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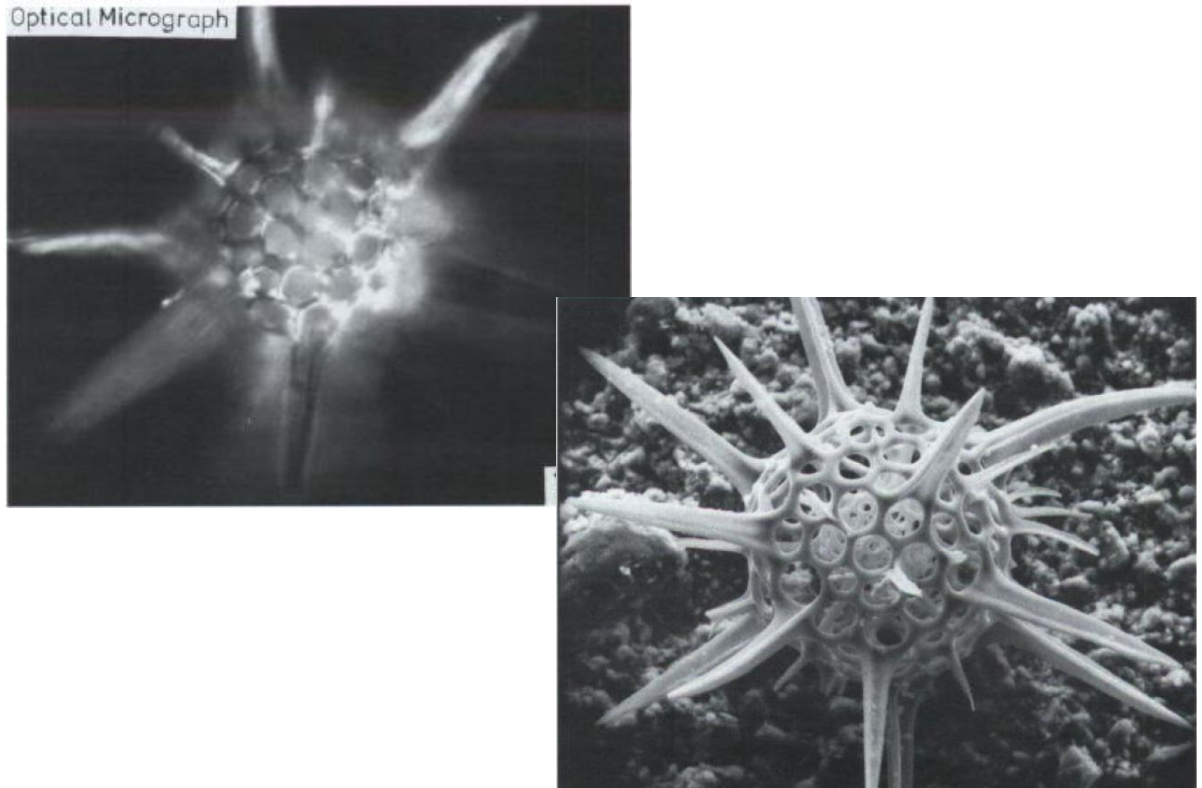
Scanning Electron Microscopy



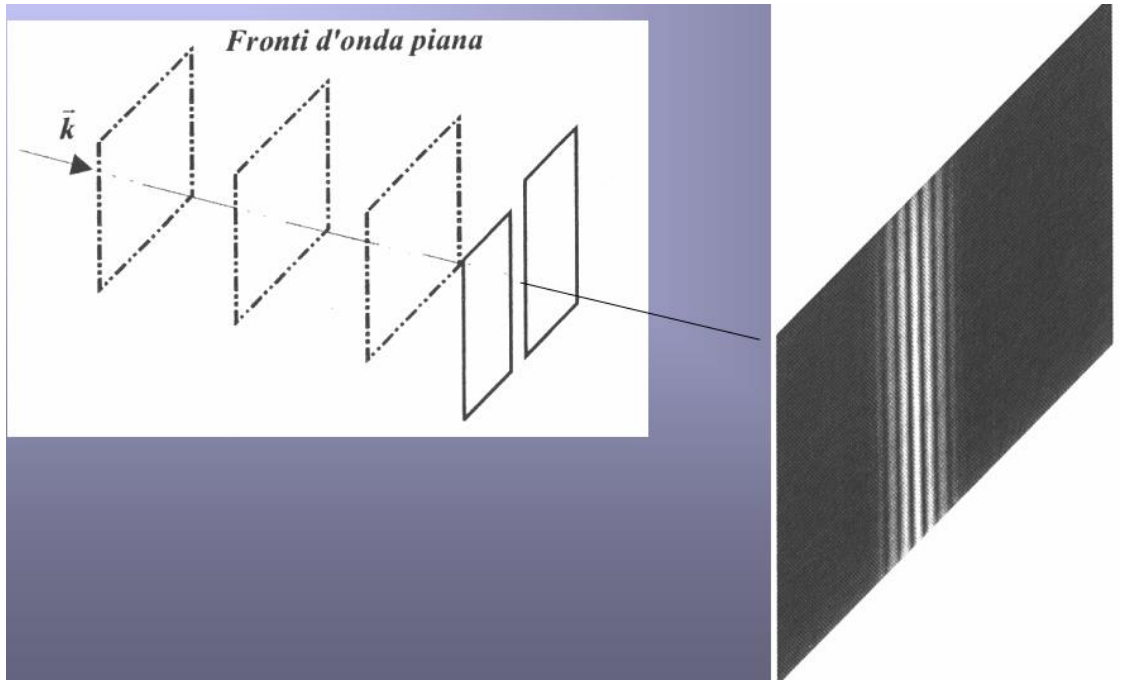
Literature:

1) L. Reimer, Scanning Electron Microscopy, ***Cap. 1****,* ***Cap. 2,*** *Cap. 3, Cap. 4, Cap. 5, Cap. 6, Cap. 10*

2) J. Goldstein et al., Scanning Electron Microscopy and X-ray Microanalysis, ***Cap. 1****, Cap. 2, Cap. 3, Cap. 4,* ***Cap. 5****, Cap. 6, Cap. 7, Cap. 8; Cap. 9,*

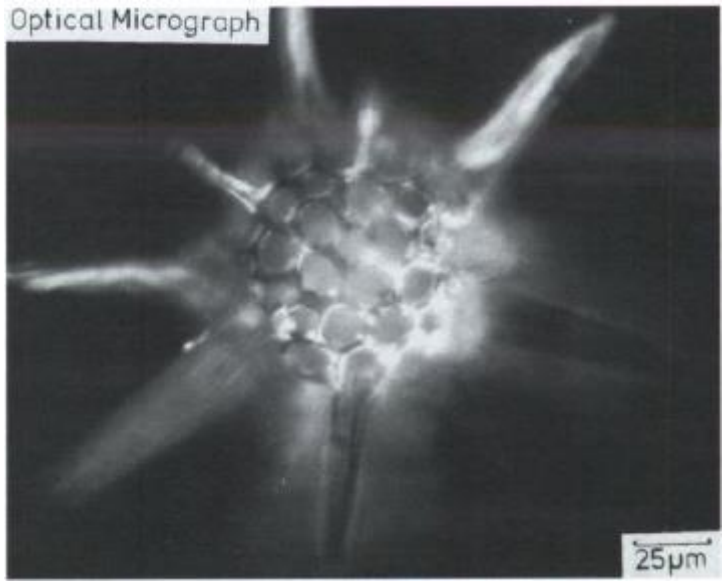
Microscopio elettronico

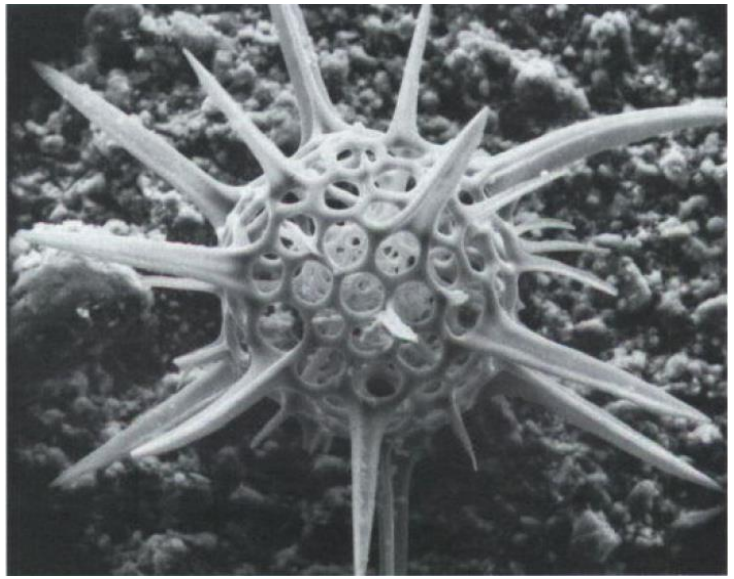
Il fenomeno della diffrazione limita l'osservazione di oggetti troppo piccoli



Microscopio elettronico

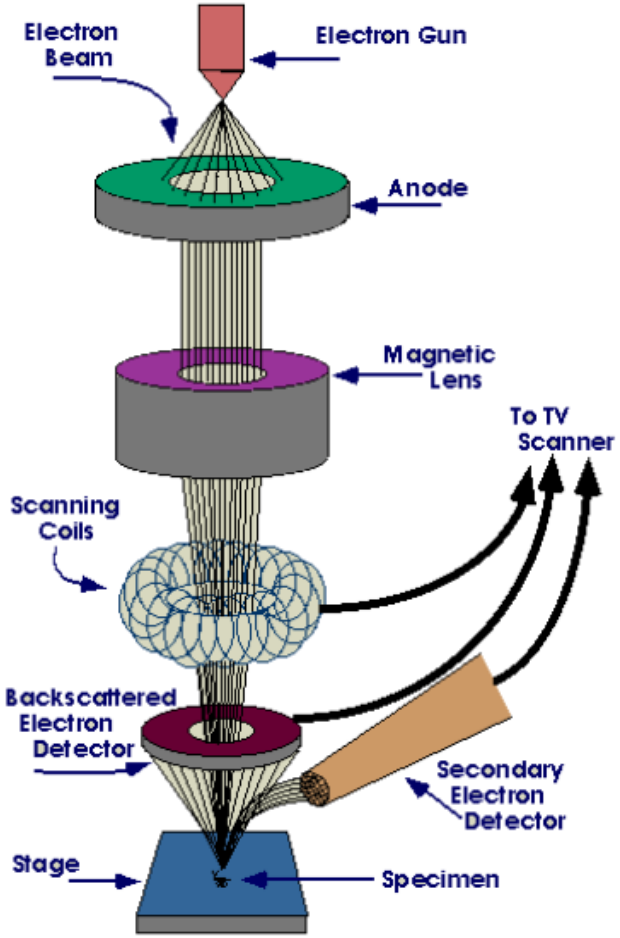
Cambiamo sonda → elettroni accelerati





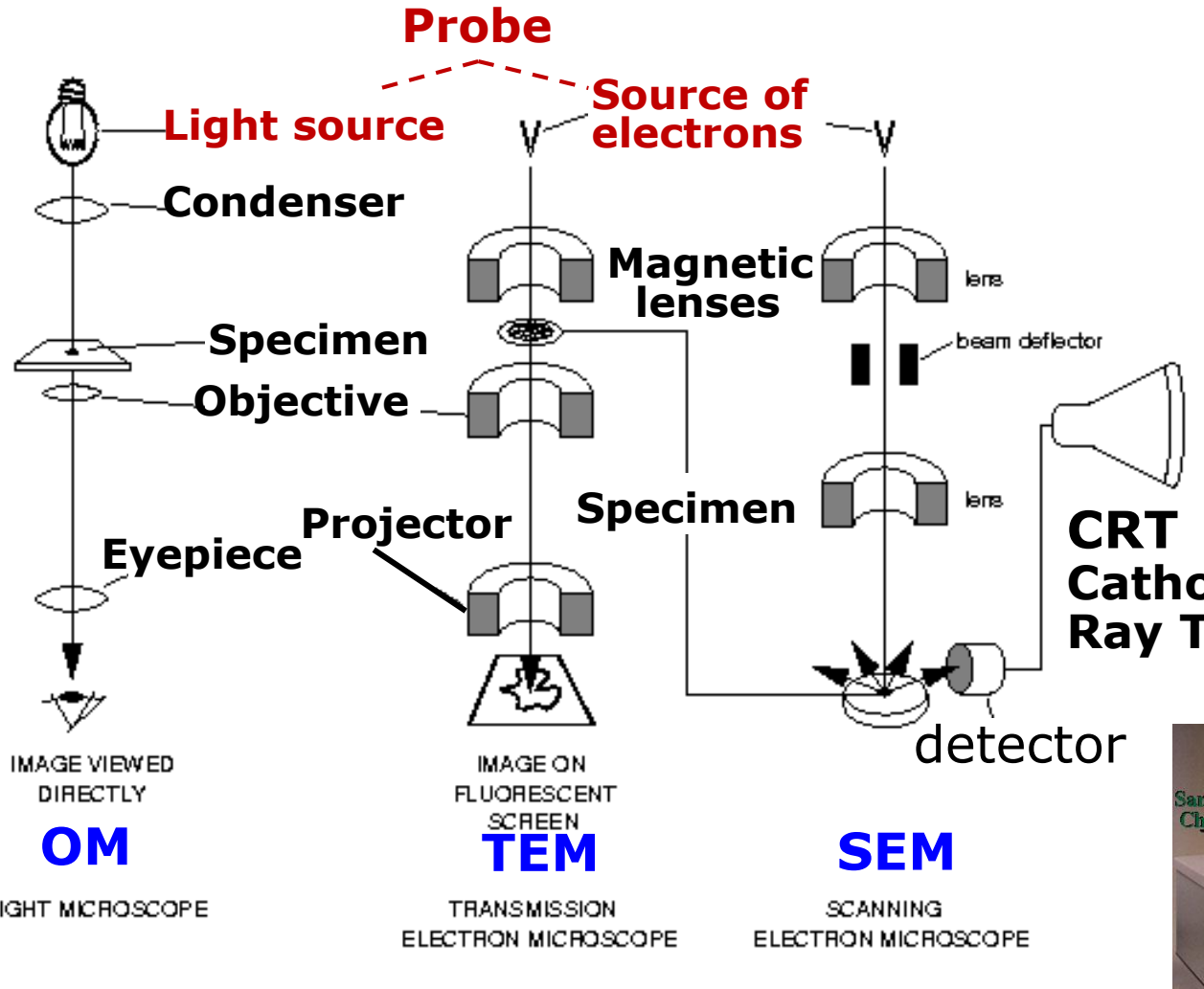
	Luce visibile	**Elettroni (30 kV)**
Lunghezza d'onda	0.7-0.4 µm	70 nm
Risoluzione	0.2 µm	1 nm

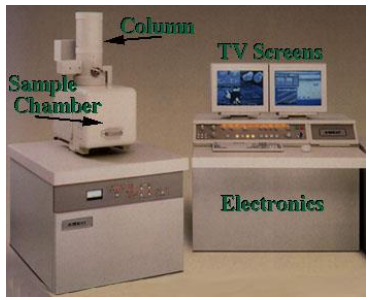
Microscopio elettronico



- Sorgente del fascio elettronico
- Lenti di focalizzazione
- Rivelazione dei segnali emessi

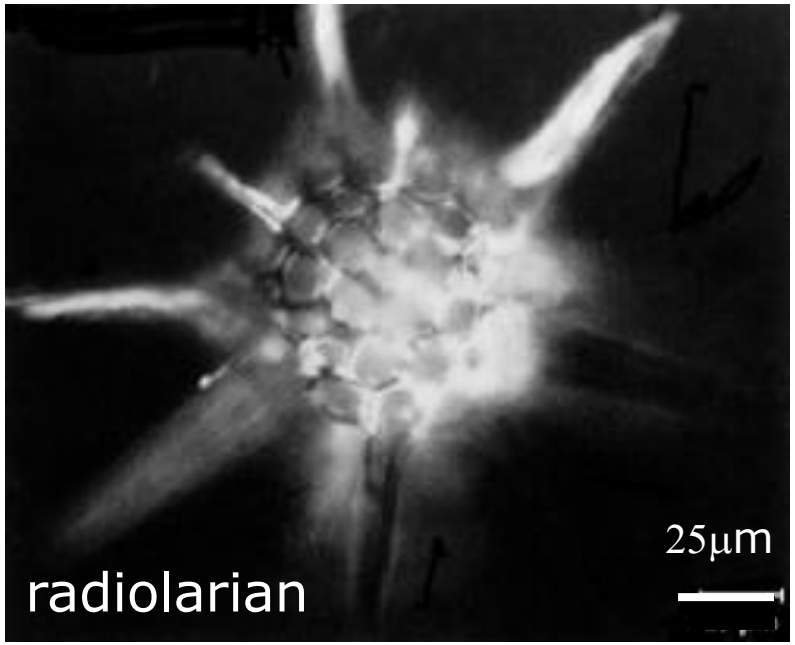
Comparison of OM,TEM and SEM





Principal features of an optical microscope, a transmission electron microscope and a scanning electron microscope, drawn to emphasize the similarities of overall design.

