

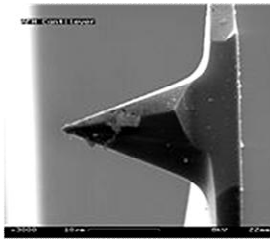
SEM and TEM

Collected by: M.Azadi

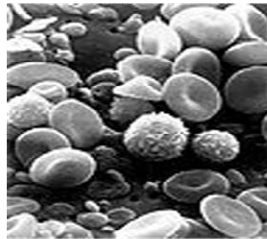
INTRODUCTION

- ❑ Electron microscopes are scientific instruments that use a beam of energetic electrons to examine objects on a very fine scale.
- ❑ Through the use of an electron microscope we can see things that we would not normally be able to see with our naked eyes and has greater magnification than light microscope.
- ❑ This required 10,000x plus magnification which was not possible using current optical microscopes

What does it look like....



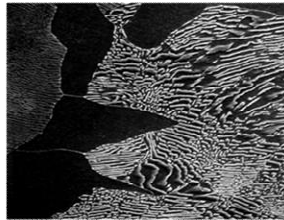
AFM Cantilever Tip



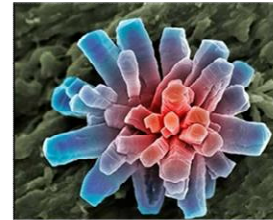
Blood Cells



Diamond Thin Film
(Numerous Multifaceted Micro-
crystals)



Microstructure of a plain carbon
steel that contains 0.44 wt% of
carbon



Calcium Phosphate
Crystal

Section 1:

Fundamentals of Electron Microscopy

- Why electrons instead of photons?
- De Broglie wavelength and resolution limits
- Electron–matter interactions
- Comparison: Optical vs SEM vs TEM

Questions

- To establish why **electrons** are necessary and what **problems they solve**.
- Why **can't we just build a better light microscope** to see atoms?

- **The Concept:** Explain that light behaves as a wave. When light passes through an aperture or around an object, it bends (diffracts). This diffraction creates interference patterns that blur the image.
 - **Abbe's Diffraction Limit:** Introduce Ernst Abbe's equation:
 - $d = \frac{\lambda}{2n \sin \theta}$
 - Where d is the resolution the smallest distance between two points we can distinguish, λ is the wavelength of light, and $n \sin \theta$ is the numerical aperture. ~ 400 nm-760 nm
 - **The Takeaway:** Resolution is directly proportional to wavelength. The best light microscopes are limited to about **200 nm** (0.2 micrometers).
 - Cannot resolve:
 - Nanostructures
 - Grain boundaries
 - Dislocations
 - Atomic lattices
- ☞ **Electrons provide much smaller wavelengths**

1.2 The Electron Solution

- **de Broglie's Wave-Particle Duality:** Introduce the concept that electrons, despite being particles, also have a wavelength. **p: momentum**
- **The Equation:** $\lambda = \frac{h}{p} = \frac{h}{mv}$ (where h is Planck's constant, m is mass, v is velocity). **$p = mv = (2mV)^{1/2}$**
- **Accelerating Voltage:** Explain that by accelerating an electron through a voltage (e.g., 5kV, 20kV, 200kV), we increase its velocity (v), which decreases its wavelength (λ).

Resolution in Electron Microscopy

Practical Resolution Limit

$$d \approx \frac{0.61\lambda}{\alpha}$$

Where:

- α = semi-angle of convergence

- **The Result:**

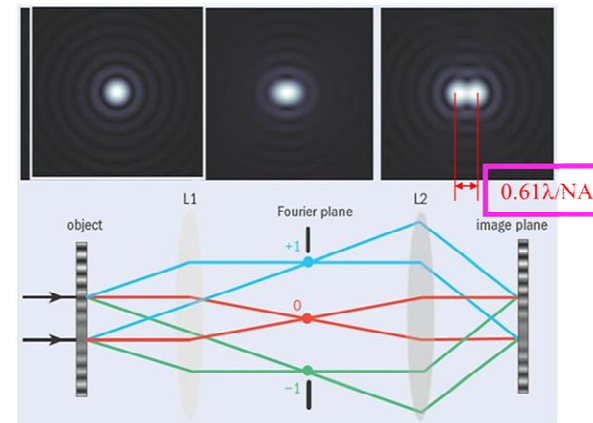
- At 10 kV, an electron wavelength is ~ 0.012 nm.
- At 200 kV (TEM), an electron wavelength is ~ 0.0025 nm.
- This is **100,000x shorter** than visible light, theoretically allowing us to resolve atoms.

TEM uses **100–300 keV**

Modern corrected TEM:

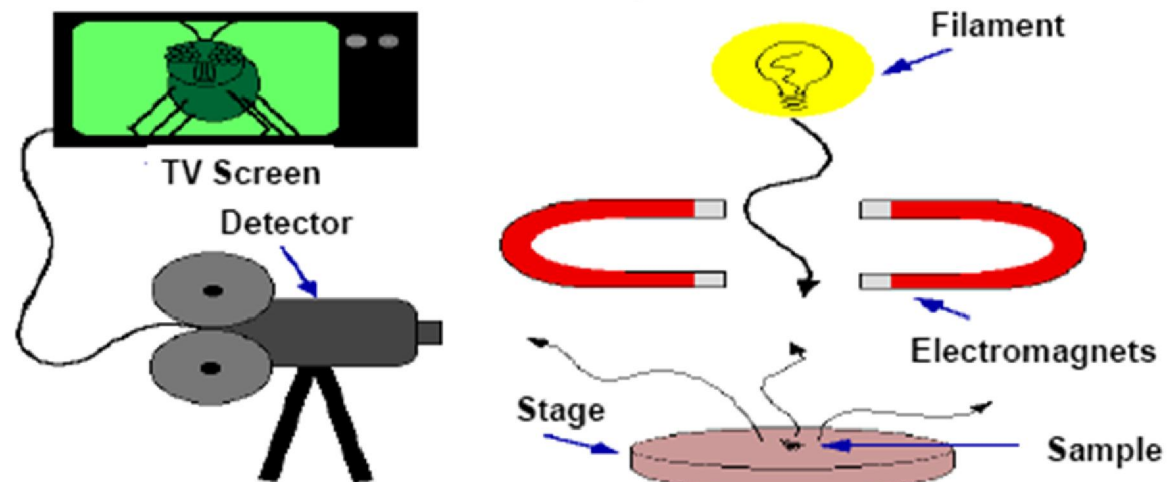
👉 Resolution < 0.5 Å

Resolution of a microscope



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

Breakdown of an Electron Microscope



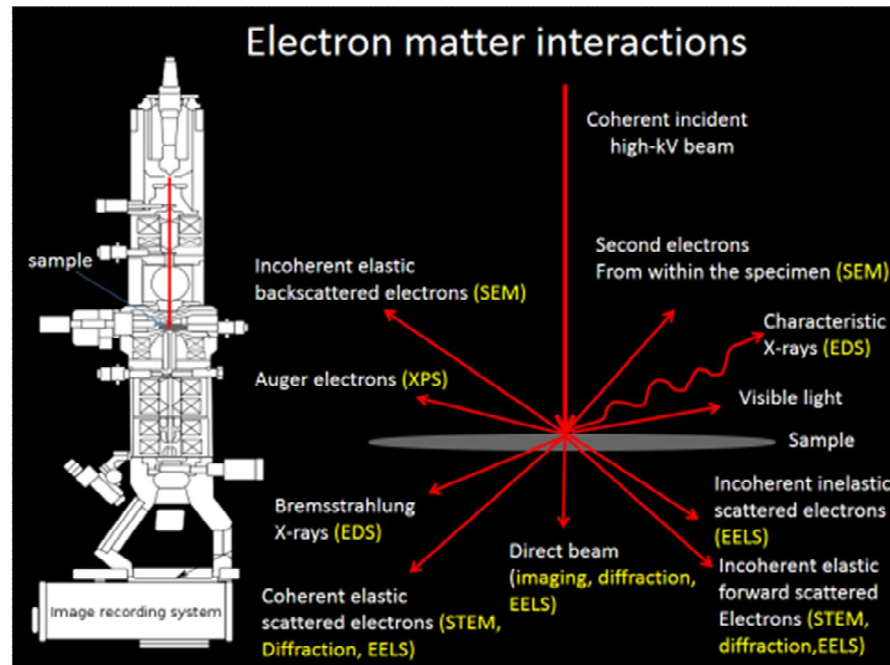
In simplest terms, an SEM is really nothing more than a television. We use a filament to get electrons, magnets to move them around, and a detector acts like a camera to produce an image.

Electron-Matter Interactions

When electrons hit matter:

- Elastic scattering
- Inelastic scattering

☞ All SEM & TEM contrast originates here



1. Elastic Scattering

In this type of collision, the **beam electrons** interact with the **nucleus of the sample's atoms** or **the electron cloud around it**. Due to the very high mass of the nucleus compared to the electron, the incoming electron **loses almost no kinetic energy**. The only thing that **changes** is the **path** of the electron.

The electron is deflected at **a wide angle** and sometimes even bounces back. This is the same electron that later exits the sample as a **Backscattered Electron (BSE)** .

This phenomenon is the basis for imaging with backscattered electrons. The heavier the atom (**higher atomic number**), the **stronger the electric field** of the nucleus, and the higher the probability of **elastic scattering** and thus BSE production.

This is why heavier materials appear brighter in BSE images.

2. Inelastic Scattering

In this type of collision, the beam electrons **interact** with the **electrons of the sample's atoms** (either **inner shell or outer shell electrons**). Since the masses of the two particles are equal (electron-electron), the incoming electron loses a **significant amount of its energy**. This energy is transferred to the sample atom's electron.

Electron–Matter Interactions

Elastic vs Inelastic Scattering

- **Elastic scattering:**
- No energy loss
- Direction changes (0-180 degree)
- Produces:
 - Diffraction
 - Backscattered electrons

Inelastic scattering:

- Energy loss
- Produces:
 - Secondary electrons
 - X-rays
 - Auger electrons
 - Plasmons (EELS)

Interaction Volume

The **interaction volume** is the region in which electrons scatter inside the material.

Key dependencies:

- Accelerating voltage (E_0)
- Atomic number (Z) or Density (ρ)
- Radiation angle

Typical size:

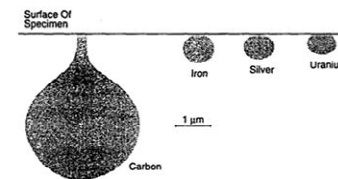
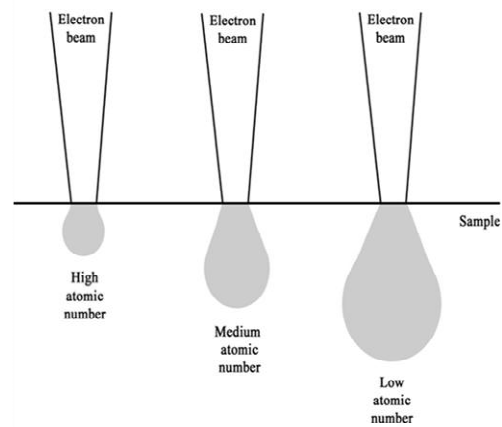
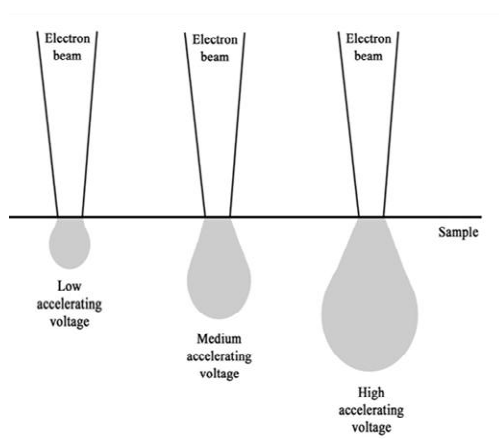
- 1 keV $\rightarrow$ few nm
- 20 keV $\rightarrow$ several μm

✦ This explains **why SEM resolution depends strongly on kV.**

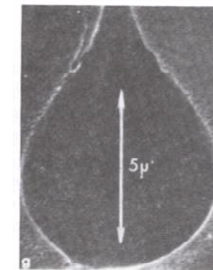
Higher voltage $\rightarrow$ larger volume
Higher $Z \rightarrow$ smaller volume
Higher angle $\rightarrow$ smaller volume

Electron beam-sample interactions

- Interaction volume increases with increasing acceleration voltage and decreases with increasing atomic number.



Pear shape



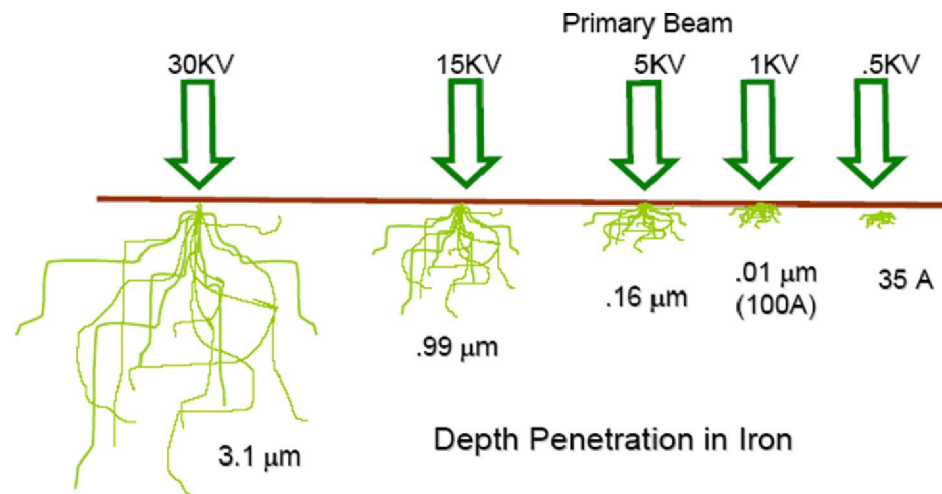
Kanaya-Okayama Depth Penetration

Formula

$$R = \frac{0.0276 A E^{1.67}}{(Z^{0.89} \rho)} \quad \mu\text{m}$$

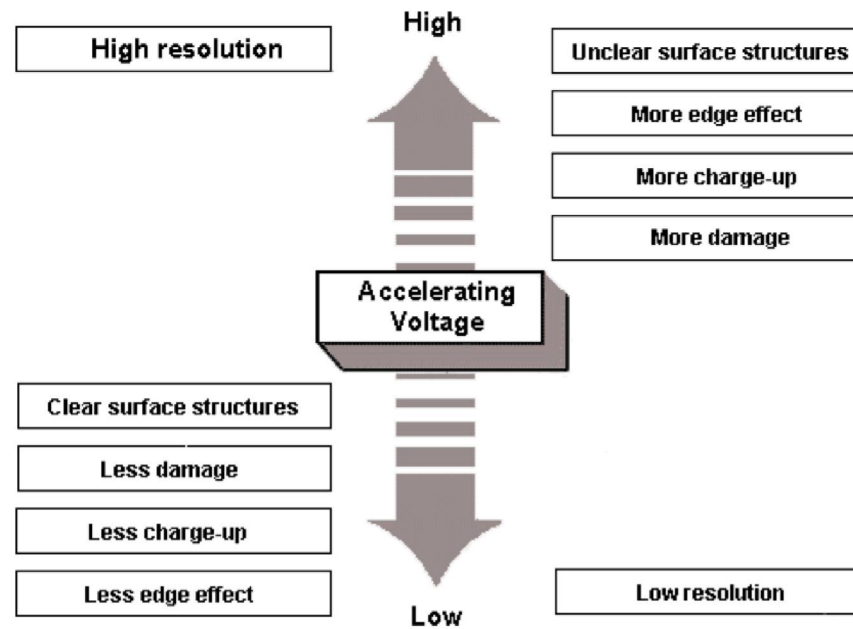
R= Depth Penetration
 A= Atomic Weight (g/mole)
 E= Beam Energy (KV)
 Z= Atomic number
 ρ = density (g/cm)³

The Affect of Accelerating Voltage

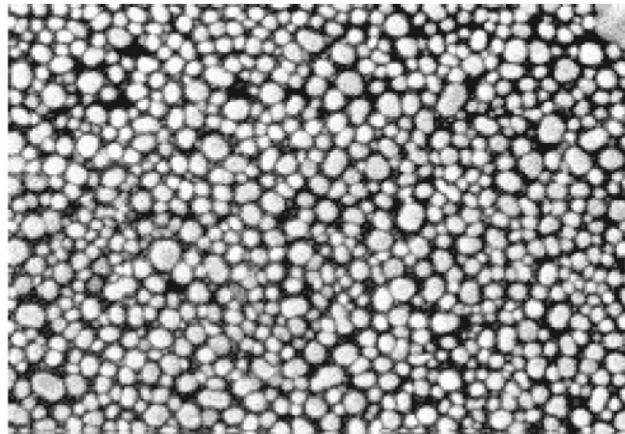


(predictions from the KO formula)

Effect of Accelerating Voltage on Image Quality

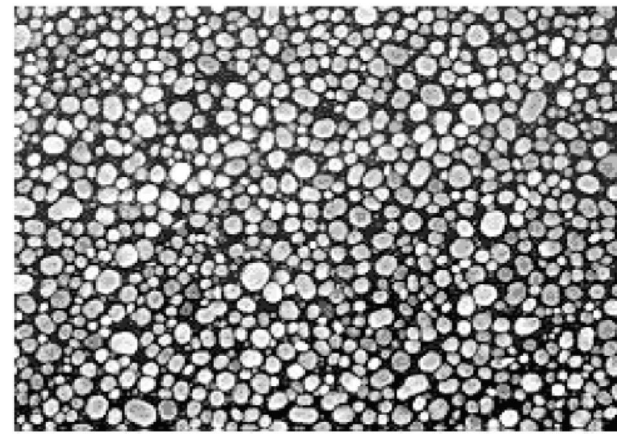


Effect of Accelerating Voltage on Observed Image



5.0 kV

36,000 X



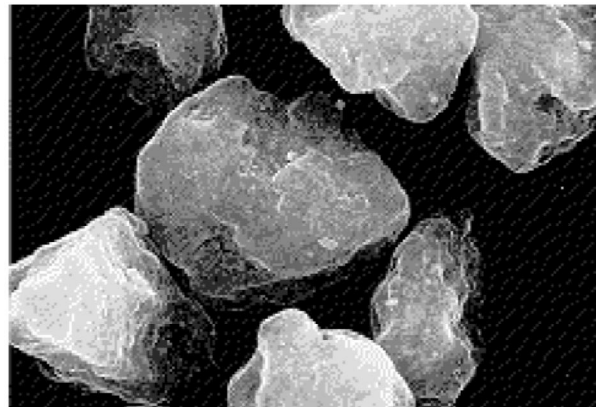
25 kV

36,000 X

Evaporated Au particles

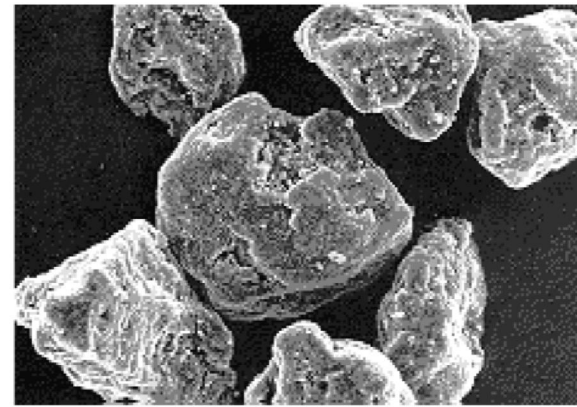
The image sharpness and resolution are better at the higher accelerating voltage, 25 kV

Effect of Accelerating Voltage on Observed Image



(a) 30 kV

2,500 X



(b) 5.0 kV

2,500 X

When high accelerating voltage is used as at (a), it is hard to obtain the contrast of the specimen surface structure. The specimen surface is also easily charged up. The surface microstructures are clearly seen at (b).

Types of Electron 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.
- **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 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.

History

- TEM constructed in 1931
- Von Ardenne first STEM in 1938 by rastering the electron beam in a TEM
- Zworykin et al. 1942, first SEM for bulk samples
- 1965 first commercial SEM by Cambridge Scientific Instruments

Resolution at that time ~ 50 nm : Today < 1 nm

Morphology only at that time : Today analytical instrument

Electron Microscope vs. Optical Microscope

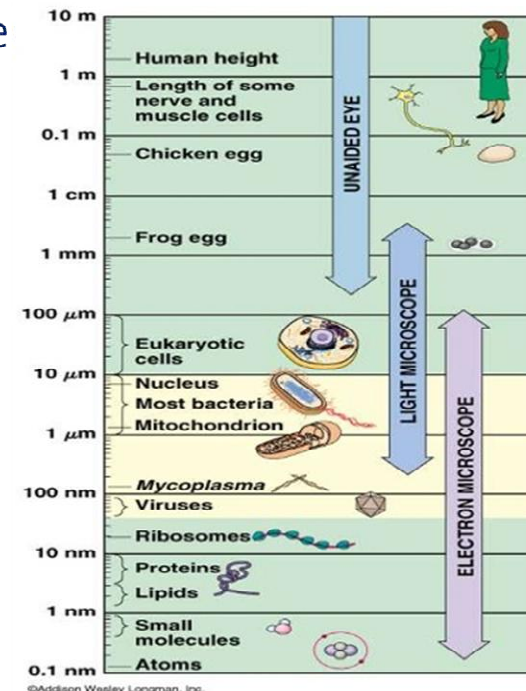
(first one built in 1931 by Ruska and Knoll) eeuwenhoek in 17th century)

Electron vs. Photon

Electron: charged, not visible.

Photon: neutral, visible at the wavelength

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



Electron Microscope vs. Optical Microscope

DIFFERENCES BETWEEN OM AND EM	
OPTICAL MICROSCOPE	ELECTRON MICROSCOPE
1. The source of light. 2. The specimen. 3. The lenses that makes the specimen seem bigger. 4. The magnified image of the specimen that you see.	1. The light source is replaced by a beam of very fast moving electrons. 2. The specimen usually has to be specially prepared and held inside a vacuum chamber from which the air has been pumped out (because electrons do not travel very far in air). 3. The lenses are replaced by a series of coil-shaped electromagnets through which the electron beam travels. 4. The image is formed as a photograph (called an electron micrograph) or as an image on a TV screen.

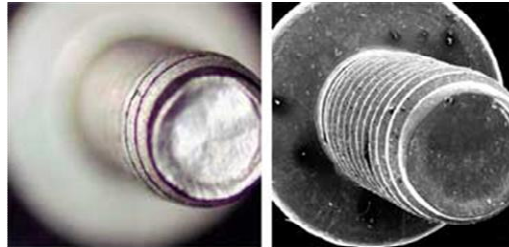
Comparison: Optical vs SEM vs TEM

Property	Optical	SEM	TEM
Resolution	~200 nm	~1–2 nm	<0.1 nm
Depth of field	Low 15.5 μm – 0.19 μm	Very high 4 mm – 0.4 μm	Very low
Sample thickness	Bulk	Bulk	<100 nm
Imaging	Surface	Surface	Internal
Magnification	4x – 1000x	10x – 3000 kx	

Depth of focus

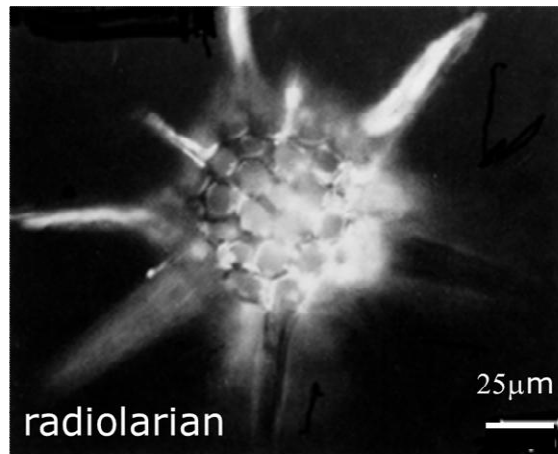
Simple Definition: Depth of field is the vertical distance (the "depth") within a sample that remains in acceptably sharp focus at the same time.

Optical microscopy vs SEM



- A SEM typically has *orders of magnitude better depth of focus* than a optical microscope making SEM suitable for studying rough surfaces.

Optical Microscopy vs SEM



OM

Small depth of field
Low resolution



SEM

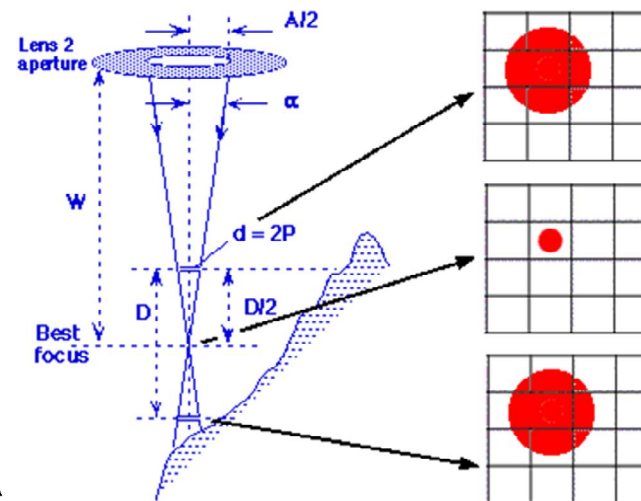
Large depth of field
High resolution

Depth of Field

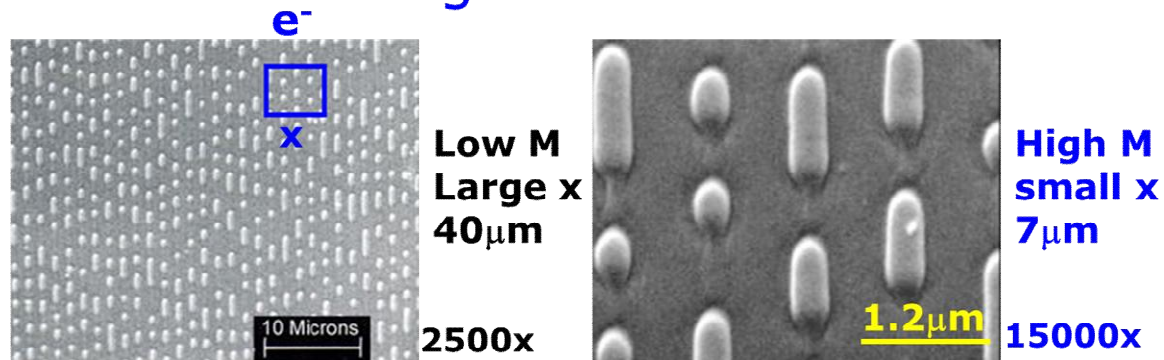
Depth of Field

$$D = \frac{4 \times 10^5 W}{AM} (\mu\text{m})$$

To increase **D**
 Decrease **aperture size**, A
 Decrease **magnification**, M
 Increase **working distance**, W (mm)



Magnification



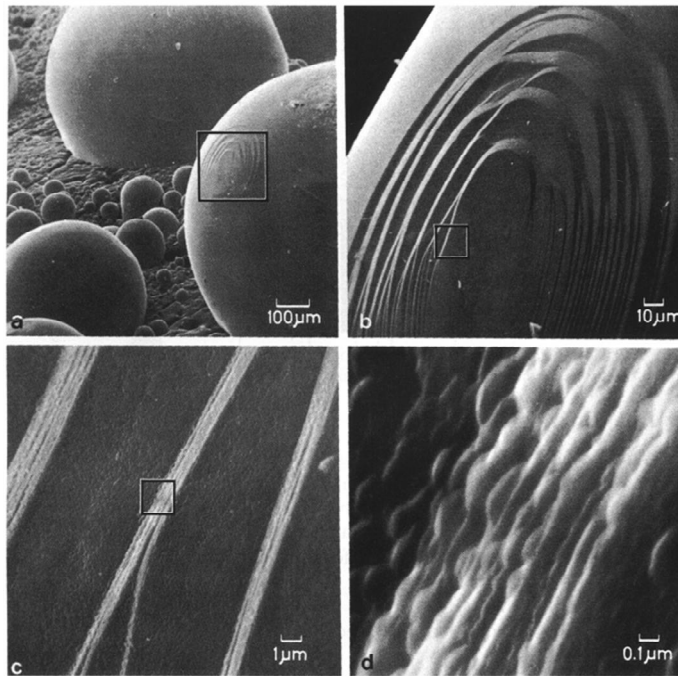
The magnification is simply the ratio of the length of the scan **C** on the Cathode Ray Tube (CRT) to the length of the scan **x** on the specimen. For a CRT screen that is 10 cm square:

$$M = C/x = 10\text{cm}/x$$

Increasing M is achieved by decreasing x.

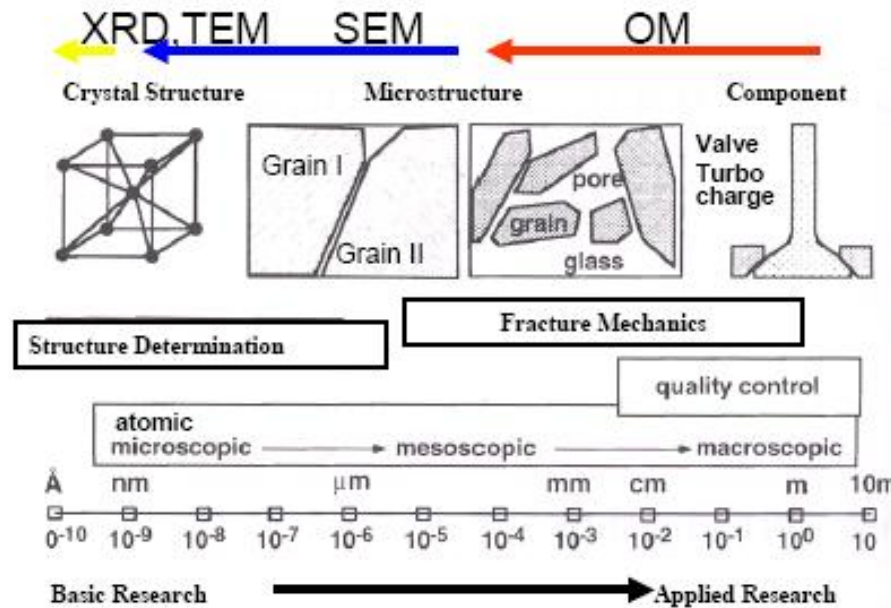
M	x	M	x
100	1 mm	10000	10 μm
1000	100 μm	100000	1 μm

Image Magnification



Example of a series of increasing magnification (spherical lead particles imaged in SE mode)

Scale and Microscopy Techniques



Microstructure ranging from crystal structure to engine components
(Si_3N_4)

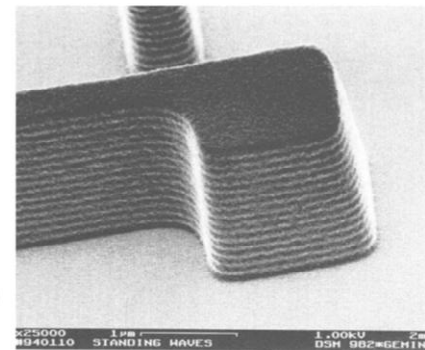
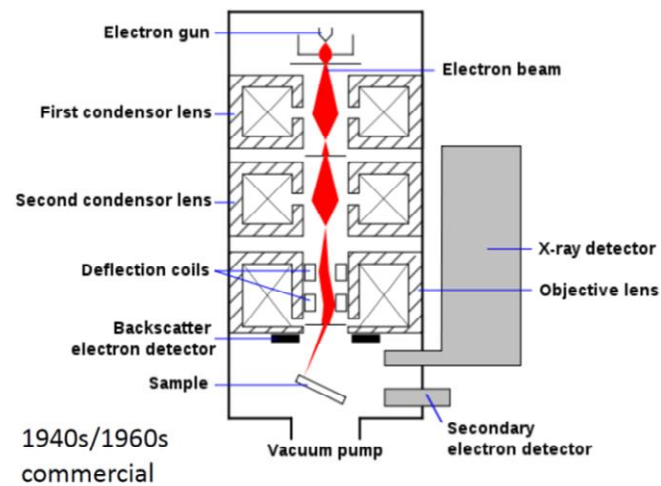
Scanning electron microscopy (SEM)

Section 2: Fundamentals of SEM

- Basis of SEM
- Secondary electrons (SE)
- Backscattered electrons (BSE)
- X-Ray
- Auger electrons
- Cathodo-luminescence (light)

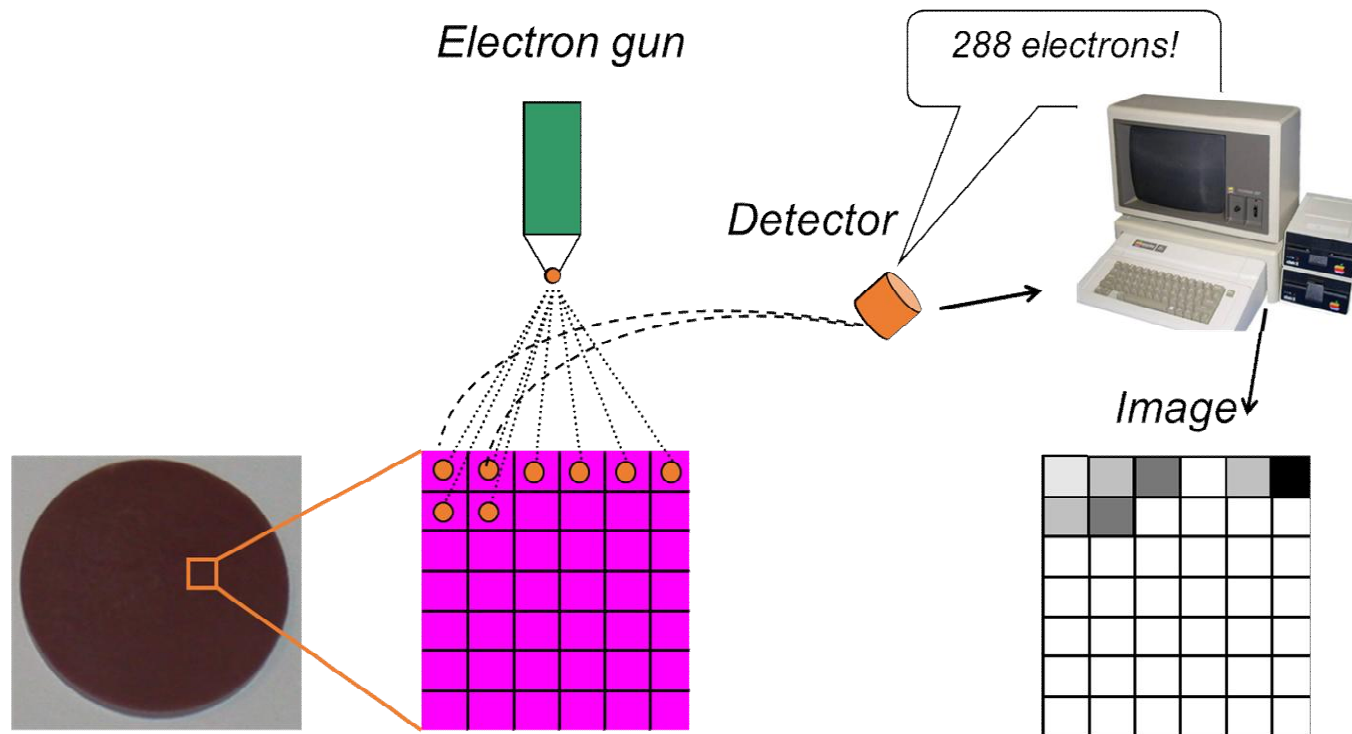
Scanning Electron Microscopy

It is a microscope that produces an image by using an electron beam that scans
the surface of a specimen inside a vacuum chamber.



