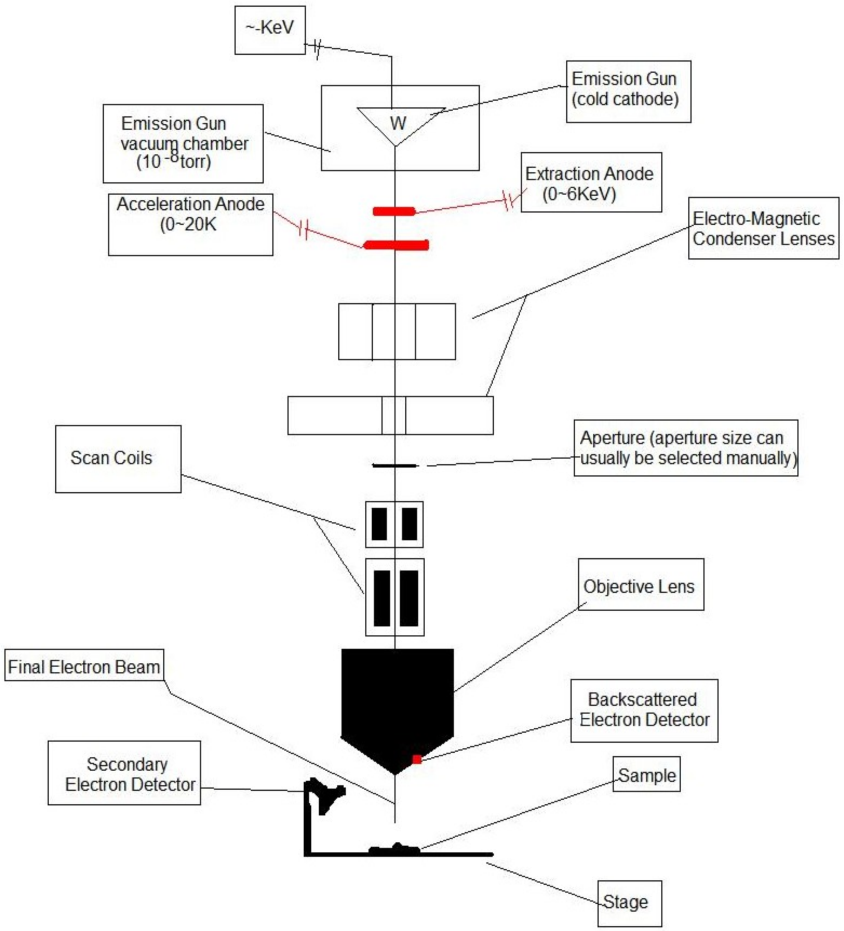
Scanning Coils



- 2 separate scanning coils.
- First scanning coil
  - Deflects the beam back and fourth in a "scanning" fashion over the sample.
- Second scanning coil
  - Referred to as <u>stigmator coil</u>.
  - Corrects problems that may have been caused by the XY deflection of the beam

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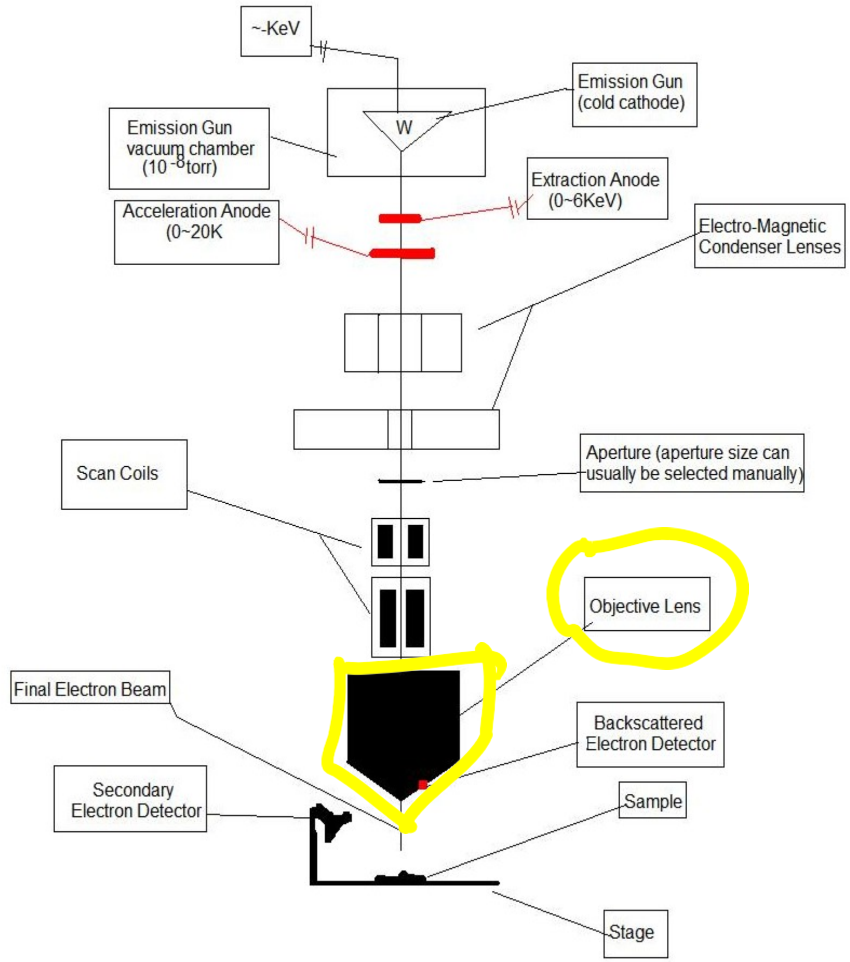
Objective Lens