Optical Microscopy (OM) vs Scanning Electron Microscopy (SEM)

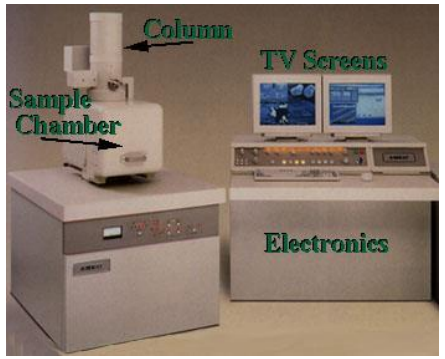


OM

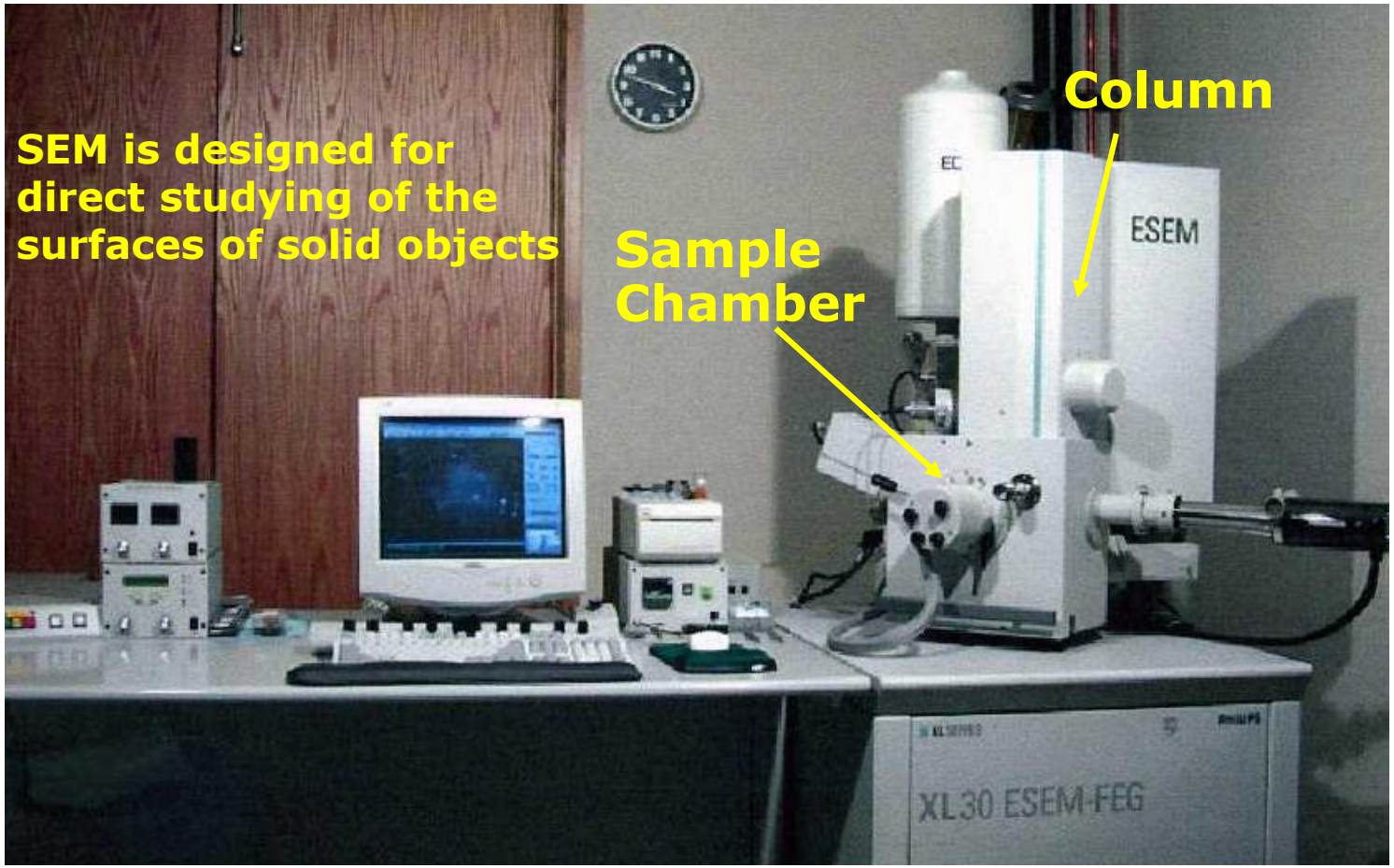
SEM

Small depth of field
Low resolution

Large depth of field
High resolution



What is SEM



Scanning electron microscope (SEM) is a microscope that uses electrons rather than light to form an image. There are many advantages to using the SEM instead of a OM.

Advantages of Using SEM over OM

	Magnification	Depth of Field	Resolution
OM	4x – 1000x	**15.5μm – 0.19μm**	**~ 0.2μm**
SEM	10x – 3000000x	**4mm – 0.4μm**	**1-10nm**

The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time and produces an image that is a good representation of the three-dimensional sample. The SEM also produces images of high resolution, which means that closely features can be examined at a high magnification.

The combination of higher magnification, larger depth of field, greater resolution and compositional and crystallographic information makes the SEM one of the most heavily used instruments in research areas and industries, especially in semiconductor industry.

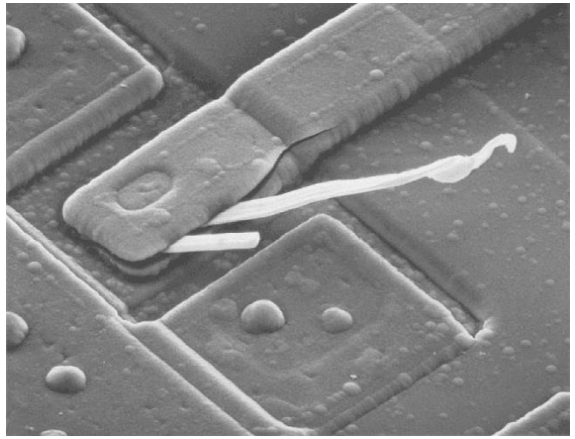
Scanning Electron Microscope
– a Totally Different Imaging Concept

Instead of using the full-field image, a **point-to-point measurement strategy** is used.

High energy **electron beam** is used to **excite** the **specimen** and the signals are collected and analyzed so that an **image** can be **constructed**.

The signals carry **topological**, **chemical** and **crystallographic** information, respectively, of the **samples surface**.

Main Applications



- **Topography**

  The surface features of an object and its texture (hardness, reflectivity… etc.)

- **Morphology**

  The shape and size of the particles making up the object (strength, defects in IC and chips…etc.)

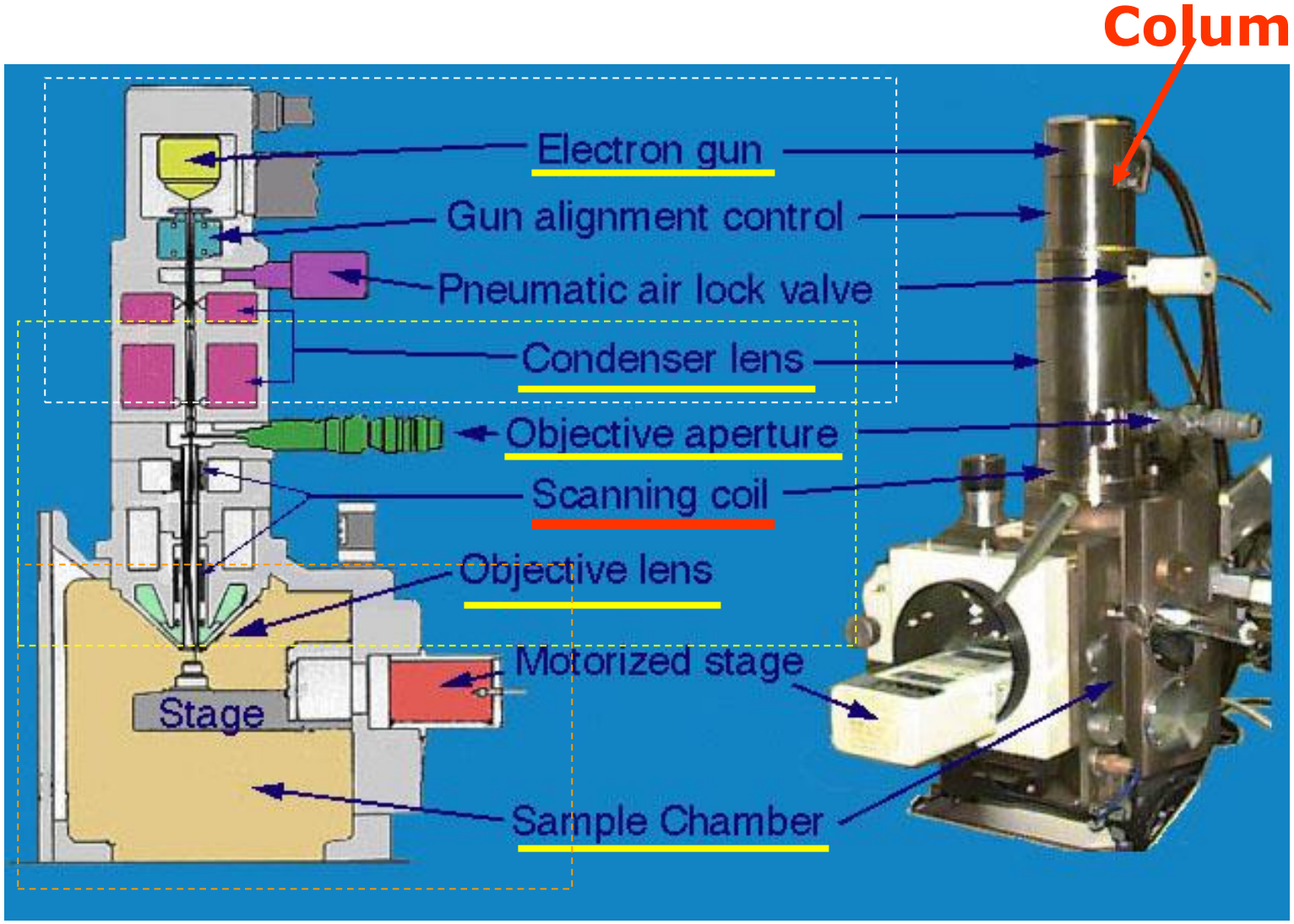
- **Composition**

  The elements and compounds that the object is composed of and the relative amounts of them (melting point, reactivity, hardness…etc.)

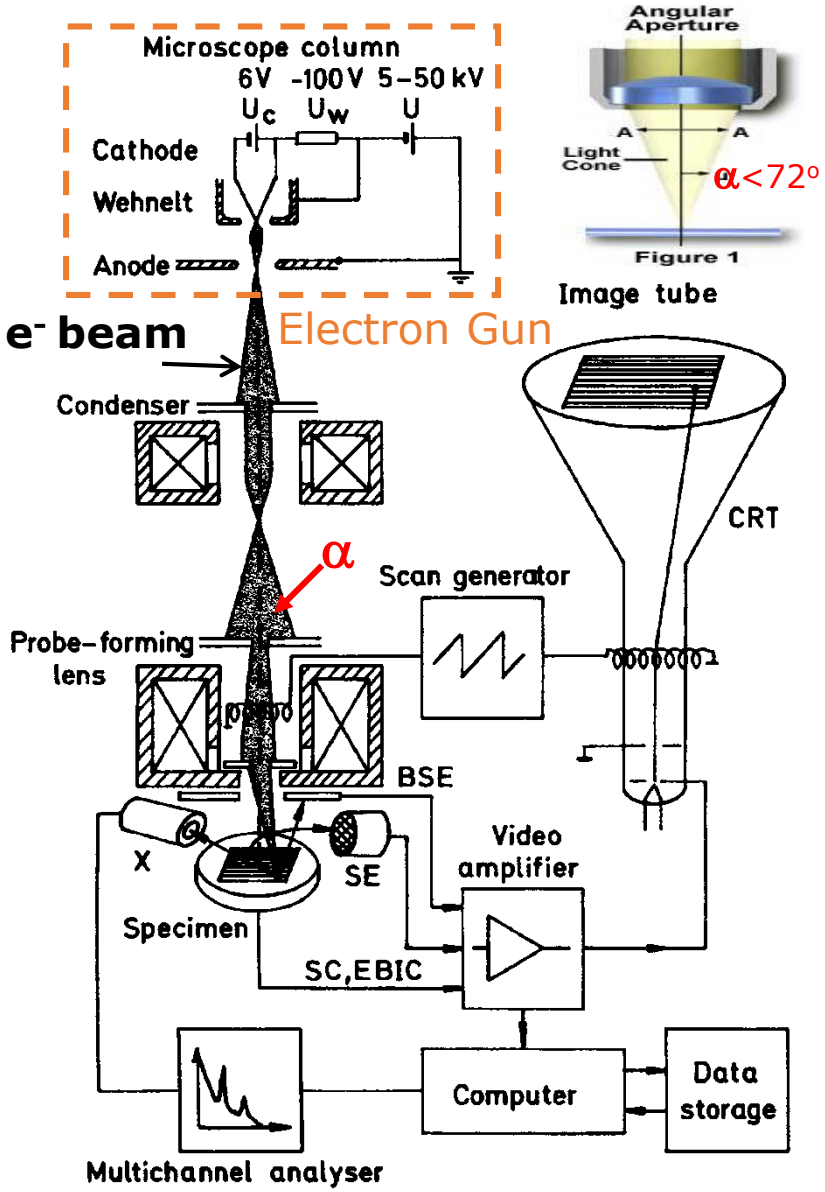
- **Crystallographic Information**

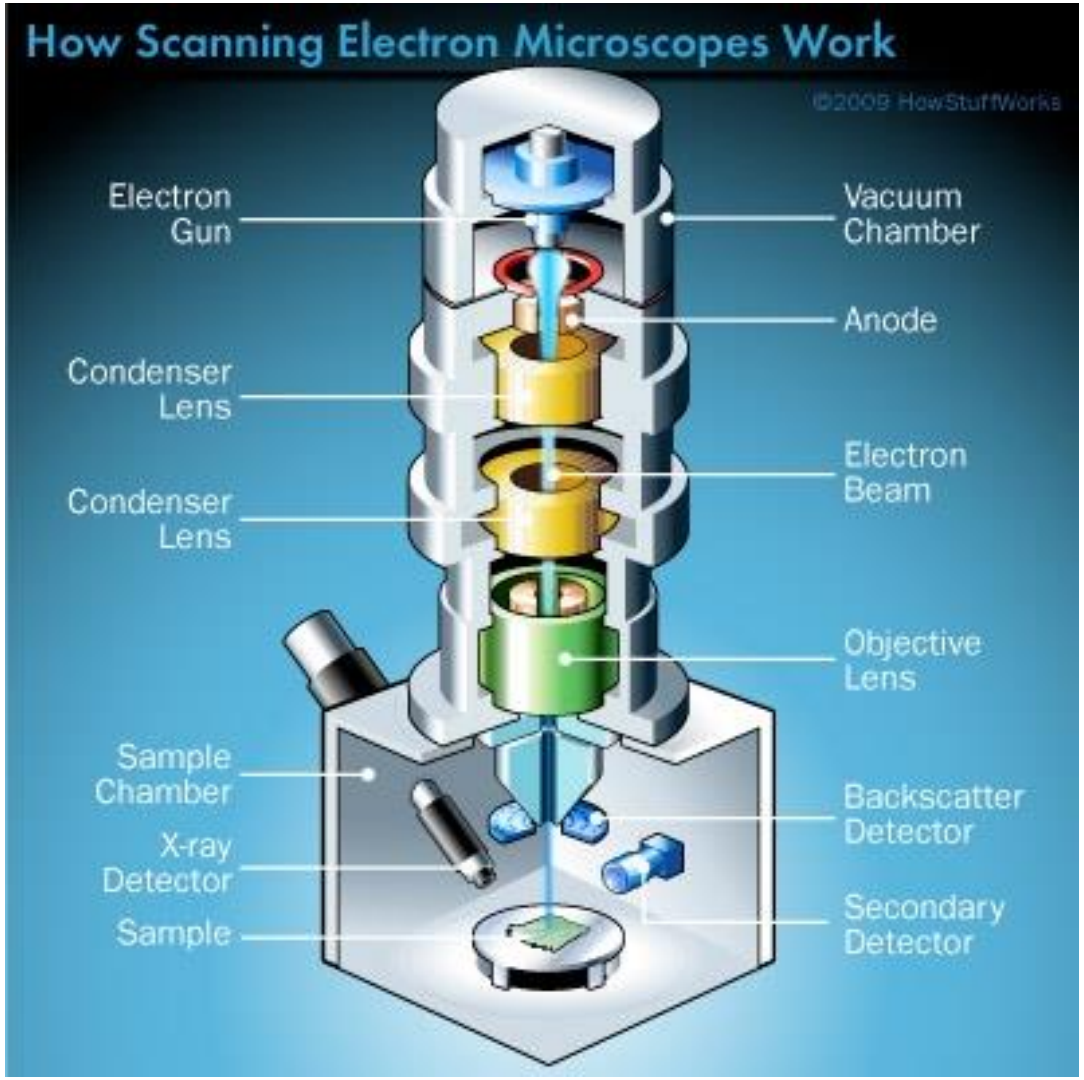
  How the grains are arranged in the object (conductivity, electrical properties, strength…etc.)

A Look Inside the Column



A more detailed look inside





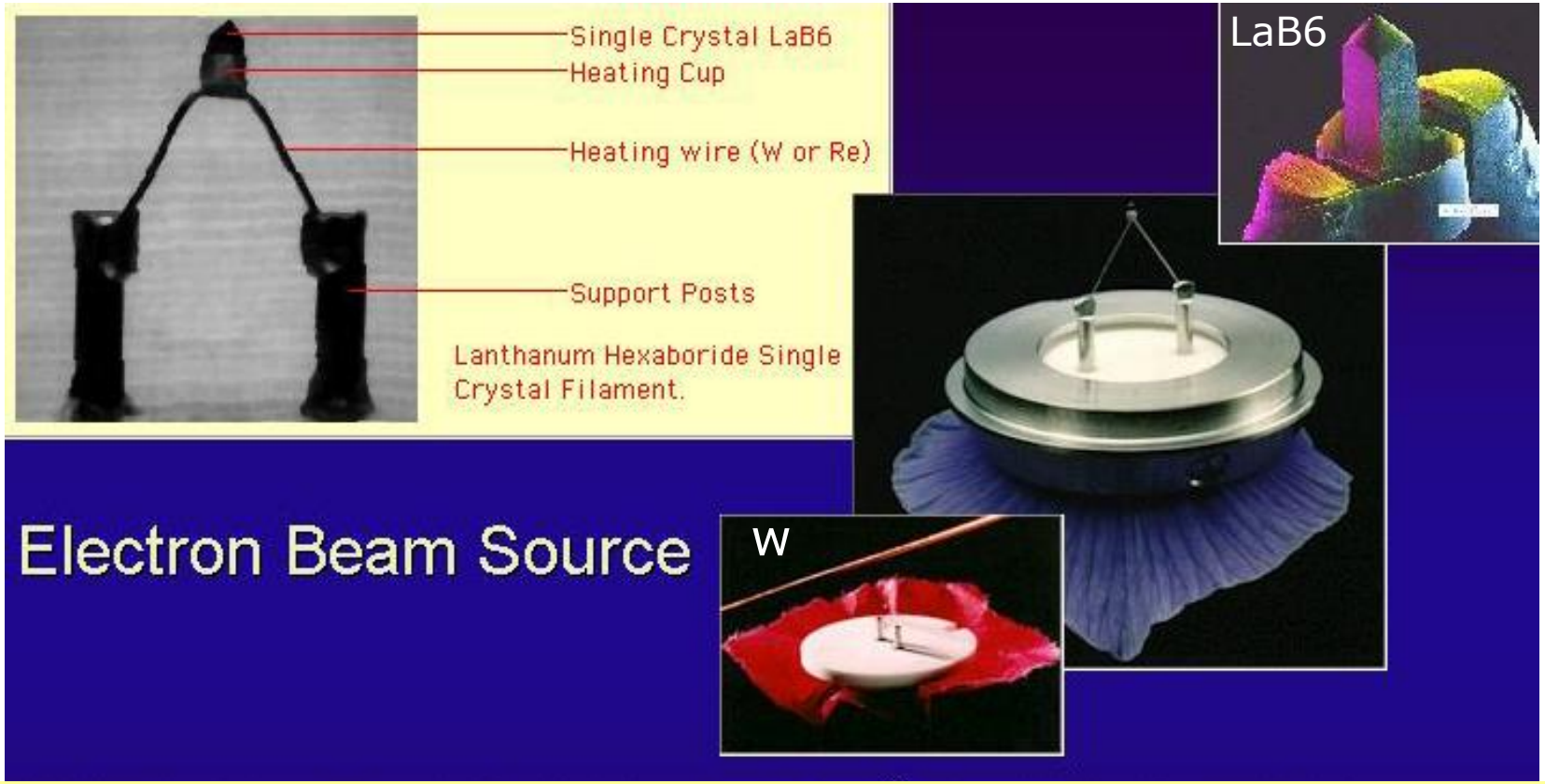
Source: L. Reimer, "Scanning Electron Microscope", 2nd Ed., Springer-Verlag, 1998, p.2

α - beam convergence

How an Electron Beam is Produced?