Photoresist layer on Si.

How do we get an image?



The most versatile instrument for a materials scientist?

What can we study in a SEM?

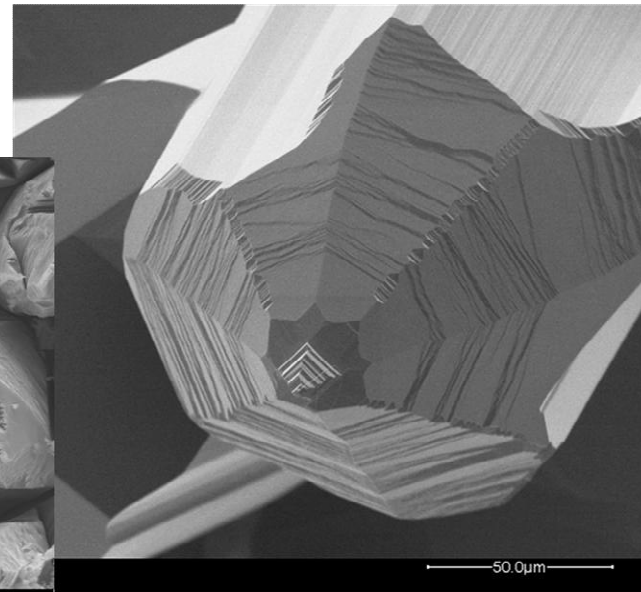
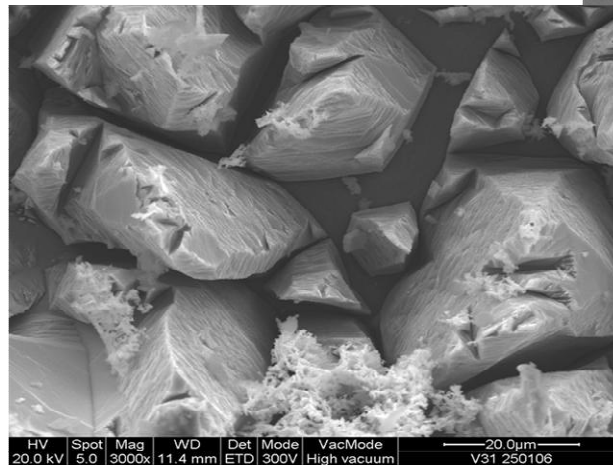
- Topography and morphology
- Chemistry or composition
- Crystallography
- Orientation of grains
- Electrical conductivity
- In-situ experiments:
 - Reactions with atmosphere
 - Effects of temperature

“Easy” sample preparation!!

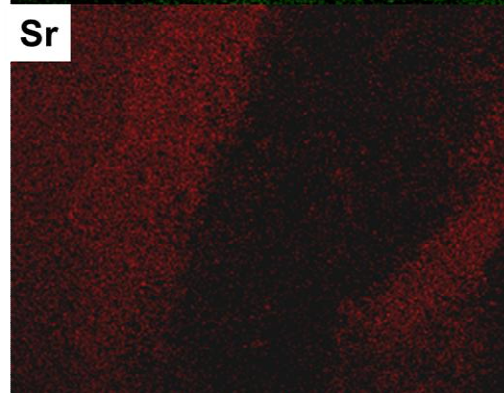
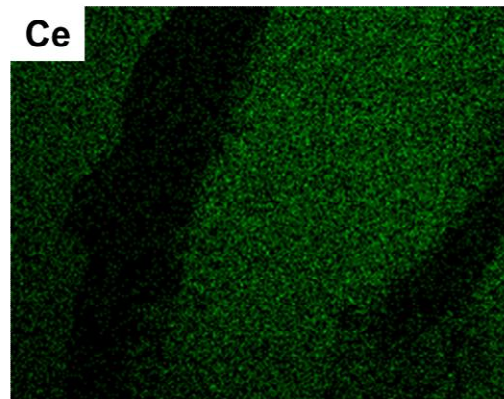
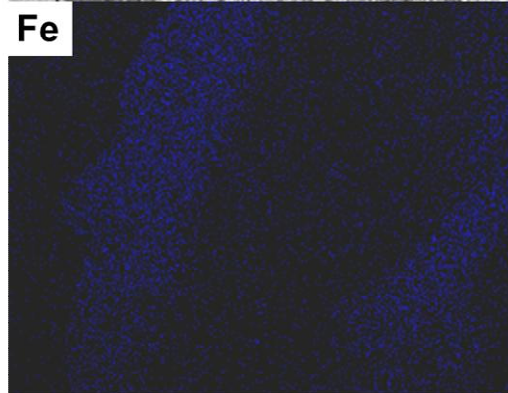
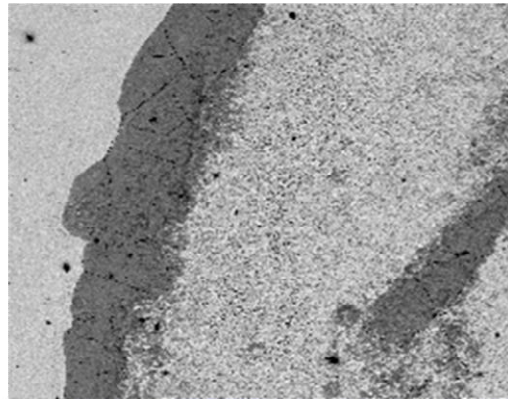
“Big” samples!

Topography and morphology

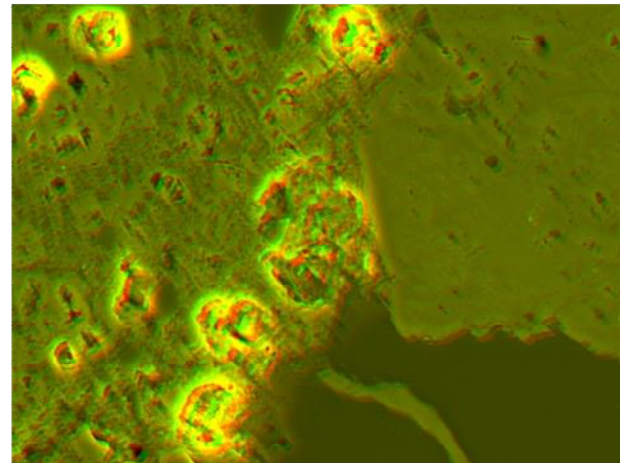
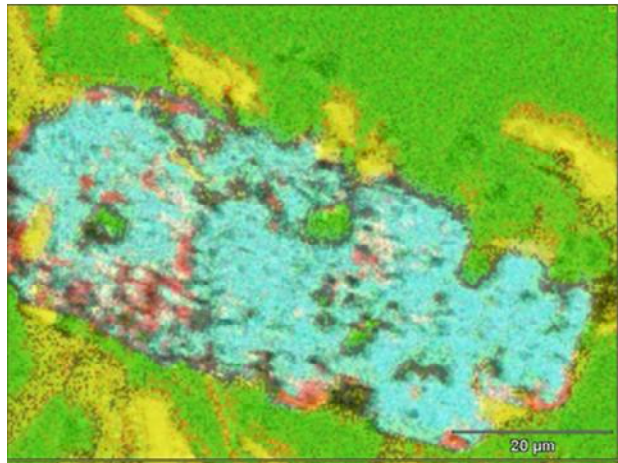
- High depth of focus



Chemistry



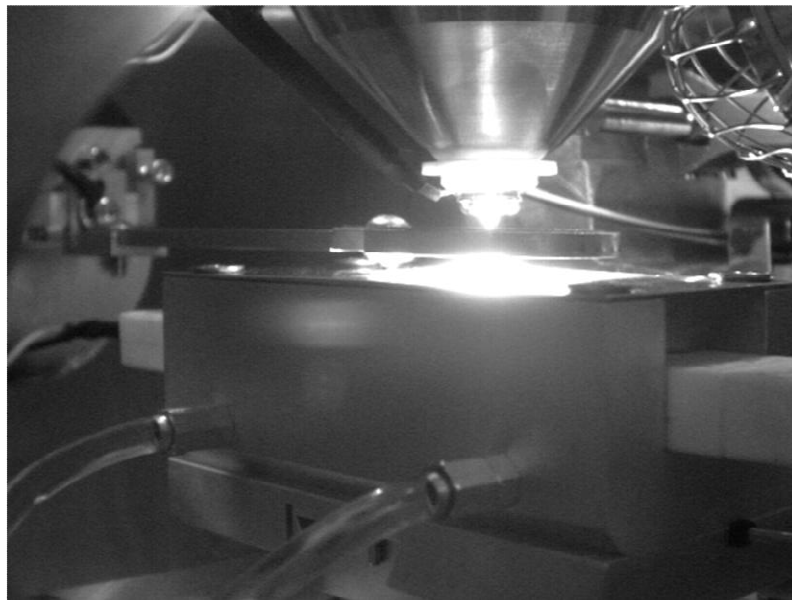
Chemistry



porous sandstone

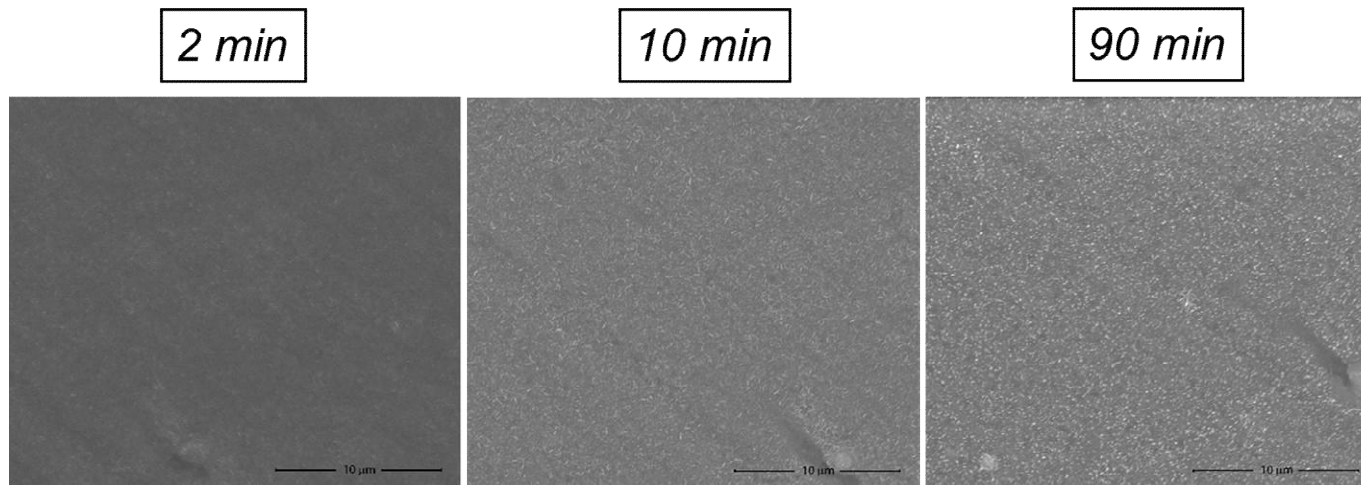
In-situ imaging

- A modern SEM can be equipped with various accessories, e.g. a hot stage

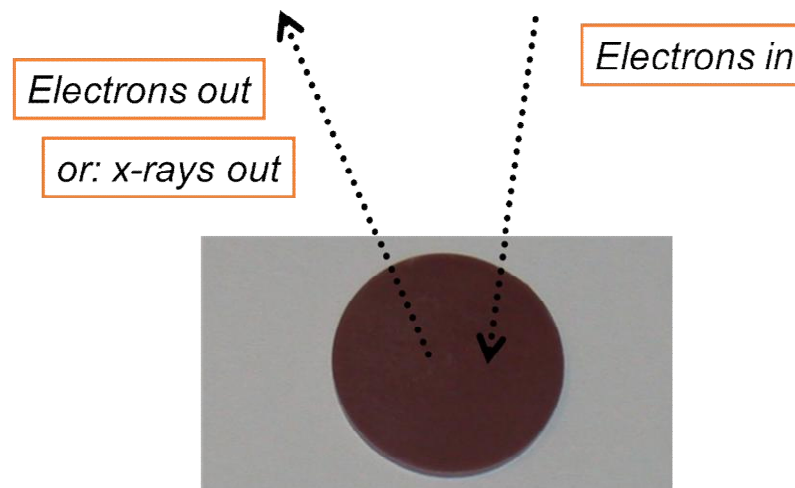


In-situ imaging: oxidation of steel at high temperatures

- 800 °C, $p_{\text{H}_2\text{O}} = 667 \text{ Pa}$
- Formation of Cr_2O_3

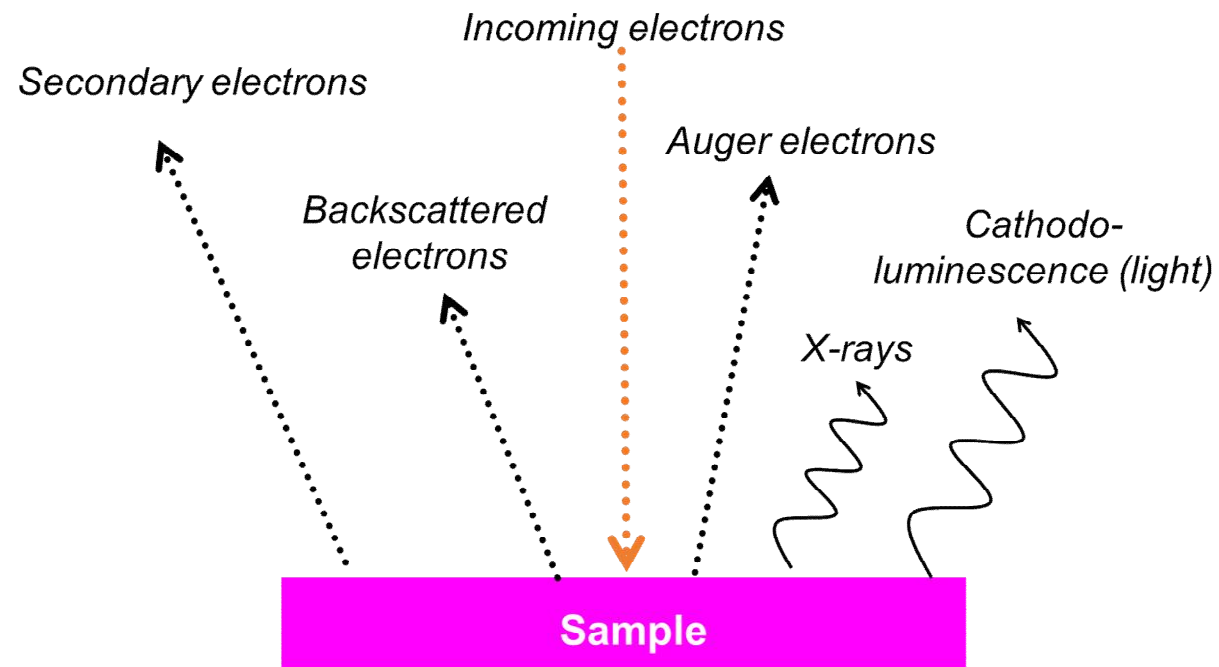


How do we get an image?



- In brief: we shoot high-energy electrons and analyze the outcoming electrons/x-rays

Signals from the sample



Signals from the sample

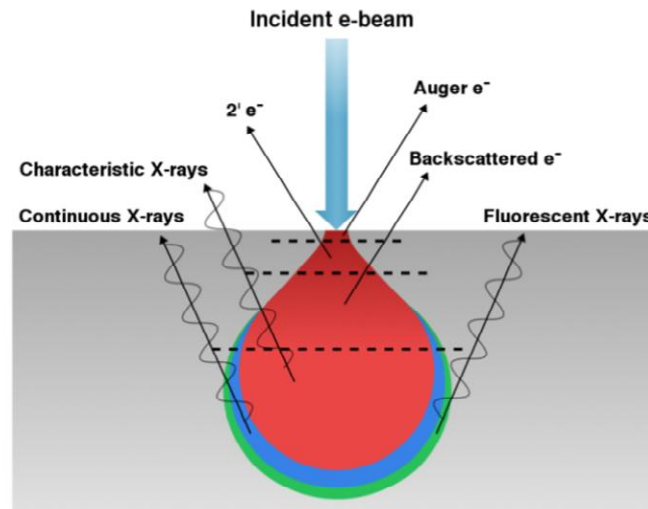
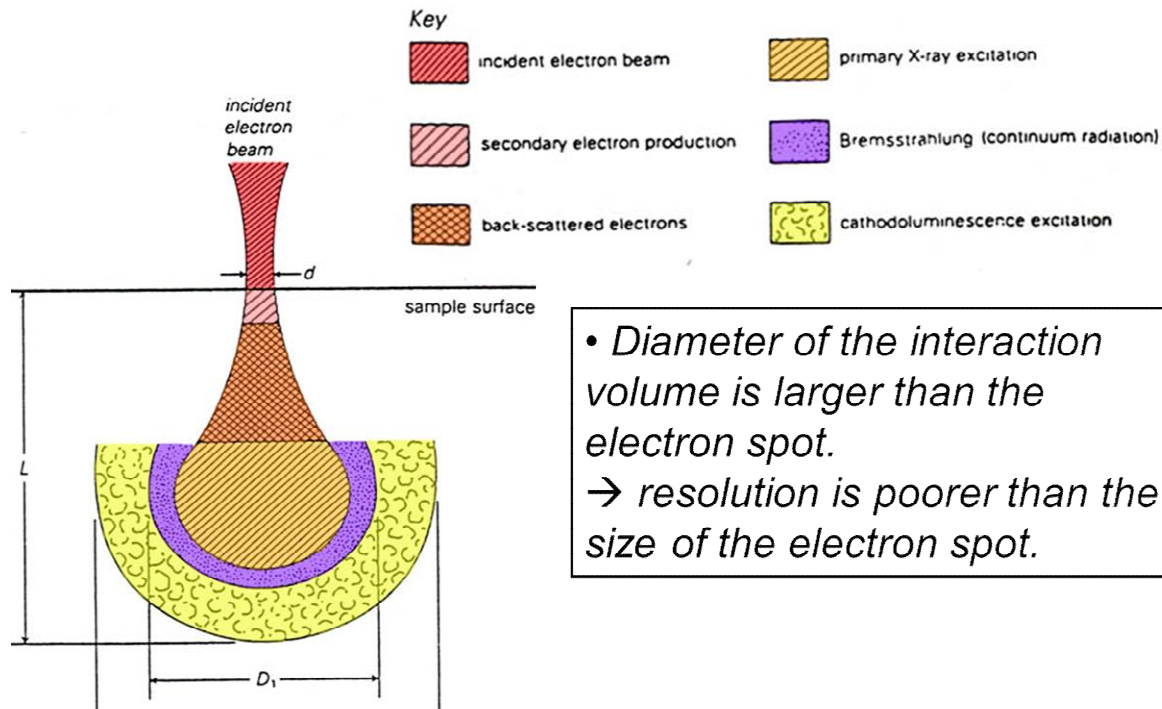


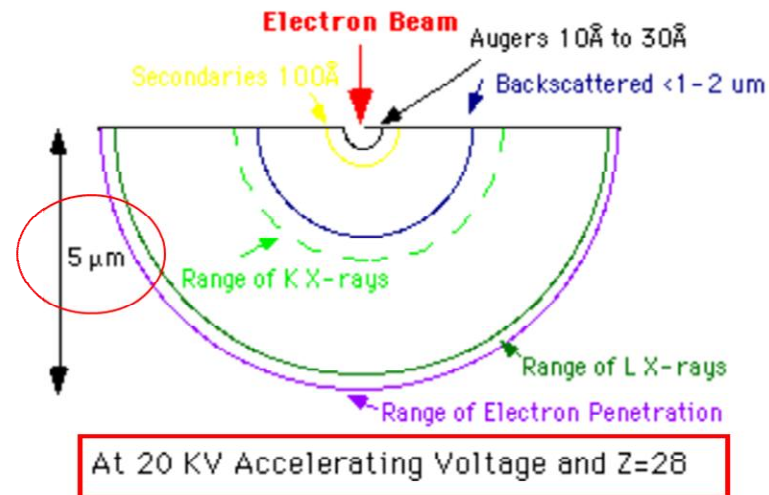
Figure 11. The interaction volume of an incident beam of electrons produces a variety of effects at different depths. SEM utilizes the secondary 2' electrons for surface imaging, whereas EDS utilizes characteristic X-rays for elemental analysis

Where does the signals come from?



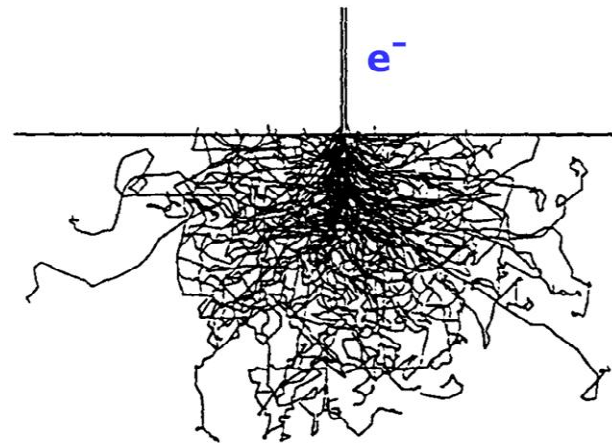
- Diameter of the interaction volume is larger than the electron spot.
→ resolution is poorer than the size of the electron spot.

Specimen interaction volume



- Atomic number of the material being examined; higher atomic number materials **absorb or stop more electrons** and so have a smaller interaction volume.
- Accelerating voltage being used; higher voltages penetrate farther into the sample and generate larger interaction volumes .
- Angle of incidence for the electron beam; the greater the angle (**further from normal**) the smaller the volume.

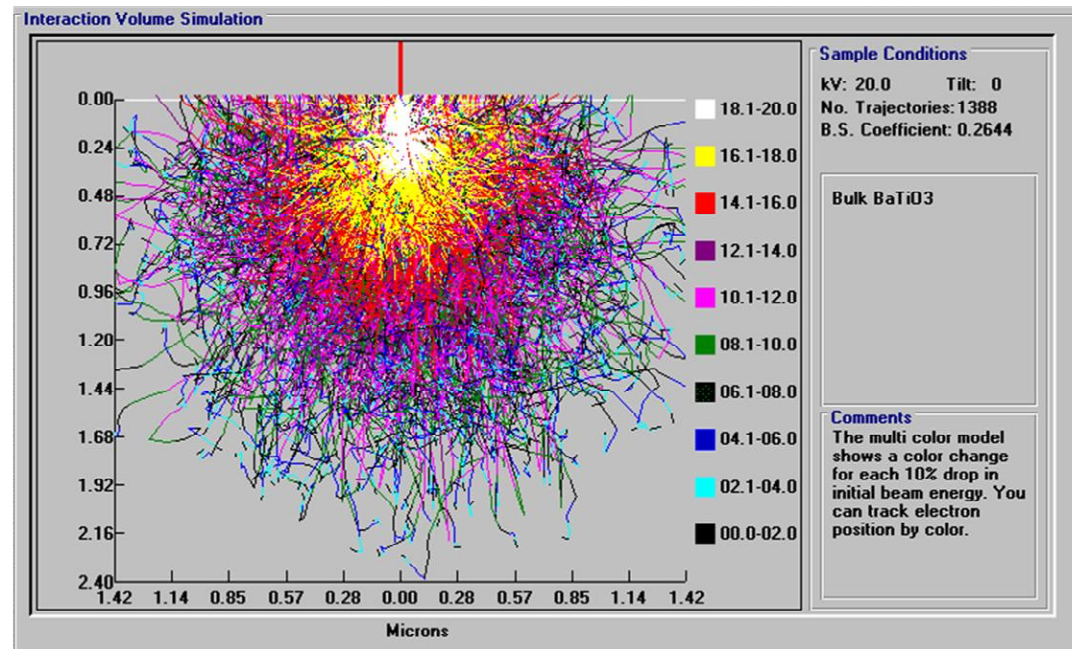
Interaction Volume



Monte Carlo simulations of 100 electron trajectories

The incident electrons do not go along a straight line in the specimen, but a zig-zag path instead.

Monte Carlo simulation of the interaction volume

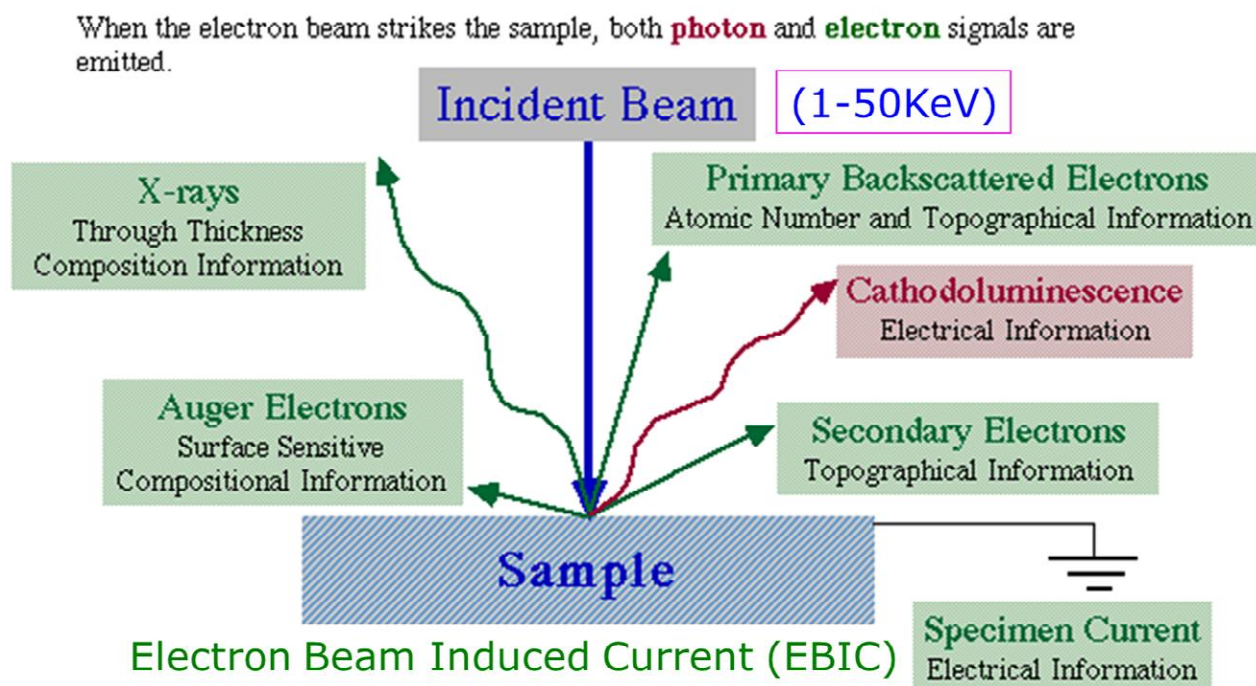


Try the online simulation at:

http://www.matter.org.uk/tem/electron_scattering.htm

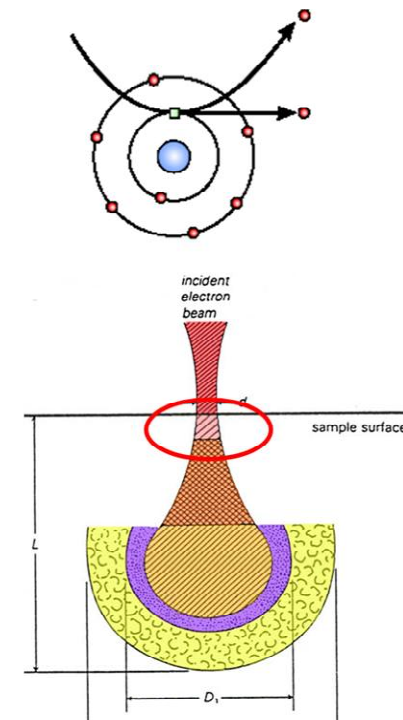
Electron Beam and Specimen Interactions

Sources of Image Information



Secondary electrons (SE)

- Generated from the **inelastic collision** between the incoming electrons and the loosely bonded **outer (valence) electrons**.
- **Low** energy electrons ($\sim 10\text{-}50\text{ eV}$)
- Only SE generated close to surface escape (topographic information is obtained)
- Number of SE is greater than the number of incoming electrons (Each incident electron can produce several secondary electrons).
- We differentiate between SE1 and SE2 based on their origin.

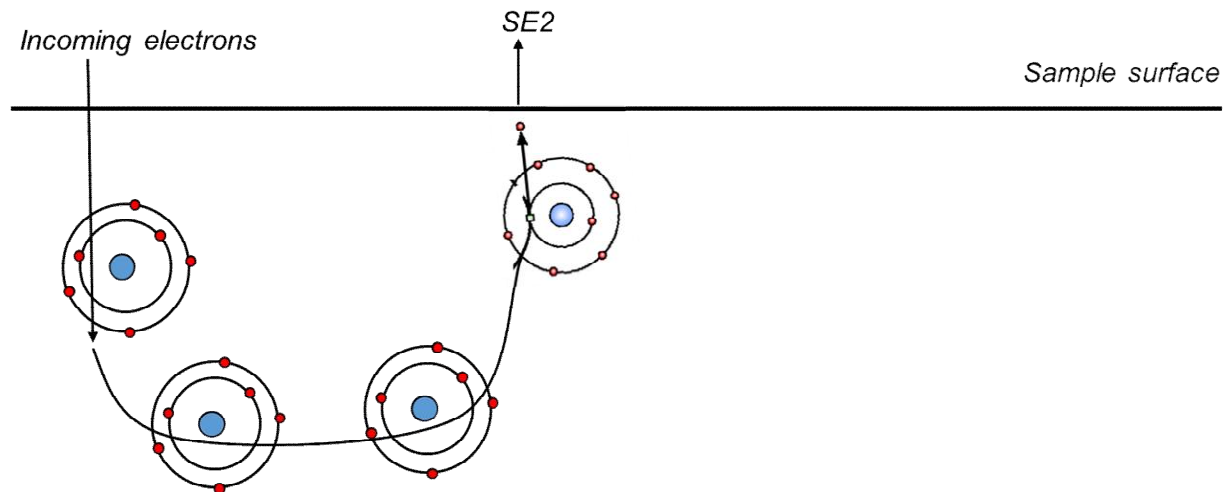


SE1

- These electrons are generated by the direct interaction of the **incoming primary electron beam** with the sample surface. That is, *at the very moment the beam electrons hit the sample*, secondary electrons are ejected from its surface.
- The signal from these electrons has **very high resolution**. The reason is that *the region where these electrons are generated is approximately the same size as the diameter of the primary electron beam (which is very narrow)*. *Therefore, the image resolution is limited only by the diameter of this beam.*

SE2

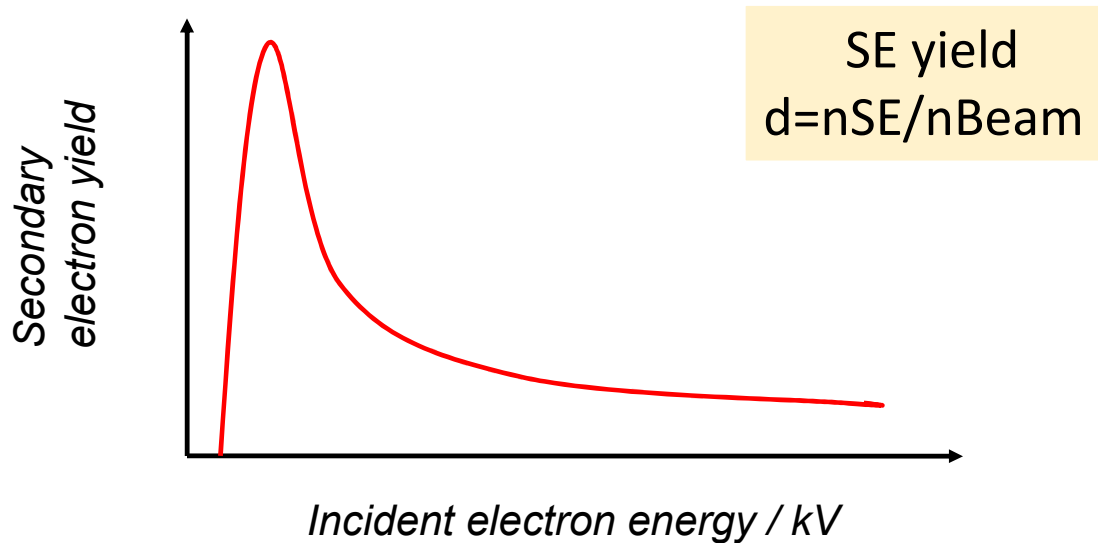
- These electrons are produced indirectly. First, the primary beam electrons, after interacting with the sample, undergo processes like **elastic scattering** and become **Backscattered Electrons (BSE)**. These backscattered electrons, **after traveling a certain path within the sample**, return to the surface. At the moment of exiting the surface, these high-energy electrons generate secondary electrons.
- SE2 come from a surface area that is bigger than the spot from the incoming electrons → resolution is poorer than for SE1 exclusively.