- Closest part of the FESEM to the sample.
- Focuses the final beam onto the sample.
- Distance between lens and sample (working distance) determines resolution. Short working distance yields better resolution and finer beam diameter.



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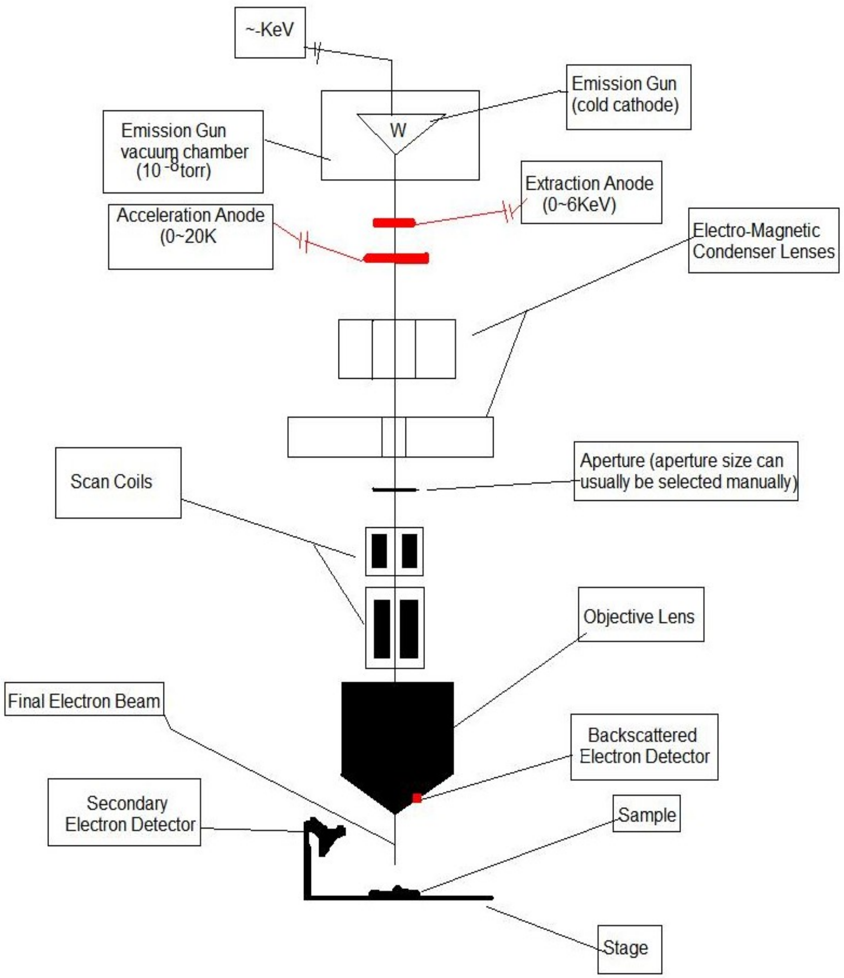
Electron Detection



- Secondary Electron Detector
  - Secondary electrons generally provide most of the image.
- Backscattered Electron Detector:
  - Backscattered electrons reflect back up toward the objective lens
  - Give topographical information as well as chemical composition information.