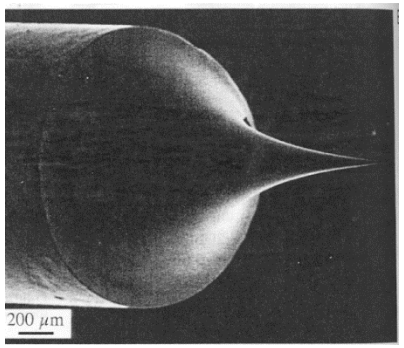
- **Electron guns are used to produce a fine, controlled beam of electrons which are then focused at the specimen surface.**
- **The electron guns may either be thermionic gun or field-emission gun**

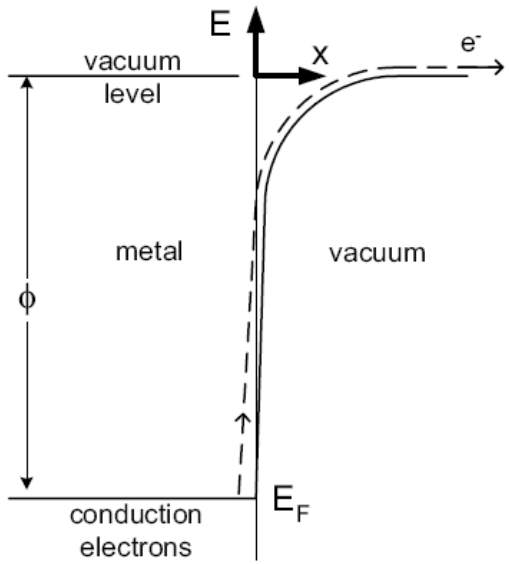
Electron beam Source

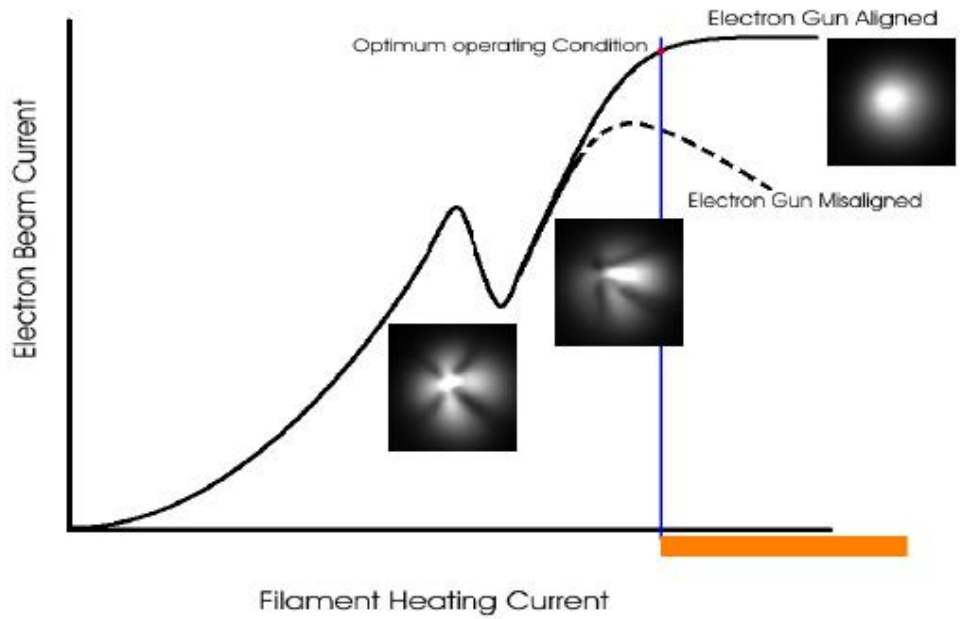


W or LaB_6 Filament
Thermionic or Field Emission Gun

Thermionic emission







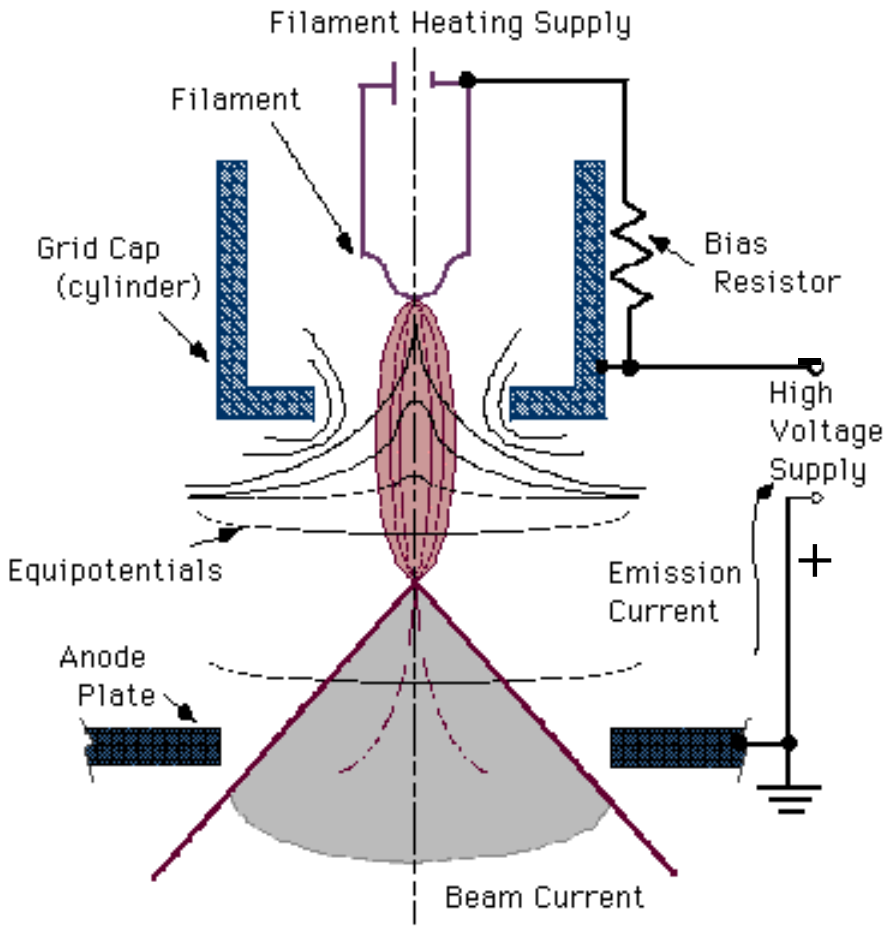
$$J_e = A\,T^2 \exp(-\phi/kT)$$

Work function Φ

W = 4.5 eV
LaB_6 = 2.5 eV
W/ZrO = 4.5 -> 2.8 eV

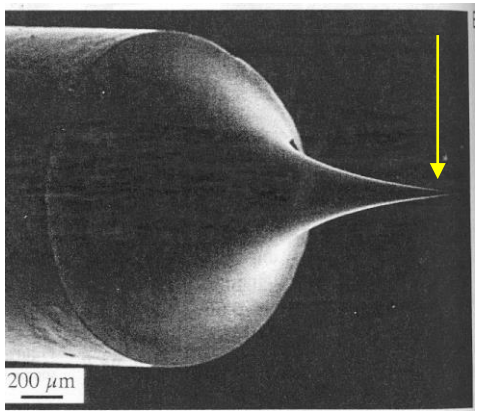
Thermionic Emission Gun

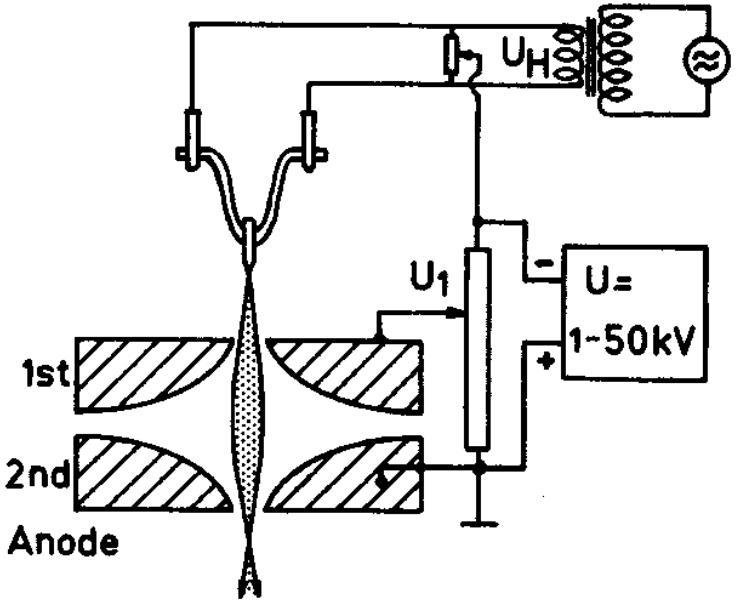
- A tungsten filament heated by DC to approximately 2700K or LaB_6 rod heated to around 2000K
- A vacuum of 10^{-3} Pa (10^{-4} Pa for LaB_6) is needed to prevent oxidation of the filament
- Electrons "boil off" from the tip of the filament
- Electrons are accelerated by an acceleration voltage of 1-50kV



Field Emission Gun

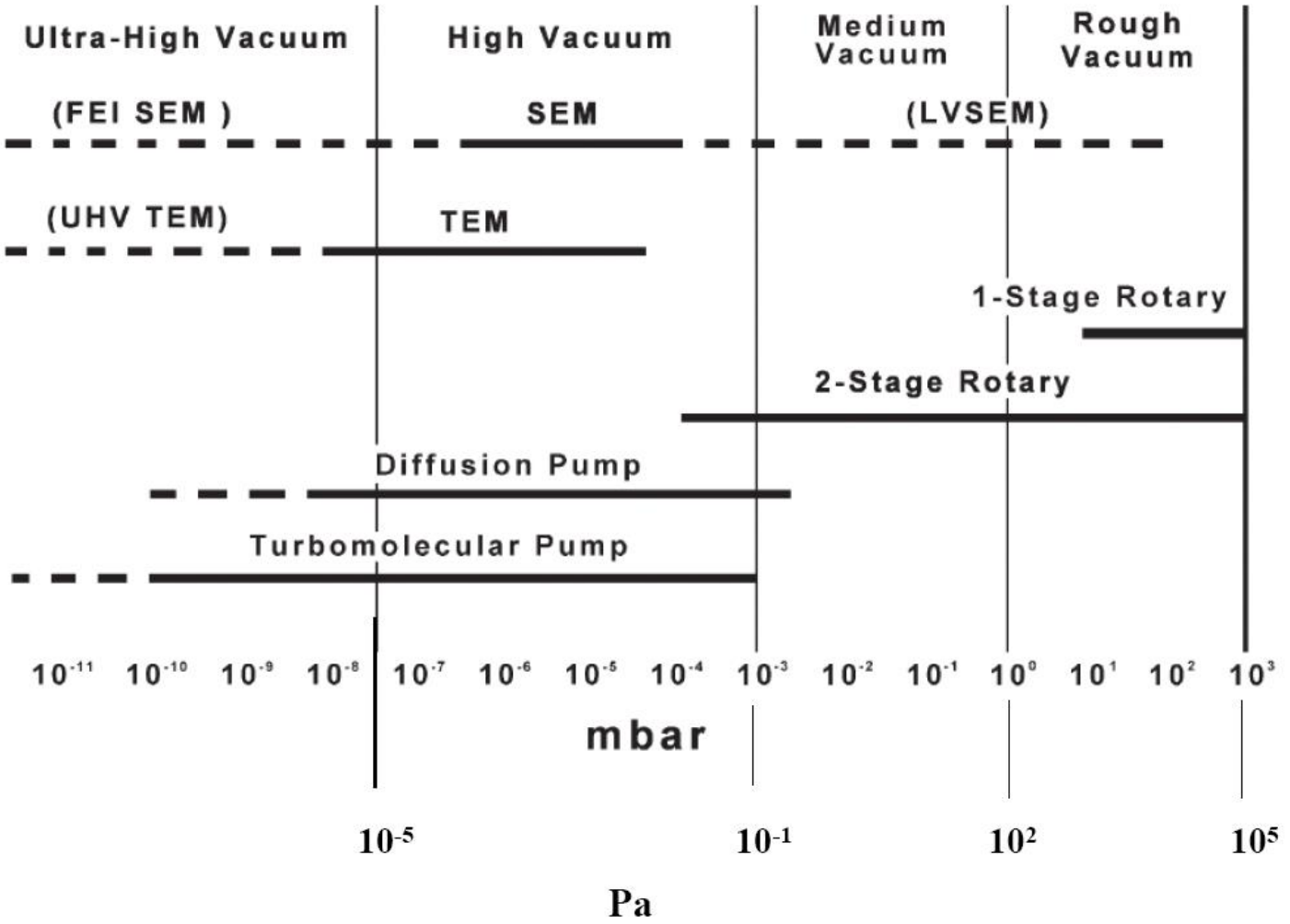
- The tip of a tungsten needle is made very sharp (radius < 0.1 μm)
- The electric field at the tip is very strong (> 10^7 V/cm) due to the sharp point effect
- Electrons are pulled out from the tip by the strong electric field
- Ultra-high vacuum (better than 10^{-6} Pa) is needed to avoid ion bombardment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





Field-emission gun

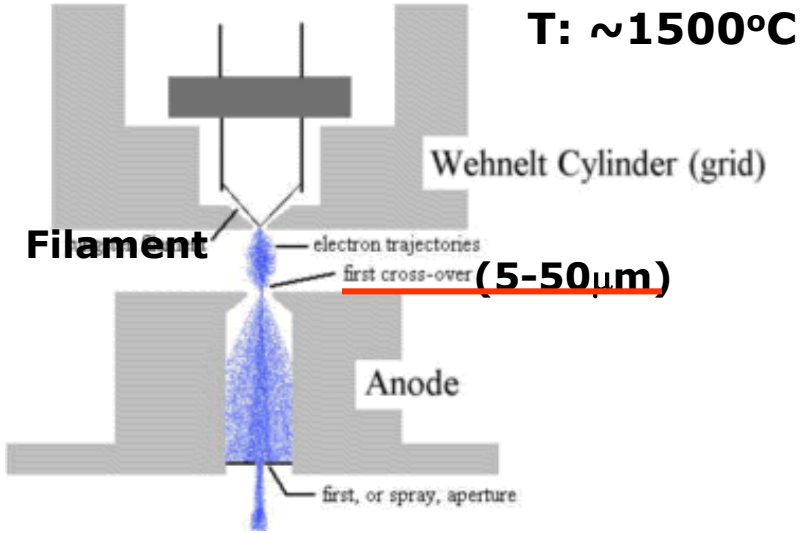
Vacuum range



Source of Electrons

Thermionic Gun

T: ~1500°C

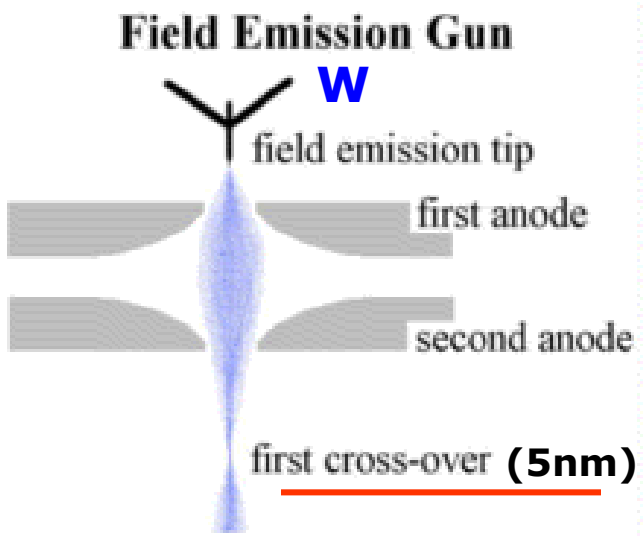


W and LaB_6

Field Emission Gun

E: >10MV/cm

W



Cold- and thermal FEG

Electron Gun Properties

Source	Brightness	Stability(%)	Size	Energy spread	Vacuum
W	$3X10^5$	~1	50μm	3.0(eV)	10^{-5} (τ)
LaB_6	$3x10^6$	~2	5μm	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

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.**

Major components and their functions

Magnetic Lenses

- **Condenser lens – focusing**

  controls the spot size and convergence (α) of the electron beam which impinges on the sample.