Factors that affect SE emission

1. Beam energy and beam current

- Electron yield goes through a maximum at low acc. voltage, then decreases with increasing acc. voltage



2. Work function of the surface

Work function

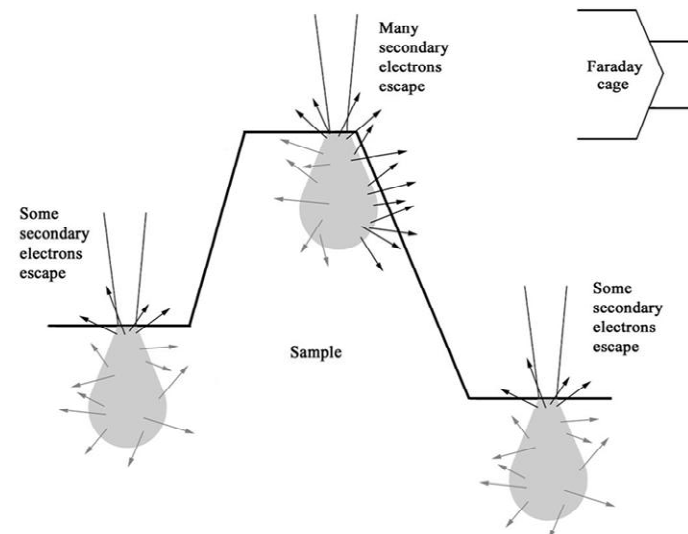
- The **work function** (symbolized by ϕ or W) is the **minimum thermodynamic work (or energy) needed to remove an electron from a solid (usually a metal) to a point in the vacuum immediately outside the solid's surface.**
- **Where does this energy barrier come from?**

It's a combination of two things:

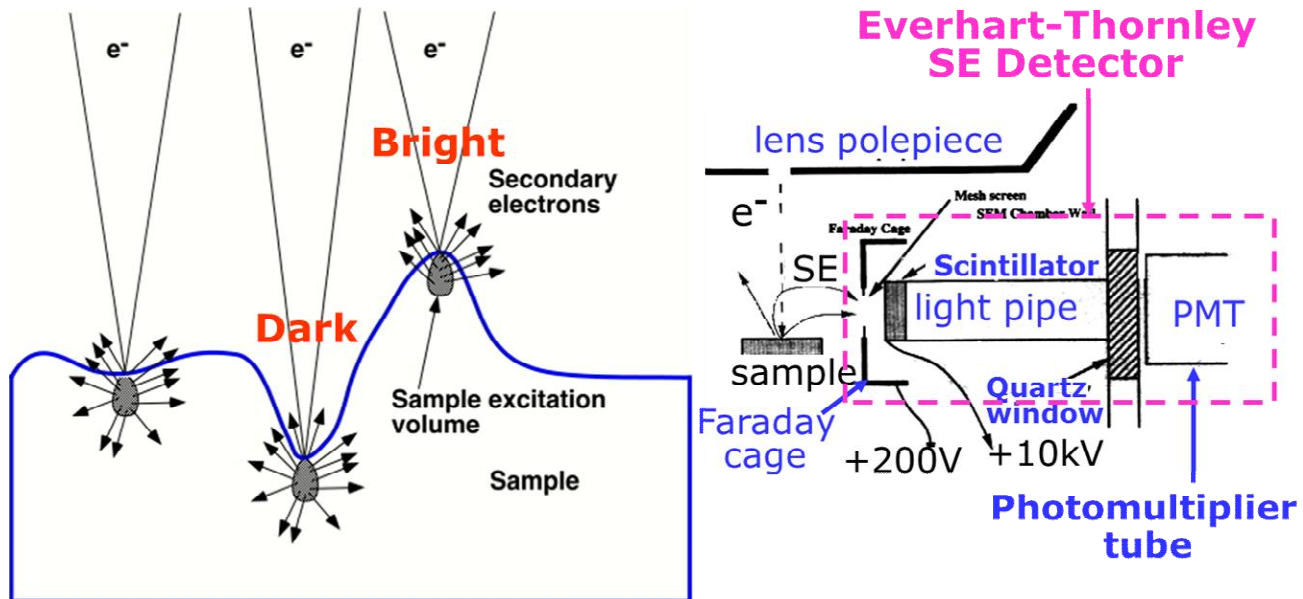
- The electron is negatively charged and is attracted to the positive charges (atomic nuclei) inside the material.
- When an electron tries to leave, it induces a positive "image charge" on the surface, which pulls it back.

Factors that affect SE emission

3. Atomic number (Z)
 - More SE2 are created with increasing Z.
 - The Z-dependence is more pronounced at **lower beam energies**.
4. The local curvature of the surface (the most important factor)

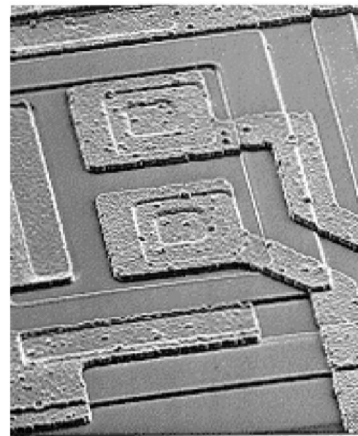


Topographical Contrast



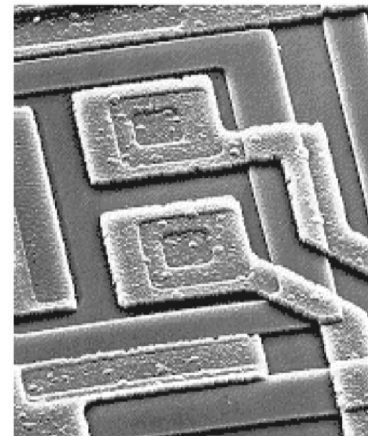
Topographic contrast arises because SE generation depend on the angle of incidence between the beam and sample.

Effect of Surface Topography on Observed Image



5 kV 720 X

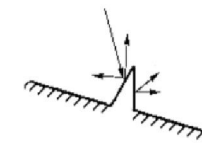
Tilt Angle = 50°



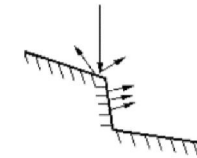
25 kV 720 X

Tilt Angle = 50°

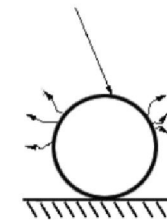
Electron penetration is increased
at higher beam energy



(a) Protrusion

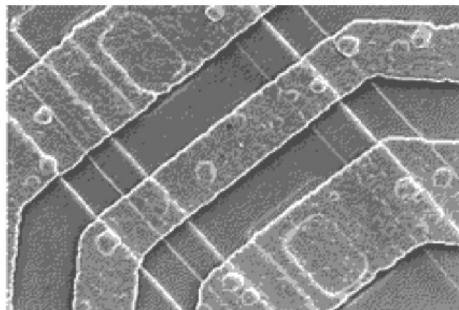


(b) Edge

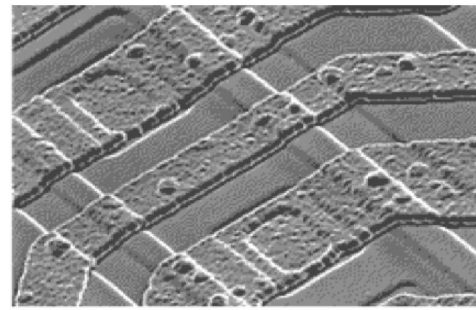


(c) Circumference

Effect of Sample Orientation on Observed Image



Tilt Angle = 0°



Tilt Angle = 45°

IC Chip

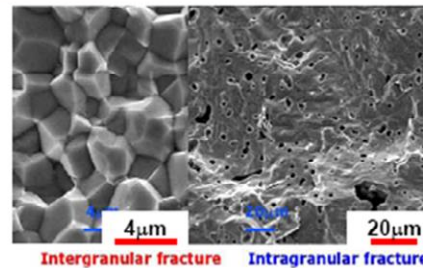
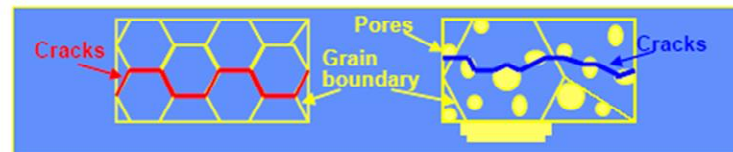
5 kV and 1,100 X Magnification

The sides of patterns are viewed by tilting the specimen. The amount of secondary electron electrons that are collected is increased.

Application: High resolution image setup

- By placing the secondary electron detector *inside* the lens, mainly SE1 are detected
 - Resolution of 1 – 2 nm is possible

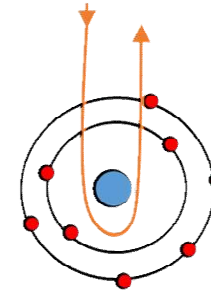
Identification of Fracture Mode



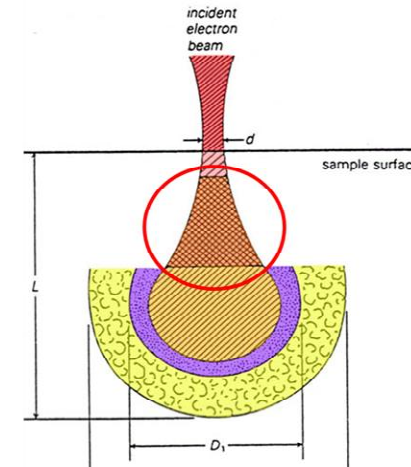
SEM micrographs of fractured surface of two BaTiO₃ samples.

Backscattered electrons (BSE)

A fraction of the incident electrons is retarded by the electro-magnetic field of the nucleus and if the scattering angle is greater than 180° the electron can escape from the surface.



- High energy electrons (elastic scattering)
- We differentiate between BSE1 and BSE2



BSE1

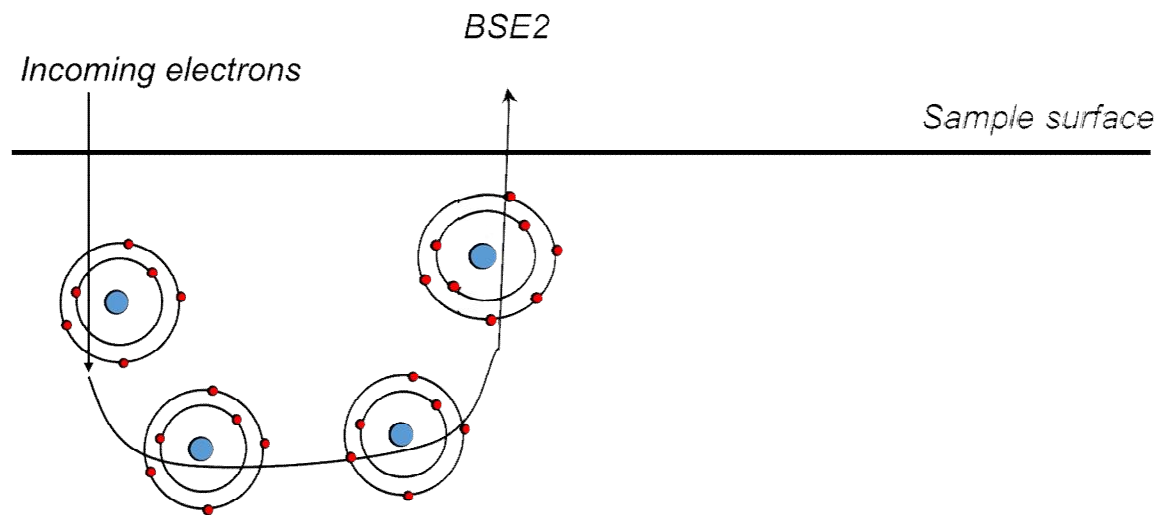
A BSE1 electron enters the sample and undergoes a **single, large-angle elastic scattering event** (close to the nucleus of an atom) that redirects it back toward the surface, allowing it to escape.

They tend to escape at a high angle relative to the sample surface. Since they come from **a very narrow region directly under the beam**, BSE1 carry **high-resolution information**.

BSE2

A BSE2 electron undergoes **multiple scattering events** within the sample.

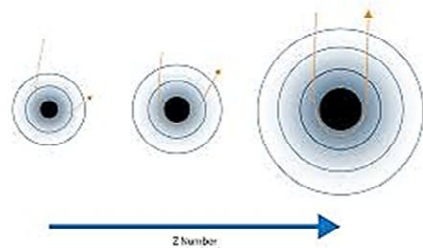
- *Most BSE are of BSE2 type*



Factors that affect BSE emission

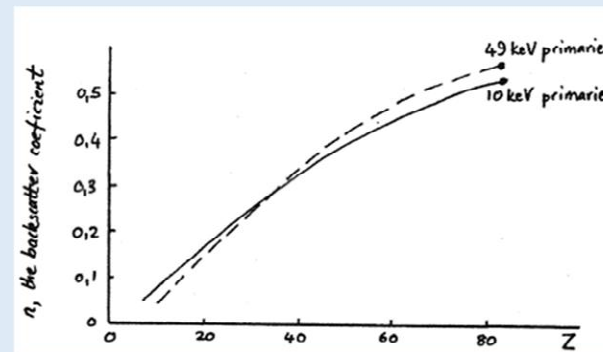
- Direction of the irradiated surface
 - More electrons will hit the BSE detector when the surface is aligned towards the BSE detector
- Average atomic number

$$\eta = \frac{I_{BSE}}{I_P}$$



BSE as a function of atomic number

- For phases containing more than one element, it is the average atomic number that determines the backscatter coefficient η

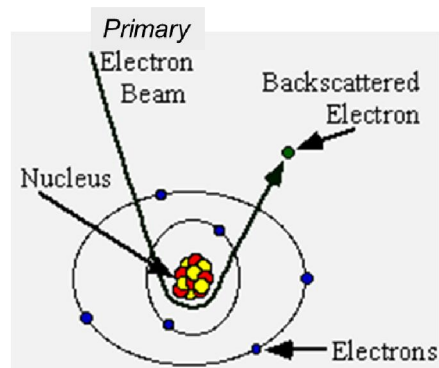


The Backscattering Coefficient (η)

The following table illustrates this relationship for a few common elements:

Element	Atomic Number (Z)	Approx. Backscattering Coefficient (η)	BSE Image Brightness
Carbon (C)	6	0.05 - 0.06	Dark
Aluminum (Al)	13	~0.15	Dark Gray
Copper (Cu)	29	~0.30	Light Gray
Silver (Ag)	47	~0.40	Bright
Gold (Au)	79	~0.50	Very Bright

Backscattered Electrons (BSE)



BSE image from flat surface of an Al ($Z=13$) and Cu ($Z=29$) alloy

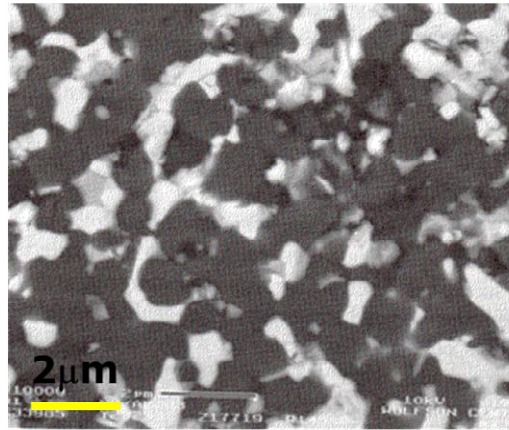
BSE are produced by **elastic interactions** of beam electrons with nuclei of atoms in the specimen and they have **high energy** and **large escape depth**.

BSE yield: $\eta = n_{BS}/n_B \sim$ function of atomic number, **Z**

BSE images show characteristics of **atomic number contrast**, i.e., high average Z appear brighter than those of low average Z. η **increases with tilt**.

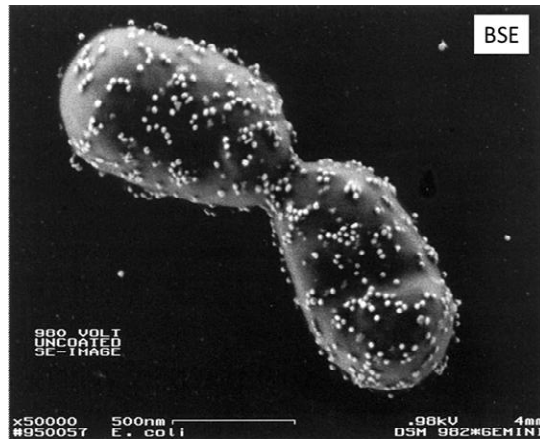
BSE Image – Atomic Number Contrast

$$\eta \approx -0.0254 + 0.016Z - 0.000186Z^2$$

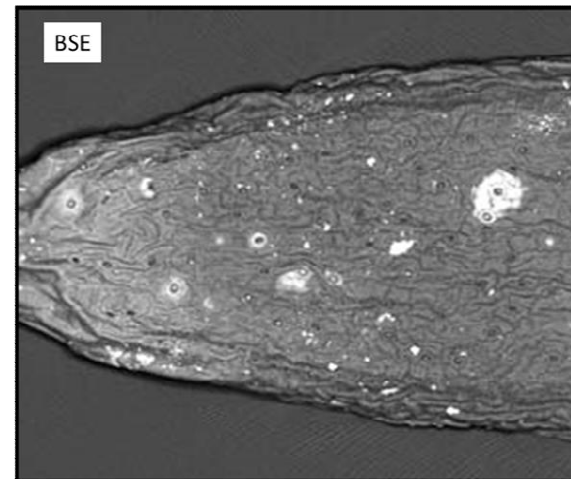


BSE atomic number contrast image showing a niobium-rich intermetallic phase (bright contrast) dispersed in an alumina matrix (dark contrast).

$Z(\text{Nb}) = 41$, $Z(\text{Al}) = 13$ and $Z(\text{O}) = 8$
Alumina- Al_2O_3



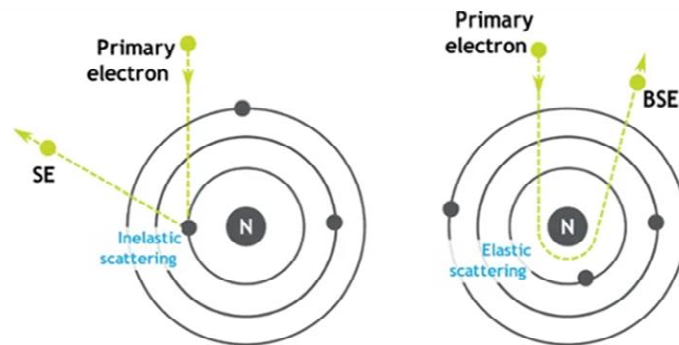
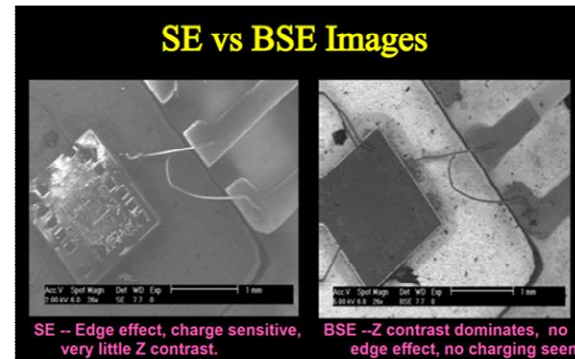
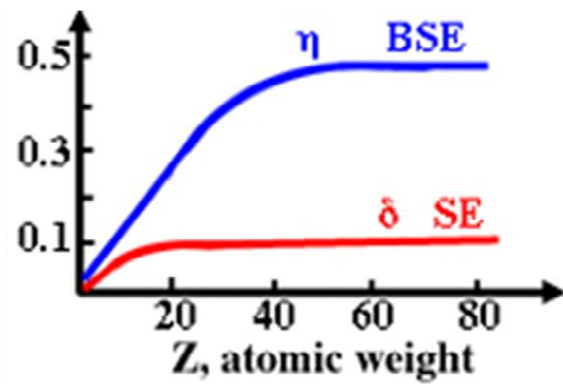
Gold particles on *E. coli* appear as bright white dots due to the higher percentage of backscattered electrons compared to the low atomic weight elements in the specimen.



Backscatter image of Nickel in a leaf

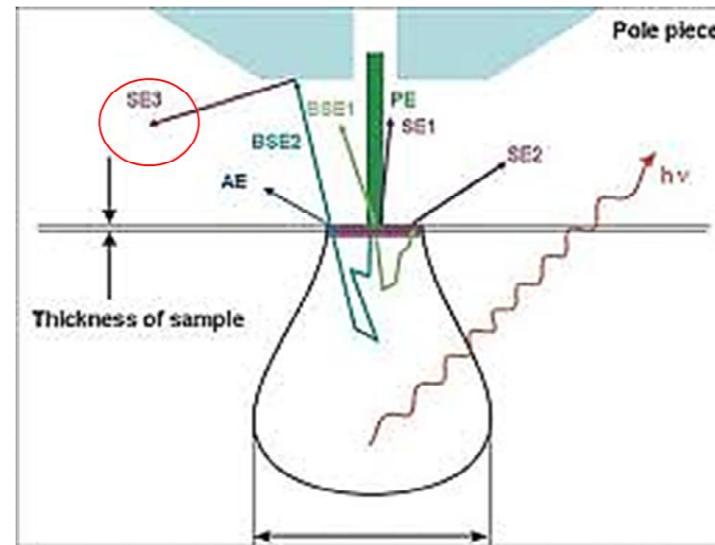
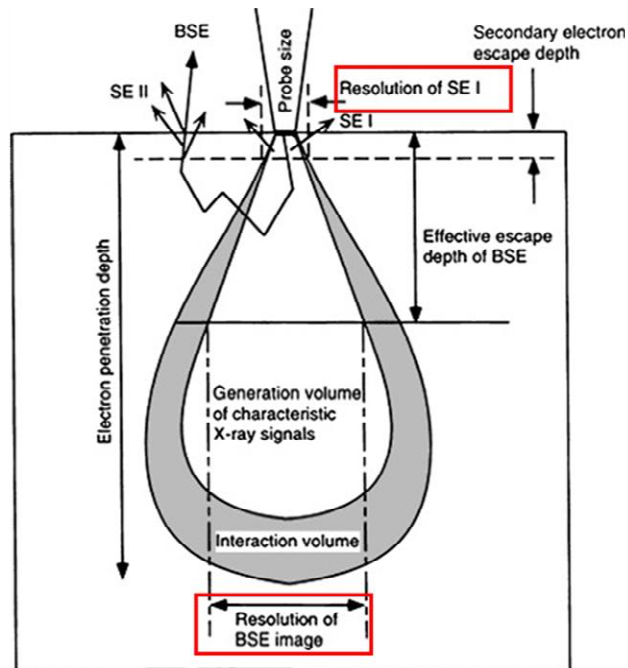
BSE vs SE

Effect of Atomic Number, Z, on BSE and SE Yield



BSE vs SE

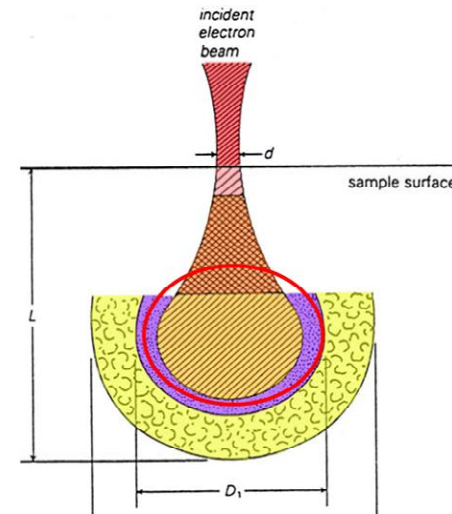
Best resolution that can be obtained: size of the electron spot on the sample surface.



The resolution of SE mode normally is higher than that of BSE.

X-rays

- *Photons* not electrons
- Each element has a *fingerprint* X-ray signal
- Poorer resolution than BSE and SE
- Relatively few X-ray signals are emitted and the detector is inefficient
 - relatively long signal collecting times are needed



Characteristic X-rays

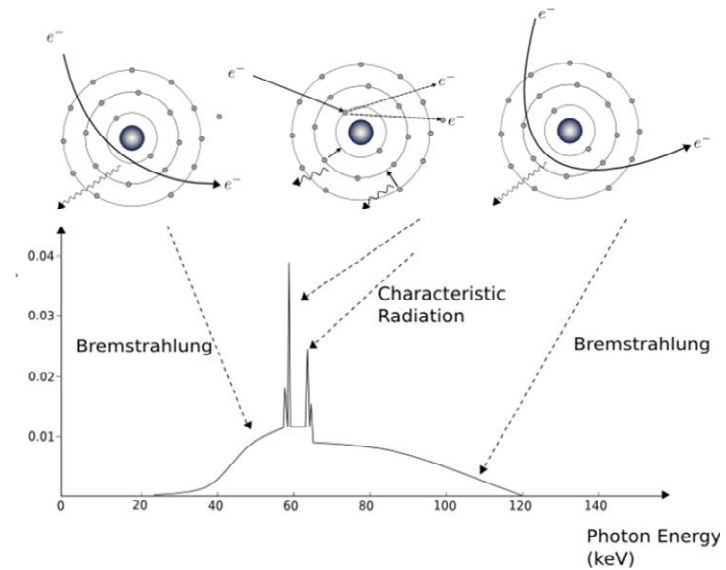
- 1.The Incident Electron:** A high-energy electron from the beam strikes an atom in the sample.
- 2.Ejection (Ionization):** If the beam electron **has enough energy**, it can knock out an electron from an *inner* shell (e.g., the K-shell), creating a vacancy (a "hole"). The atom is now in an excited, ionized state and is unstable.
- 3.Relaxation (De-excitation):** To return to a stable, low-energy state, an electron from a *higher energy shell* (e.g., the L-shell or M-shell) "falls down" to fill the vacancy in the lower K-shell.
- 4.Energy Release:** When the outer electron falls to the inner shell, *it loses a specific amount of energy. This energy is released in the form of an X-ray photon.*

Continuous X-rays (**Bremsstrahlung**)

- 1.The Incident Electron:** A high-energy electron from the beam travels through the sample.
- 2.Coulomb Attraction:** As it passes close to the positively charged nucleus of an atom, **the electron is attracted** and its path **is bent by the strong electric field** (Coulomb field) of the nucleus. **It decelerates and changes direction.**
- 3.Energy Loss (The "Brake"):** When a charged particle decelerates or changes direction, **it must lose energy**. The law of conservation of energy dictates that this lost energy must go somewhere.
- 4.Photon Emission:** The energy lost by the electron **is emitted as a new photon**: an X-ray.

Characteristic X-rays and Continuous X-rays

Feature	Characteristic X-rays	Continuous X-rays (Bremsstrahlung)
Origin	Inner shell ionization (electron ejected)	Deceleration of electron in nuclear field
Mechanism	Electron transition between shells	"Braking" of the electron
Energy	Discrete (specific peaks)	Continuous (smooth background)
Dependence	Specific to the element (Z)	Depends on beam energy (E_0)
Usefulness	Useful - Used for elemental ID and quantification	Noise - Contributes to background, reduces peak-to-background ratio
Spectrum	Sharp Peaks	Curved Background



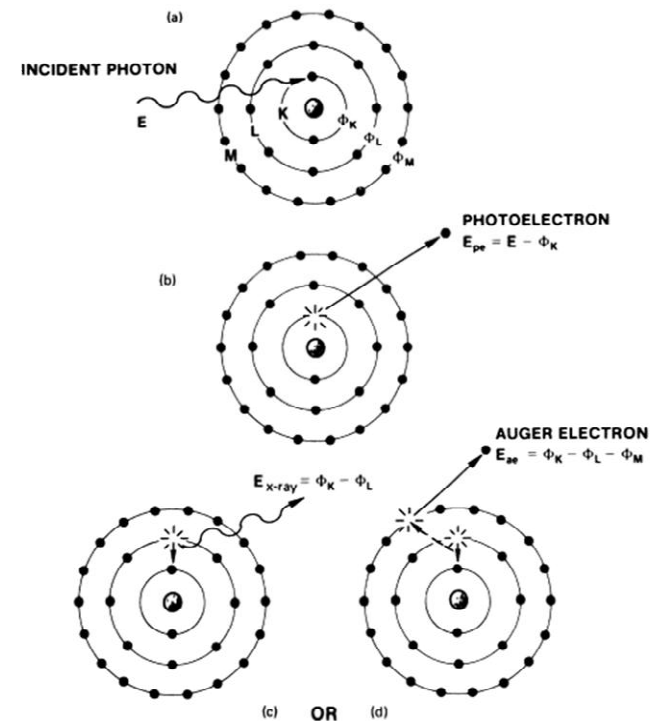
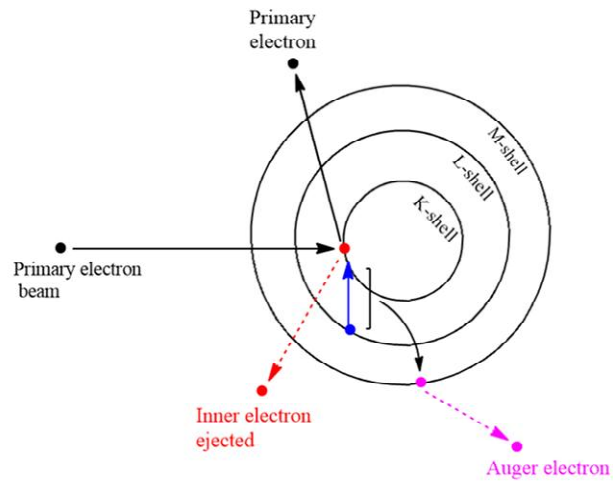
Application of X-rays

- No data of topography
- Most common spectrometer: EDS (energy-dispersive spectrometer)
- Signal overlap *can* be a problem
- We can analyze our sample in different modes
 - spot analysis
 - line scan
 - chemical concentration map (elemental mapping)

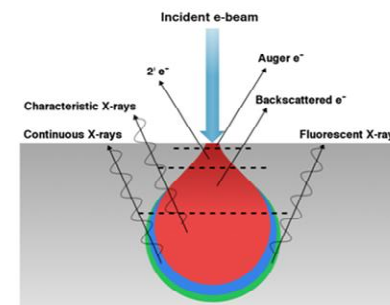
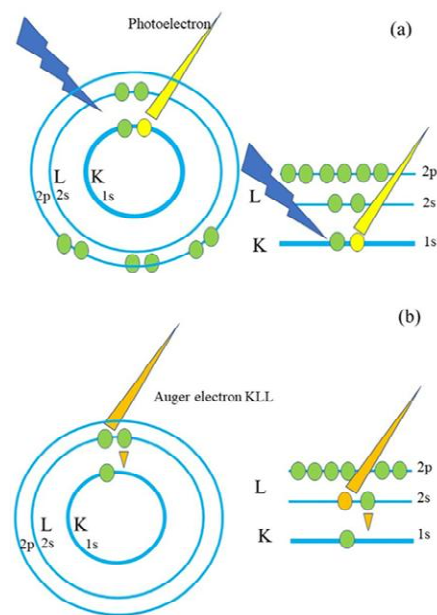
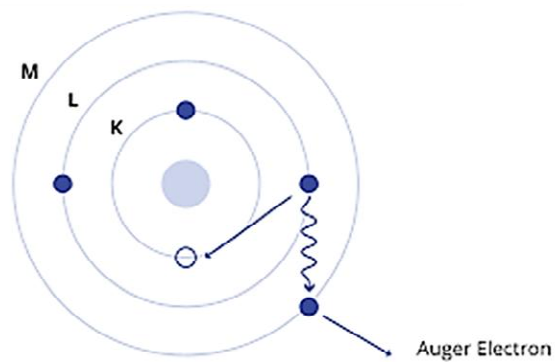
The Auger electron

- The Auger effect is a **three-step** process that **begins exactly** the same way as characteristic X-ray generation.
- **Ionization (The Initiation):**
 - A high-energy electron from the beam strikes an atom and ejects an electron from an **inner shell** (e.g., the K-shell), creating a core hole. The atom is now in an excited, **ionized state**.
- **Relaxation (The Filling):**
 - An electron from a **higher energy shell** (e.g., the L_1 shell) falls down to fill the vacancy in the K-shell.
- **Energy Transfer & Emission (The Auger Electron):**
 - Here is where it differs from X-ray generation. The energy released by the electron falling from L_1 to K is **not** released as an X-ray photon. Instead, this energy is simultaneously transferred to **another electron** in the same atom (e.g., in **the L_2 or L_3 shell**).
 - This second electron uses that transferred energy to overcome its binding energy and is ejected from the atom.
 - This ejected electron is called an **Auger electron**.