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Outline

- Scanning Electron Microscopy
- Field Emission Scanning Electron Microscopy
- Transmission Electron Microscopy
- Energy Dispersive Spectroscopy

Transmission Electron Microscopy

- Similar to the SEM, differing in that the electron beam is transmitted through an extremely thin sample.
- The interaction of the electrons traveling through the sample is analyzed.

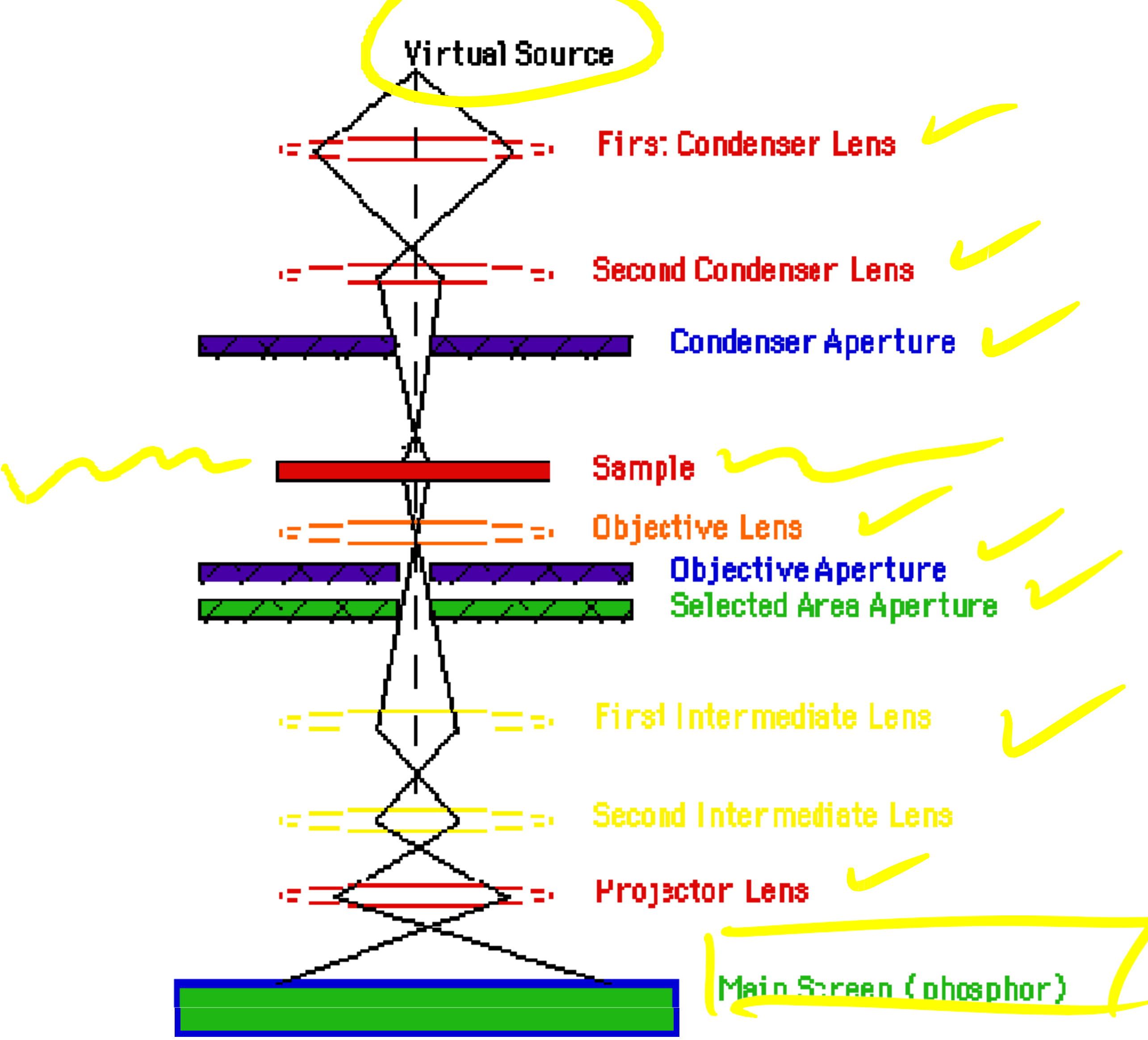
TEM Lens System

- Samples thicknesses between 50-200nm can be viewed with a TEM.
- An electron gun produces monochromatic electrons that are finely focused into a beam with magnets.
- There are multiple electro-magnetic lenses that focus the electrons into a fine beam.