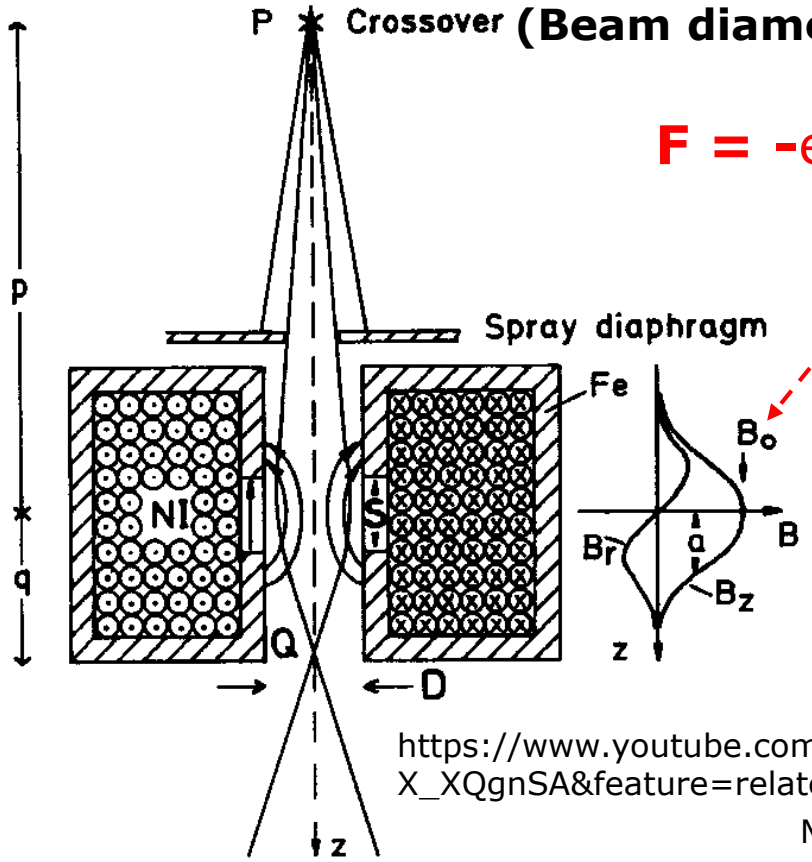
- **Objective lens – final probe forming**

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

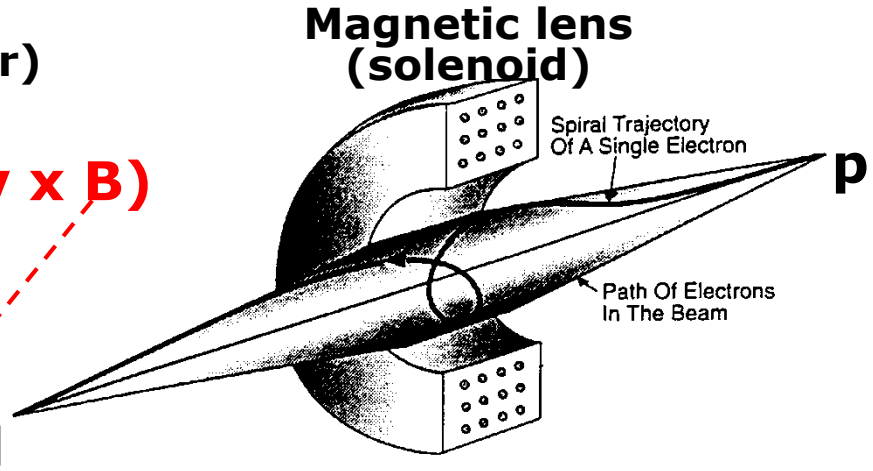
How Is Electron Beam Focused?

http://www.matter.org.uk/tem/lenses/electromagnetic_lenses.htm

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



$F = -e(v \times B)$



Lens formula: $1/f = 1/p + 1/q$

Demagnification: $M = q/p$

https://www.youtube.com/watch?v=sCYX_XQgnSA&feature=related 6:13-6:23

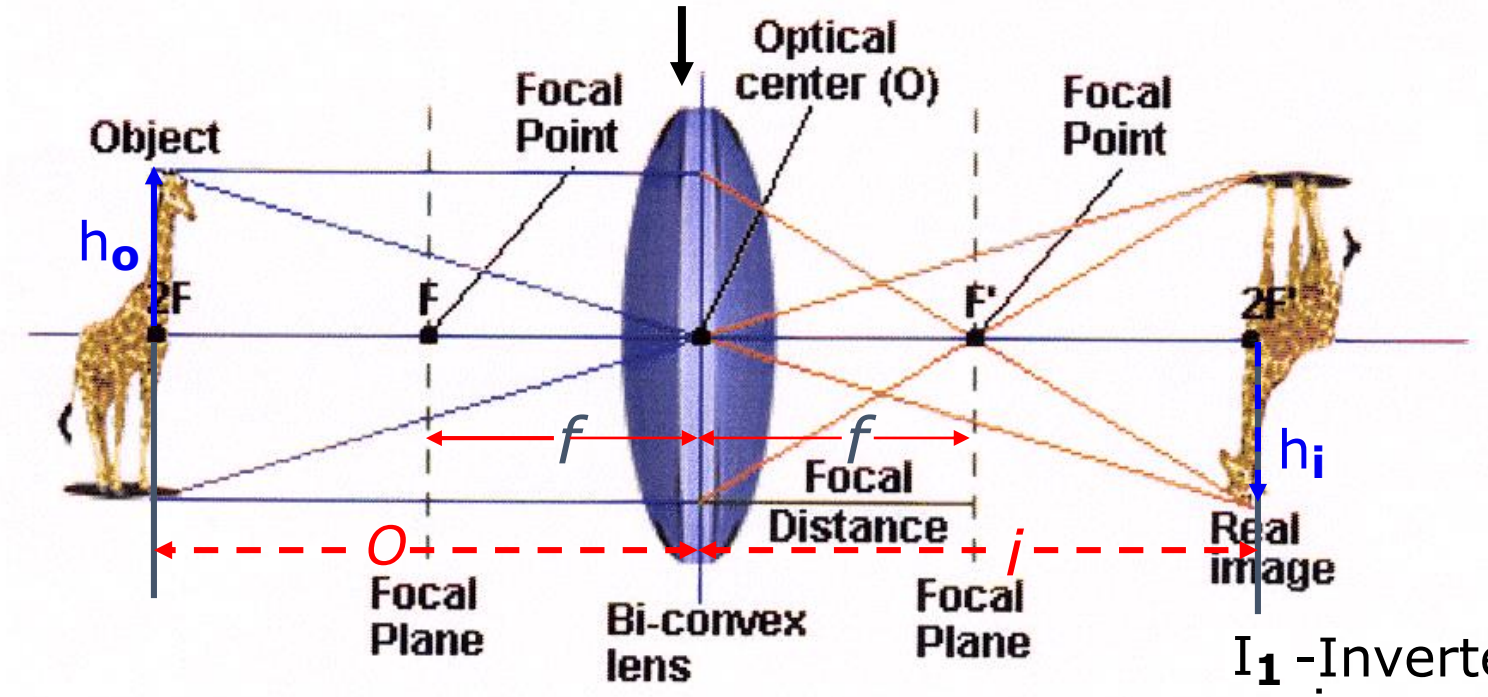
Magnetic lens

$f \propto B_o^2$

f can be adjusted by changing B_o, i.e., changing the current through coil. **B_o - magnetic field**

Lens formula and magnification

Objective lens



I_1 -Inverted image

Lens Formula $\frac{1}{f} = \frac{1}{O} + \frac{1}{i}$

***f*-focal length (distance)**

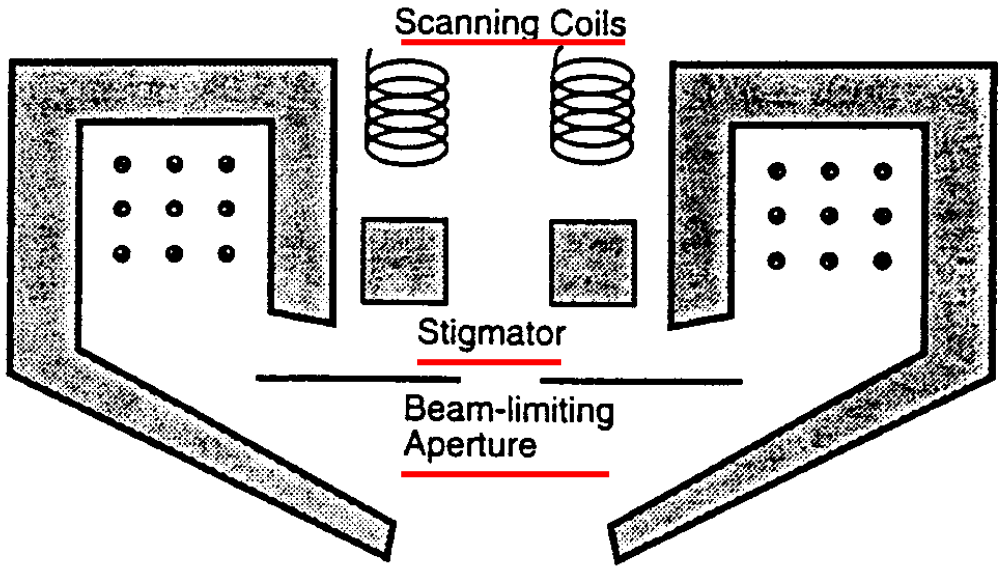
***O*-distance of object from lens**

Magnification by objective $m_o = \frac{h_i}{h_o} = \frac{i}{O}$

***i*-distance of image from lens**

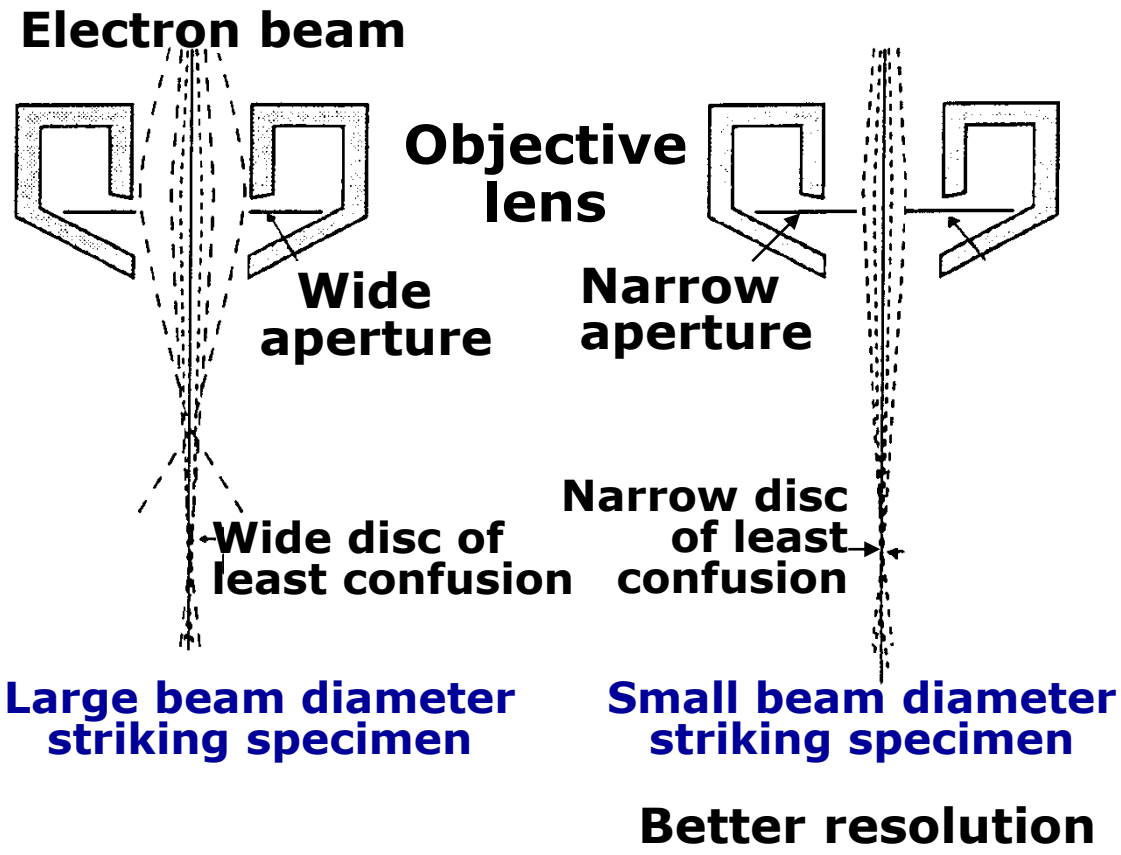
The Objective Lens

- 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 ~**10nm** beam spot which carries a beam current of approximately **10^{-9}– 10^{-12} A.**



The Objective Lens – Aperture

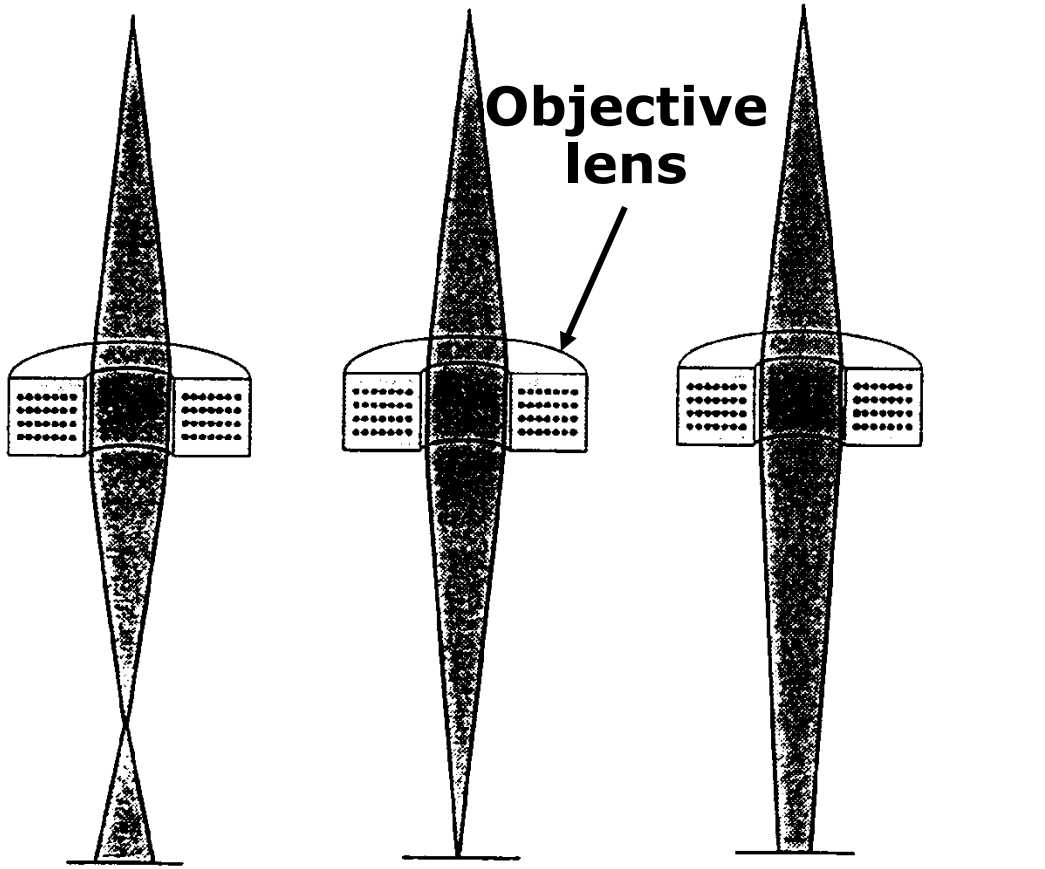
- Since the electrons coming from the electron gun have spread in kinetic energies and directions of movement, they may not be focused to the same plane to form a sharp spot.
- By inserting an aperture, the stray electrons are blocked and the remaining narrow beam will come to a narrow



"**Disc of Least Confusion**"

The Objective Lens - Focusing

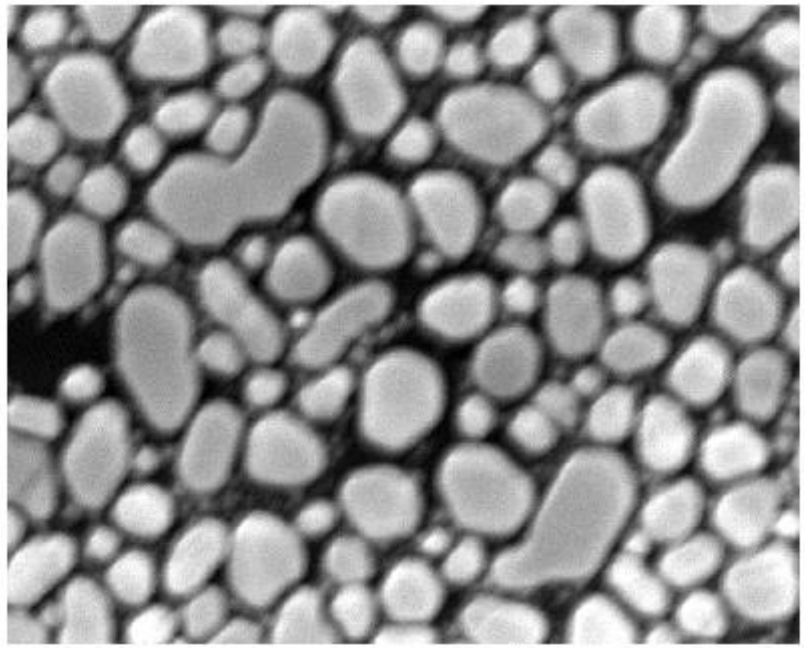
- By changing the current in the objective lens, the magnetic field strength changes and therefore the **focal length** of the objective lens is changed.



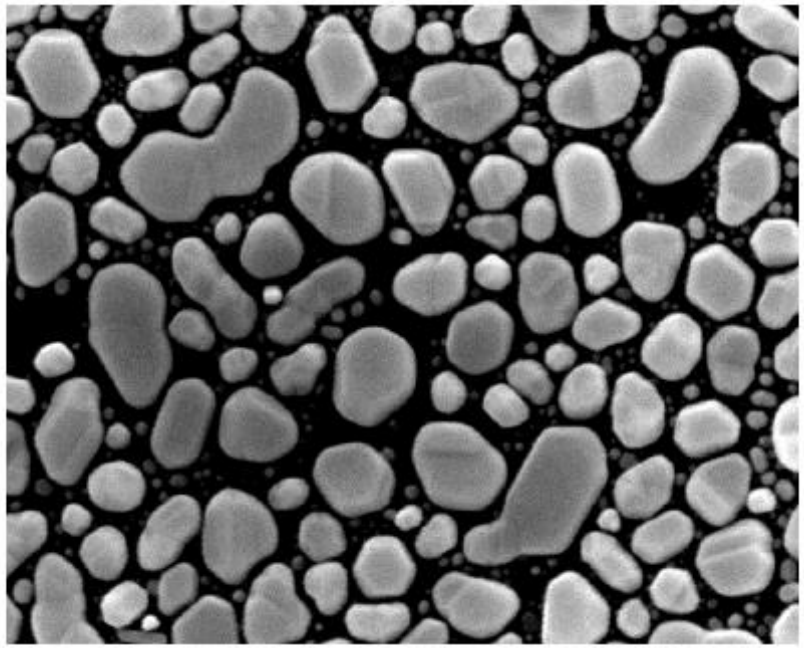
Objective lens $\Rightarrow$ Focus

The lens can be controlled by changing the current in the lens coil which changes the focal length (focus). The higher the current, the shorter the focal lenght, making the lens stronger.