The Auger electron



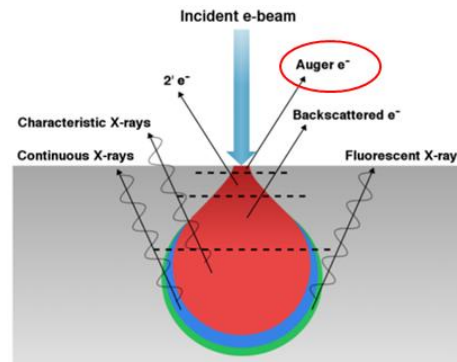
The Auger electron



The Auger electron

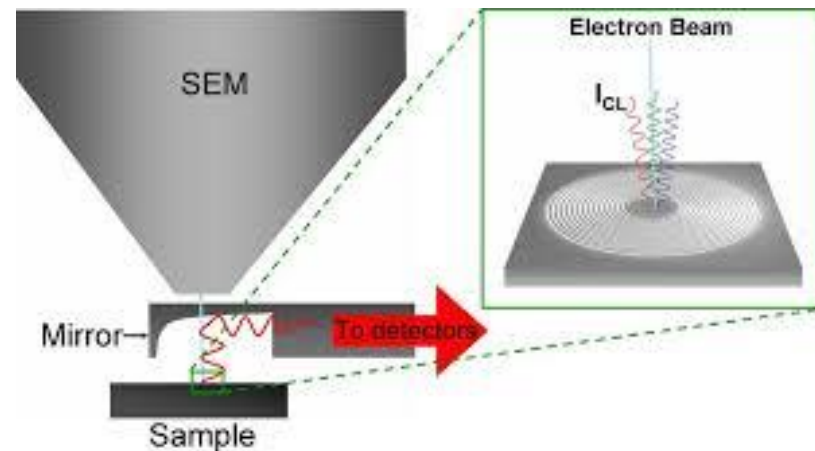
Utilization

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.



Cathodoluminescence (CL)

CL is the emission of **photons of light** (ultraviolet, visible, or infrared) from a material (semiconductor or insulator) when it is excited by an electron beam.



Cathodoluminescence (CL)

The energy (wavelength/color) of this light is not random. It is governed by the equation:

$$\lambda = \frac{hc}{E}$$

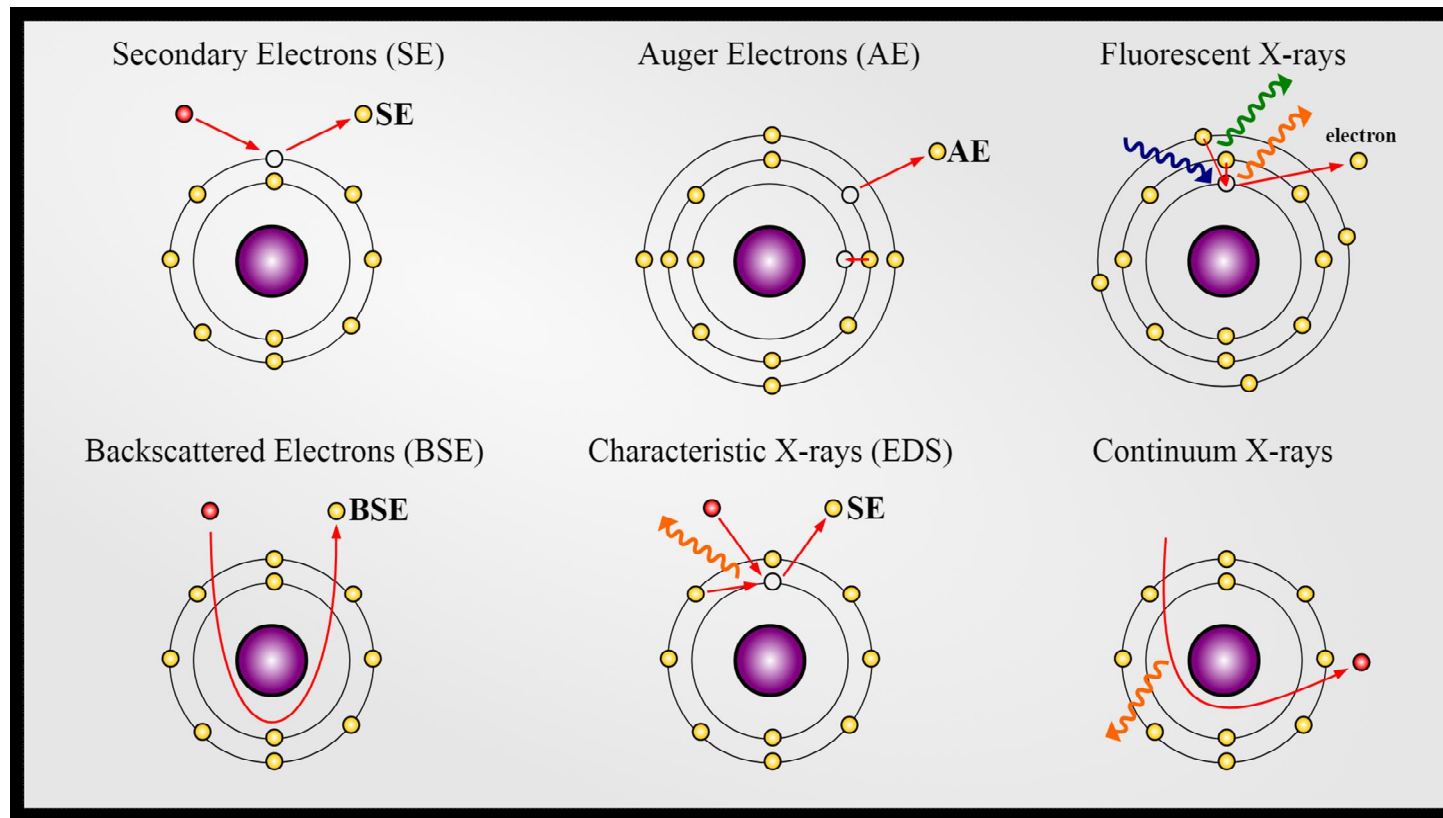
Where **λ** is the wavelength, **h** is Planck's constant, **c** is the speed of light, and **E** is the energy of the transition. Therefore, by measuring the wavelength of the emitted light with a spectrometer, we can directly probe the energy levels within the material. This is called **Spectroscopic CL** or **Hyperspectral CL**.

Cathodoluminescence (CL)

Utilization

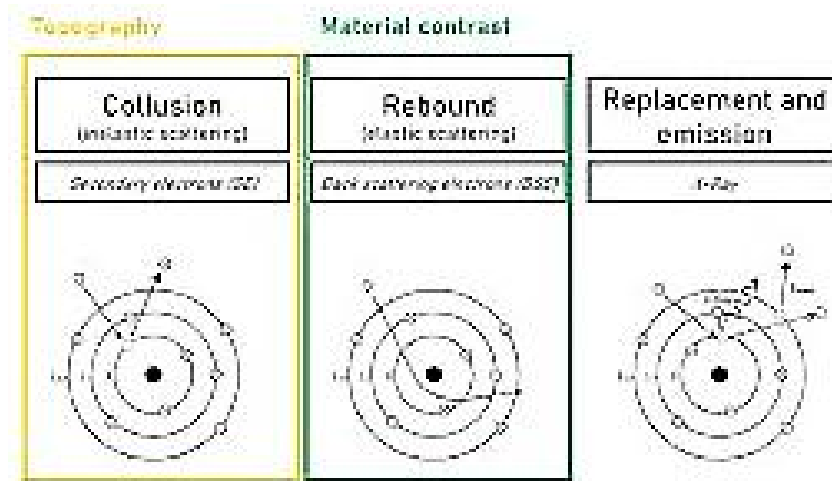
Light emitted from the atom will have a characteristic energy which is unique to the element from which it originated.

1. Phase Identification (different materials, different colors)
2. Trace element mapping (the "fingerprint" of activators)
3. Stress and strain mapping (piezospectroscopy)
4. Band gap measurement (compositional mapping in semiconductors)



SEM Signal Types

Signal	Energy	Depth	Information
Secondary electrons (SE)	<50 eV	1–5 nm	Topography
Backscattered electrons (BSE)	High	50–500 nm	Z-contrast
X-rays (EDS)	keV	μm	Composition



Secondary Electrons (SE)

Properties:

- Low energy (<50 eV)
- Escape depth: 1–5 nm

Provide:

- Surface morphology
 - High-resolution topography
- ☞ Main imaging mode in SEM

Backscattered Electrons (BSE)

Properties:

- High energy
- Strong Z-dependence

Contrast:

- High Z → bright
 - Low Z → dark
- ☞ Used for **phase identification & compositional contrast**

X-ray Generation (EDS)

Mechanism:

- Inner shell ionization
- Characteristic X-ray emission

Applications:

- Elemental identification
- Chemical mapping
- Quantitative microanalysis

TEM: Transmitted Electron Signals

Transmitted electrons:

- Bright-field imaging
- Dark-field imaging
- Diffraction

Information:

- Crystal structure
- Defects
- Interfaces
- Atomic positions

Section 3:

SEM Instrumentation & Imaging Physics

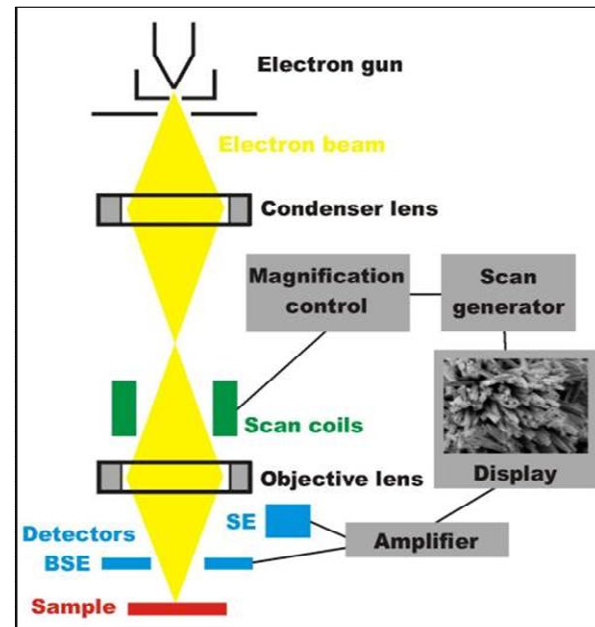
- **The architecture of an SEM column**
- **electron gun operation principles**
- **electron optics and beam control**
- **signal detection**

SEM Column Overview

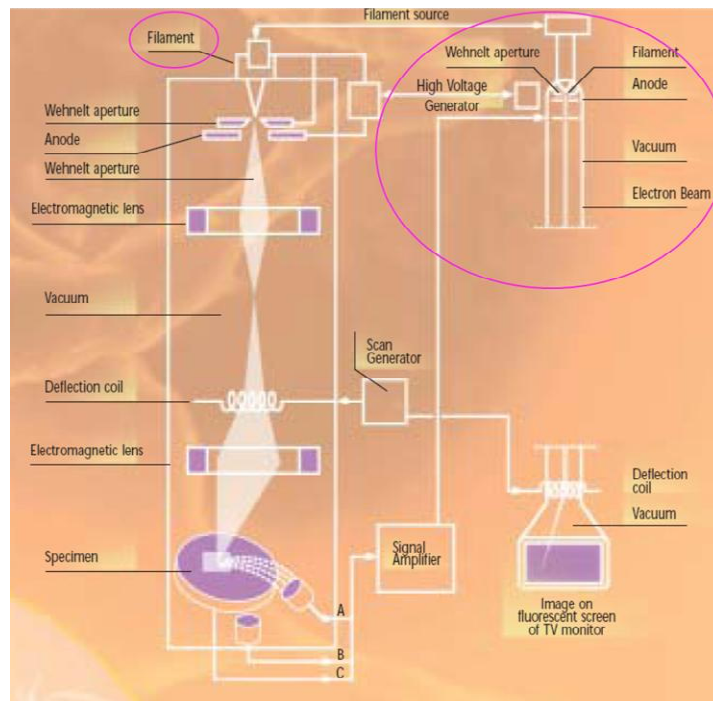
Main components:

- Electron source (gun)
- Condenser lenses
- Apertures
- Scan coils
- Objective lens
- Detectors
- Vacuum system

The instrument in brief



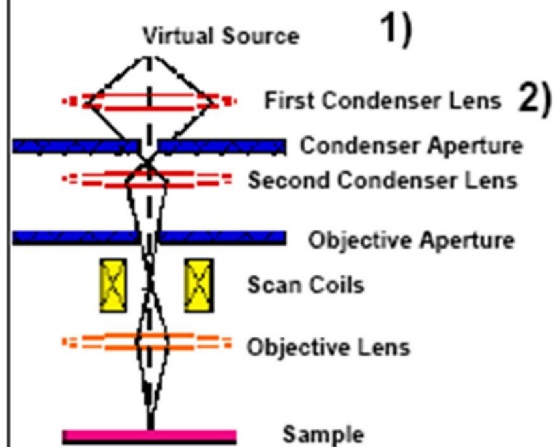
Components of the instrument



- *electron gun (filament)*
- *electromagnetic optics*
- *scan coils*
- *sample stage*
- *detectors*
- *vacuum system*
- *computer hardware and software*



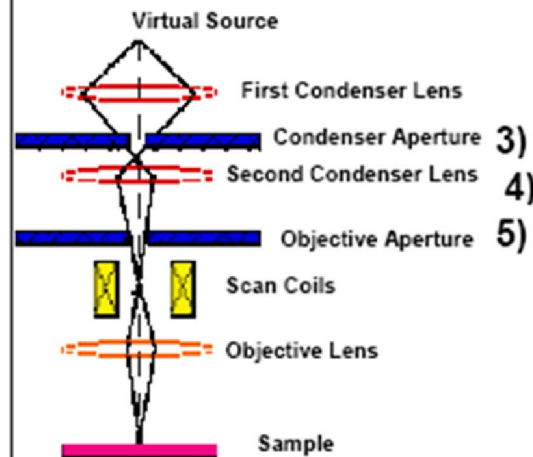
Scanning Electron Microscope



1) The "Virtual Source" at the top represents the electron gun, producing a stream of monochromatic electrons.

2) The stream is condensed by the first condenser lens (usually controlled by the "coarse probe current knob"). This lens is used to both form the beam and limit the amount of current in the beam. It works in conjunction with the condenser aperture to eliminate the high-angle electrons from the beam.

Scanning Electron Microscope

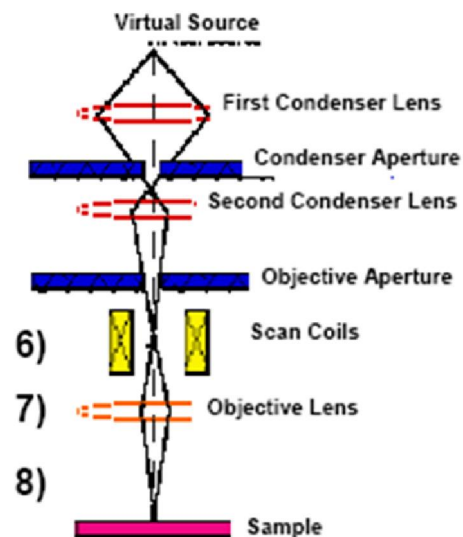


3) The beam is then constricted by the condenser aperture (usually not user selectable), eliminating some high-angle electrons.

4) The second condenser lens forms the electrons into a thin, tight, coherent beam and is usually controlled by the "fine probe current knob".

5) A user selectable objective aperture further eliminates high-angle electrons from the beam.

Scanning Electron Microscope

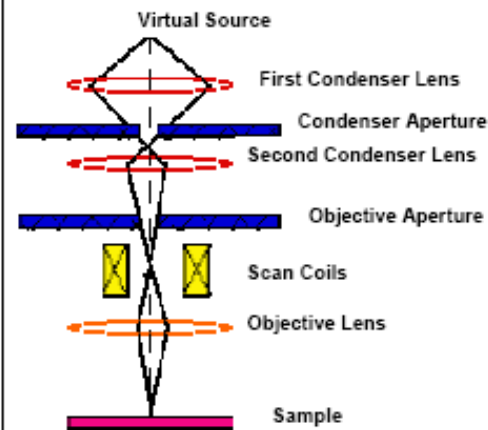


6) A set of coils then "scan" or "sweep" the beam in a grid fashion (like a television), dwelling on points for a period of time determined by the scan speed (usually in the microsecond range).

7) The final lens, the objective, focuses the scanning beam onto the part of the specimen desired.

8) When the beam strikes the sample (and dwells for a few microseconds) interactions occur inside the sample and are detected with various instruments.

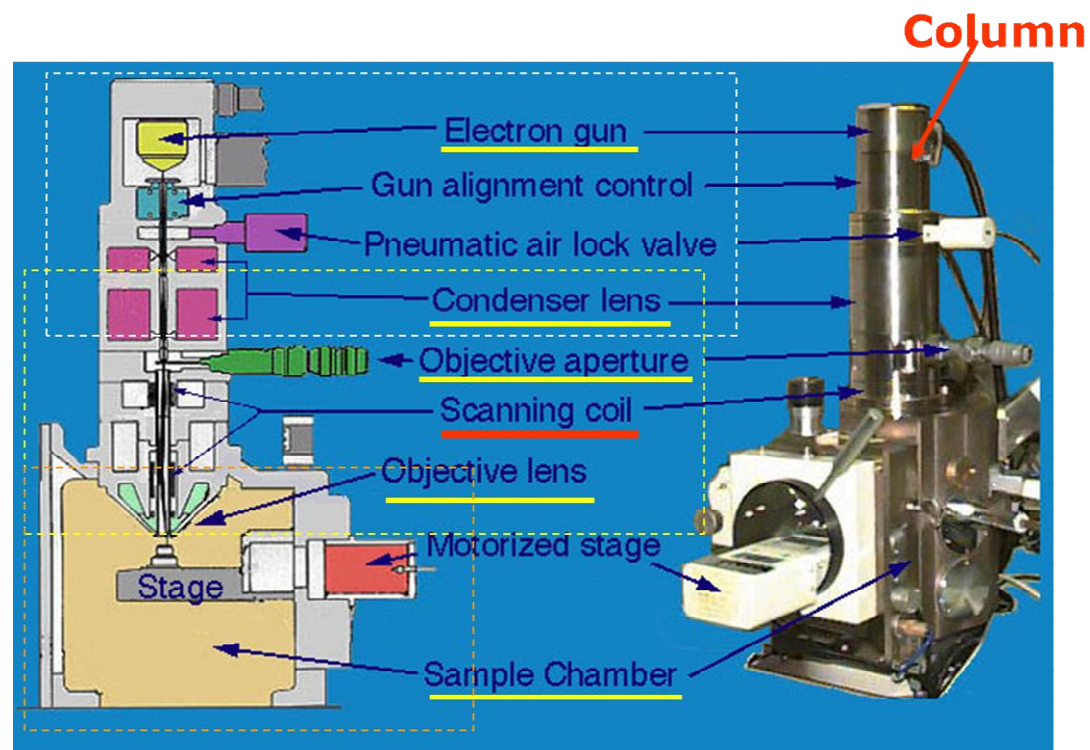
Scanning Electron Microscope



9) Before the beam moves to its next dwell point these instruments count the number of e^- interactions and display a pixel on a CRT whose intensity is determined by this number (the more reactions the brighter the pixel).

10) This process is repeated until the grid scan is finished and then repeated, the entire pattern can be scanned 30 times/sec.

A Look Inside the Column



How an Electron Beam is Produced?

- **Electron guns** are used to produce a fine, controlled beam of electrons which are then focused at the specimen surface.
- We want **many electrons per time unit per area** (high current density) and as small electron spot as possible.
- **The electron guns may either be thermionic gun or field-emission gun.**

Electron guns

- Traditional guns: thermionic electron gun (electrons are emitted when a solid is heated)
 - W-wire, LaB₆-crystal
- Modern: field emission guns (FEG) (cold guns, a strong electric field is used to extract electrons)
 - Single crystal of W, etched to a thin tip



- **How it works:** A sharp needle (tip radius ~100 nm) is placed in a strong electric field. The field lowers the work function, allowing electrons to "tunnel" out without heat.

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)

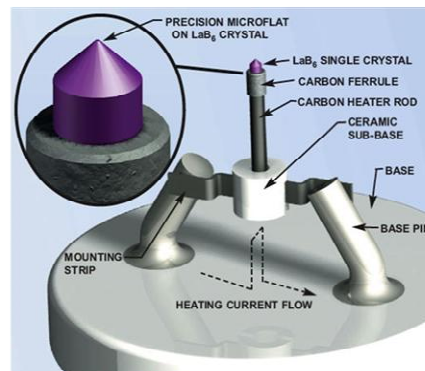
The LaB_6 filament is also a thermal filament. However, its work function is lower than for a tungsten filament, so it is more efficient.

Tungsten field emission gun (FEG)

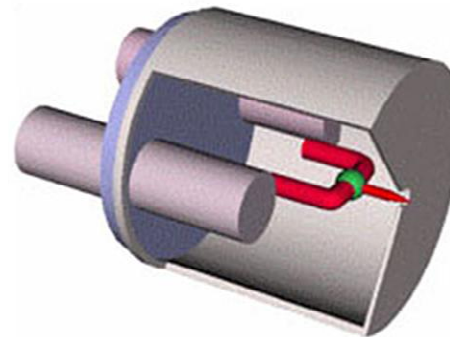
The FEG gun is not a thermal filament. Instead, electrons are expelled by applying a very powerful electric field very close to the filament tip. The size and proximity of the electric field to the electron reservoir in the filament causes the electrons to tunnel out of the reservoir.



W hairpin



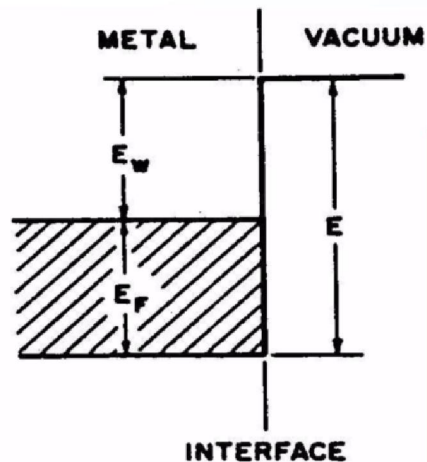
LaB_6 crystal



FEG

Concept of Work Function

Energy Model of Thermionic Emission



$$E_W = E - E_F$$

The emission current density is given by Richardson equation

$$J_c = A_c T^2 \exp(-E_W/kT) \text{ A/cm}^2$$

$$A_c - (\text{A/cm}^2 \text{K}^2); T - (\text{K})$$

At sufficiently high temperatures, a certain % of the electrons become sufficiently energetic to overcome the work function (E_W) of the cathode material and escape the source.

The Richardson-Dushman Equation

The Richardson-Dushman equation is the core of the energy model, defining the maximum thermionic current density a material can emit at a given temperature

$$J_{RD} = A_{RD} \cdot T^2 \cdot e^{-\frac{\Phi}{k_B T}}$$

Where:

- J_{RD} is the emission current density (A/cm²).
- A_{RD} is the Richardson-Dushman constant (theoretical value ~120 A/cm²/K²)
- T is the absolute temperature (K).
- Φ is the work function of the cathode material (eV)
- k_B is the Boltzmann constant.
- $e^{-\frac{\Phi}{k_B T}}$ is the Boltzmann factor, representing the probability that an electron has enough thermal energy to escape

Calculation of Current Density Tungsten Cathode

The filament current necessary to reach the operating temperature, and to obtain filament saturation decrease with the age of the filament.

$$J_c = A_c T^2 \exp(-E_w/kT) \text{ A/cm}^2$$

$$\text{If } A_c = 60 \text{ A/cm}^2 \text{K}^2$$

$$E_w = 4.5 \text{ eV}; k = 8.617 \times 10^{-5} \text{ eV/K}$$

$$T = 2700 \text{ K}$$

$$J_c = ?$$

- **W:** Work function ~4.5 eV, requires heating to ~2700K, offering a brightness of $\sim 10^5 \text{ A}\cdot\text{cm}^{-2}\cdot\text{sr}^{-1}$
- **LaB₆:** Work function ~2.7 eV, operates at ~1900K, achieving a much higher brightness of $\sim 10^6 \text{ A}\cdot\text{cm}^{-2}\cdot\text{sr}^{-1}$

Emission current density and current at the crossover

- The distinction between **emission current density** (a property of the cathode material) and the **current at the crossover** (a property of the gun geometry and biasing) is critical for understanding SEM performance.
- The **current at the crossover** is the total electron current passing through that tiny spot. It is always **less than** the total emission current from the filament.

Beam Current Density

The J_c in the e-beam at crossover point represents the current that could be concentrated into a focused spot on the sample, provided there is no aberration in the electron lenses.

The J_b is the maximum intensity of electrons in the beam at the crossover, can be defined as

$$J_b = \frac{i_b}{\pi\left(\frac{d_o}{2}\right)^2}$$

i_b - is the total beam or emission current measured from filament

Beam current usually varies between 100-200 μ A.

In practice, maximum current density in the final image is desired.

d = Probe diameter at the sample

Electron beam Source



W Filament - Current density $\sim 10\text{A/cm}^2$, probe size $\sim 5.0\text{nm}$
 LaB₆ - Current density $\sim 10^3\text{A/cm}^2$, probe size $\sim 1.5\text{nm}$
 FEG - Current density $\sim 10^5\text{A/cm}^2$, probe size $\sim <1.0\text{nm}$

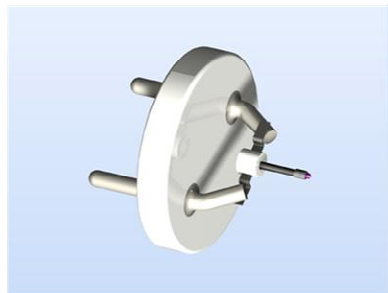


John F. Mansfield North Campus Electron Microbeam Analysis Lab., University of Michigan

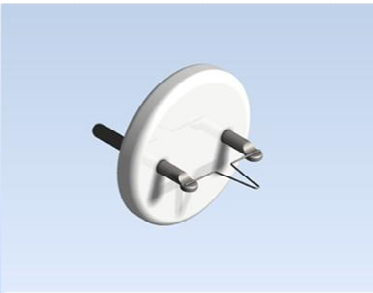
Electron guns

- With field emission guns we get a **smaller spot** and **higher current densities** compared to thermionic guns.
- **Vacuum requirements** are tougher for a field emission guns.

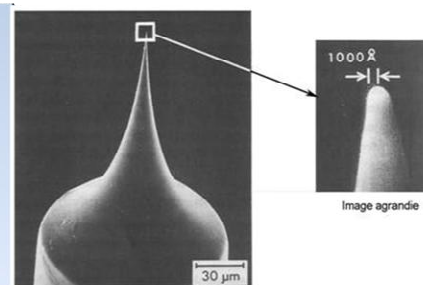
- **Tungsten:** Cheap, durable, but lower brightness, higher energy spread, requires high vacuum.
- **LaB6 (Lanthanum Hexaboride):** Brighter, longer life than tungsten, but requires better vacuum.



Single crystal of LaB₆



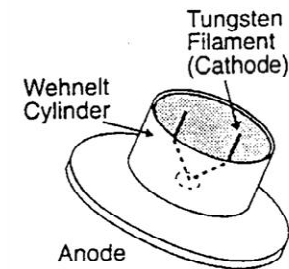
Tungsten wire



Field emission tip

Thermionic Emission Gun

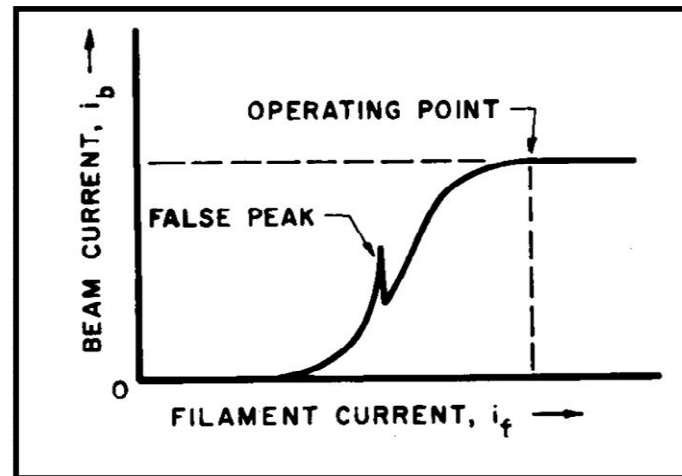
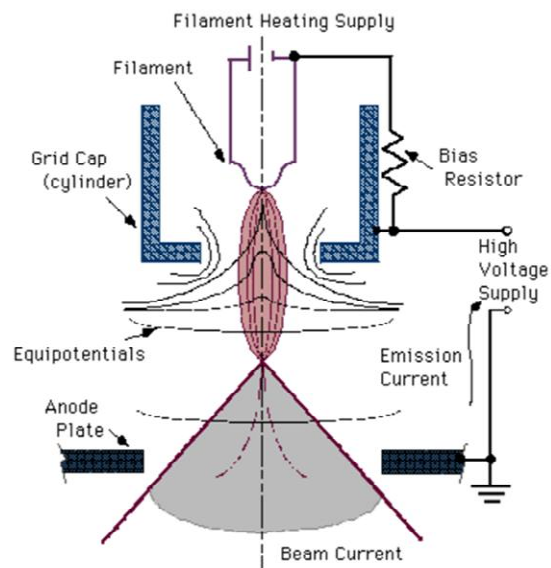
- A tungsten filament heated by DC to approximately 2700K or LaB₆ rod heated to around 2000K.
- A vacuum of 10^{-3} Pa (10^{-4} Pa for LaB₆) is needed to prevent oxidation of the filament.
- Electrons “boil off” from the tip of the filament.
- Electrons are accelerated by an acceleration voltage of 1-50kV.



Beam spot image at different stage of heating

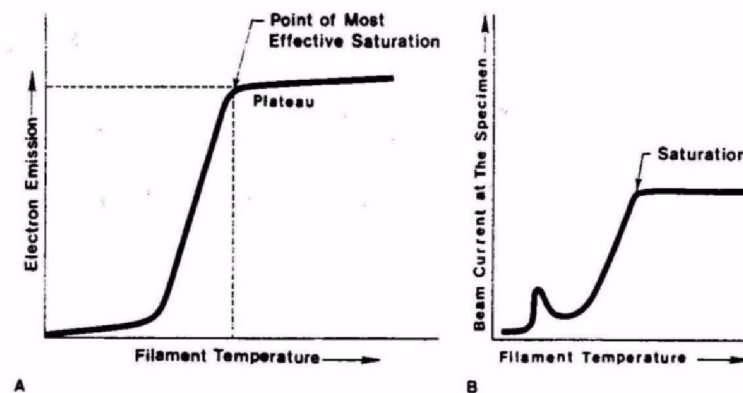


Thermionic Sources



Increasing the filament current will increase the beam current but only to the point of saturation at which point an increase in the filament current will only shorten the life of the emitter.

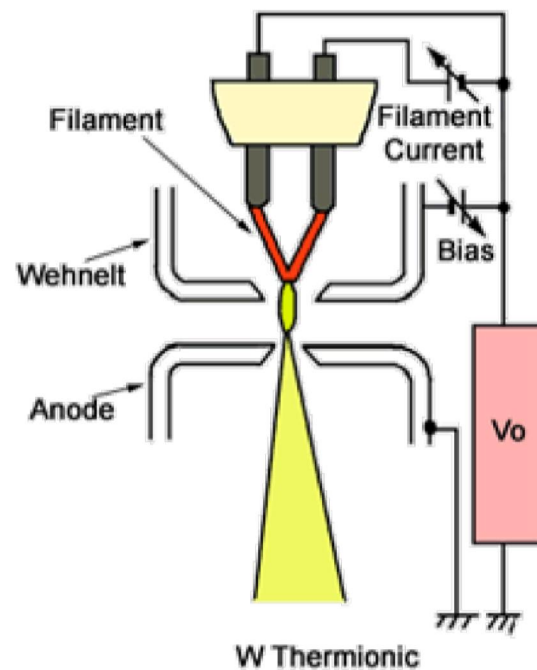
Saturation of Filament Current



At the most effective point of saturation, the highest quantity of electrons are generated for the least amount of current.

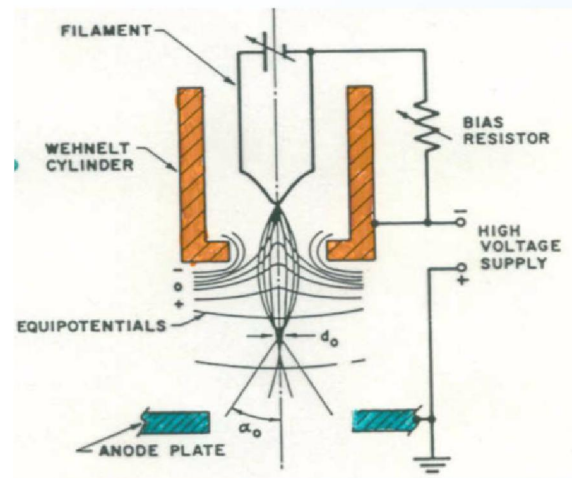
As the current flowing through the filament increases, the number of electrons emitted increases up to a specific point called Saturation Point.