TEM Lens System

- The second condenser lens determines the brightness or intensity of the beam on the sample.
  - This is accomplished by increasing or decreasing the beam diameter that hits the sample.
- The beam then passes through an aperture which filters electrons that are not tightly bound in the beam from traveling to the sample.
- The first condenser lens determines the spot size or the general diameter of the beam when it hits the sample.

TEM Lens System



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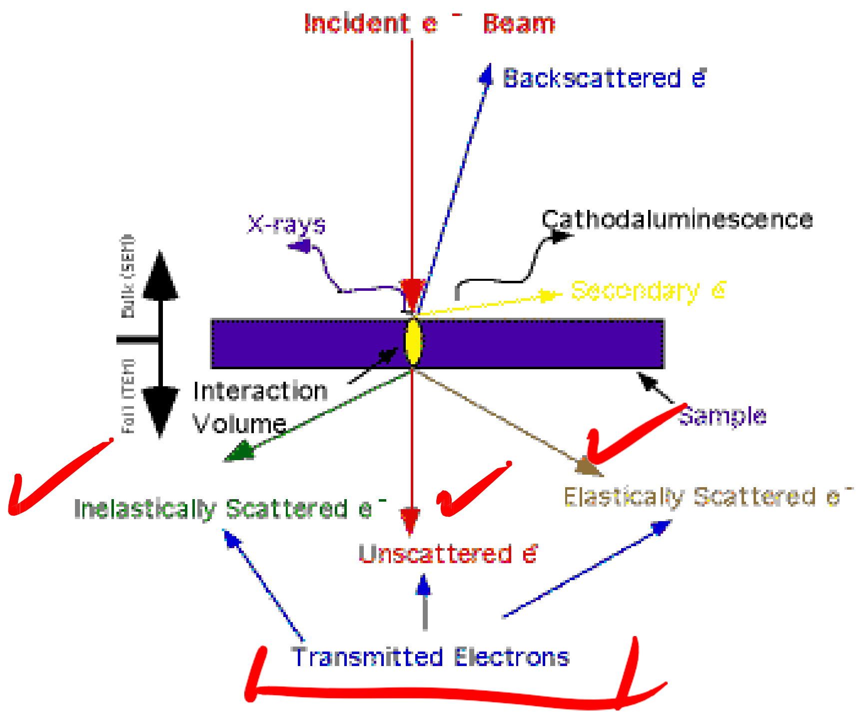
TEM Generated Species Types

These finely focused incident electrons strike the sample with a high voltage (120-200keV).

The following types of species are created once the electron beam hits the sample

- Backscattered electrons
- Secondary electrons
- Auger electrons
- X-Rays
- Unscattered electrons
- Elastically scattered electrons
- Inelastically scattered electrons



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TEM Generated Species Types

- The following species are generated in any EM microscope.
  - Backscattered electrons
  - Secondary electrons
  - Auger electrons
  - X-Rays
- These however are unique to the TEM
  - Unscattered electrons
  - Elastically scattered electrons
  - Inelastically scattered electrons

TEM Generated Species Types

X-Rays

- X-rays are produced when an incident electron causes a secondary electron to leave an atom, a vacancy is produce in an orbital near the nucleus
- An electron from an outer shell of the atom will fall to fill the vacancy near the nucleus, creating a surplus of energy
- This surplus of energy is released as an x-ray
- Emitted x-rays have unique energies associated with a specific atom and are used to determine elemental composition of the sample.

TEM Generated Species Types

Unscattered Electrons

- Incident electrons that pass through, or are transmitted through the sample
- Thinner areas yield more unscattered electrons and appear lighter than thicker areas
- Generally, electrons cannot be transmitted through samples thicker than 200nm without significantly higher energy

TEM Generated Species Types

Elastically Scattered Electrons

- Electrons that pass through the sample, changing course by being deflected by the sample without energy loss
- Bragg's Law states that similar incident electrons are scattered by the same atomic spacing
- These electrons are collected and give information on stoichiometry, atomic arrangement, and crystal lattice of a sample

TEM Generated Species Types

Inelastically Scattered Electrons

- Electrons that pass through the sample, changing course from being deflected by the sample and losing energy
- Electron Energy Loss Spectroscopy
  - The amount of energy lost is characteristic of the element that was hit by the incident electron
  - This will yield compositional and bonding information from the sample