Not in focus

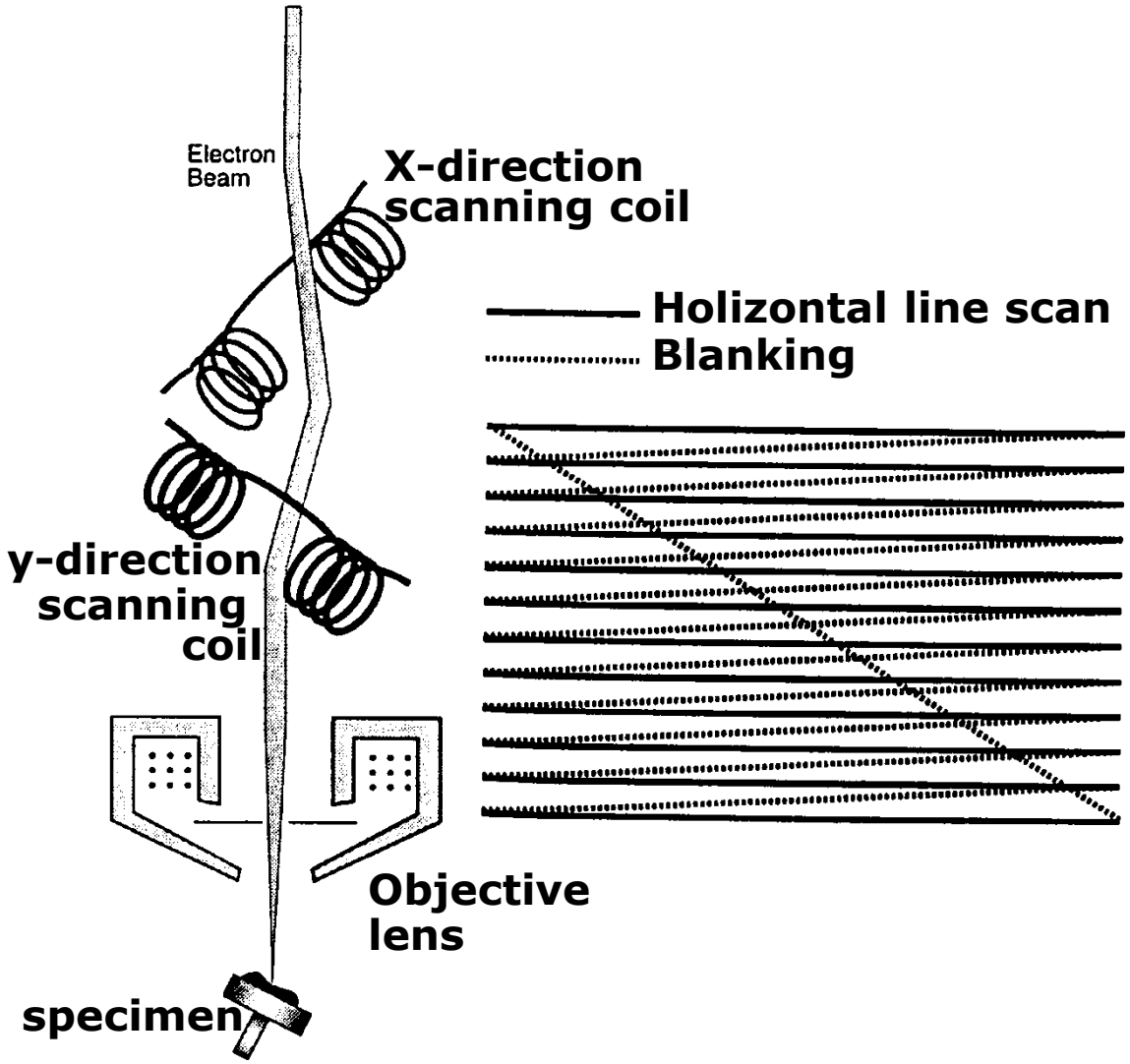


In focus

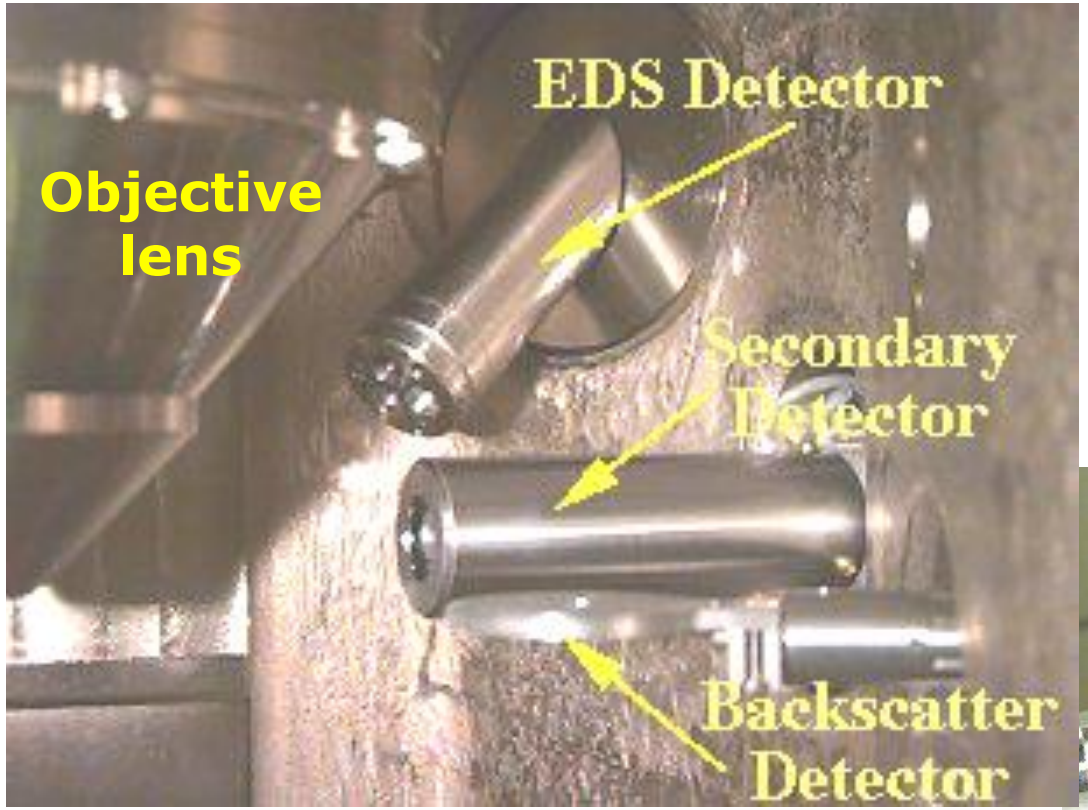


The Scan Coil and Raster Pattern

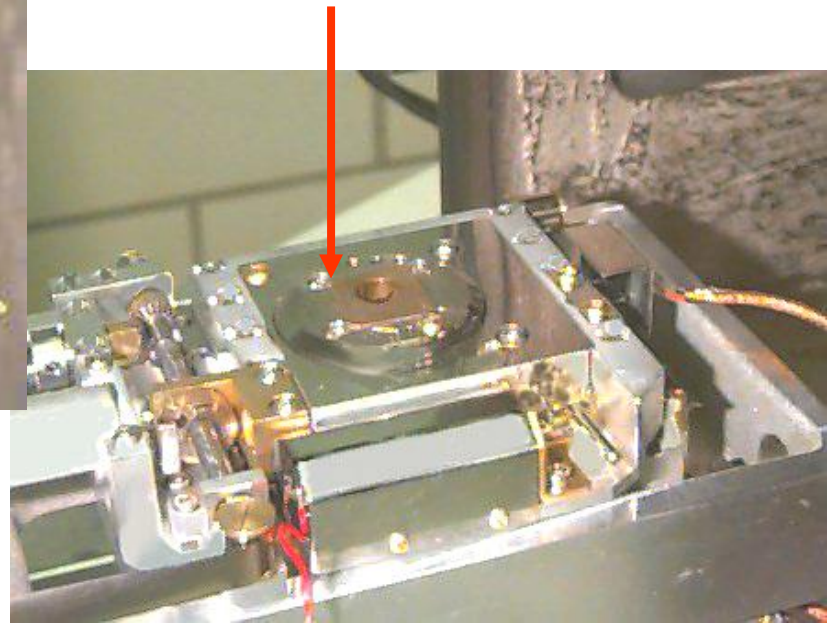
- Two sets of coils are used for scanning the electron beam across the specimen surface in a **raster** pattern similar to that on a TV screen.
- This effectively samples the specimen surface **point by point** over the scanned area.



Electron Detectors and Sample Stage



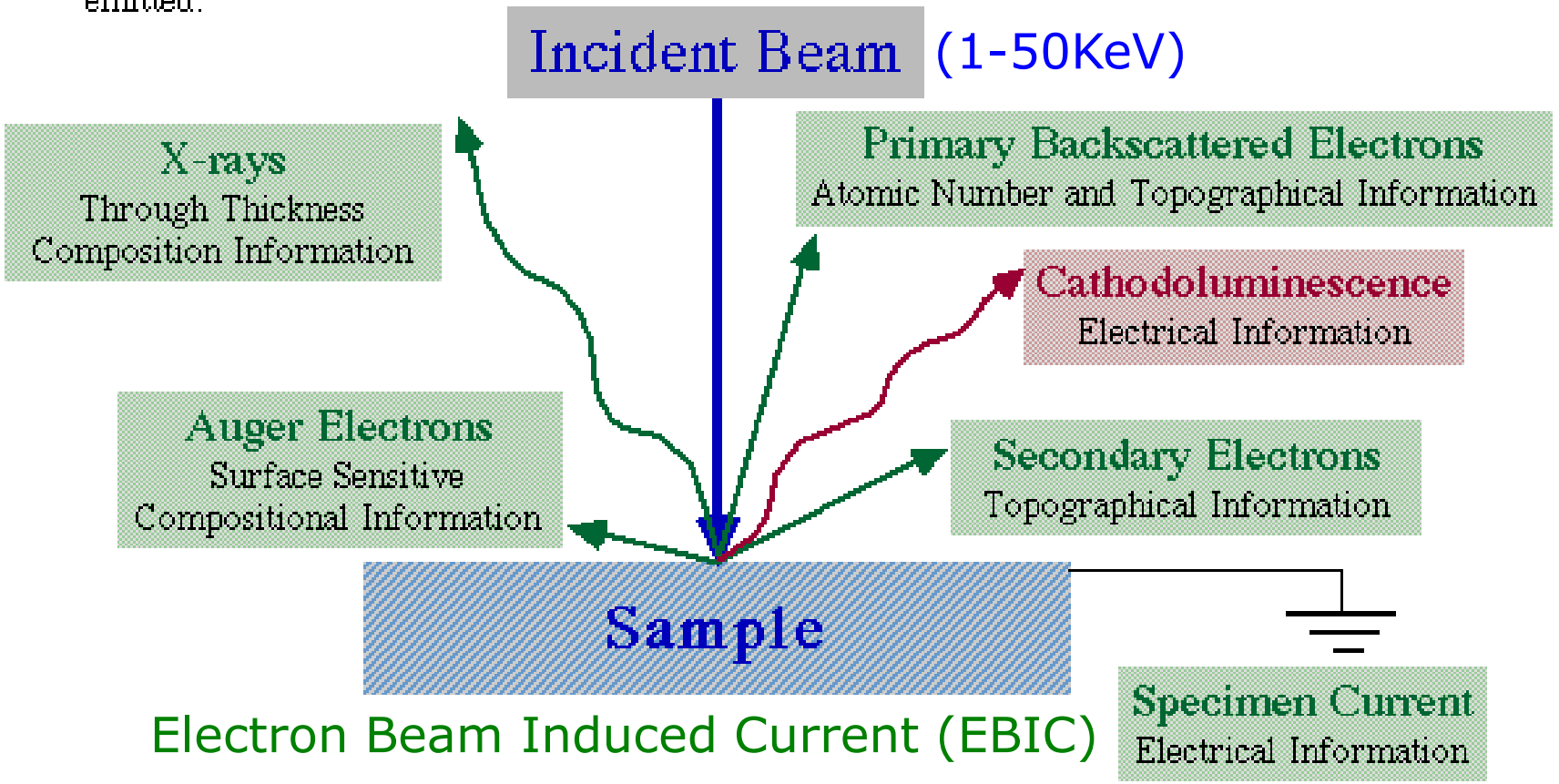
Sample stage



Electron Beam and Specimen Interactions

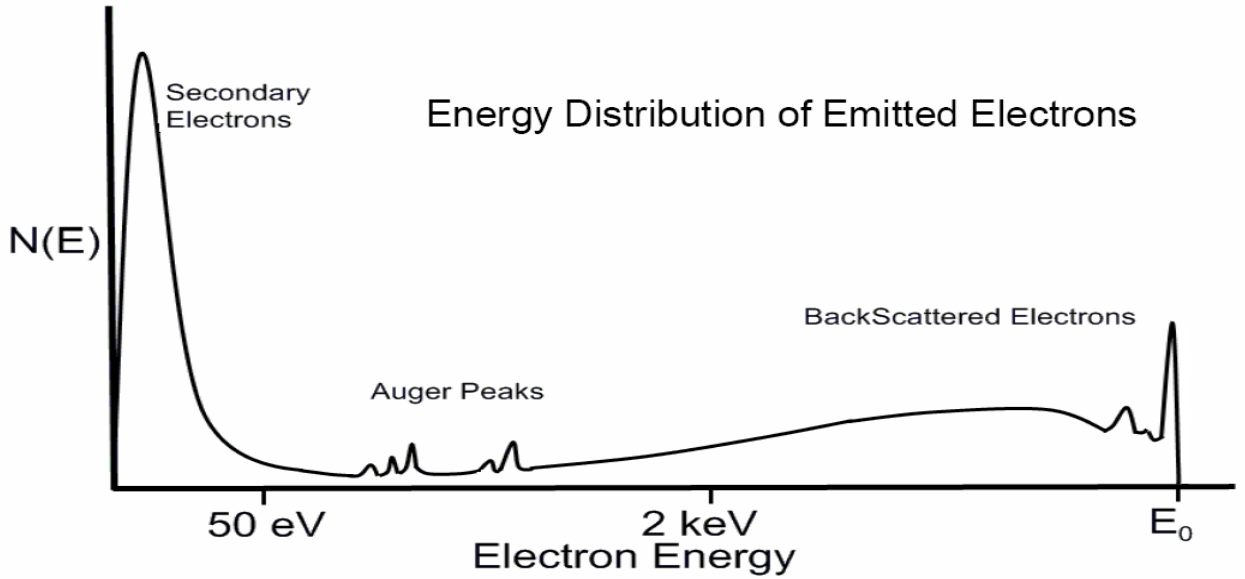
Sources of Image Information

When the electron beam strikes the sample, both **photon** and **electron** signals are emitted.



Electron Beam Induced Current (EBIC)

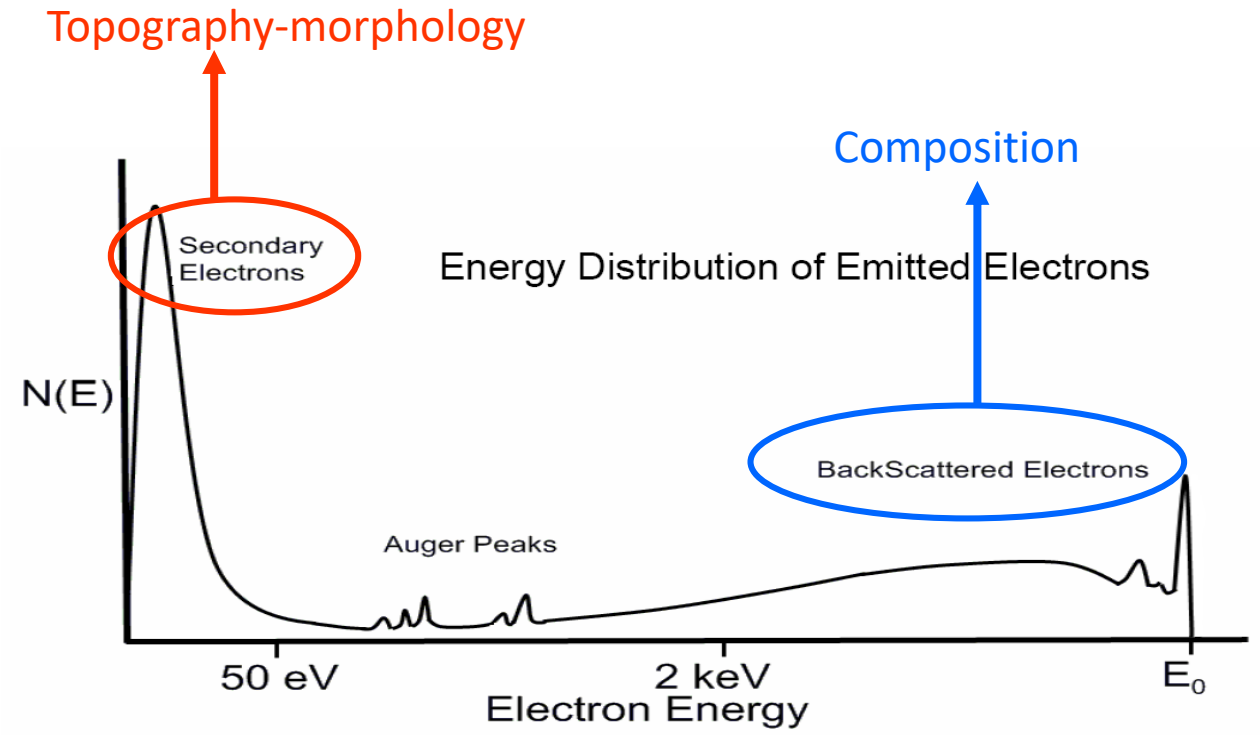
Energy distribution of emitted electrons



Secondary electrons are low energy electrons ejected from the specimen atoms by the energetic primary beam

Backscattered electrons are primary beam electrons scattered back out of the sample.

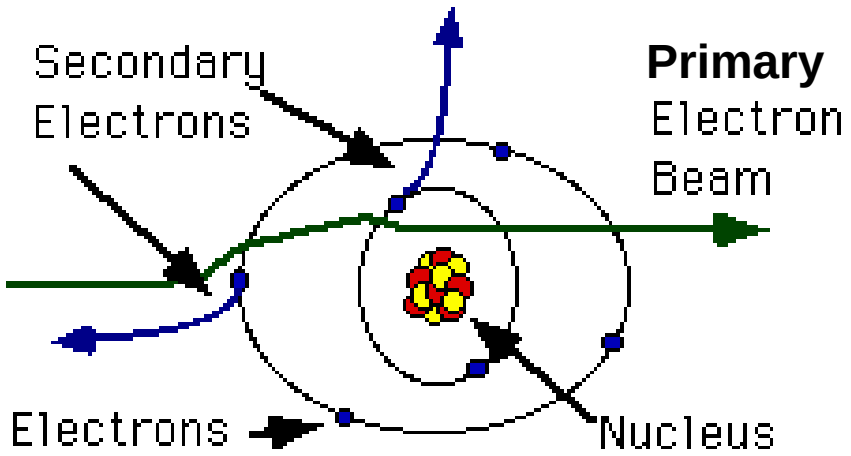
Energy distribution of emitted electrons



Secondary electrons are low energy electrons ejected from the specimen atoms by the energetic primary beam

Backscattered electrons are primary beam electrons scattered back out of the sample.