Thermionic Emission Electron Source

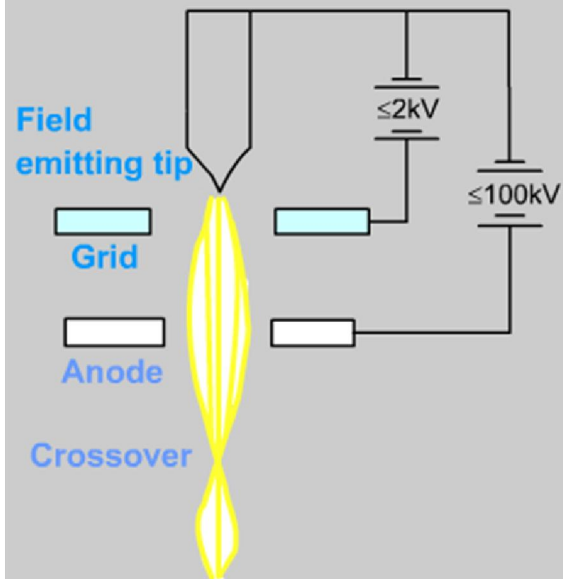


The most common emitter material for the electron beam is tungsten (W) or LaB6. The emitter is located at the top of the SEM column in the Gun assembly. The filament sits inside a housing called the Wehnelt cap. A potential is created between the sample and the gun and when the emitter is heated to a certain temperature electrons will begin streaming from the tip of the emitter and travel down the column. The electron beam is focused and defocused by the use of electromagnetic lenses.

Configuration of Self-biased Gun Wehnelt Cap Assembly



Field emission electron gun

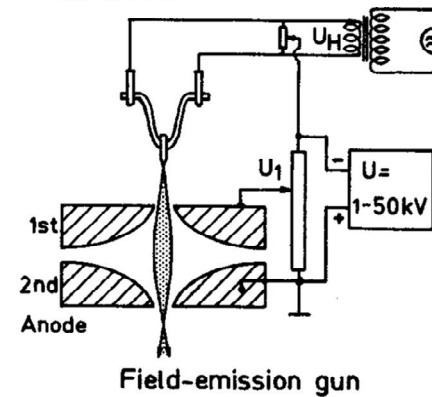
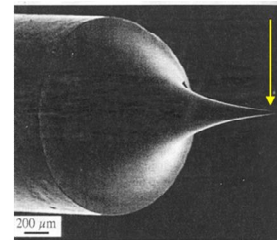


In the field emission gun, a very strong electric field (10^9 Vm^{-1}) is used to extract electrons from a metal filament. Temperatures are lower than that needed for thermionic emission.

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

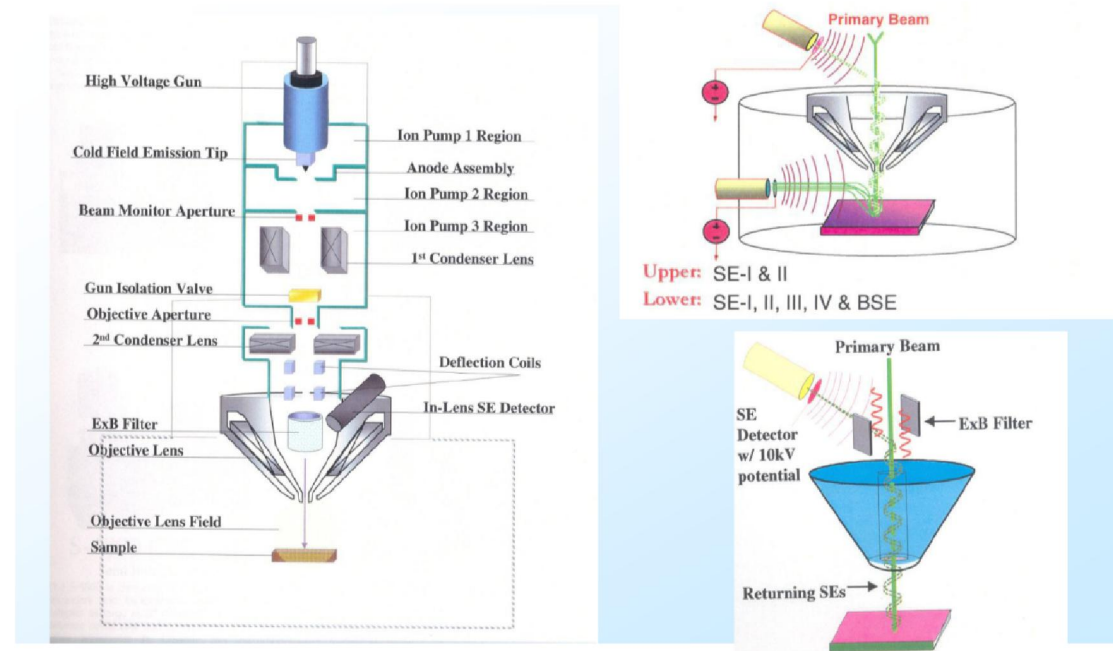
Field Emission Gun

- The tip of a tungsten needle is made very sharp (radius $< 0.1 \mu\text{m}$)
- The electric field at the tip is very strong ($> 10^7 \text{ V/cm}$) due to the sharp point effect.
- Electrons are pulled out from the tip by the strong electric field.
- Ultra-high vacuum (better than 10^{-6} Pa) is needed to avoid ion bombardment to the tip from the residual gas.
- Electron probe diameter $< 1 \text{ nm}$ is possible



1st anode to regulate the field strength at the tip by U_1 and hence regulate the emission current. 2nd anode: accelerates the electrons to the final kinetic energy.

Cold-Cathode Field Emission SEM S-4800 Column



Electron Sources

Types of guns:

Source	Brightness	Energy Spread	Stability
Tungsten (W)	Low	High	Moderate
LaB ₆	Medium	Medium	Good
Field Emission Gun (FEG)	Very high	Very low	Excellent

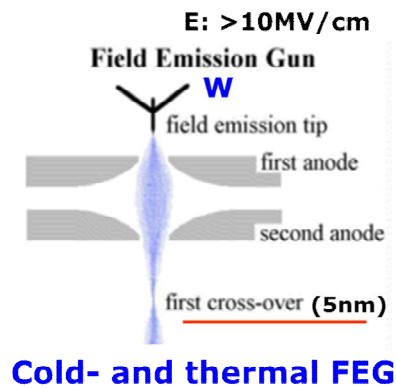
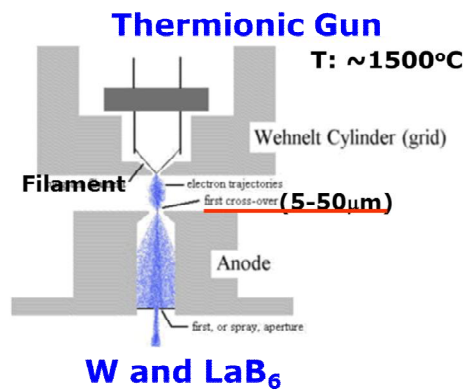
Thermionic emission:

- Heat-driven electron emission
- Lower brightness
- Lower cost

Field emission:

- Quantum tunneling
- Extremely high brightness
- High spatial coherence

Source of Electrons



Electron Gun Properties

Source	Brightness	Stability(%)	Size	Energy spread	Vacuum
W	3×10^5	~ 1	50 μm	3.0(eV)	$10^{-5} (\tau)$
LaB ₆	3×10^6	~ 2	5 μm	1.5	10^{-6}
C-FEG	10^9	~ 5	5nm	0.3	10^{-10}
T-FEG	10^9	< 1	20nm	0.7	10^{-9}

Brightness – beam current density per unit solid angle

Electron Source Performance Comparison:

Emitter Type	Thermionic	Thermionic	Schottky FE	cold FE
Cathode material	W	LaB ₆	ZrO/W (100)	W(310)
Operating temperature [K]	2,800	1,900	1,800	300
Cathode radius [nm]	60,000	10,000	< 1,000	< 100
Effective source radius [nm]	15,000	5,000	15 (*)	2.5(*)
Emission current density [A/cm ²]	3	30	5,300	17,000
Total emission current [μA]	200	80	200	5
Normalized brightness [A/cm ² .sr.kV]	1.10 ⁴	1.10 ⁵	1.10 ⁷	2.10 ⁷
Maximum probe current [nA]	1000	1000	10	0.2
Energy spread @ cathode [eV]	0.59	0.40	0.31	0.26
Energy spread @ gun exit [eV]	1.5 - 2.5	1.3 - 2.5	0.35 - 0.7	0.3 - 0.7
Beam noise [%]	1	1	1	5 - 10
Emission current drift [%/h]	0.1	0.2	< 0.5	5
Operating vacuum hPa/mbar	< 1.10 ⁻⁵	< 1.10 ⁻⁶	< 1.10 ⁻⁹	< 1.10 ⁻¹⁰
Typical Cathode life [h]	100	> 1000	> 5000	> 2000
Cathode regeneration	not required	not required	not required	every 6 to 8 hours
Sensitivity to external influence	minimal	minimal	low	high

* virtual source

Why Need a Vacuum?

When a SEM is used, the electron-optical column and sample chamber must always be at a vacuum.

- 1. If the column is in a gas filled environment, electrons will be scattered by gas molecules which would lead to reduction of the beam intensity and stability.**
- 2. Other gas molecules, which could come from the sample or the microscope itself, could form compounds and condense on the sample. This would lower the contrast and obscure detail in the image.**

Why do we need vacuum?

- Chemical (corrosion!!) and thermal stability is necessary for a well-functioning filament (gun pressure)
 - A field emission gun requires $\sim 10^{-10}$ Torr
 - LaB_6 : $\sim 10^{-6}$ Torr
- The signal electrons must travel from the sample to the detector (chamber pressure)
 - Vacuum requirements is dependant of the type of detector

2.3 The Vacuum System

- **Why so serious?** Electrons are light and easily scattered.
 - **Collision:** If an electron hits an air molecule, it scatters. This causes loss of signal and a hazy background.
 - **Contamination:** Air contains hydrocarbons (oil, grease). The electron beam "cracks" these molecules, depositing carbon on the sample and inside the column.
 - **Filament Life:** A hot filament in the presence of oxygen will oxidize and burn out in seconds.
- **The Pumps:**
 - **Rotary Vane/Dry Pump (Roughing Pump):** Gets the chamber from atmosphere (760 Torr) to "rough vacuum" ($\sim 10^{-3}$ Torr). It removes the bulk of the air.
 - **Turbomolecular Pump (Turbo):** Uses high-speed fan blades to knock gas molecules out of the chamber. Achieves high vacuum (10^{-6} to 10^{-7} Torr).
 - **Ion Pump (UHV):** Used in FEG microscopes and TEM columns. Ionizes remaining gas atoms and absorbs them into a reactive metal. Achieves Ultra-High Vacuum (10^{-9} to 10^{-10} Torr).

Electron Optics System

- Condenser lenses: control beam current
- Scan coils: raster beam scanning
- Objective lens: focuses probe on sample

Goal: Small probe size + high beam current

2.2 Electromagnetic Lenses

- **Not Glass:** Crucially explain that we cannot use glass lenses because electrons cannot pass through glass.
- **The Principle:** A coil of copper wire (the lens) carries a current. This creates a magnetic field. When electrons pass through this field, they experience the **Lorentz Force**, which bends their trajectory.
- **Lens Types:**
 - **Condenser Lenses:** Located after the gun. Their job is to gather the electrons and control the *spot size* and *current* (how many electrons hit the sample).
 - **Objective Lens:** The most important lens. It does the primary focusing. The sample sits inside or very near the magnetic field of this lens. Aberrations here ruin image quality.
 - **Projector/Intermediate Lenses (TEM):** Magnify the image formed by the objective lens and project it onto the screen/camera.

Magnetic Lenses

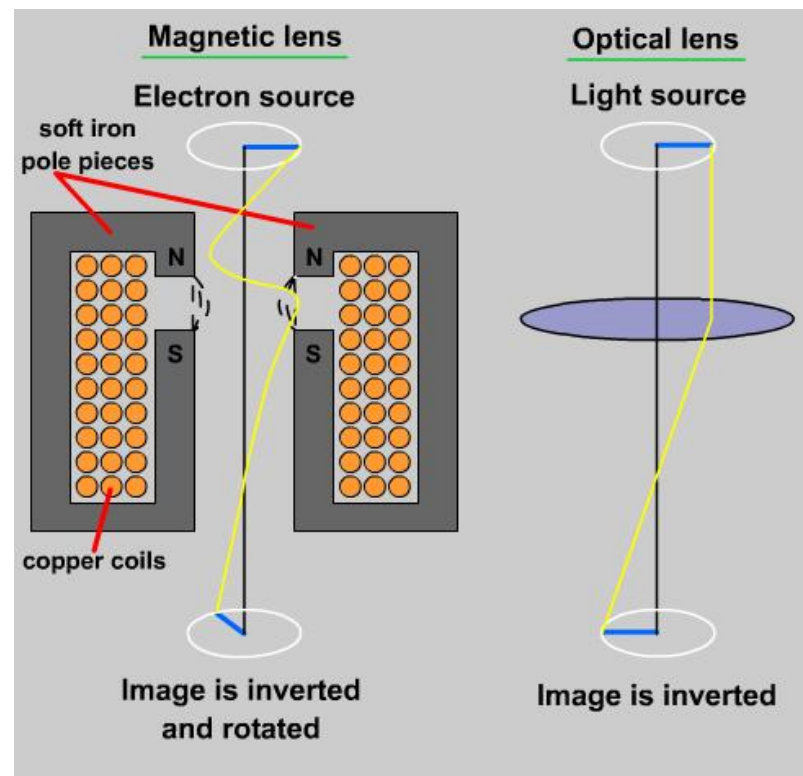
- **Condenser lens – focusing**

determines the beam current which impinges on the sample.

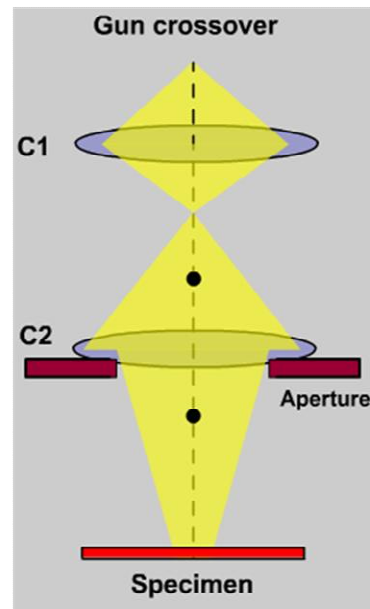
- **Objective lens – final probe forming**

determines the final spot size of the electron beam, i.e., **the resolution of a SEM.**

Electromagnetic lens

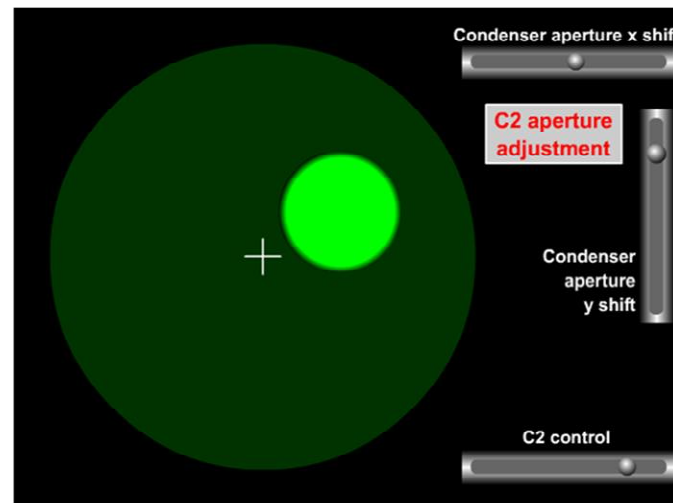


Condenser-lens system



C1 controls the spot size

C2 changes the convergence of the beam



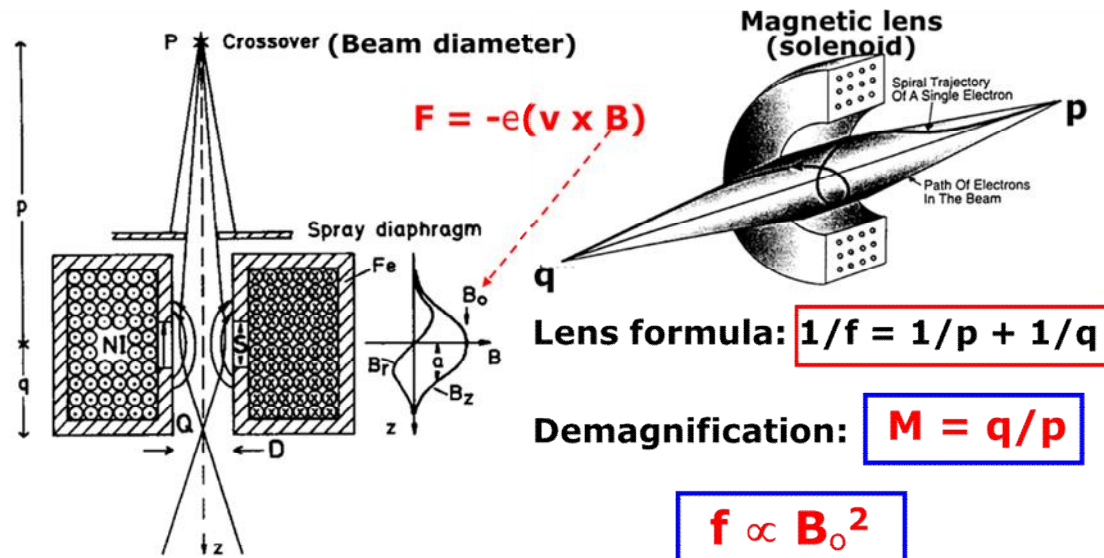
The condenser aperture must be centered

The Condenser Lens

- For a thermionic gun, the diameter of the first **cross-over point** $\sim 20\text{-}50\text{ }\mu\text{m}$
- If we want to focus the beam to a size $< \mathbf{10\text{ nm}}$ on the specimen surface, the magnification should be $\sim 1/5000$, which is not easily attained with one lens (say, the objective lens) only.
- Therefore, condenser lenses are added to **demagnify** the cross-over points.

How Is Electron Beam Focused?

A magnetic lens is a solenoid designed to produce a specific magnetic flux distribution.



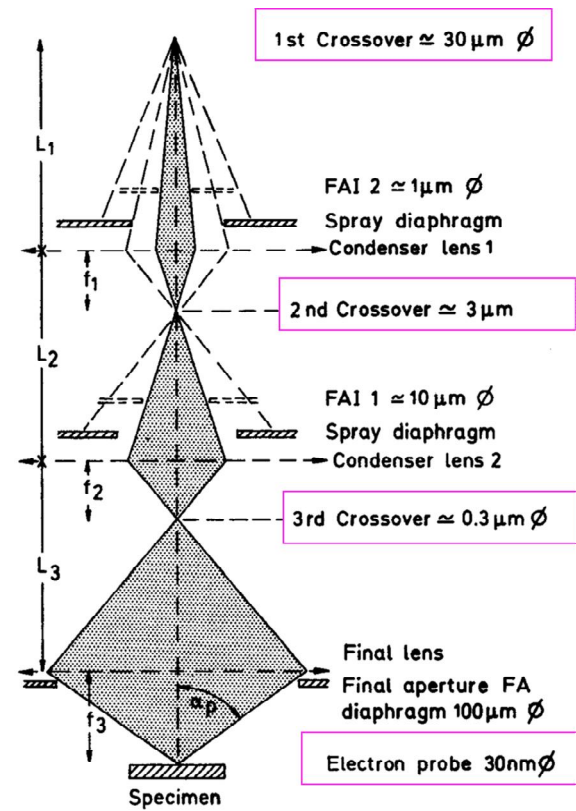
f can be adjusted by changing B_o , i.e., changing the current through coil.

v = electron speed

The Condenser Lens

Demagnification:

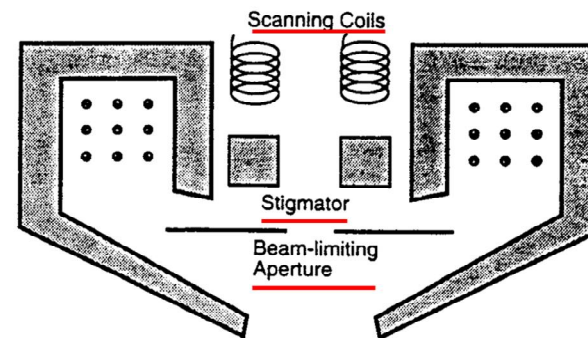
$$M = f/L$$



The Objective Lens

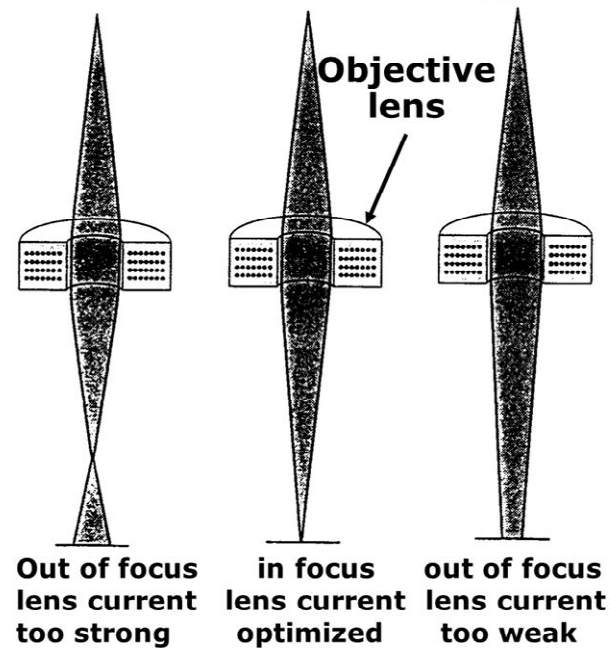
- The objective lens controls the **final focus** of the electron beam by changing the magnetic field strength
- The cross-over image is finally demagnified to an **$\sim 10\text{nm}$** beam spot which carries a beam current of approximately **10^{-9} - 10^{-12} A.**

The objective lens is machined to very high precision and the magnetic field pattern is very carefully designed. However, the precision attainable by machining cannot match that required for controlling a beam with $\varnothing 10\text{ nm}$. The stigmator, which consists of two pairs of pole-pieces arranged in the X and Y directions, is added to correct the minor imperfections in the objective lens.



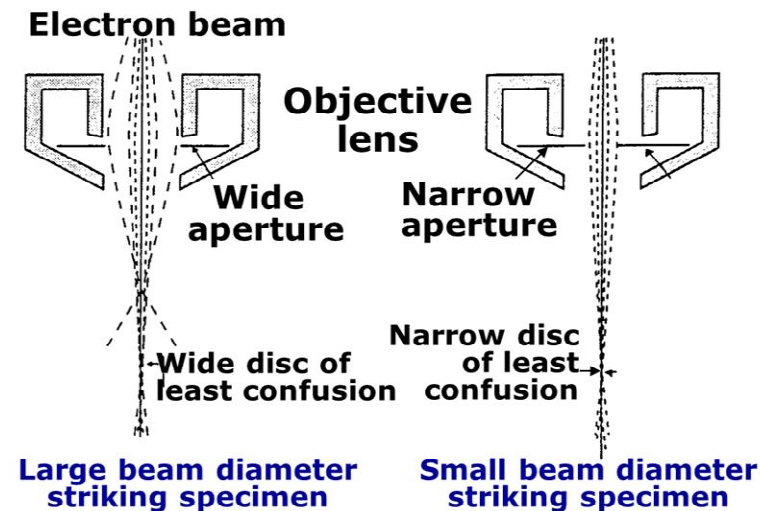
The Objective Lens - Focusing

- By changing the current in the objective lens, the magnetic field strength changes and therefore the **focal length** of the objective lens is changed.



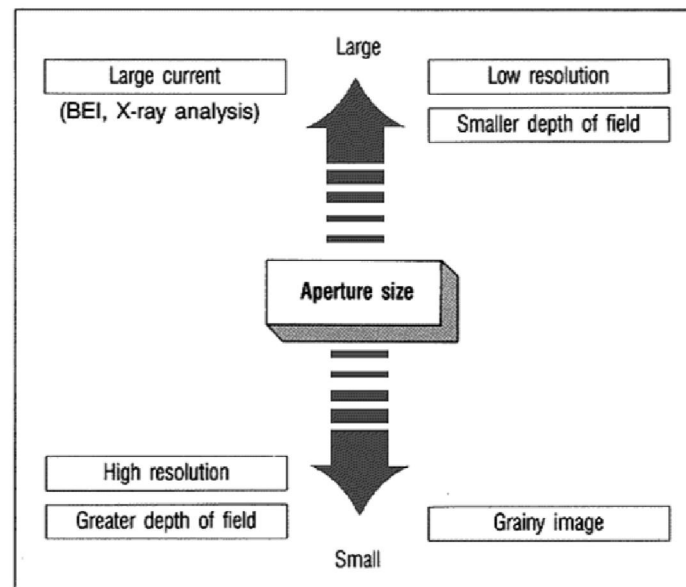
The Objective Lens – The Aperture

- Since the electrons coming from the electron gun have **spread in kinetic energies and directions of movement**, they may not be focused to the same plane to form a sharp spot.
- By inserting an aperture, the stray electrons are blocked and the remaining narrow beam will come to a narrow



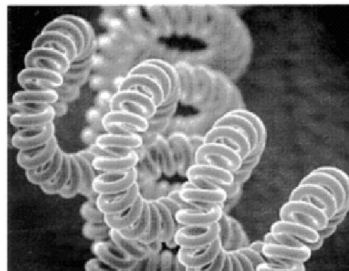
"Disc of Least Confusion"

Effect of Objective Lens Aperture Diameter on Image

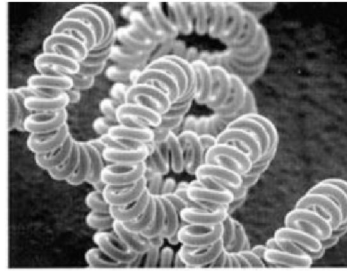


The objective lens (OL) aperture set in the SEM as standard is of the optimum size selected considering various conditions. SEM images require not only a fine electron probe, but also a sufficient amount of signal for forming an image. The aperture cannot be reduced unnecessarily. The OL aperture must be selected with consideration given to the effect on the image.

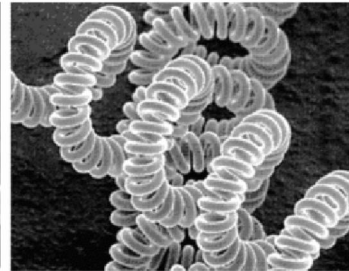
Effect of Objective Lens Aperture Diameter and WD on Image



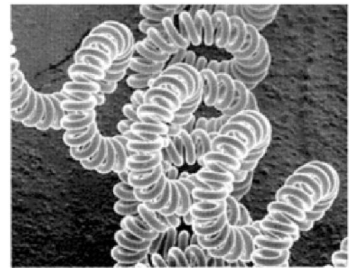
(a) Aperture diameter = 600 μm
Working distance = 10 mm



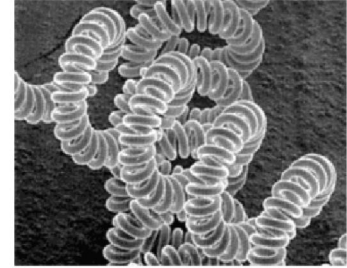
(b) Aperture diameter = 200 μm
Working distance = 10 mm



(c) Aperture diameter = 200 μm
Working distance = 20 mm



(d) Aperture diameter = 200 μm
Working distance = 38 mm



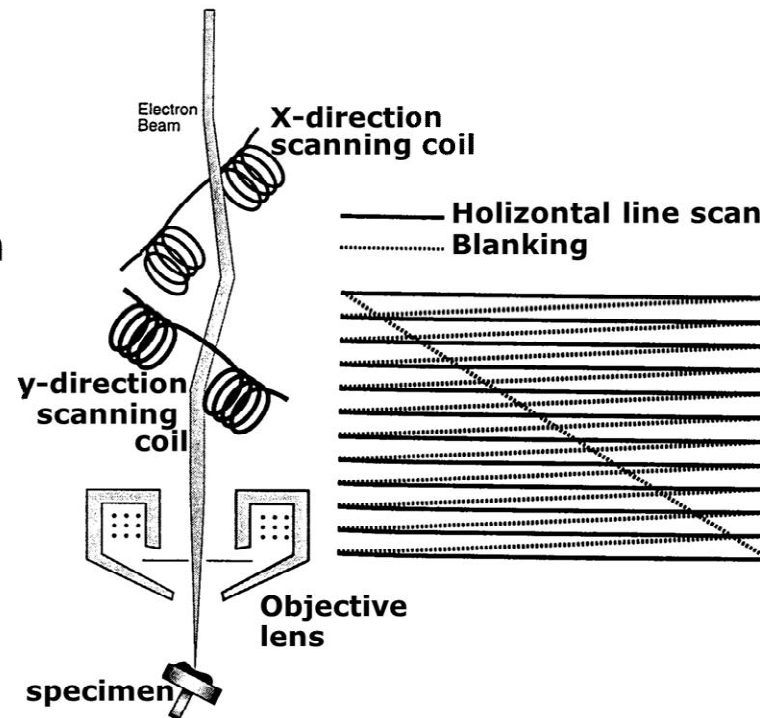
(e) Aperture diameter = 100 μm
Working distance = 38 mm

Electric light bulb
filament

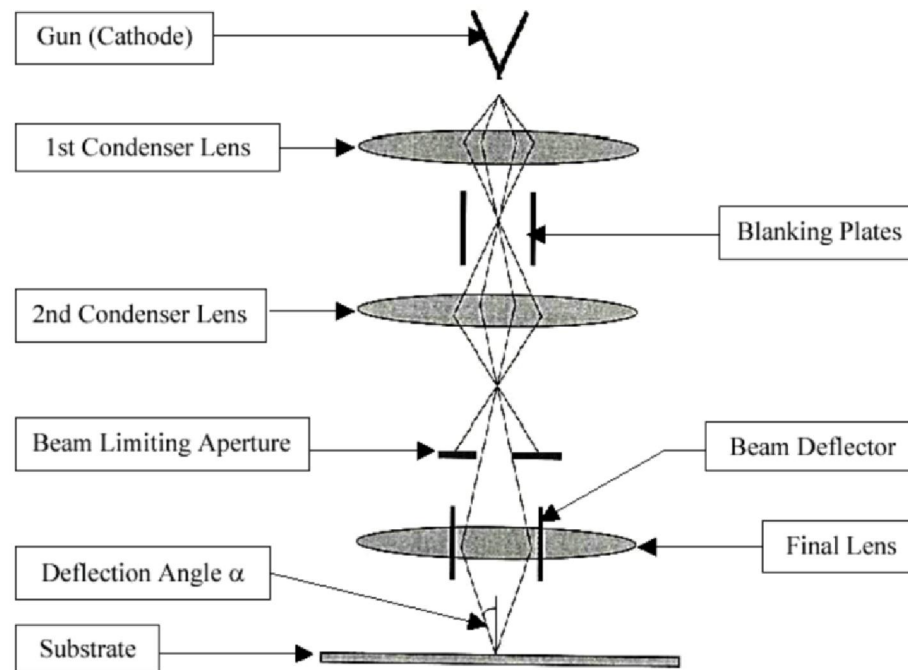
The smaller the objective
lens aperture diameter
and the longer the
working distance (WD),
the greater will be the
depth of field

The Scan Coil and Raster Pattern

- Two sets of coils are used for scanning the electron beam across the specimen surface in a **raster** pattern similar to that on a TV screen.
- This effectively samples the specimen surface **point by point** over the scanned area.



Scanning Electron Microscope



Electron Lenses & Aberrations

Major aberrations:

- Spherical aberration (C_s)
- Chromatic aberration (C_c)
- Astigmatism

Modern solutions:

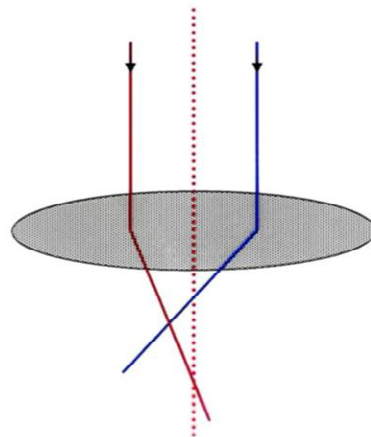
- Cs correctors
- Monochromators
- Cold field emission guns

Chromatic Aberration

Chromatic aberration occurs because the lens bends lower-energy electrons more strongly than higher-energy electrons.

Result: Lower-energy electrons are over-focused (focal point is shorter). Higher-energy electrons are under-focused (focal point is longer). This creates a blurred disk on the sample.