TEM Sample Preparation

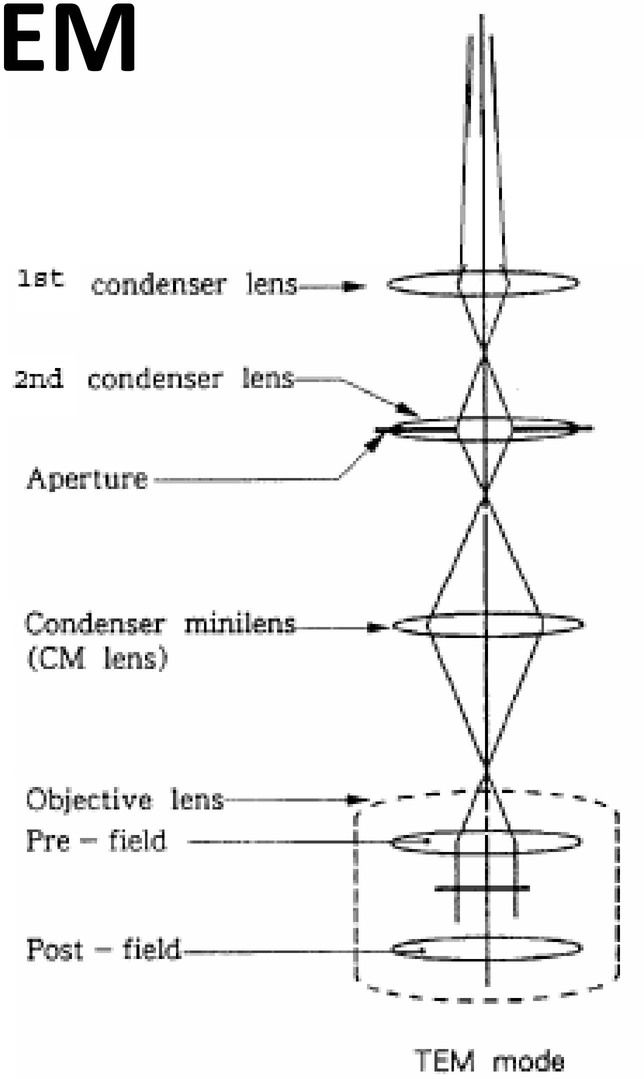
- Samples must be thinned to ~100nm
- Non-conductive samples must be coated with a conductive material to prevent charging

JEOL 2010F 200 kV, Field-emission TEM/STEM

Condenser aperture sizes

- 150 µm
- 70 µm
- 40 µm
- 30 µm
- 10 µm

1.9 Å resolution



PSU MCL JOEL 2010F Operators Manual

JEOL 2010F 200 kV, Field-emission TEM/STEM

Can operate between 100-200kV

Schottky Zirconium Oxide Tungsten (ZrO/W) field emitter

Three apertures

- 1^{st} condenser
- 2^{nd} condenser
- Condenser mini-lens

PSU MCL JOEL 2010F

Outline

- Scanning Electron Microscopy
- Field Emission Scanning Electron Microscopy
- Transmission Electron Microscopy
- Energy Dispersive Spectroscopy (EDS)

Energy Dispersive Spectroscopy (EDS)

- Energy Dispersive X-ray Spectroscopy (EDS) is an analytical capability that can be used in conjunction with SEM/TEM techniques
- This technique is also sometimes referred to as EDXA (energy dispersive x-ray analysis)
- SEM gun electrons collide with the electrons within the sample, causing some of them to be knocked out of their orbits. These electron shell positions are filled by higher energy electrons which emit x-rays in the process.

Energy Dispersive Spectroscopy (EDS)

- Sensitive for low-Z elements, (B-U), not appropriate for high Z elements. Elemental detection typically 0.1 – 1 atomic weight percentage
- Windows in front of the detector can absorb low-energy X-rays, so EDS detectors cannot detect H, He, and Li

Energy Dispersive Spectroscopy (EDS)

- The impact of the electron beam on the sample produces characteristic x-rays that can identify elemental information
- Samples must be vacuum compatible, so it may not be possible to interrogate some organic material

Energy Dispersive Spectroscopy (EDS)

- Can give quantitative results for samples that are flat and homogeneous. Relative x-ray intensity varies with the angle of the surface, so irregular surfaces can cause scattered x-rays.
- Analyzers are then used to characterize the x-ray photons for their energy (or wavelength) and abundance to determine the elemental makeup of the area under test

Energy Dispersive Spectroscopy (EDS)

- EDS can be used to gather pin point information, or to map the elemental composition of a larger area Typical spot size is 1 micron.
- The test is inexpensive and widely available on many SEM/TEM systems
- Can be used for a number of industries: aerospace, biomedical/biotechnology, electronics, pharmaceutical , photonics