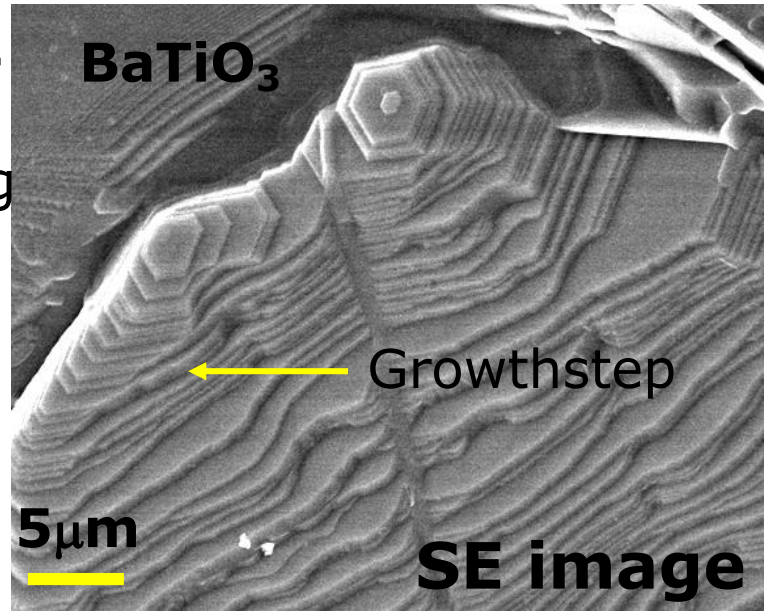
Secondary Electrons (SE)



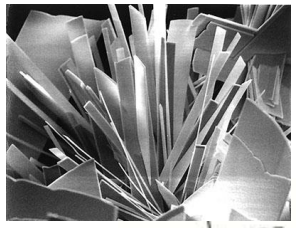
Produced by **inelastic** interactions of high energy electrons with valence (or conduction) electrons of atoms in the specimen, causing the ejection of the electrons from the atoms. These ejected electrons with energy less than **50eV** are termed "secondary electrons".
Each incident electron can produce several secondary electrons.

SE yield: $\delta = n_{SE}/n_B$ independent of **Z**
δ decreases with increasing beam energy and increases with decreasing glancing angle of incident beam

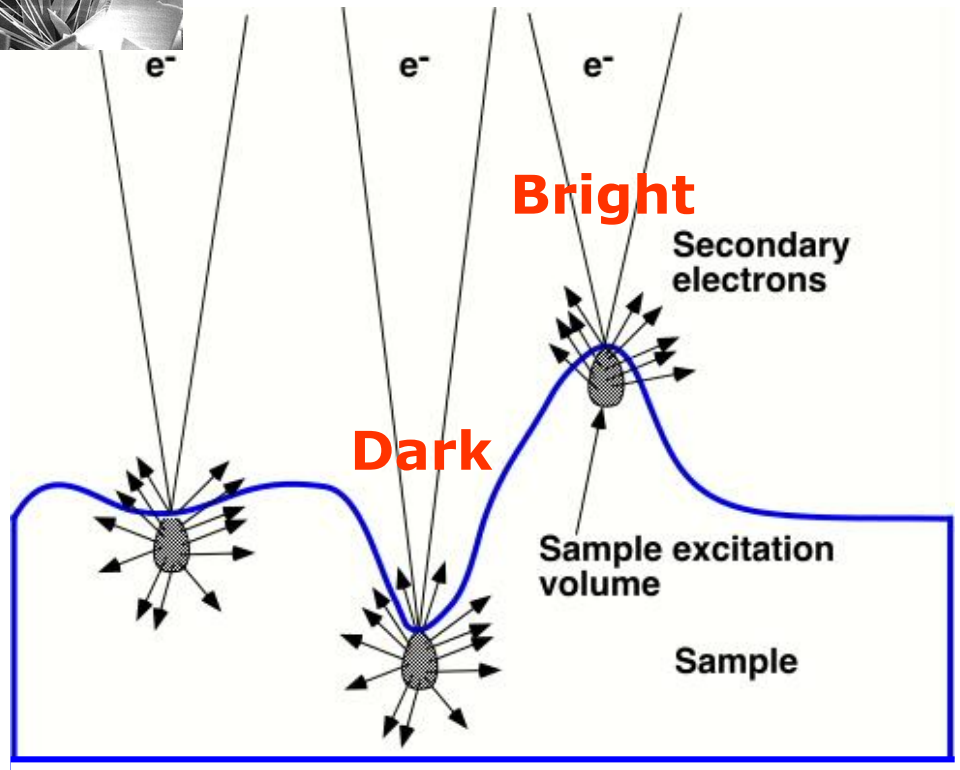
Production of SE is very **topography** related. Due to their low energy, only SE that are very near the surface (**<10nm**) can exit the sample and be examined (**small escape depth**).



Z – atomic number

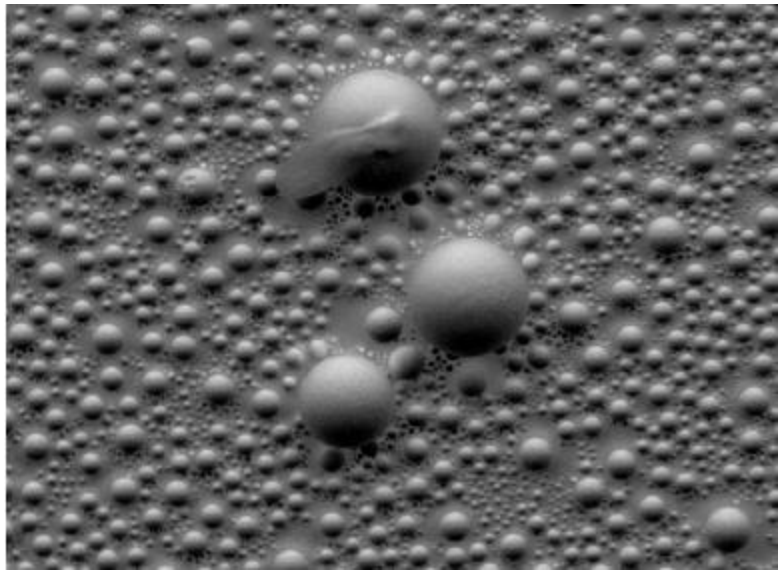


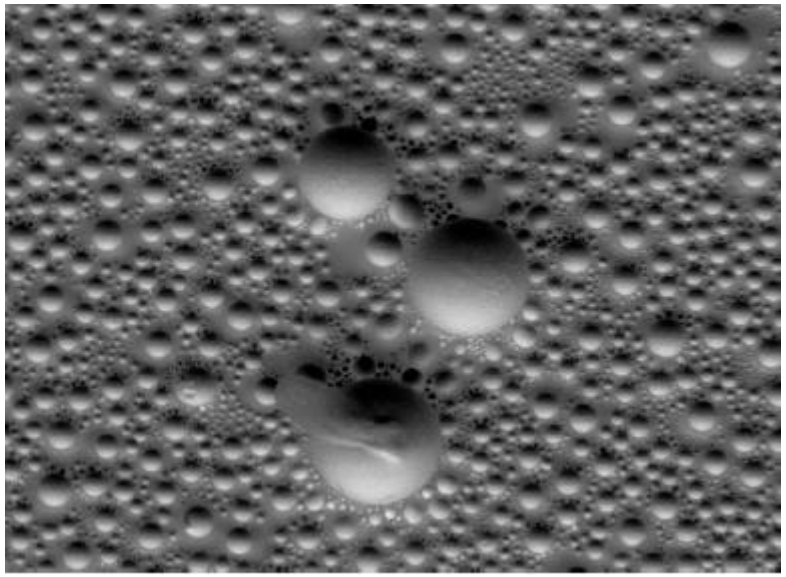
Topographical Contrast



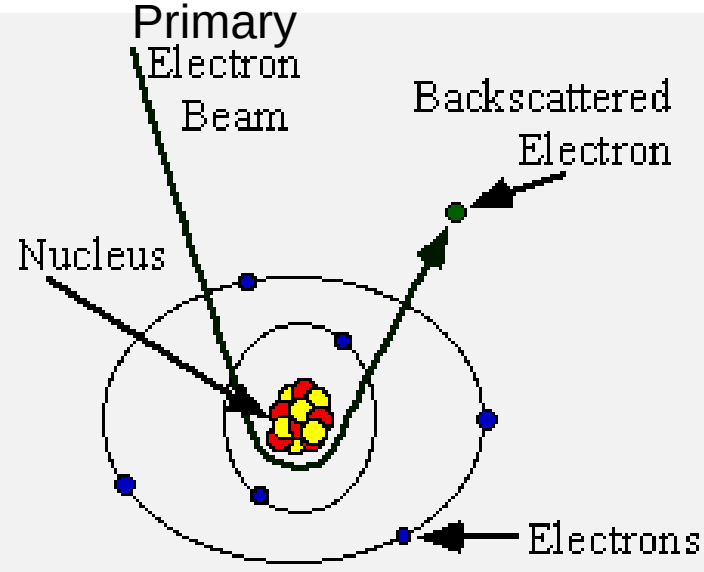
Topographic contrast arises because **SE generation** depend on the **angle of incidence** between the beam and sample. Thus local variations in the angle of the surface to the beam (**roughness**) affects the numbers of electrons leaving from point to point. The resulting "**topographic contrast**" is a function of the **physical shape** of the specimen.

What is real at SEM?





Backscattered Electrons (BSE)



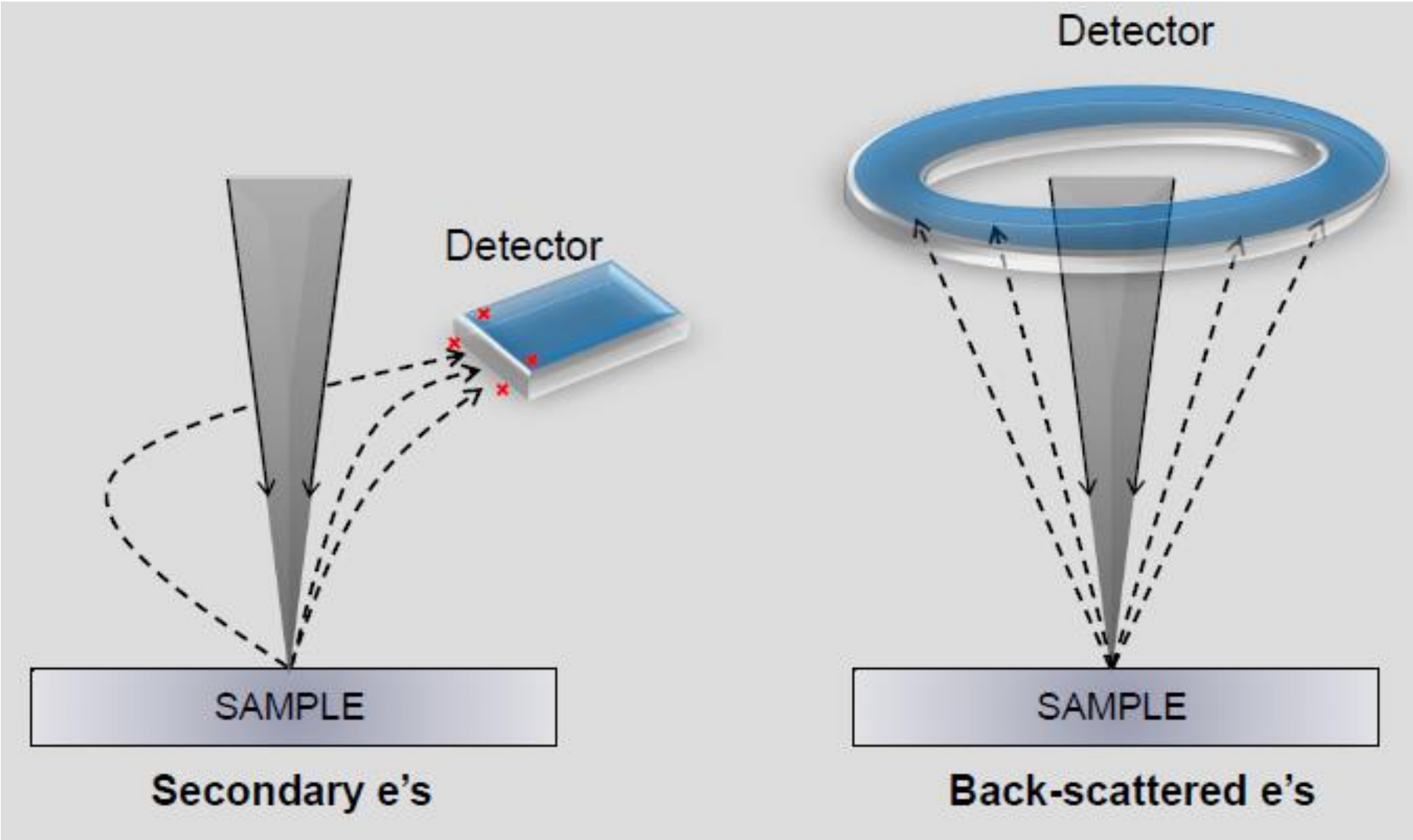
BSE image from flat surface of an Al **(Z=13)** and Cu **(Z=29)** alloy

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

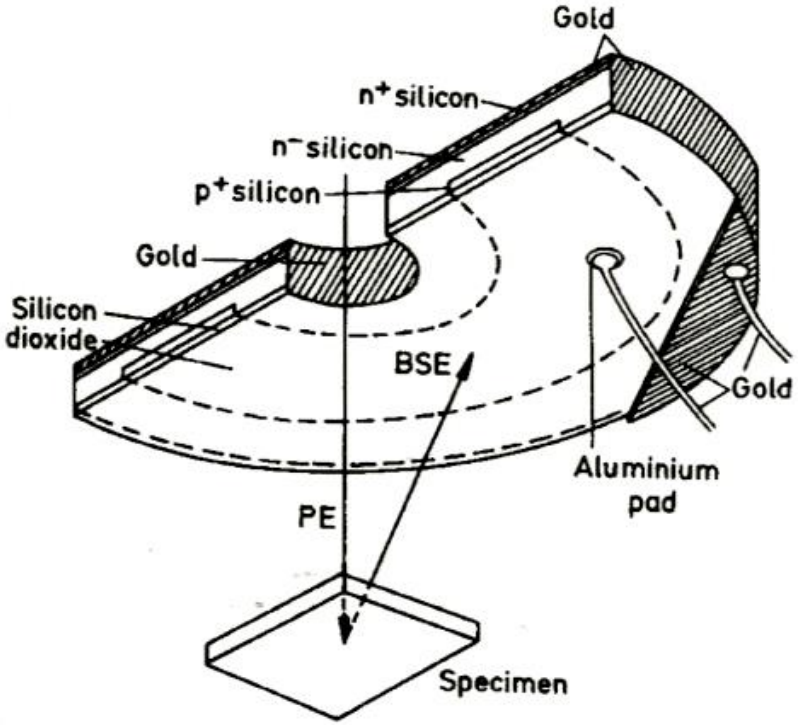
BSE images show characteristics of **atomic number contrast**, i.e., high average Z appear brighter than those of low average Z. η increases with **tilt**.

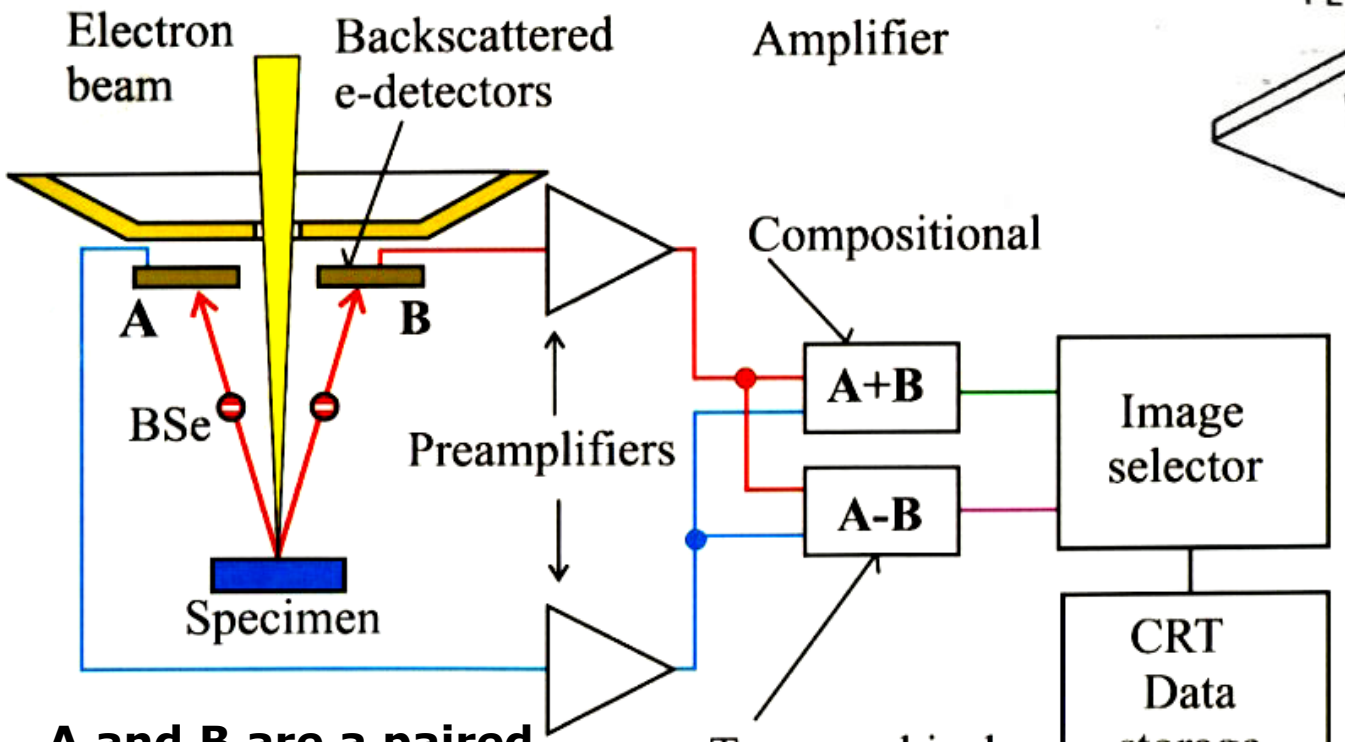
Detection of secondary and back-scattered electrons



Semiconductor Detector for Backscattered Electrons

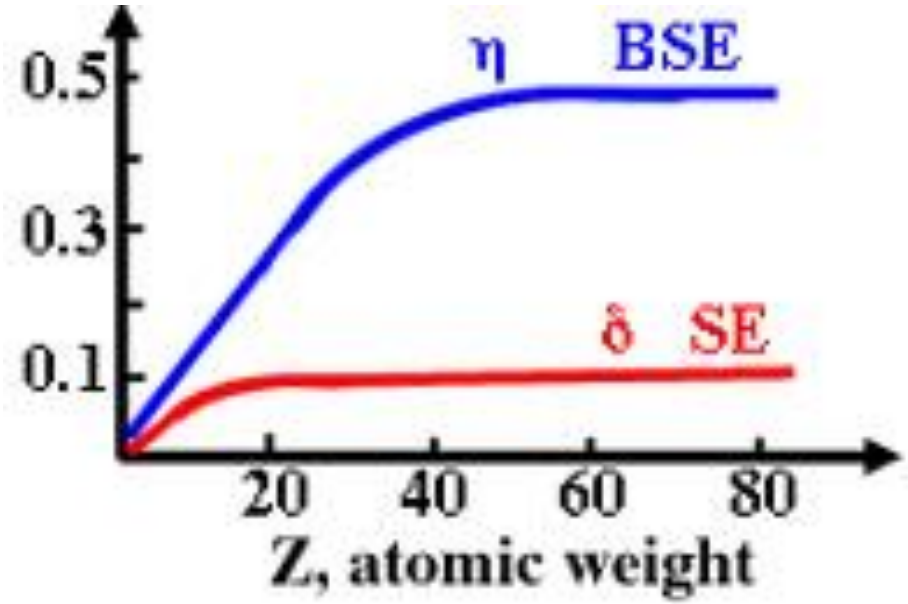
High energy electrons produce electron-hole pairs (charge carriers) in the semiconductor, and generate a current pulse under an applied potential.