Chromatic Aberration



$$r_{chr} = C_c \frac{\Delta E}{E_0} \beta$$

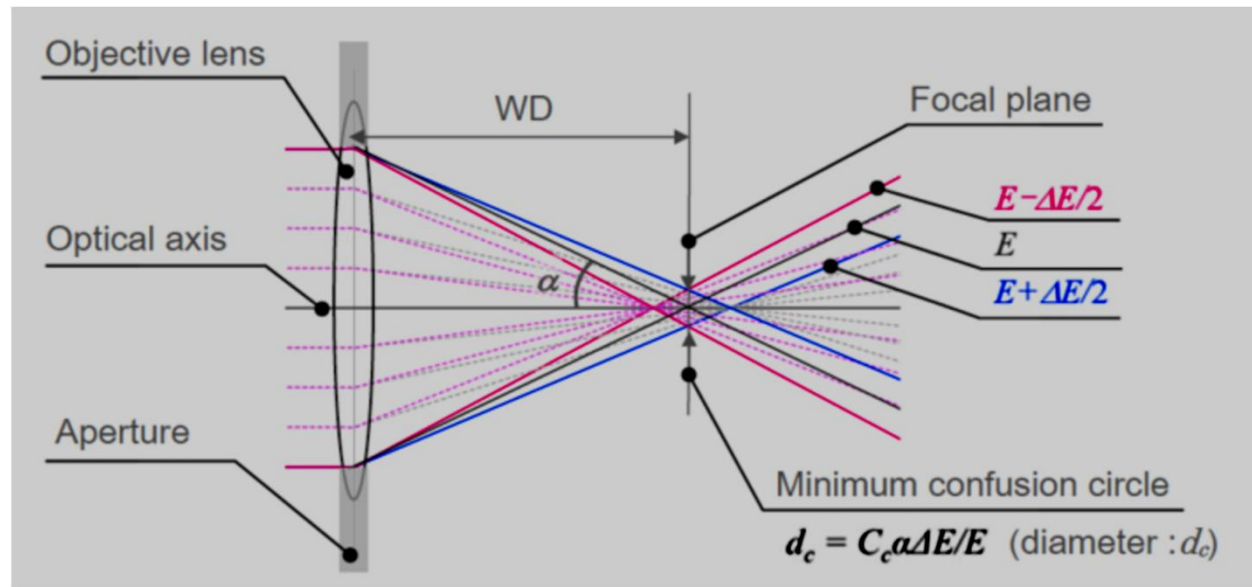
- Faster (higher energy) electrons focused less strongly than slower electrons

$$d_c = C_c \alpha \frac{\Delta V}{V}$$

- Aberration coefficient is roughly equal to the focal length of the lens

d_c = Chromatically limited Beam Diameter
 C_c = Chromatic Aberration Coefficient of the Final Lens
 α = Convergence Half Angle of the Beam at the target
 V = Beam Voltage
 ΔV = Energy Spread

d_c : Diameter of the blur disk due to chromatic aberration (meters)



Schematic diagram of the spread of electron beams due to chromatic aberration of the objective lens

Methods to Resolve (Correct/Minimize) Chromatic Aberration

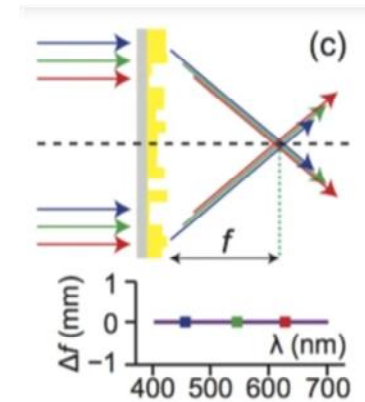
1: Use a High-Brightness, Low-Energy-Spread Source

Electron Source	ΔE (eV)	Chromatic Blur (d_c)
Tungsten (W) thermionic	1.5 – 2.5	Large
LaB ₆ thermionic	0.8 – 1.5	Medium
Cold FEG	0.2 – 0.3	Very small
Schottky FEG	0.5 – 0.8	Small

$$\frac{d_c(\text{FEG})}{d_c(\text{W})} = \frac{0.3}{2.5} \approx \frac{1}{8}$$

2: Reduce Aperture Angle (α)

3: Use a Monochromator



Spherical aberration

Spherical aberration is the single most important lens defect **limiting the resolution of modern** SEMs (especially those without aberration correctors).

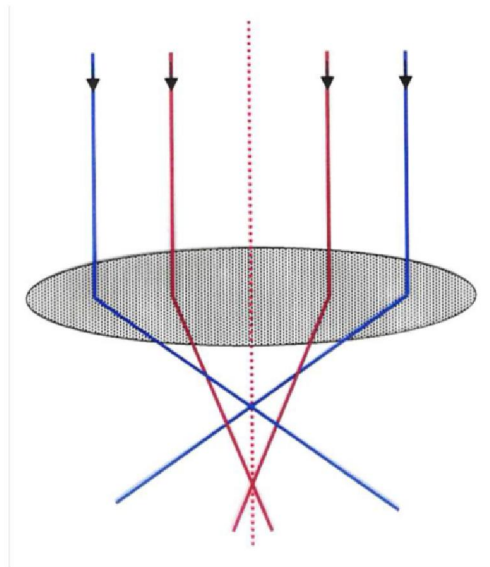
It occurs because the electromagnetic **lens is stronger at its periphery (edges) than at its center.**

Electrons passing through the **outer edges** of the lens are bent (focused) **too strongly.**

Electrons passing near the center are bent correctly.

Result: Instead of all electrons converging at the same focal point, they converge at slightly different points along the optical axis. This creates a disk of least confusion (the smallest possible focused spot) that is significantly larger than the theoretical ideal.

Spherical Aberration



- Off-axis electrons focused more strongly

$$d_s = \frac{1}{2} C_s \alpha^3$$

- Aberration coefficient is approximately equal to the focal length of the lens

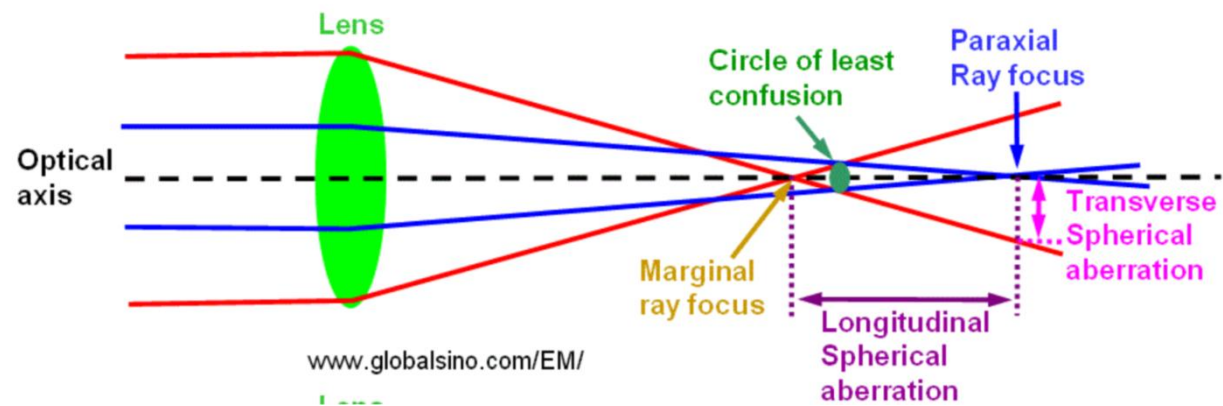
d_s = Beam Diameter

C_s = Spherical Aberration Coefficient of the Final Lens

α = Convergence Half Angle of the Beam at the target

α = beam semi-angle (radians) at the specimen

Spherical Aberration Formula



Resolving Spherical Aberration in SEM

Unlike chromatic aberration, spherical aberration cannot be eliminated by aperture alone (only reduced). True "resolution" of spherical aberration requires active correction.

1. Reduction (Not Elimination) – Using a Small Aperture

2. True Correction – Spherical Aberration Correctors

Modern high-resolution SEMs and STEMs use **multipole correctors** (hexapole or quadrupole-octupole systems).

Numerical Example for Comparison (SEM at 20 kV, $\alpha = 0.01$ rad)

Parameter	Approximate Value	Formula	Result
$C_s = 10 \text{ mm}, \alpha = 0.01$	$d_s = C_s \cdot \alpha^3$	$10 \times (10^{-2})^3 = 10 \times 10^{-6}$	10 nm
$C = 10 \text{ mm}, \Delta E = 1 \text{ eV (FEG)}, E = 20,000 \text{ eV}$	$d_c = C \cdot (\Delta E/E) \cdot \alpha$	$10 \times (1/20000) \times 0.01 = 10 \times 5 \times 10^{-7}$	5 nm
$C = 10 \text{ mm}, \Delta E = 2.5 \text{ eV (W)}, E = 20,000 \text{ eV}$	$d_c = C \cdot (\Delta E/E) \cdot \alpha$	$10 \times (2.5/20000) \times 0.01 = 10 \times 1.25 \times 10^{-6}$	12.5 nm

Result: At high voltage, spherical and chromatic aberrations are comparable. At low voltage (e.g., 1 kV), d_c becomes much larger (approximately 250 nm for thermionic tungsten in this example).

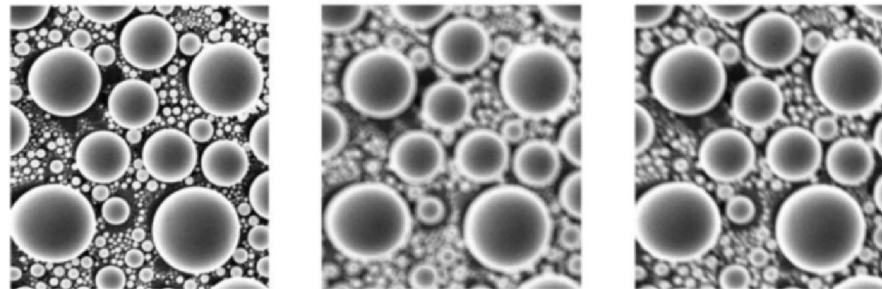
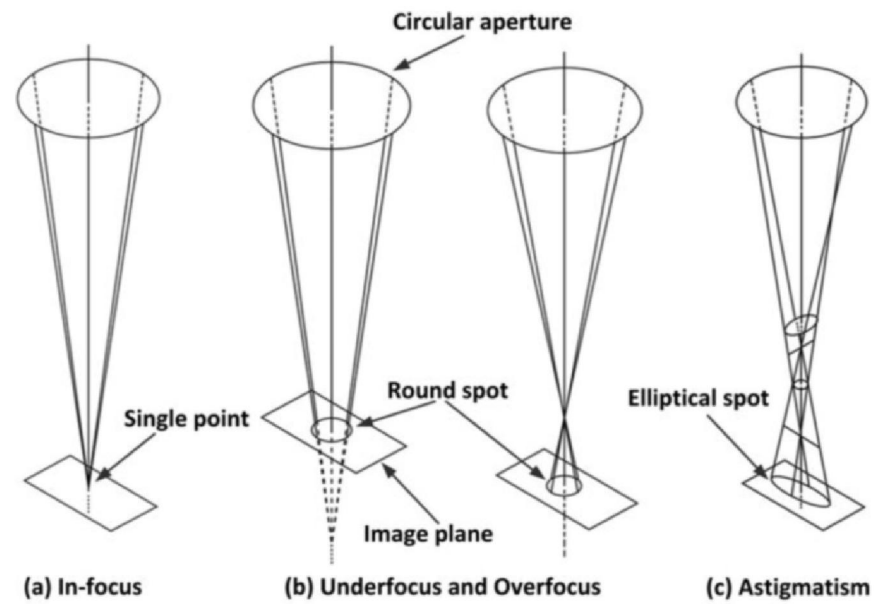
Astigmatism in an SEM

In an ideal electromagnetic lens, the magnetic field is perfectly **symmetrical** around the optical axis. It focuses electrons equally in all radial directions, producing a circular spot.

In reality, the lens is never perfect. Small imperfections cause the lens to focus electrons more strongly in one plane (say, the X-Z plane) than in the perpendicular plane (Y-Z plane).

Effect on Image.

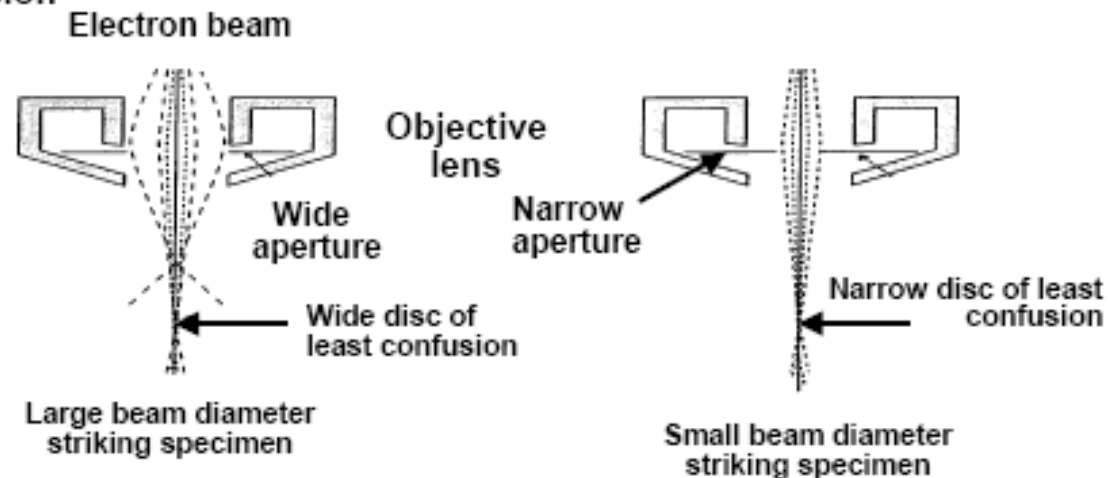
Round features appear **elongated (stretched) in one direction** and **compressed in the perpendicular direction**.



Electromagnetic Lenses

The Objective Lens - Aperture

- Since the electrons coming from the electron gun have spread in kinetic energies and directions of movement, they may not be focused to the same plane to form a sharp spot.
- By inserting an aperture, the stray electrons are blocked and the remaining narrow beam will come to a narrow "Disc of Least Confusion"



Astigmatism vs. Other Aberrations (The Full Comparison)

Aberration	Cause	Can user correct?	Dominates at...
Spherical	Lens focuses edges too strongly	No (factory/correctors)	High kV, large apertures
Chromatic	Lens focuses slow e^- more strongly	No (gun/monochromator)	Low kV
Astigmatism	Asymmetric lens (different X/Y focus)	YES (via stigmator)	All conditions (if present)

$$d_{total} \approx \sqrt{d_0^2 + d_s^2 + d_c^2 + d_d^2}$$

Where:

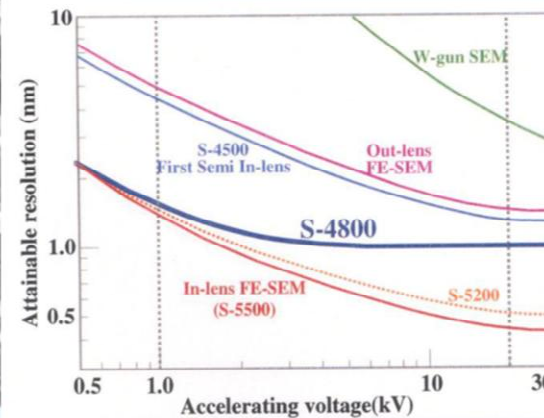
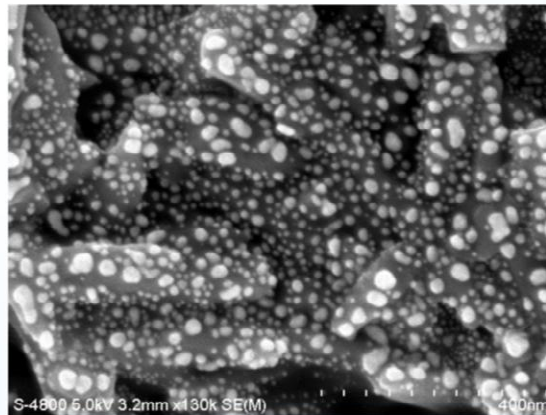
- d_0 = Effective source diameter (after demagnification by lenses)
- d_s = Spherical aberration disk diameter
- d_c = Chromatic aberration disk diameter
- d_d = Diffraction-limited disk diameter

Resolution

We can also improve the resolution by:

- Increasing the strength of the condenser lens
- Decreasing the size of the objective aperture
- Decreasing the working distance (WD = the distance the sample is from the objective lens)

Resolution of SEMs



SEM provides high image magnification and resolution!

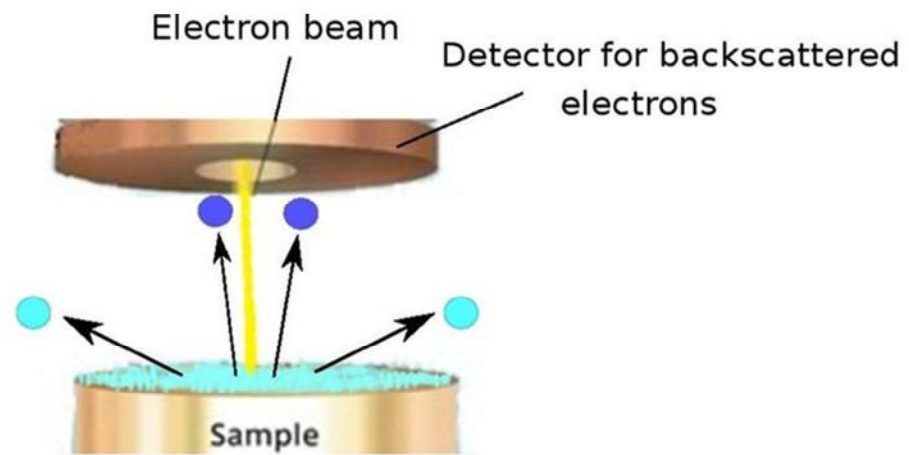
5.0 nm (50 Å) - common for commercial instruments

2.0 nm (20 Å) - for Advanced Research instruments

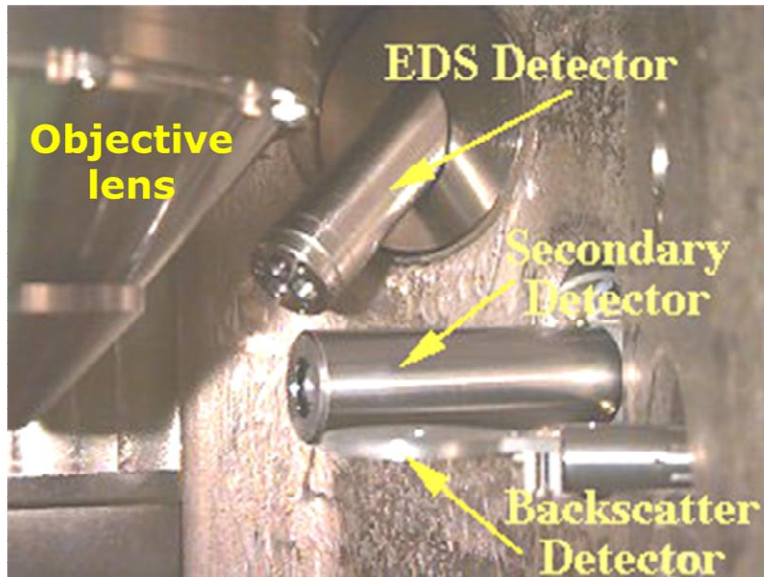
Some comments on resolution

- Best resolution that can be obtained: size of the electron spot on the sample surface
 - The introduction of FEG has dramatically improved the resolution of SEM's
- The volume from which the signal electrons are formed defines the resolution
 - SE image has higher resolution than a BSE image
- Scanning speed:
 - a weak signal requires slow speed to improve signal-to-noise ratio
 - when doing a slow scan drift in the electron beam can affect the accuracy of the analysis .

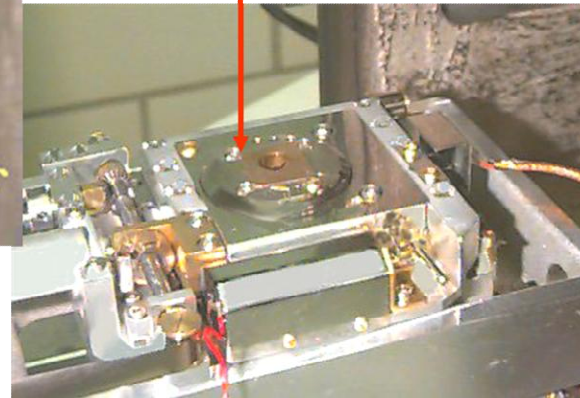
Electron detector



Electron Detectors and Sample Stage



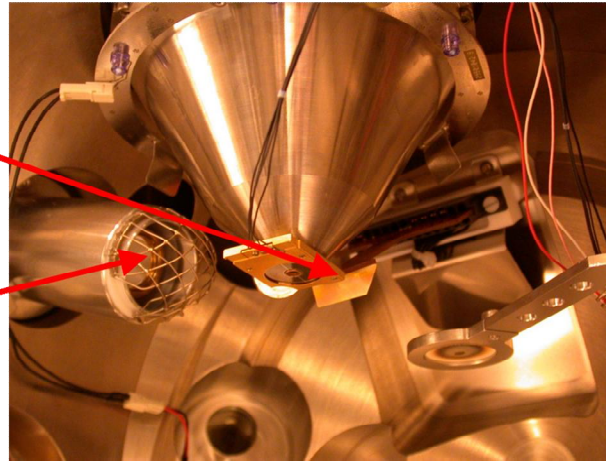
Sample stage



Detectors

*Backscattered electron
detector:
(Solid-State Detector)*

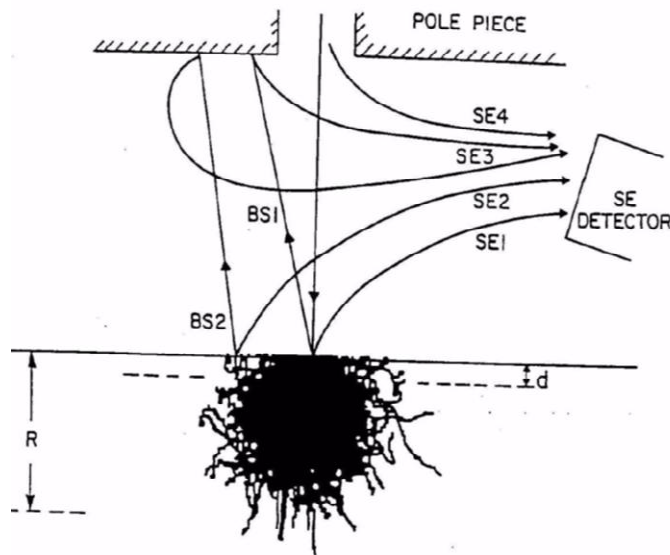
*Secondary electron detector:
(Everhart-Thornley)*



Our traditional detectors

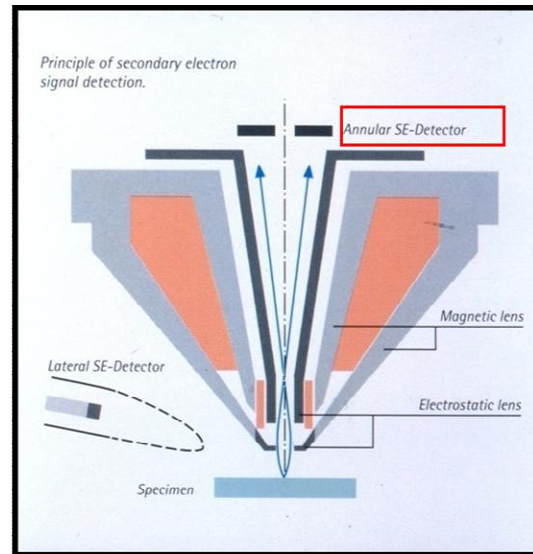
- Different detectors capture different **types** of signals (e.g., secondary electrons, backscattered electrons, X-rays). Each signal reveals different information about the sample.
- Secondary electrons: Everhart-Thornley Detector
- Backscattered electrons: Solid State Detector
- X-rays: Energy dispersive spectrometer (EDS)

Origin of SE Emission



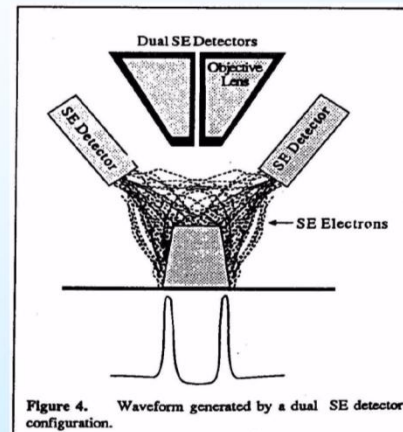
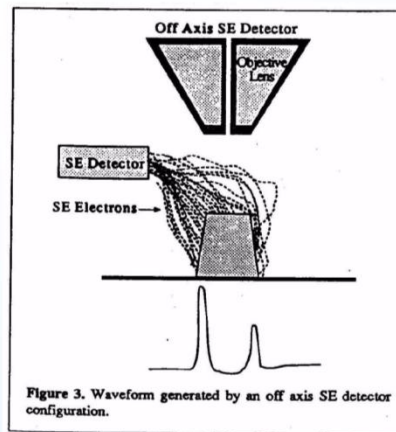
The size, sensitivity and position of the detector drastically affect its collection efficiency and the appearance of the image, and also the results of any measurements made from the image.

Metrology schemes must take into account the type & characteristics of the detector and their effect on the observed signal.

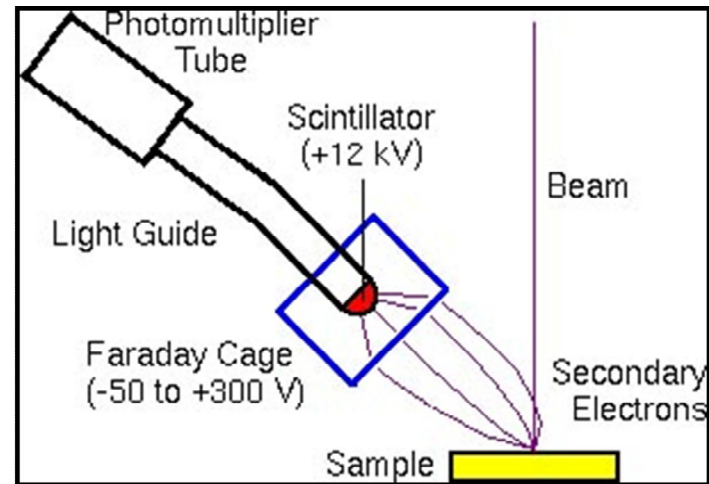


The position of the secondary electron detector affects signal collection and shadow. An in-lens detector within the column is more efficient at collecting secondary electrons that are generated close to the final lens (i.e. short working distance).

Waveforms Generated from a Single and Dual SE Detectors



Uneven sample irradiation or signal collection will affect the measurement results.



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.

1. Secondary Electron Detector (SED) - The "Topography" Detector

What it detects: Low-energy electrons (< 50 eV) ejected from the *surface* atoms of the sample.

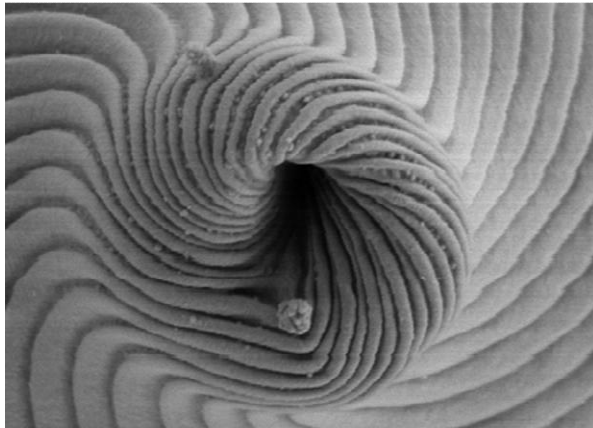
Where they come from: The top few nanometers of the sample.

How it works (Everhart-Thornley type): A positively biased grid (scintillator) attracts the low-energy secondary electrons, accelerates them, and converts them into **light**. A photomultiplier tube amplifies that light into **an electrical signal**.

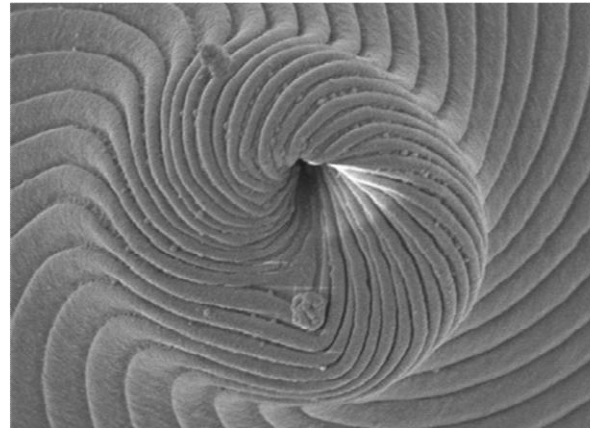
What the image looks like: Extremely high-resolution, strong edge brightness, excellent depth perception.

Best for: Seeing fine surface detail (texture, cracks, pores, nanoparticles) at high magnification.

Secondary Electron Detector



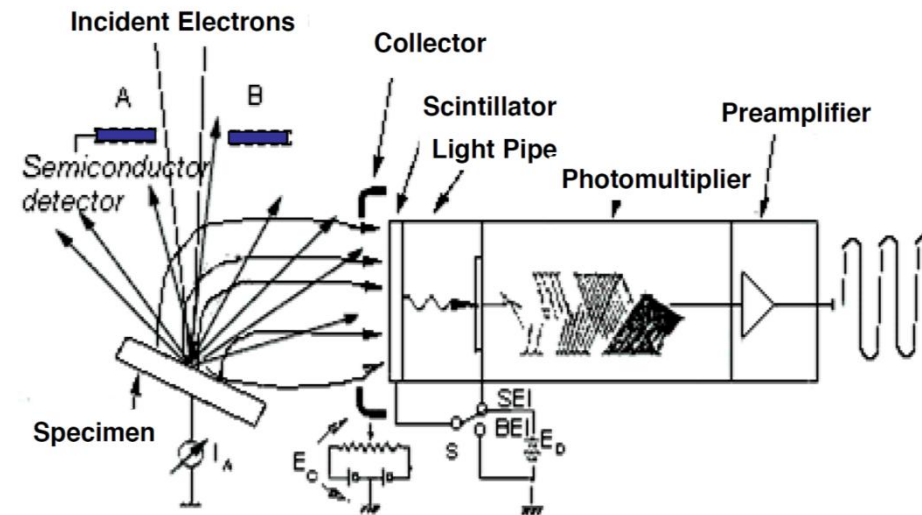
Side Mounted



In-Lens

What are the differences between these two images?

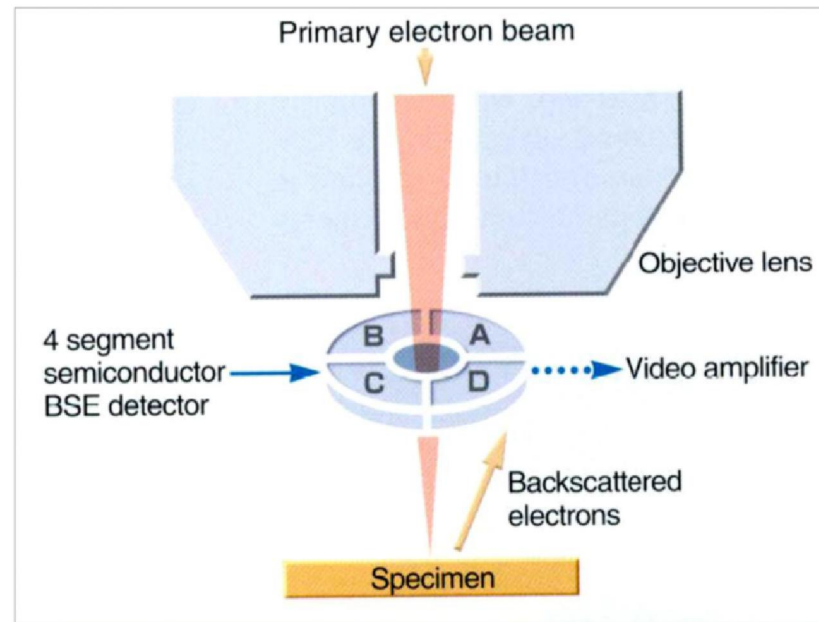
Secondary Electron Detector



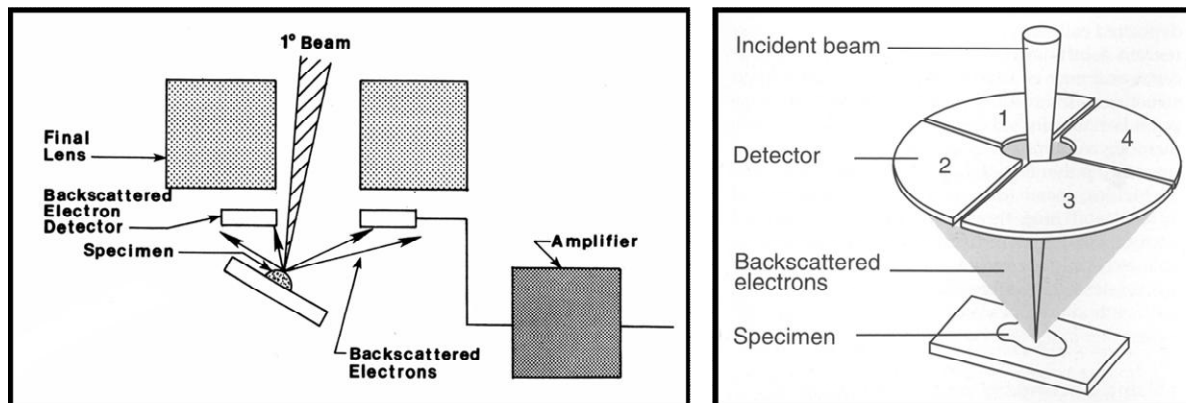
2. Backscattered Electron Detector (BSD) - The "Composition" Detector

- **What it detects:** High-energy electrons (> 50 eV, up to beam energy) that bounce out of the sample after elastic scattering.
- **Where they come from:** A larger interaction volume (tens to hundreds of nanometers deep).
- **How it works:** Usually a solid-state detector (a semiconductor diode) placed directly above the sample, often in a "annular" (ring) shape around the beam. When a backscattered electron hits the diode, it creates electron-hole pairs, generating a current.
- **Key physics:** The probability of backscattering increases with the **atomic number (Z)** of the element.
 - High-Z materials (e.g., gold, lead, tungsten) appear **bright**.
 - Low-Z materials (e.g., carbon, aluminum, biological tissue) appear **dark**.
- **What the image looks like:** Atomic number contrast (Z-contrast). A polished cross-section of a circuit board will show bright solder pads, medium-bright copper traces, and dark epoxy.
- **Best for:** Identifying different phases or elements, finding heavy metal particles, imaging uncoated or charging samples (BSD is less affected by charging).