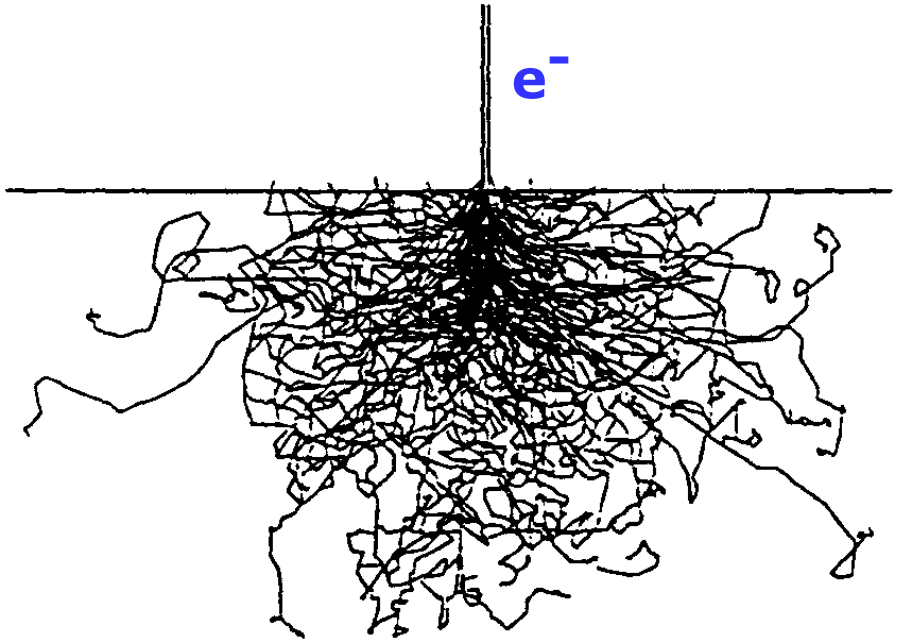


A and B are a paired semiconductor detectors

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



Interaction Volume: I

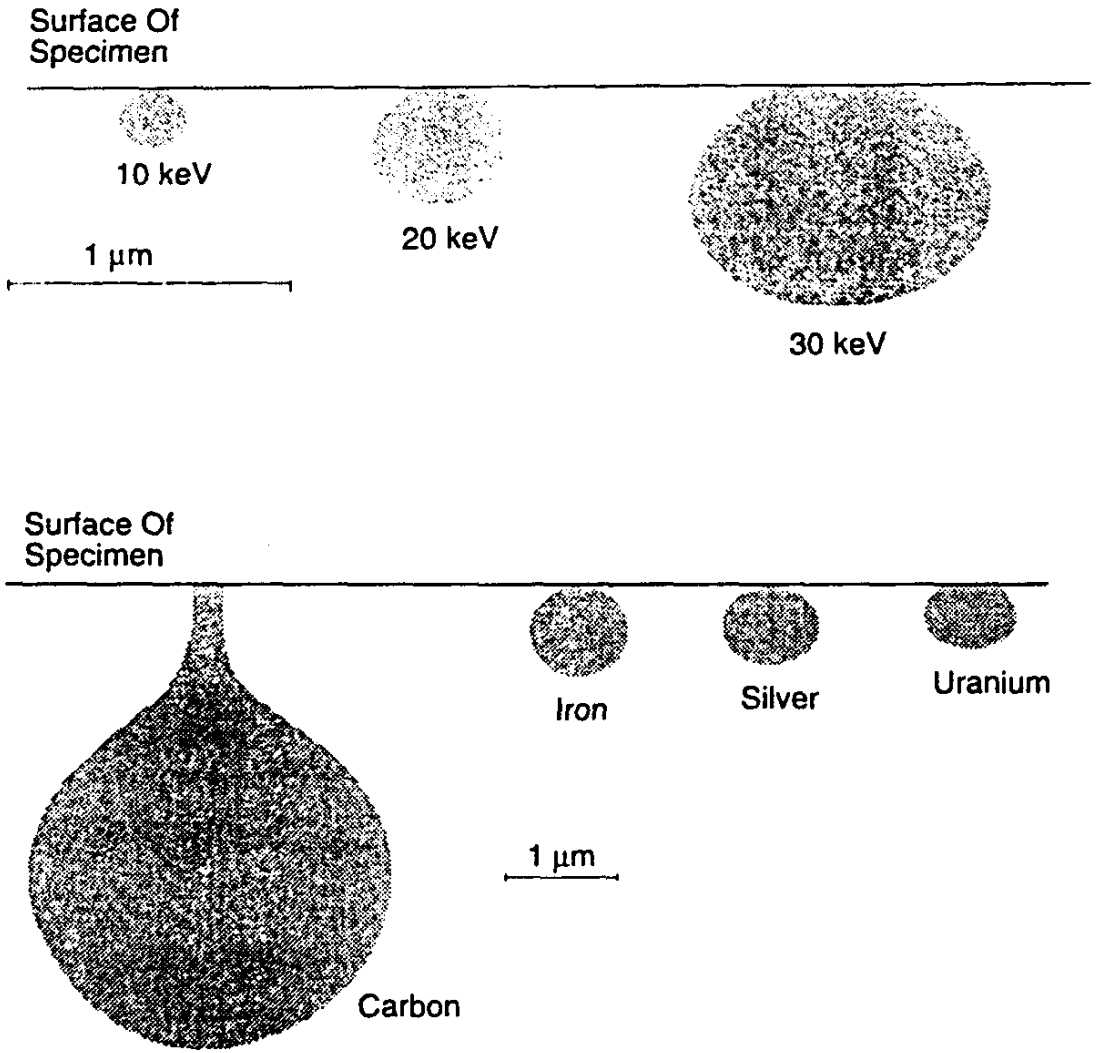


Monte Carlo simulations of 100 electron trajectories

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

Interaction Volume: II

The penetration or, more precisely, the **interaction volume** depends on the **acceleration voltage** (energy of electron) and the **atomic number** of the specimen and e^- beam size



Escape Volume of Various Signals

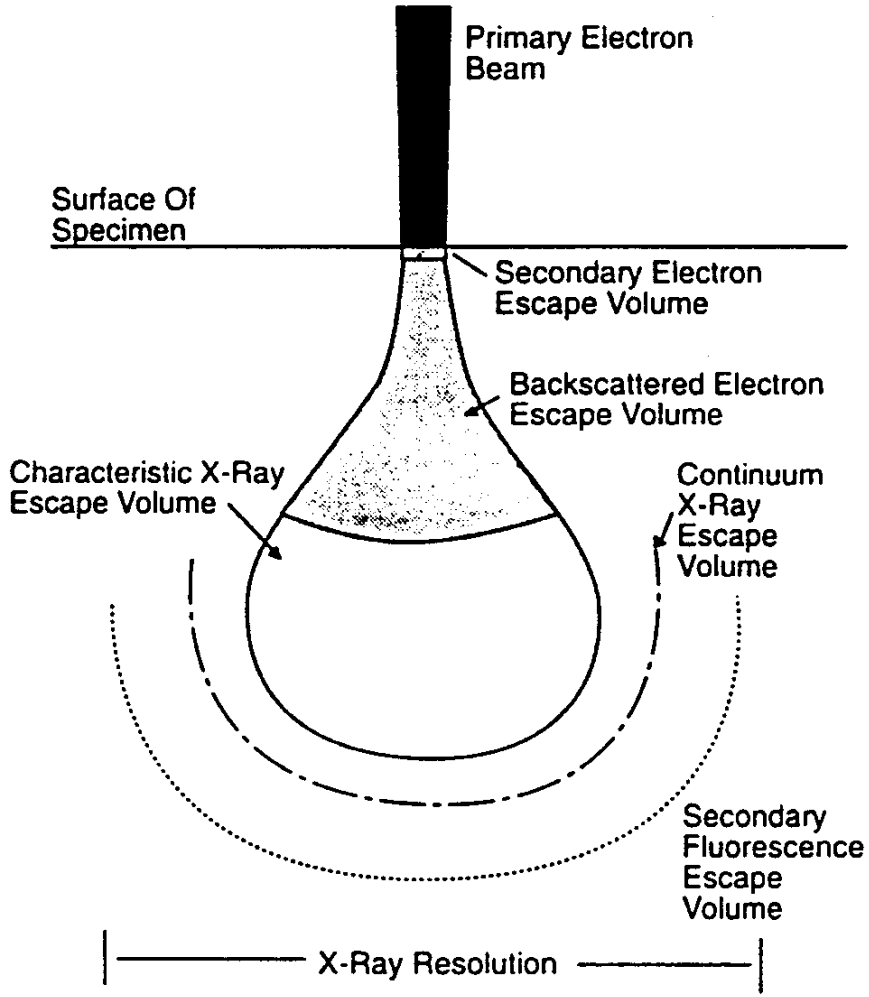
- The incident electrons interact with specimen atoms along their path in the specimen and generate various signals.
- Owing to the **difference in energy** of these signals, their '**penetration depths**' are different
- Therefore **different signal** observable on the specimen surface comes from **different parts** of **the interaction volume**
- **The volume responsible for the respective signal** is called the **escape volume of that signal.**

Escape Volumes of Various Signals

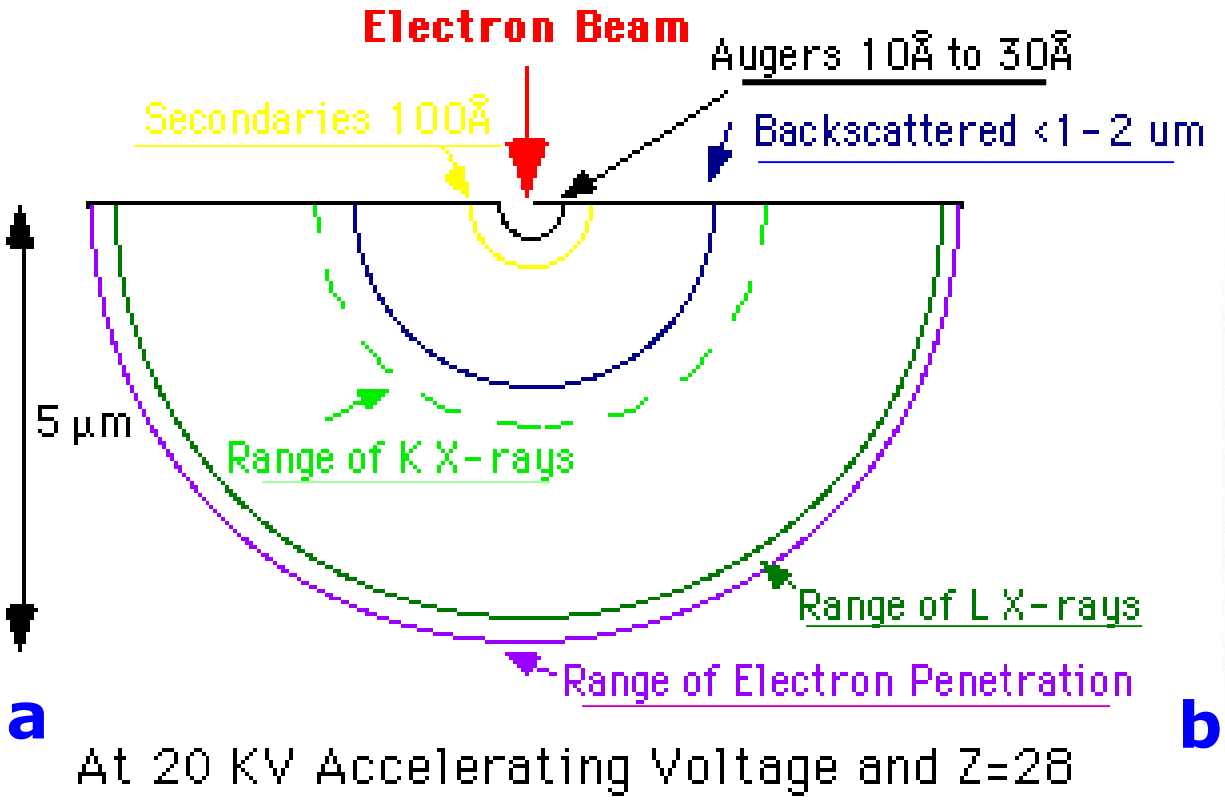
If the diameter of primary electron beam is ~5nm

- Dimensions of escape zone of

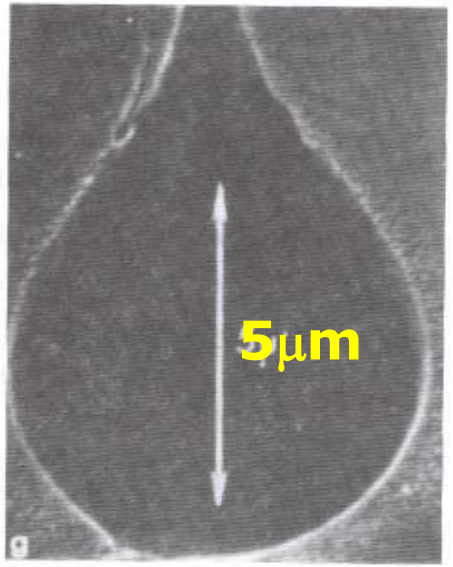
- **Secondary electron:** diameter~**10nm**; depth~**10nm**
- **Backscattered electron**: diameter~**1μm**; depth~**1μm**
- **X-ray**: from the whole interaction volume, i.e., ~**5μm** in diameter and depth



Electron Interaction Volume



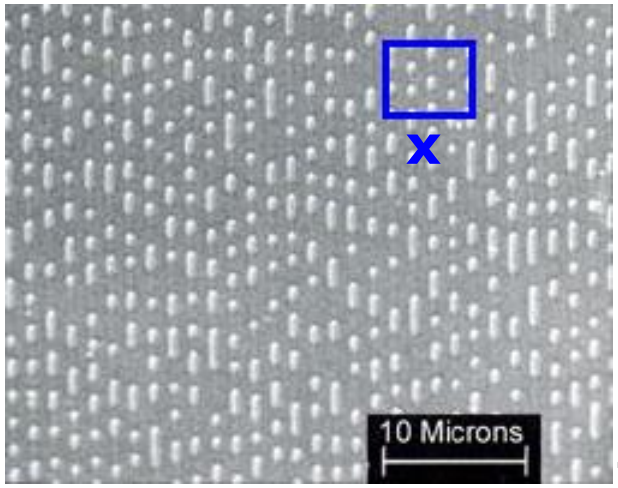
Pear shape



b

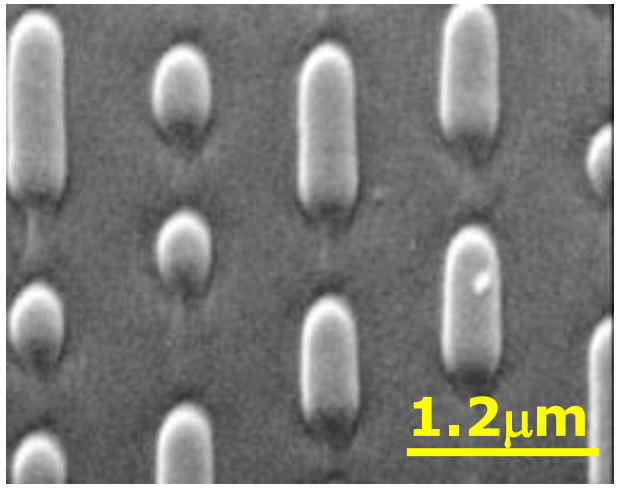
a.Schematic illustration of electron beam interaction in Ni
b.Electron interaction volume in polymethylmethacrylate (plastic-a low Z matrix) is indirectly revealed by etching

Magnification



Low M
Large x
40μm

2500x



High M
small x
7μm

15000x

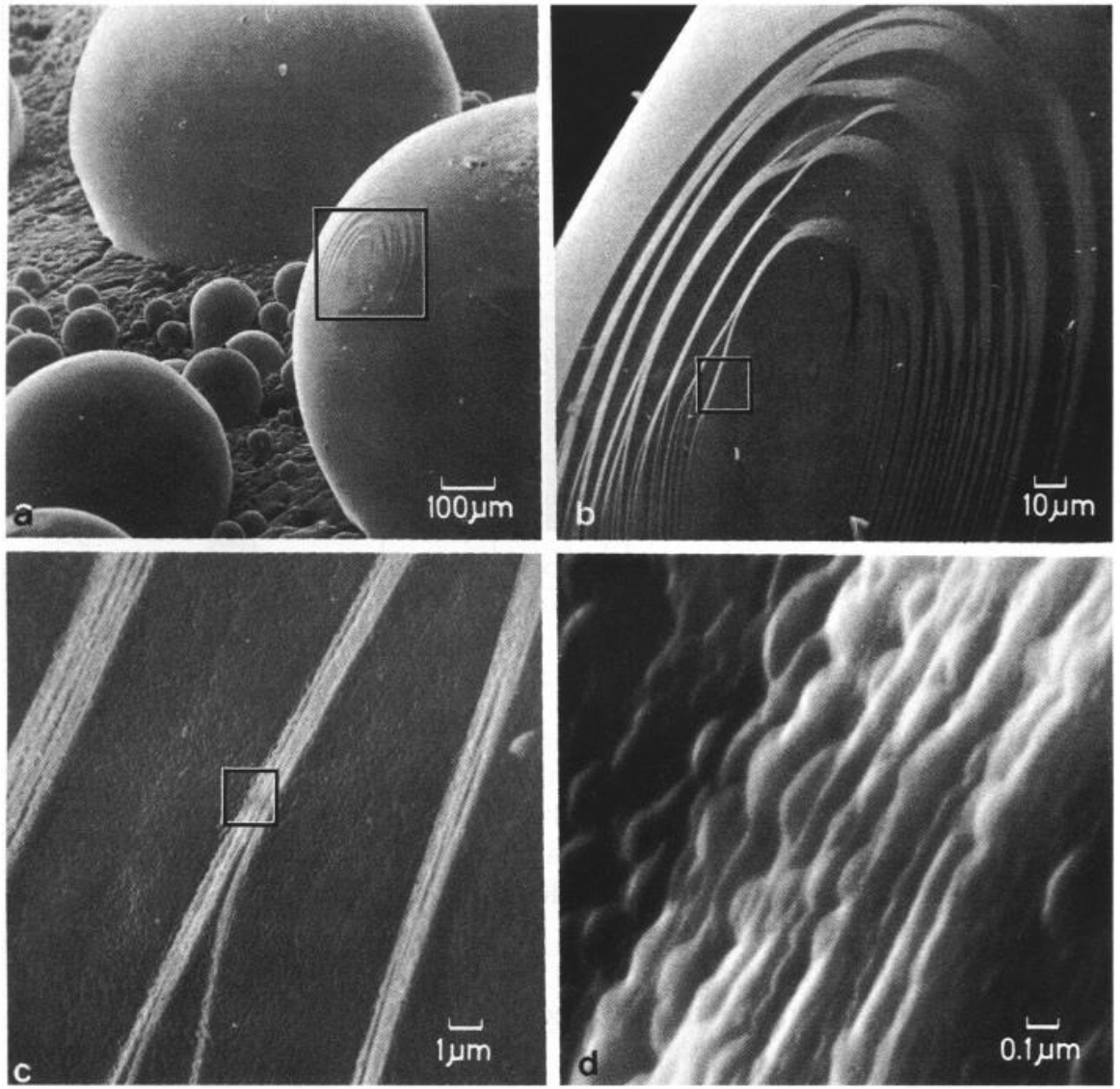
The magnification is simply the ratio of the length of the scan C on the Cathode Ray Tube (CRT) to the length of the scan x on the specimen. For a CRT screen that is 10 cm square:

$$M = C/x = 10cm/x$$

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)

Ultimate resolution obtainable in an SEM image can be limited by:

$\lambda \sim 500nm$ $NA = n\sin\alpha$

$d_{min} = 0.61\lambda/NA$ for OM

1. **Electron Optical limitations**
   Diffraction: $d_d = 1.22\lambda/\alpha$
   for a 20-keV beam, $\lambda = 0.0087nm$ and $\alpha = 5x10^{-3}$ $d_d = 2.1nm$
   Chromatic and spherical aberrations: $d_{min} = 1.29\lambda^{3/4} C_s^{1/4}$
   A SEM fitted with an FEG has an **achievable resolution** of ~1.0nm at 30 kV due to smaller C_s (~20mm) and λ.
2. **Specimen Contrast Limitations**

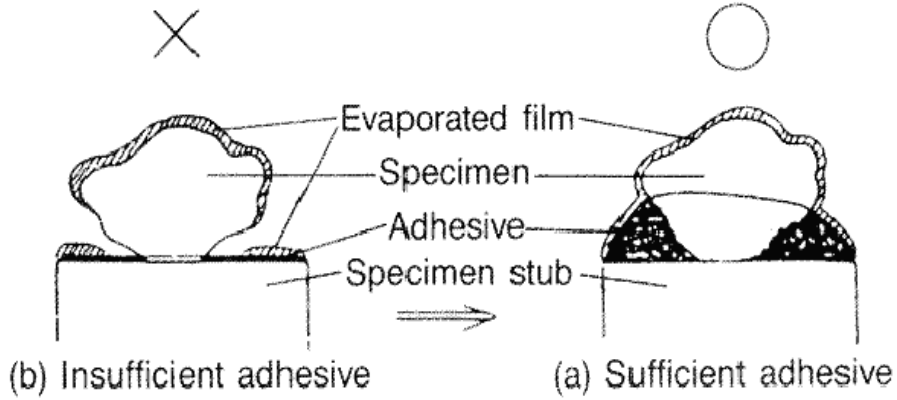
Contrast	d_{min}
1.0	2.3nm
0.5	4.6nm
0.1	23nm
0.01	230nm

3. **Sampling Volume Limitations (Escape volume)**

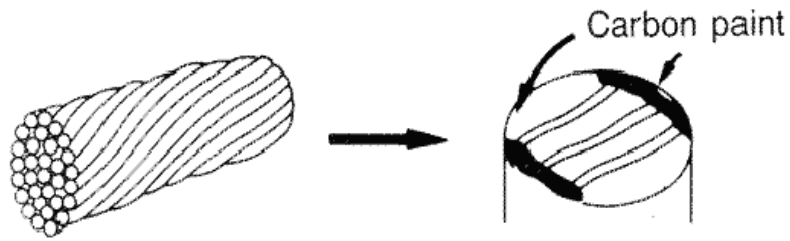
http://www.youtube.com/watch?v=SVK4OkUK0Yw at~1:47-3:07

C_s – coefficient of spherical aberration of lens (~mm)

Sample needs to be conductive

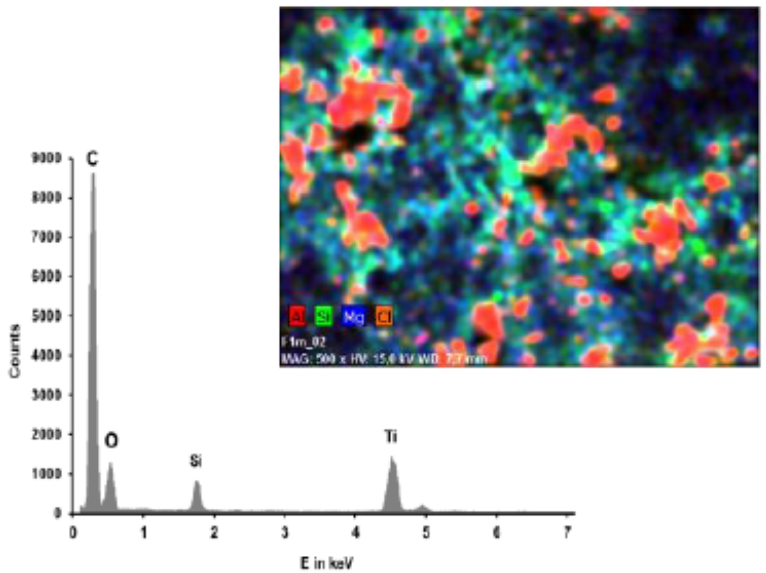


Fixing of a bulk specimen

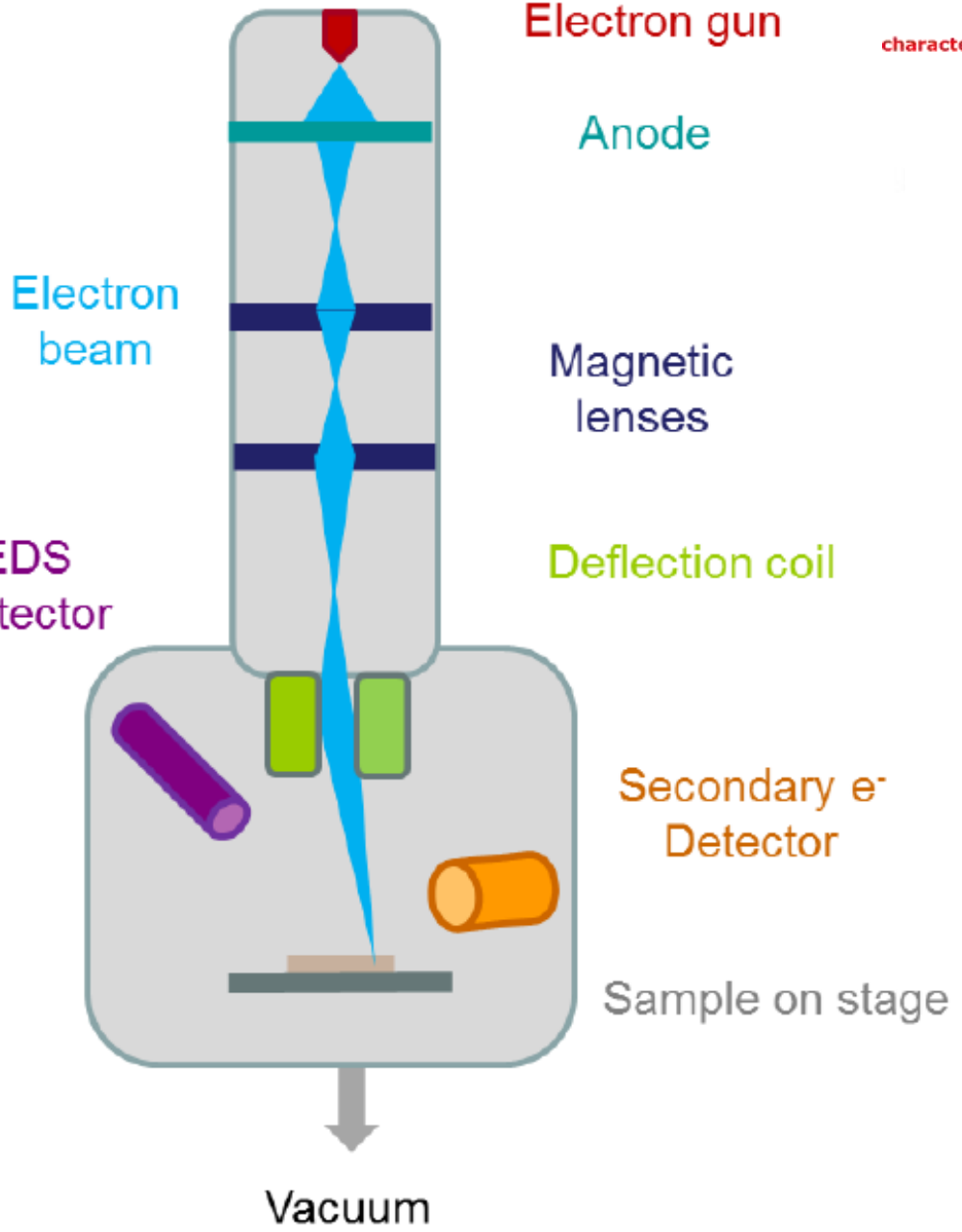


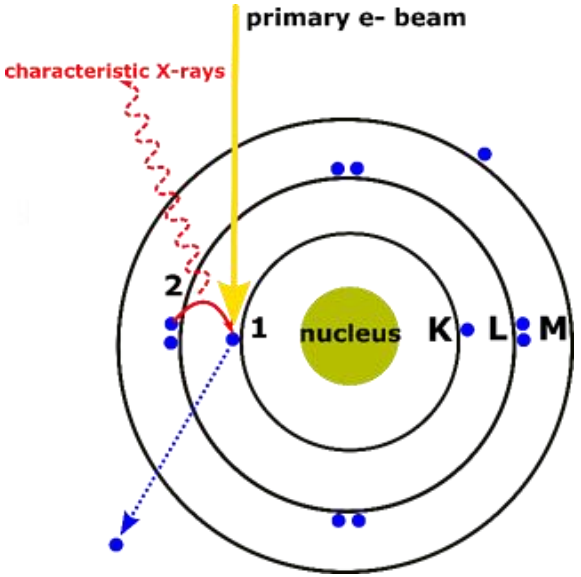
Fixing of fiber

In addition to secondary electrons imaging, Energy Dispersive X-ray Spectroscopy (EDS or EDX) analysis is used for chemical analysis.
The EDS system detects the X-photons emitted by the sample after excitation by the electron beam. The energies of the emitted X-photons are characteristic of each chemical element.

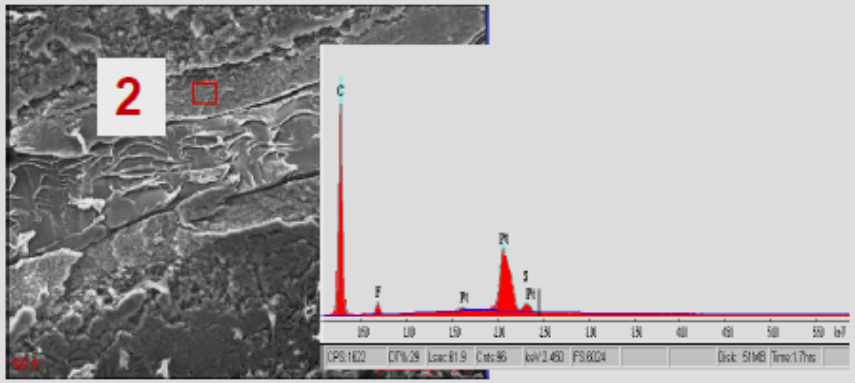


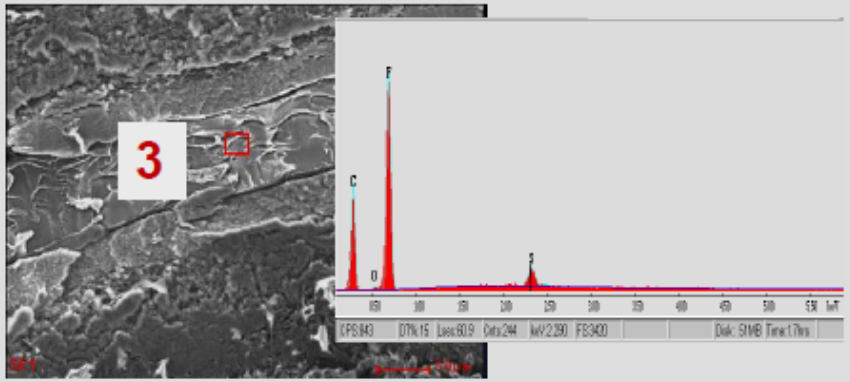
X-ray spectra give quantitative information about the elemental composition of the sample surface. 1D scans and 2D maps can be generated.



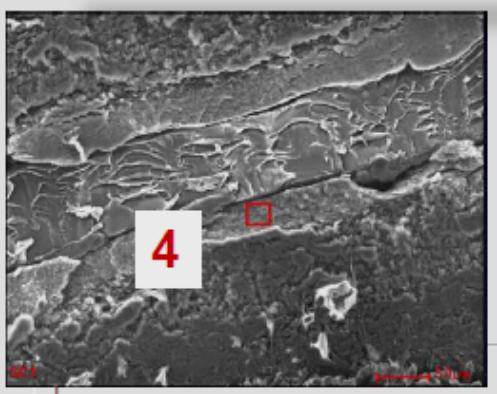


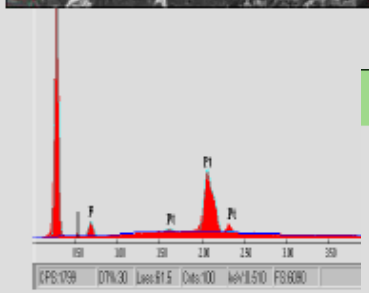
Chemical analysis using EDS





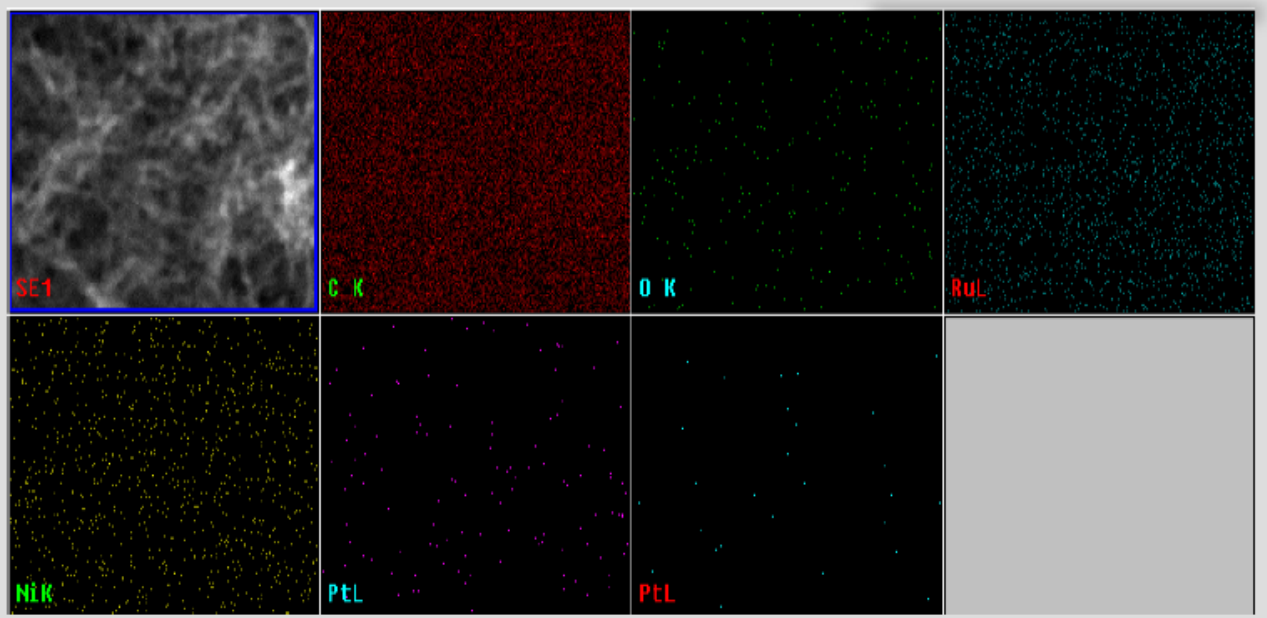
Membrane electrode assembly **(MEA)**





Elemental Mapping using EDS

5% Pt - Ru / NiCNT



SEM and EDS techniques

Strengths

- Rapid, high-resolution imaging
- Quick identification of elements present
- Good depth of field
- Versatile platform that supports many other tools

Limitations

- Vacuum compatibility typically required
- May need to etch for contrast
- SEM may spoil sample for subsequent analyses
- Size restrictions may require cutting the sample
- Ultimate resolution is a strong function of the sample and preparation
- Some elements can not be detected in the EDS

SEM and EDS techniques

Main Uses

- Reveal topographical surface details
- High resolution images
- Detect compositional differences
- Elemental microanalysis and particle characterization

Relevant Industries

- Aerospace
- Automotive
- Biomedical/biotechnology
- Compound Semiconductor
- Electronics
- Industrial Products
- Pharmaceutical
- Photonics
- Polymer
- Semiconductor
- Solar Photovoltaics
- Telecommunications