Backscattered Electron Detector



Backscatter Detector



The most common design is a four quadrant solid state detector that is positioned directly above the specimen

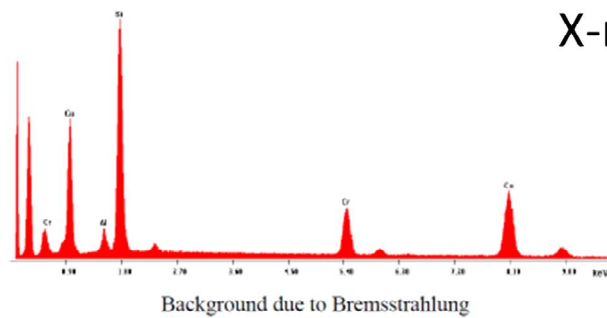
3. Energy-Dispersive X-ray Spectrometer (EDS or EDX)

- **What it detects:** Characteristic X-rays emitted when the beam ejects an inner-shell electron and an outer-shell electron falls in to fill the hole. Each element has a unique "fingerprint" of X-ray energies.
- **How it works:** A cooled [silicon drift detector](#) (SDD) [measures the energy of each](#) incoming X-ray and builds a spectrum ([counts vs. energy](#)).
- **What you get:** **Not an image** (though you can map it). You get:
 - A spectrum showing which elements are present.
 - A semi-quantitative composition (e.g., "This particle is 60% Si, 40% O" → likely SiO₂).
 - Elemental maps (color overlays on the SEM image).
- **Best for:** Identifying unknown materials, checking purity, elemental analysis.

SEM EDS and WDS

- Microanalysis [EDS and WDS](#) (material of an other course)
- Energy dispersive (EDS)
 - Operate just like a solar cell.
 - Charge generated by one X-ray
- Wavelength dispersive (WDS)
 - Bent LiF crystal acts like a conventional glass lens
 - Degree of deviation in bent crystal depends on X-ray wavelength

EDS spectrum



X-ray Spectroscopy

EDS: Energy Dispersive Spectroscopy
EDXS: Energy Dispersive X-ray Spectroscopy
X-EDS: X-ray Energy Dispersive Spectroscopy
EDX: Energy Dispersive X-ray analysis



4. Cathodoluminescence (CL) Detector

- **What it detects:** Photons (visible light, UV, IR) emitted when the electron beam excites certain materials (e.g., semiconductors, minerals, phosphors).
- **How it works:** A parabolic mirror or light guide collects the light and sends it to a photomultiplier tube or spectrometer.
- **What you get:** Information about electronic properties, defects, bandgap, and trace elements.
- **Best for:** Studying quantum dots, LED materials, and defects in semiconductors.

5. Scanning Transmission Electron Detector (STEM-in-SEM)

- **What it detects:** *Electrons that pass through* a very thin sample (like a TEM sample).
- **How it works:** A bright-field detector (collects unscattered electrons) and one or more annular dark-field detectors (collects scattered electrons).
- **What you get:** TEM-like images (bright-field and dark-field) at lower voltage (e.g., 30 kV) than a conventional TEM.
- **Best for:** Nanoparticles, thin films, biological sections, and when you need internal structure of thin specimens.

6. Electron Backscatter Diffraction Detector (EBSD)

- **What it detects:** Backscattered electrons that have been diffracted by the crystal lattice of the sample.
- **How it works:** A special phosphor screen and camera mounted at a steep angle (typically 70° tilt). The beam creates a "Kikuchi pattern" (a banded pattern unique to the crystal structure and orientation).
- **What you get:** Crystal orientation maps, grain size and boundary information, phase identification.
- **Best for:** Metals, ceramics, and geological samples—anything where crystal structure matters.

Sample Preparation in SEM

Sample preparation is one of the most critical steps in Scanning Electron Microscopy. A poorly prepared sample can ruin image quality, damage the microscope, or produce misleading results.

Fundamental Requirements for SEM Samples

Requirement	Why it matters
Vacuum compatibility	SEM operates under high vacuum (10^{-4} to 10^{-6} Pa). Samples must not outgas.
Conductivity	Non-conductive samples charge up, causing image distortion, beam deflection, and damage.
Stability	Samples must withstand electron beam without melting, cracking, or moving.
Size compatibility	Must fit within chamber limits (typically <50 mm diameter for standard stages).
No volatile components	Water, oil, or solvents evaporate → vacuum loss + contamination.

Sample Preparation

Sample Coating

Q: Why ?

A: Charging:

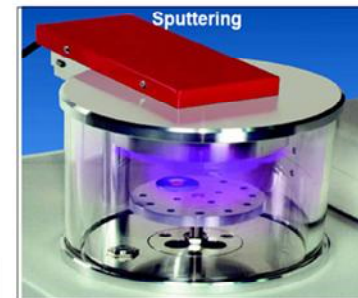
SEM works by scanning a beam of negatively charged electrons across the sample. On conductive samples, the charge flows to ground. On insulators, electrons accumulate on the surface.

Solutions:

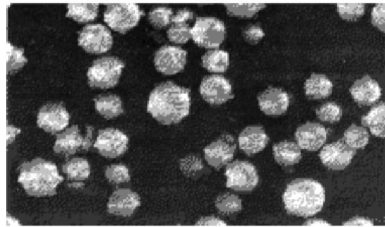
- Sputter coating with C, Cr, or Au-Pd
- Carbon tape, carbon paint, In foil

Coating methods:

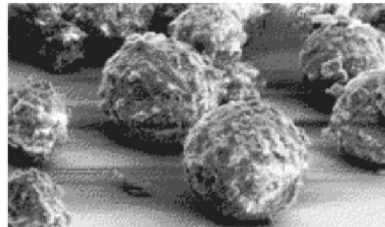
- **Sputter coating:** Most common for metals (Au, Pt, Au/Pd). Argon plasma knocks atoms from target onto sample.
- **Evaporation:** High vacuum thermal or e-beam evaporation. Cleaner but slower.
- **Carbon evaporator:** For carbon coating (EDX samples).



Sample Preparation – Toner Powder

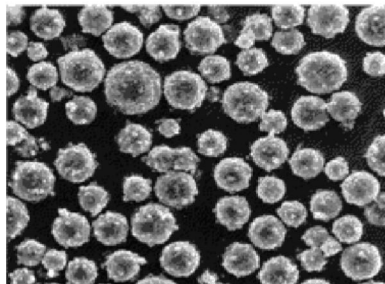


15 kV Tilt = 0 degrees 720 X

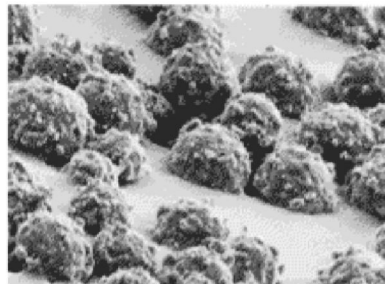


5 kV Tilt = 45 degrees 1,400 X

Sample charging
due to insufficient
electrical contact to
sample holder



5 kV Tilt = 0 degrees 720 X



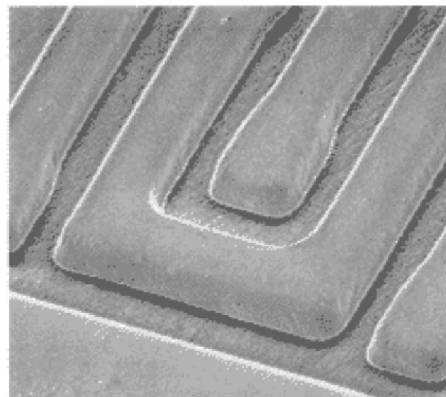
5 kV Tilt = 45 degrees 1,400 X

Properly prepared
sample

Influence of Sample Charging on Image Quality

Non-conductive Material

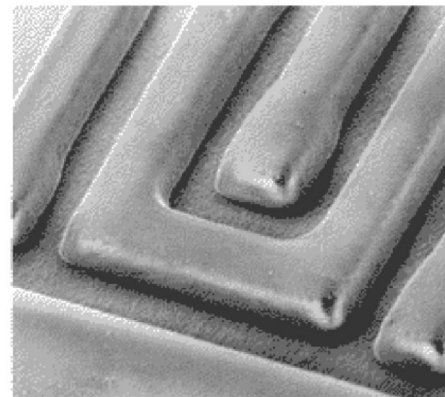
Image with No Surface Charging



1.0 kV

3,200 X

Image with Surface Charging

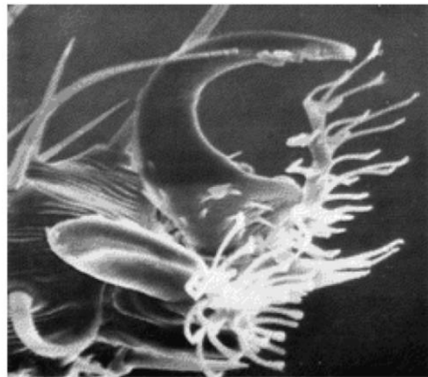


1.3 kV

3,200 X

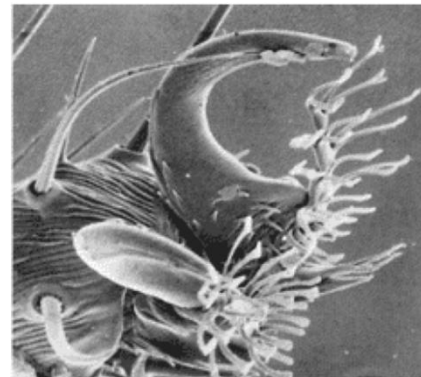
Biological Samples

Vinegar Fly Foreleg



10 kV

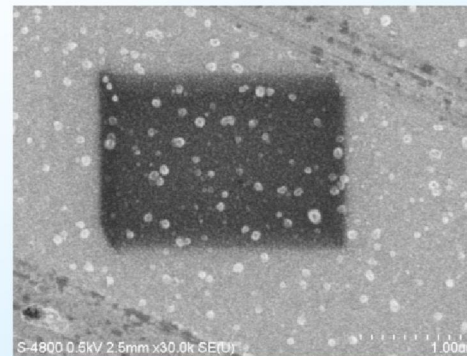
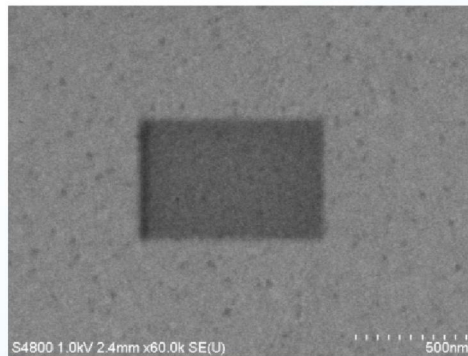
Sample charging causes extremes in image contrast at 10kV reducing detail



4 kV

Reducing beam voltage to 4 kV equalizes image contrast and improves detail

Sample Contamination



Residual gas or the presence of organic contaminants can affect imaging, reducing the sharpness of the image. Requires careful handling of specimens!

FESEM and ESEM

FESEM

A **FESEM** is simply an SEM that uses a **Field Emission Gun (FEG)** as its electron source instead of a conventional thermionic gun (tungsten or LaB₆).



A standard SEM is typically used for low-to-medium magnification (10-150,000X) imaging of conductive samples, usually metals.

For non-conductive samples, a conductive coating of carbon, gold, chromium, etc. should be applied to avoid charging effects.

A variable pressure SEM (low vacuum or environmental) is used for non-conductive specimens like glass, polymers/paint, and biological materials.

A field-emission SEM (FESEM) is used for medium-high magnification (20,000- 800,000X) and high resolution at low electron beam accelerating voltages (0.5-2 kV) resulting in excellent surface texture images even on non-conductive materials.

Feature	SEM (Thermionic)	FESEM (Field Emission)
Electron Source	Heated tungsten filament or Lanthanum Hexaboride (LaB ₆) crystal	Sharp tungsten tip under high electric field (cold or thermally assisted)
Operating Temperature	~2700 K (Tungsten) or ~1900 K (LaB ₆) – runs hot	Room temperature or slightly warm (very low thermal drift)
Brightness	Lower (limits resolution and signal at high magnification)	100–1000 times higher (very bright, coherent beam)
Beam Diameter	Larger (nanometer scale)	Much smaller (sub-nanometer possible)
Resolution	Typically 3–10 nm (standard SEM)	Typically 0.5–2 nm (much higher)
Vacuum Requirement	High vacuum (easier to achieve)	Ultra-high vacuum (UHV, more demanding)
Sample Charging	More problematic (often requires conductive coating like gold/palladium)	Less problematic; can image uncoated insulators at low voltages
Cost & Maintenance	Lower initial cost; filament replacement is cheap but frequent (50–200 hours for W)	Higher initial cost; tip lasts longer (1000s hours) but requires UHV maintenance

Environmental SEM: ESEM

- Traditional SEM chamber pressure: $\sim 10^{-6}$ Torr
- ESEM: 0.08 – 30 Torr
- Various gases can be used
- Requires different SE detector

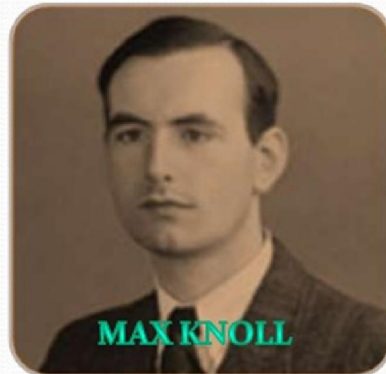
Why ESEM?

- To image challenging samples such as:
 - insulating samples
 - vacuum-sensitive samples (e.g. biological samples)
 - irradiation-sensitive samples (e.g. thin organic films)
 - wet samples (oily, dirty, greasy)
- To study and image chemical and physical processes in-situ such as:
 - mechanical stress-testing
 - oxidation of metals
 - hydration/dehydration (e.g. watching paint dry)

Feature	Standard SEM	ESEM (Environmental SEM)
Chamber Pressure	High Vacuum (10^{-4} to 10^{-6} Pa)	Low Vacuum to Moderate Gas Pressure (10 to 4000 Pa)
Sample State	Must be completely dry and vacuum-stable	Can be wet, hydrated, oily, or outgassing
Sample Conductivity	Must be conductive or coated (Au/Pt/C)	Can be non-conductive (no coating needed)
Water/Liquids	Impossible (water boils instantly in vacuum)	Possible (water remains liquid at room temp under gas pressure)
Detection Method	Everhart-Thornley (ET), In-lens, etc.	Gaseous Secondary Electron Detector (GSED) + Pressure Limiting Apertures (PLAs)
Resolution	Excellent (1–10 nm)	Good but slightly lower (3–20 nm, due to beam scattering in gas)

TEM

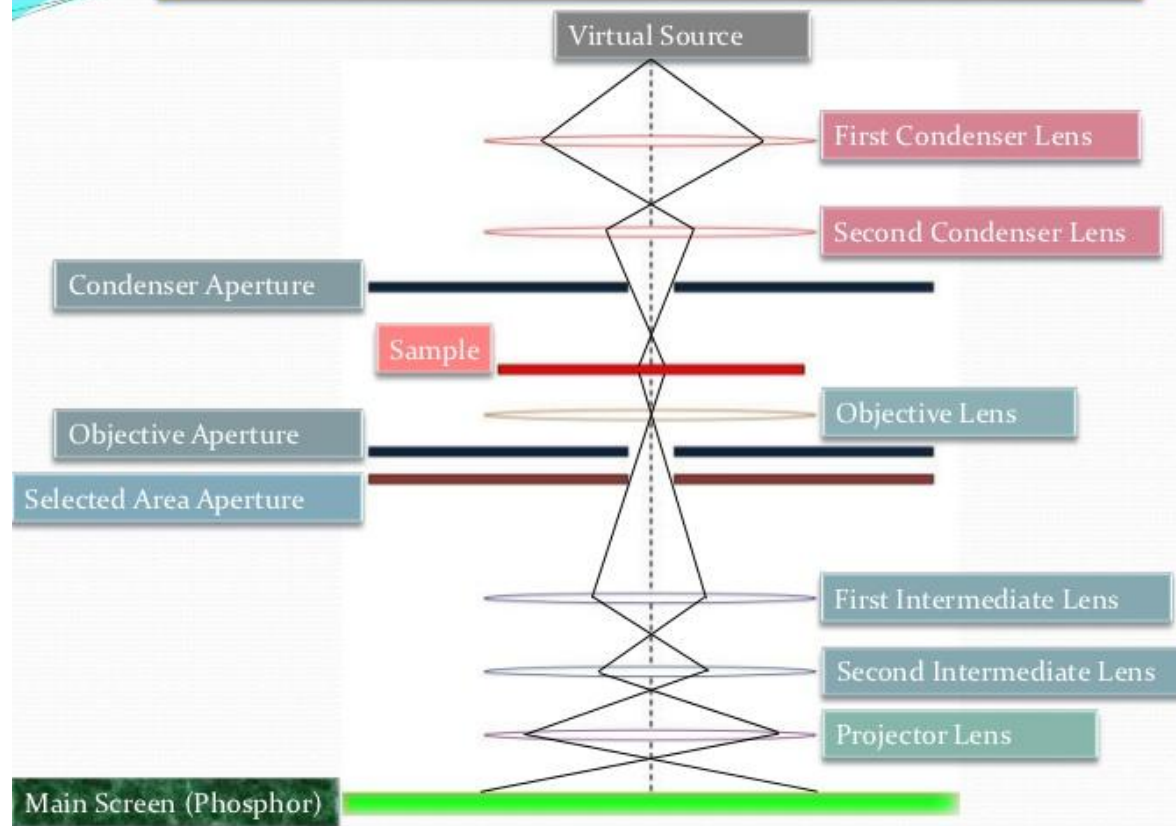
➤The **transmission electron microscope (TEM)** was the first type of Electron Microscope to be developed and is patterned exactly on the light transmission microscope except that a focused beam of electrons is used instead of light to "see through" the specimen. It was developed by Max Knoll and Ernst Ruska in Germany in 1931.



TRANSMISSION ELECTRON MICROSCOPY (TEM)

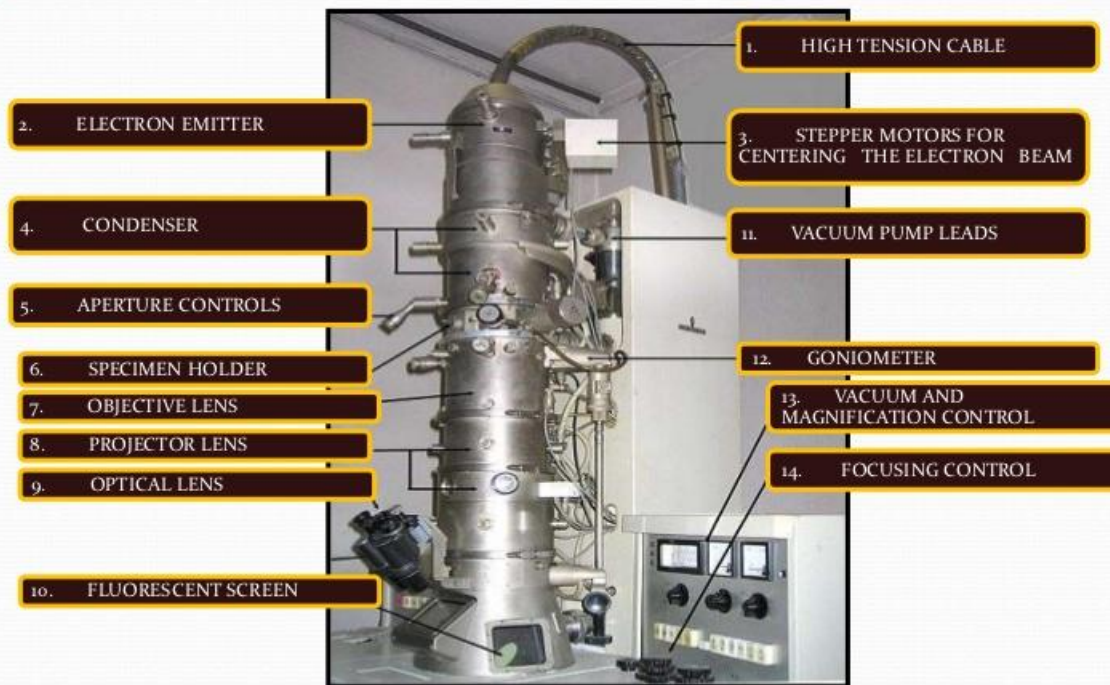
- **Transmission electron microscopy (TEM)** is a microscopy technique whereby a beam of electrons is transmitted through an ultra thin specimen, interacting with the specimen as it passes through. An image is formed from the interaction of the electrons transmitted through the specimen; the image is 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.

DIAGRAM TO REPRESENT TEM'S WORKING

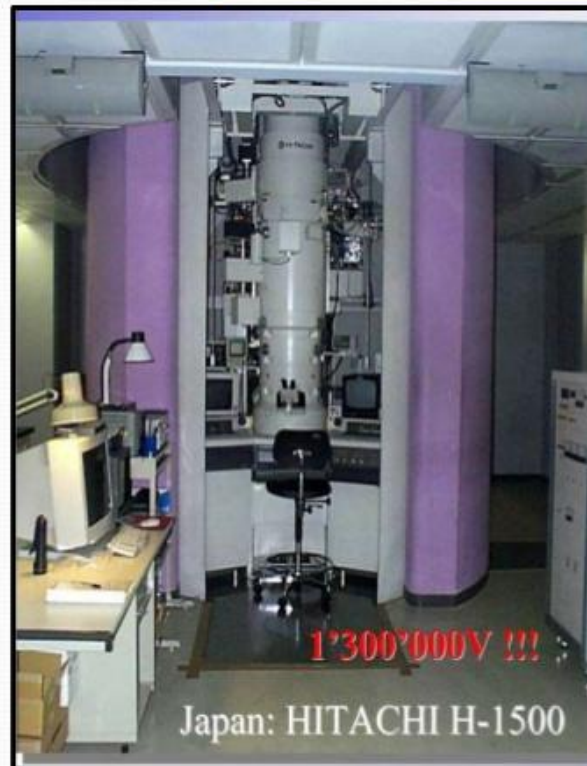


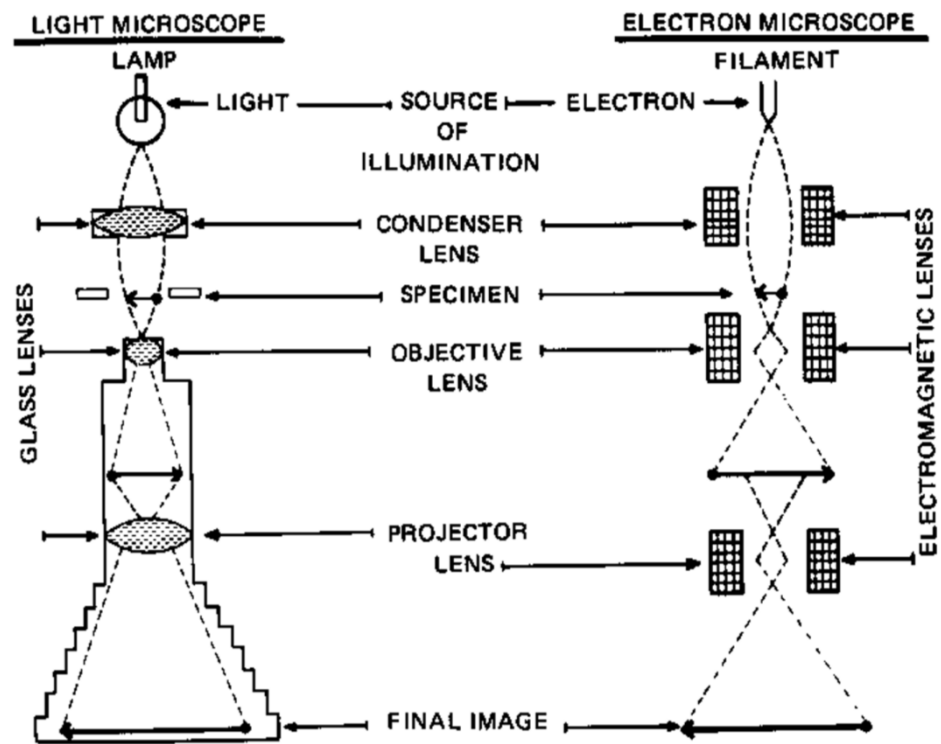
Component	Function
Electron gun	Produces electrons via thermionic (LaB_6) or field emission (FEG)
Condenser lenses	Control beam convergence, spot size, and illumination area
Sample (specimen)	Must be <100–200 nm thick for electrons to transmit
Objective lens	The most important lens — forms the first image and diffraction pattern
Objective aperture	Selects which scattered electrons contribute to the image
Intermediate & projector lenses	Magnify the image onto the detector
Detector	Converts electrons to light (scintillator) or directly to digital signal

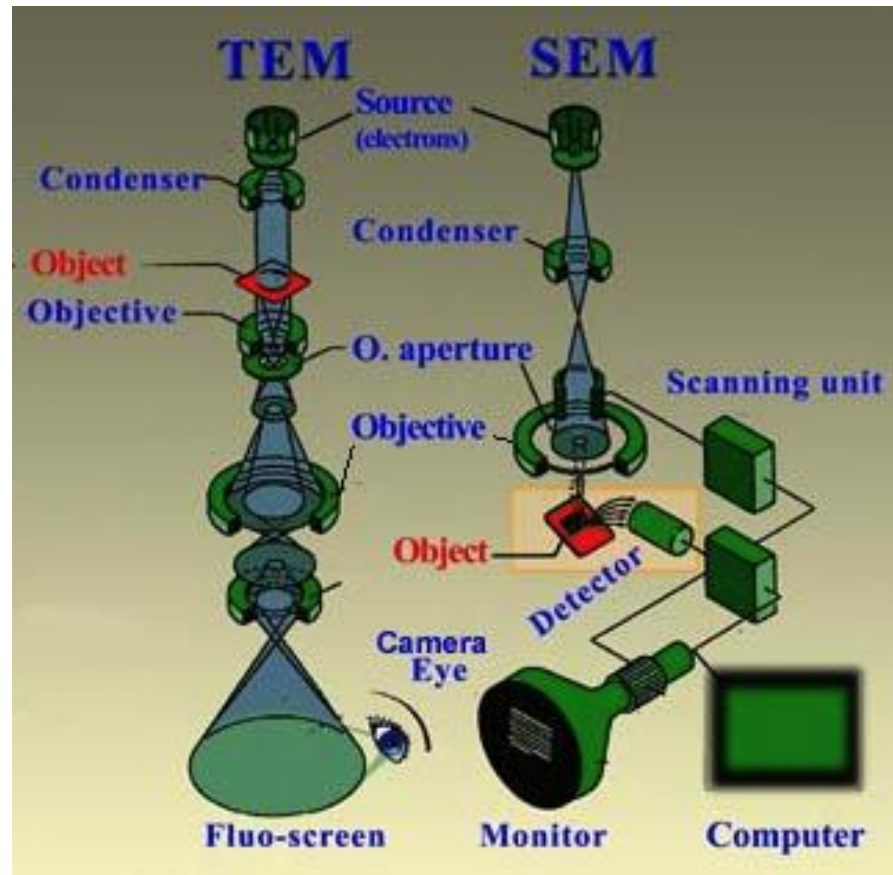
DIFFERENT COMPONENTS OF TEM



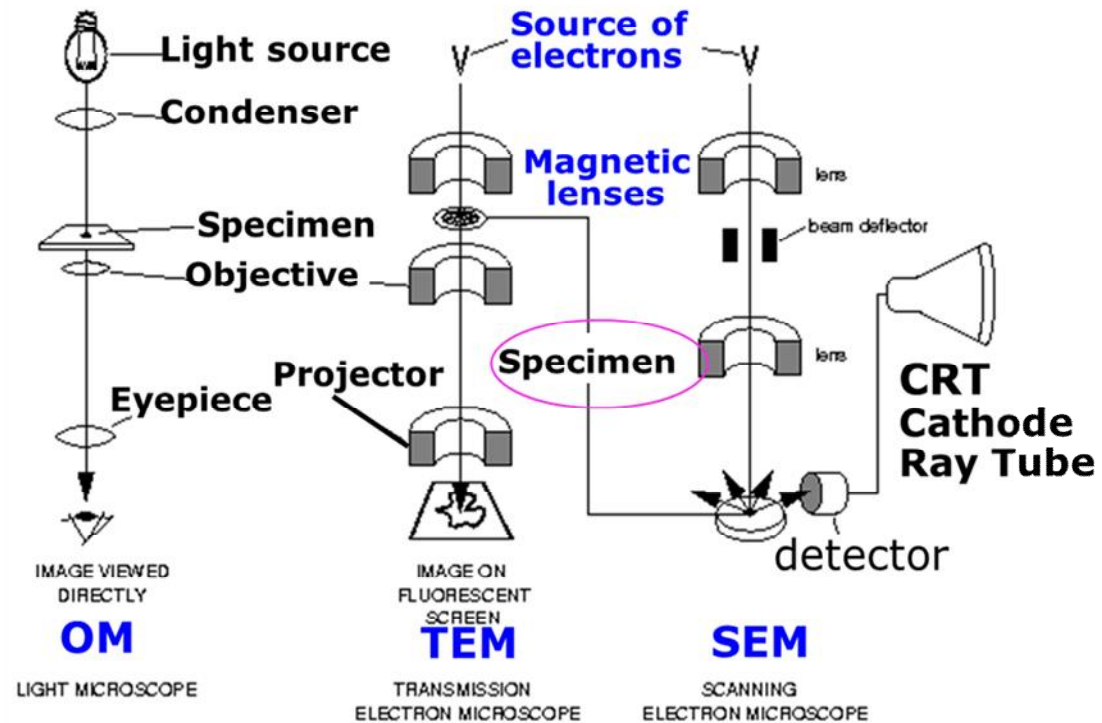
SOME TYPICAL TEM MODELS



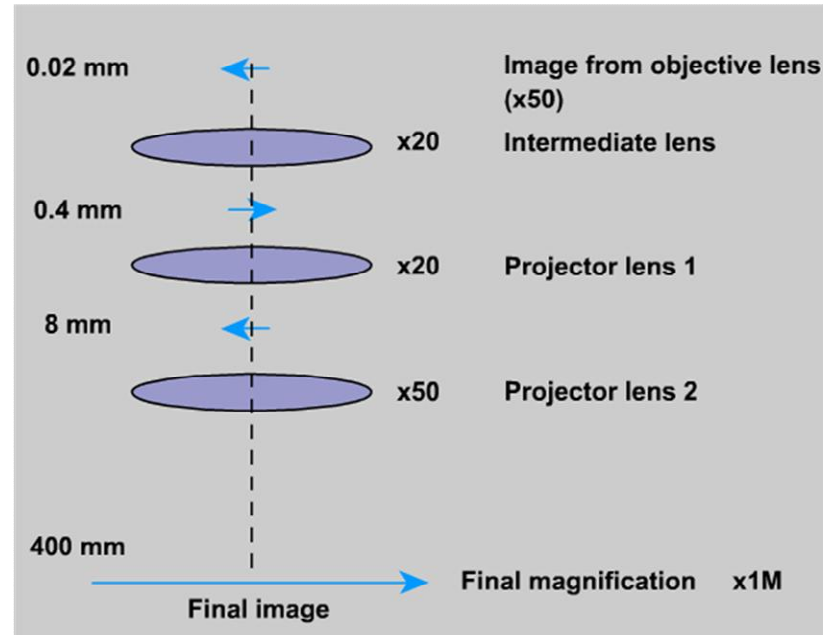




Comparison of OM, TEM and SEM



Magnification in TEM



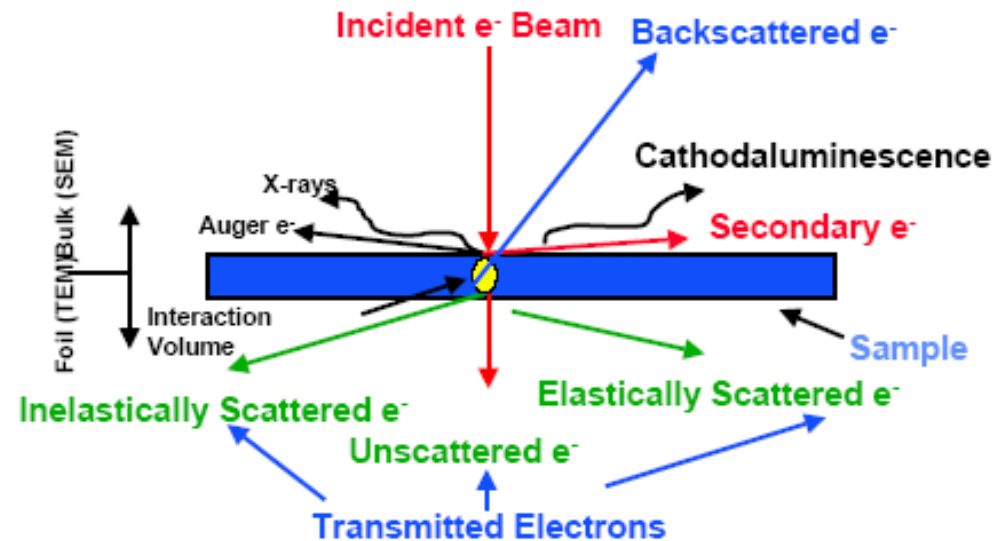
$$M_{\text{ob}} \times M_{\text{int}} \times M_{\text{proj}} = \text{Total Mag}$$

Depends on the magnification, some lens may not be used

Lens group	Function	Explanation
Condenser 1 (C1)	Controls beam crossover (spot size)	Smaller spot = less current = less damage but dimmer image
Condenser 2 (C2)	Controls beam convergence angle (illumination)	Large convergence = more diffraction but worse contrast
Objective	Forms the first image and diffraction pattern	Most important lens — focus changes contrast dramatically
Intermediate	Switches between image and diffraction modes	A few percent change toggles between modes!
Projector	Final magnification	Rarely touched by user — set by software

Electron-Solid Interactions

When an electron beam strikes a sample, a large number of signals are generated.



We can divide the signals into two broad categories:

a) electron signals, b) photon signals

Electron-Sample Interactions (What the Beam Does)

When a high-energy electron hits a thin sample, several things can happen:

Interaction	Result	What we detect
No interaction (transmitted straight)	Bright field signal	Mass-thickness contrast
Elastic scattering (changes direction, no energy loss)	Diffraction pattern	Crystallographic info, phase contrast
Inelastic scattering (loses energy)	X-rays, secondary electrons, energy loss	EDS, EELS (composition, bonding)
Absorption (rare in TEM)	Heating, damage	Avoid if possible

Key concept: The *objective aperture* allows us to choose which of these electrons form the image.

Bright Field (BF): You collect only the direct, **unscattered electron beam**.

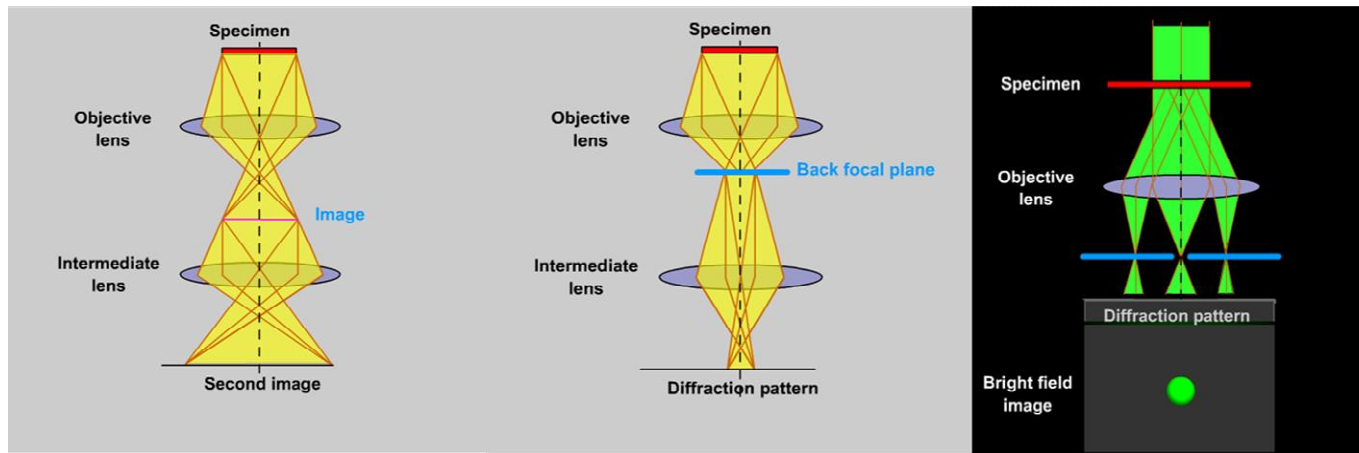
Result: The image is formed by the direct beam. Areas that scatter electrons (dense, crystalline) appear **dark**. Areas that don't scatter (vacuum, holes, amorphous carbon) appear **bright**.

Dark Field (DF): You block the direct beam and collect **only the scattered electrons**.

Result: The direct beam is now blocked (it hits the aperture). Only electrons scattered to that specific angle form the image. Crystal planes that caused that scatter appear **bright**; everything else is **dark**.

Feature	Bright Field (BF)	Dark Field (DF)
What is collected?	Direct, unscattered beam	Scattered beams (diffracted or high-angle)
What is blocked?	Scattered beams	Direct beam
Image appearance	Dense/crystalline = Dark Hole/amorphous = Bright	Crystalline/defects = Bright Amorphous/holes = Dark
Contrast mechanism	Mass-thickness & diffraction	Primarily diffraction (or Z-contrast in STEM)
Resolution	Higher (more electrons used)	Lower (fewer electrons)
Best for...	General morphology, holes, edges	Crystals, defects, nanoparticles, heavy atoms

TEM imaging modes



Bright field/dark field depends on the aperture position. Modern way for diffraction tilts the beam instead of moving the aperture



TEM SAMPLE PREPARATION

- *Cleaning the surface of the specimen*

The proper cleaning of the surface of the sample is important because the surface can contain a variety of unwanted deposits, such as dust, silt, and detritus, media components, or other contaminants.

The best way to clean the surface of specimen from contaminants is to carefully rinse them three times for 10 min in 0.1 M cacodylic acid buffer (pH 7.3) at room temperature.

Sample Preparation

The TEM sample should be thin (due to the absorption of the electron in the material), so special care must be taken while cutting **a thin slice of the metal** so that the **specimen is not deformed during its preparation**. This is because plastic deformation can introduce **unwanted structural defects** in the microstructure that are visible in TEM images.

The various techniques are:

- a) SPARK CUTTER:** - In this, electric discharge between a wire and the specimen are used to cut the metal by removing small particles of metal from the surface of the specimen.

- b) FOCUSED ION BEAM (FIB):**- A thin slice of the sample was cut by an ion beam on a scanning ion microscope. The main advantage of this method is that it allows for selective thinning at desired locations by cutting trenches in the sample.

TEM - transmission electron microscopy

Specimen preparation

Types

replicas

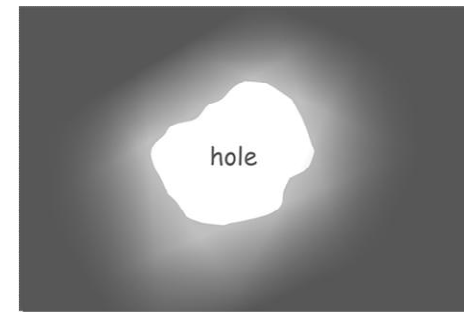
films

slices

powders, fragments

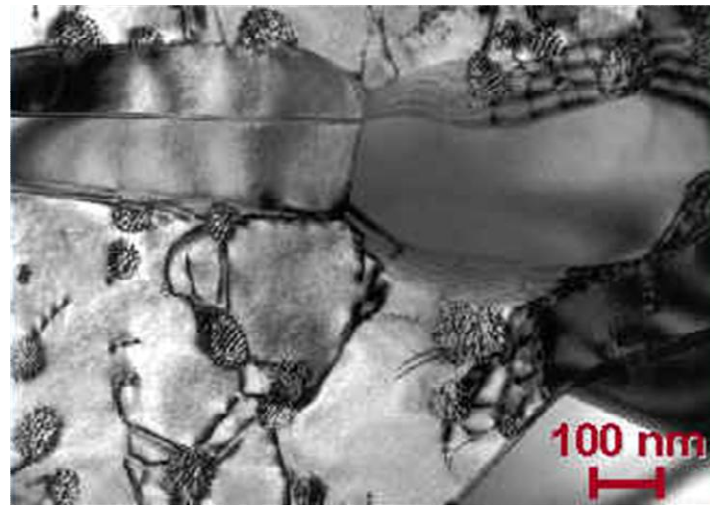
foils

examine region
around perforation



TEM - transmission electron microscopy

Examples



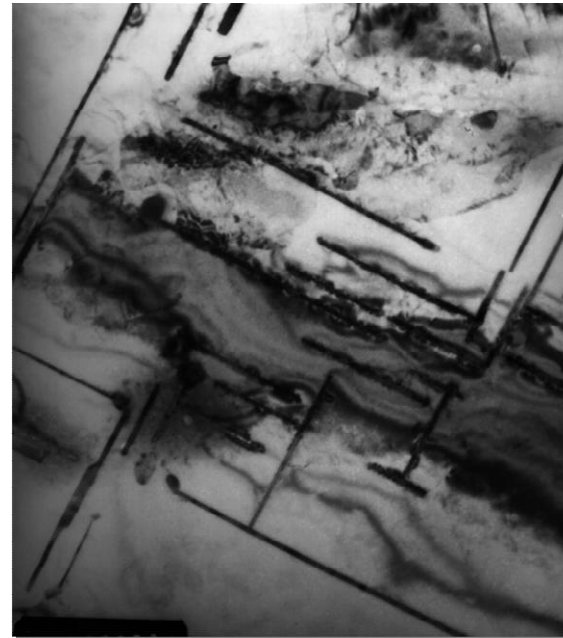
Matrix - β' -Ni₂AlTi

Precipitates - twinned L1₂ type γ' -Ni₃Al

TEM - transmission electron microscopy

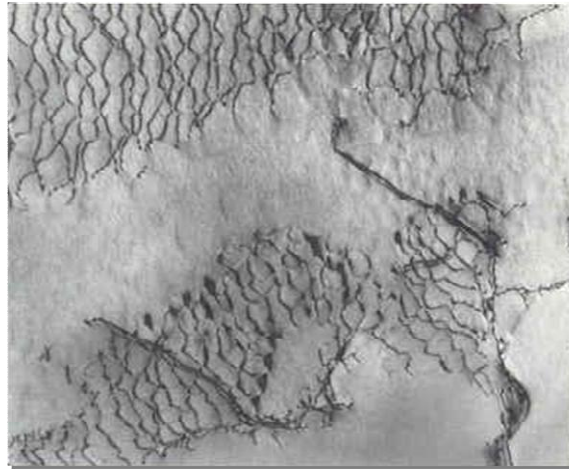
Examples

Precipitation in an
Al-Cu alloy

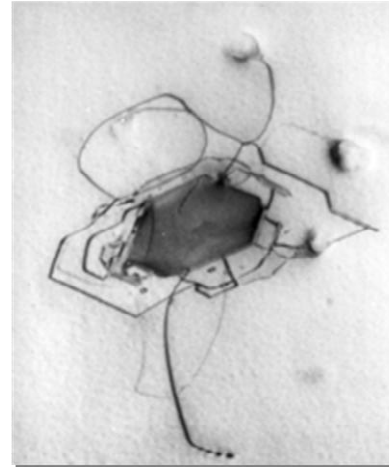


TEM - transmission electron microscopy

Examples

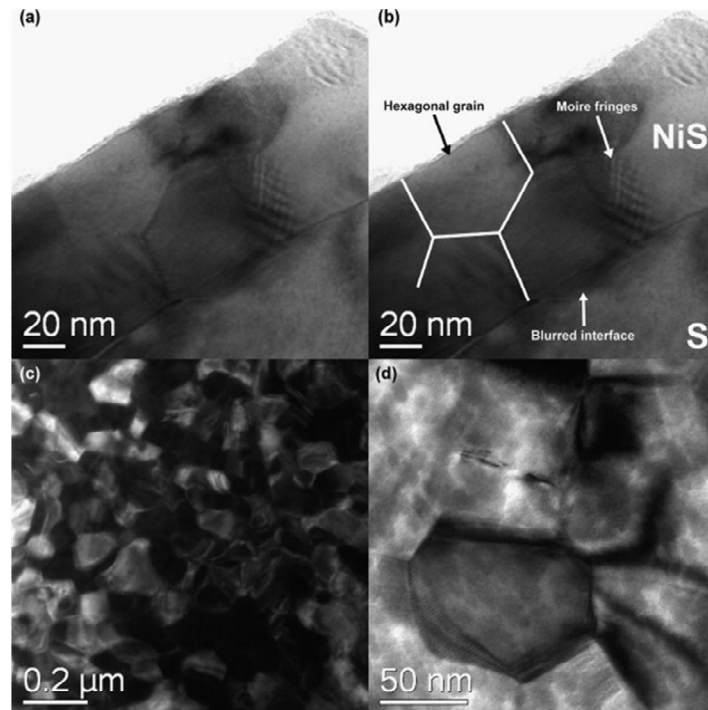


dislocations
in superalloy



SiO₂ precipitate
particle in Si

NiSi thin film



Results from TEM analysis of NiSi thin films: (a) XTEM highlighting equiaxed grains in the NiSi film in which Moiré (interference) fringes due to orientation differences between grains can be observed; (b) notable features in the as-obtained image (a) are indicated; (c) plan view, elastic hollow cone dark field image of the film, highlighting individual grains with diameters of 60–200 nm; and (d) plan view TEM image showing polygonal NiSi grains.

M. Bhaskaran et al. / Micron 40 (2009) 11–14

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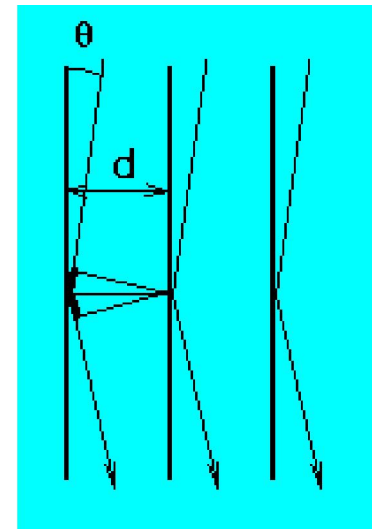
Diffraction

Use Bragg's law - $\lambda = 2d \sin \theta$

But λ much smaller

($0.0251\text{\AA}$ at 200kV)

if $d = 2.5\text{\AA}$, $\theta = 0.288^\circ$



Types of Diffraction Patterns in TEM

Pattern Type	Appearance	Formation Mechanism	Key Information Obtained	Typical Materials	Common Pitfall / Note
Single Crystal (Spot Pattern)	Array of discrete, sharp spots	Parallel beam diffracted by a single crystal grain	Crystal structure (fcc/bcc/hcp), zone axis orientation, lattice parameters	Metals, ceramics, large grains, epitaxial films	Misindexing due to ignoring systematic absences
Polycrystalline (Ring Pattern)	Concentric, continuous circles	Superposition of spot patterns from randomly oriented grains	Phase identification (d-spacings from ring radii), grain size (ring sharpness), texture (non-uniform intensity)	Nanomaterials, powders, polycrystalline thin films	Ring broadening from small grains or strain
Nanocrystalline	Broad, diffuse rings	Very small grains (<10 nm) cause significant reciprocal lattice point broadening	Average crystallite size, confirmation of nanocrystalline state	Early-stage nucleation, ball-milled powders, some thin films	Can be mistaken for amorphous if too broad
Amorphous (Halo)	One or two very broad, featureless halos	No long-range periodic order → no sharp Bragg peaks	Absence of crystallinity, average nearest-neighbor distance (from halo position)	Polymers, oxide glasses, amorphous silicon, most biomaterials	Halo position is <i>not</i> equivalent to a d-spacing
Kikuchi Pattern	Pairs of parallel lines crossing the pattern	Inelastically scattered electrons that subsequently Bragg diffract	Ultra-precise orientation (<<0.1°), crystal perfection assessment, lattice parameter (via HOLZ)	Single crystals, deformed metals, semiconductors	Sharp lines = good crystal; diffuse/absent = high defect density or damage

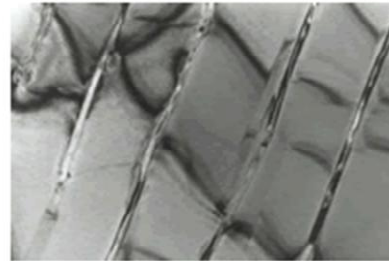
Types of Diffraction Patterns in TEM

Pattern Type	Appearance	Formation Mechanism	Key Information Obtained	Typical Materials	Common Pitfall / Note
Higher-Order Laue Zone (HOLZ)	Faint ring of discrete spots (or lines in CBED) at large radius	Diffraction from reciprocal lattice planes not parallel to the beam	3D reciprocal lattice information, precise lattice parameter (0.1% strain), local composition	Thicker single crystals, alloys, strained layers	Requires careful pattern indexing & often simulation
Convergent Beam (CBED)	Discs (not spots) with internal intensity variations (HOLZ lines)	Focused, convergent beam scanned over a small probe (<10 nm)	Space group determination, sample thickness, local strain, crystal polarity, site occupancy	Advanced structural analysis, semiconductors, ferroelectrics	Complex to interpret; requires simulation tools (e.g., JEMS, multislice)
Double Diffraction Pattern	Extra spots at vector sums of allowed reflections	A diffracted beam acts as an incident beam for another set of planes	Can appear as "forbidden" spots — warning: do not misinterpret as a new phase	Graphite, oxides, two-phase materials, thicker samples	Always check if extra spots = sum of two strong spots
Twin Pattern	Extra spots at rational fractions (e.g., $1/3(422)$ in fcc)	Two crystal orientations related by a twin operation	Twin law, twin boundary orientation	Deformed metals, some ceramics (e.g., calcite), some nanoparticles	Can be confused with double diffraction; check symmetry
Moiré Pattern (real-space fringes, not a diffraction pattern per se)	Periodic fringes superimposed on the image	Overlap of two crystals with slightly different d-spacings or orientations	Misfit, twist angle (e.g., in bilayer graphene), heteroepitaxy	2D materials, epitaxial interfaces, thin overlayers	Fringe spacing is <i>not</i> a lattice spacing — it's a beat phenomenon

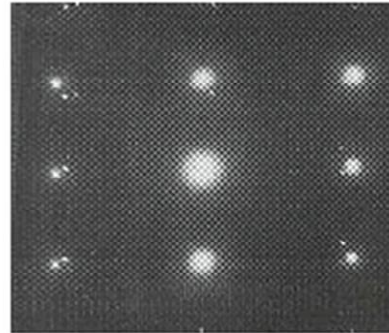
TEM - transmission electron microscopy

Examples

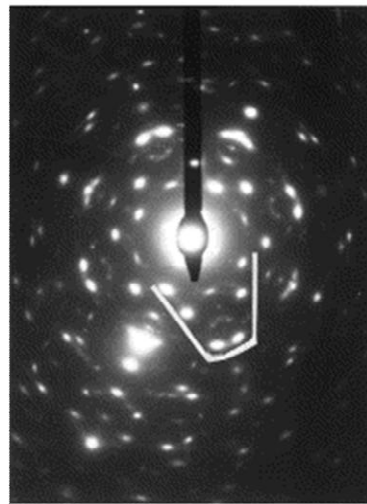
lamellar Cr_2N
precipitates in
stainless steel



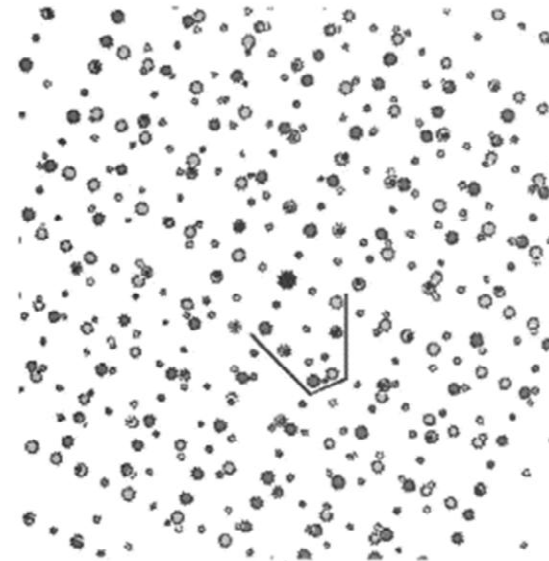
electron
diffraction
pattern



Electron diffraction



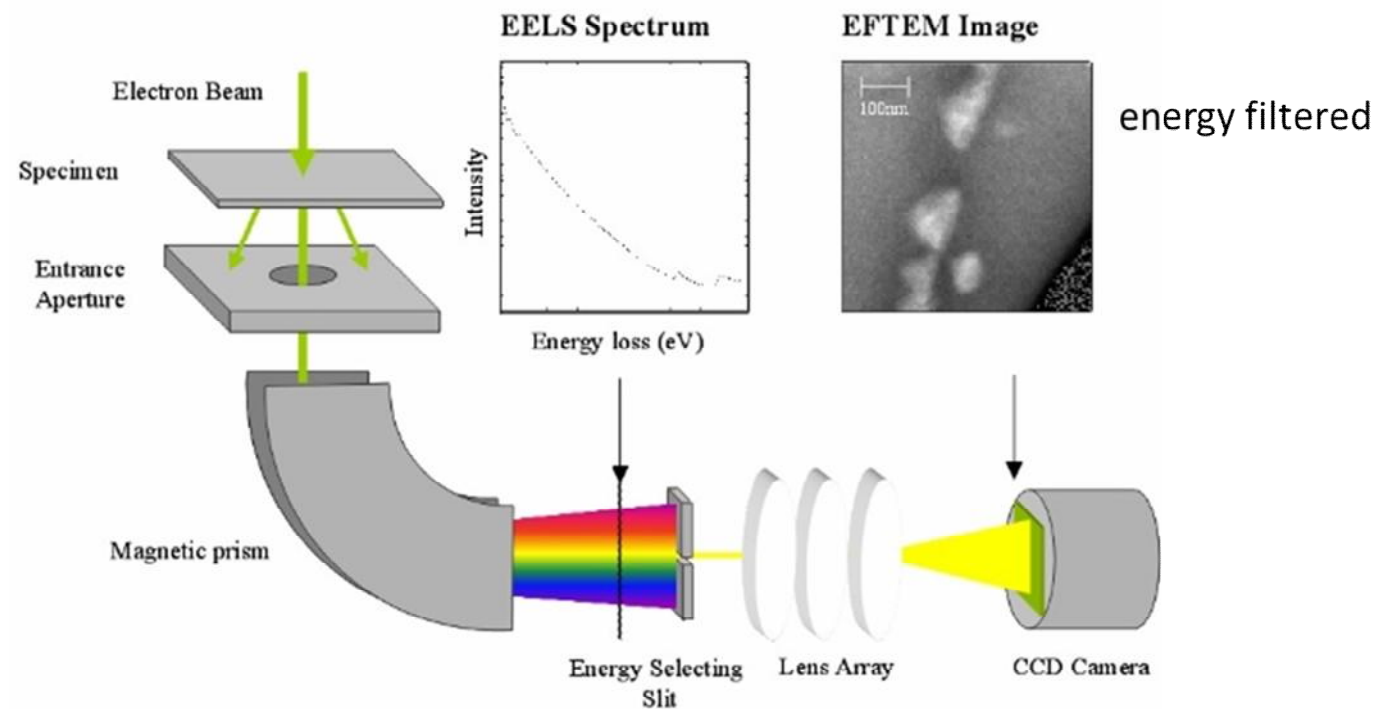
a



b

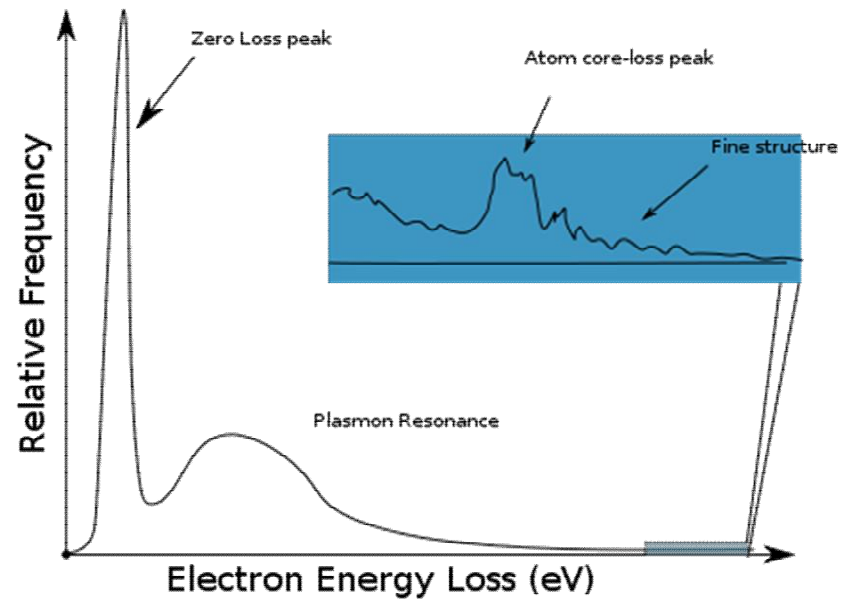
Measured diffraction pattern of a plan-view prepared $\text{ReSi}_{1.75}$ film on Si (100) (— — — guiding line for orientation); (b) theoretical diffraction diagram of $\text{ReSi}_{1.75}$ with zone axis $[0\ 1\ 0]$ and four superposed patterns, each turned around 45° .

Electron energy loss spectroscopy EELS



Electron energy loss spectroscopy EELS

- Elemental analysis
 - light elements C →
 - 3d transition metals
 - Sc, Zn
- chemical bonding e.g.
carbon sp^2/sp^3



Electron energy loss spectroscopy EELS

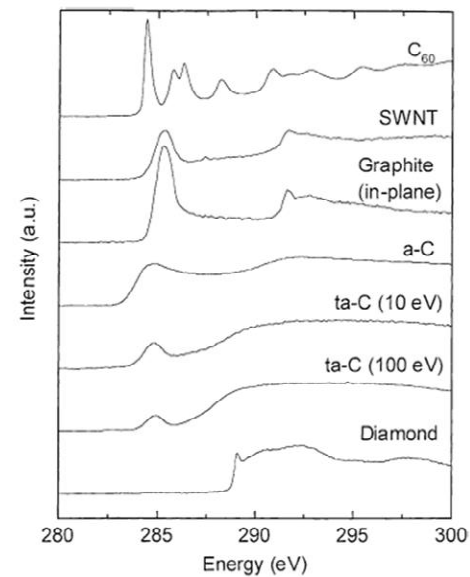
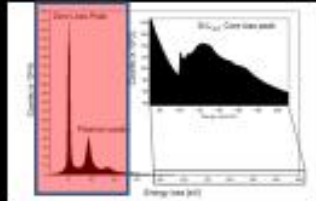
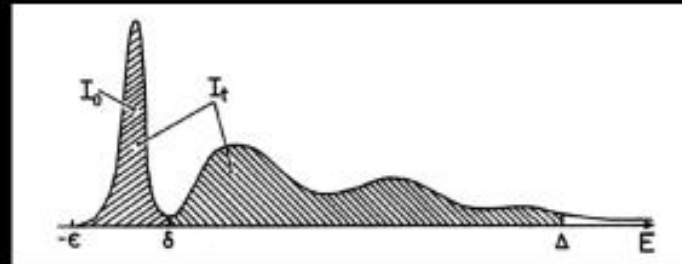


Fig. 29. Carbon K edge electron energy loss spectra of various carbon phases, after Waidmann et al. [196].

S. Waidmann, M. Knupfer, J. Fink, B. Kleinsorge, J. Robertson, J. Appl. Phys. 89 (2001) 3783.



Low-Loss EELS: Thickness



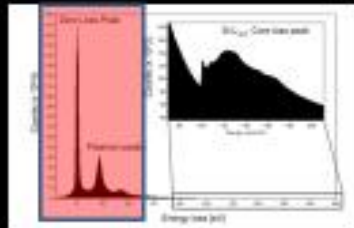
$$t = \lambda_p \frac{I_p}{I_0}$$

t = thickness

λ_p = plasmon mean free path

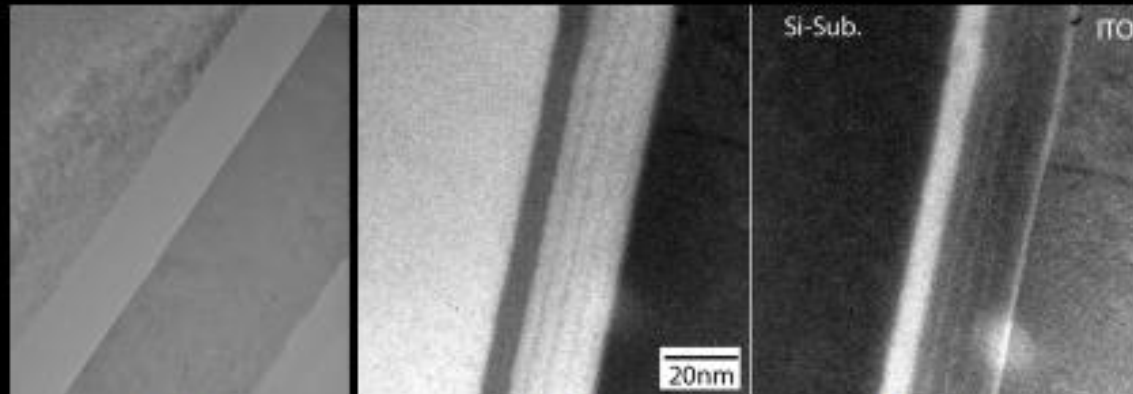
I_p = Intensity of the plasmon peak

I_0 = Intensity of the zero loss peak



Low-Loss EELS: Energy filtering

**EFTEM imaging of Si/aSi/ITO (Indium Tin Oxide)
stack sample for REC**



TEM image

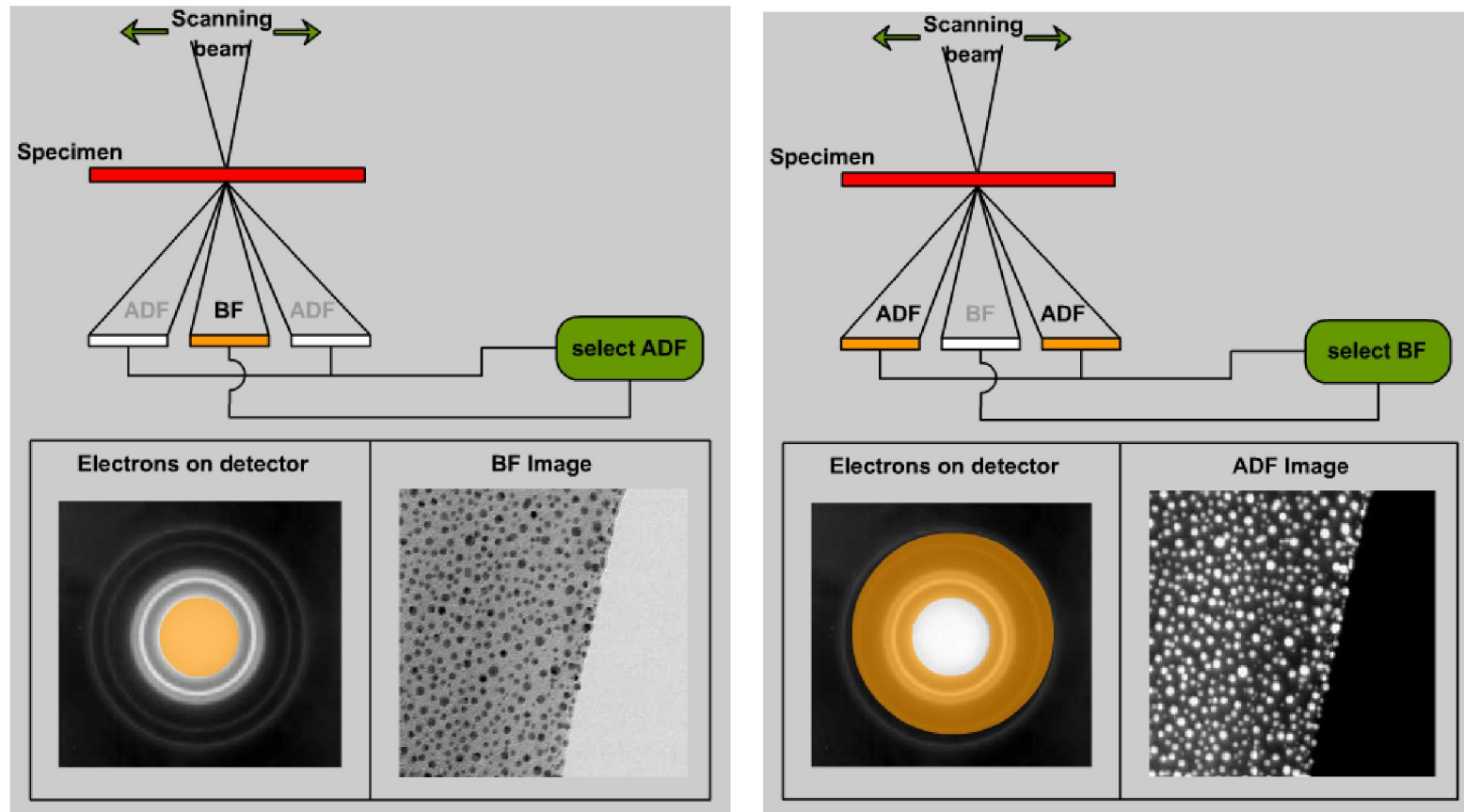
EFTEM (16 eV)

EFTEM (23 eV)

References

- **Fundamentals of materials Science and Engineering – William D. Callister**
- **Physical Metallurgy – Robert W. Cahn**
- **Physical Metallurgy and Advanced Materials – R. E. Smallman**
- **Physical Metallurgy Principles – Robert E. Reedhill**
- **Scanning electron microscope – S. pramanik, H. Kumar gupta, C. singh**

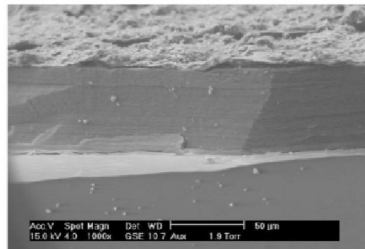
STEM image



Bright and dark field STEM image of Au particles on a carbon film

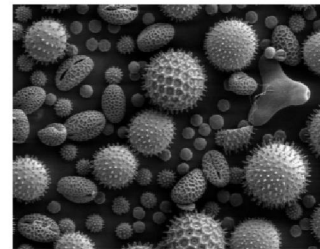
What is the differences between this dark field and the previous one?

Scanning Electron Microscopy



Metal corrosion layer

Deer tick



Pollen

Three different imaging modes: secondary electrons (low energy electrons from the surface of the material), backscattered electrons (more from bulk) and x-rays (from near-surface region).

Probes used

- **Visible light**
- **X-ray**
 - X-ray diffraction (XD)
 - X-ray photo electron spectroscopy (XPS)
- **Neutron**
 - Neutron diffraction (ND)
- **Ion**
 - Secondary ion mass spectrometry (SIMS)
 - Cleaning and thinning samples
- **Electron**
 - Scanning electron microscopy (SEM)
 - Transmission electron microscopy (TEM)
 - Electron holography (EH)
 - Electron diffraction (ED)
 - Electron energy loss spectroscopy (EELS)
 - Energy dispersive x-ray spectroscopy (EDS)
 - Auger electron spectroscopy (AES)