Electron Microscope

Reference: J. Goldstein et. al., Scanning electron microscope and X-Ray microanalysis

Origin of Alternate Microscopic Instrument

Limitations with Light microscopes:

1. Spatial resolution limited by the wavelength sensitive to eye
2. Depth of field extremely low at higher magnifications
3. Optical components cannot be tuned in-field (aberrations are determined by various factors not easily tuned)
4. Higher magnifications required to visualize sub-micron features are not practical due to different trade-offs.

SOlution is to use radiations with smaller wavelengths!

Solution: Electron microscope

Why electrons ?

Electrons in material can be ejected, accelerated to required velocities.

Fundamentally all systems have wave-particle duality. Electron velocities can be adjusted to have waves with sufficiently low wavelength

Electron beams can be converged, diverged and suitably chiseled using electrically tuned components ‘electron optics’

Immense experience exists thanks to CRT televisions

Electron Gun

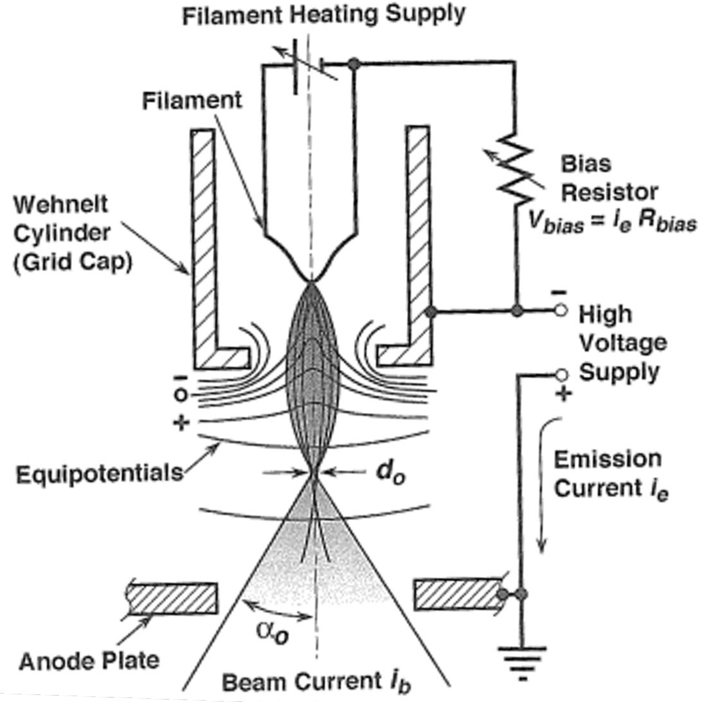
Otherwise called a Cathode Ray Tube:

Cathode is typically a metallic wire (W) typically bent like a 'V' shape

Wire heated to about 2000 - 2700 K by resistive heating.

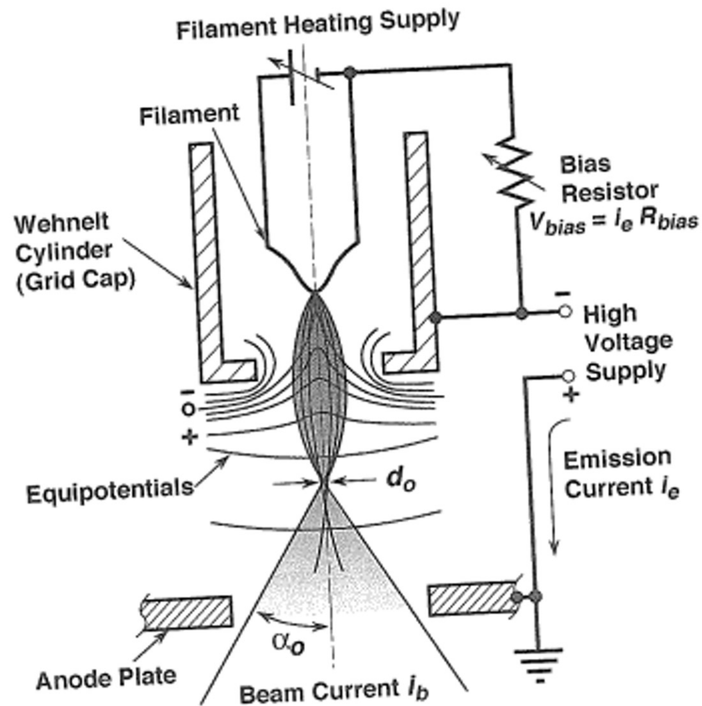
Sharpest region - largest current density - region of significant electron emission

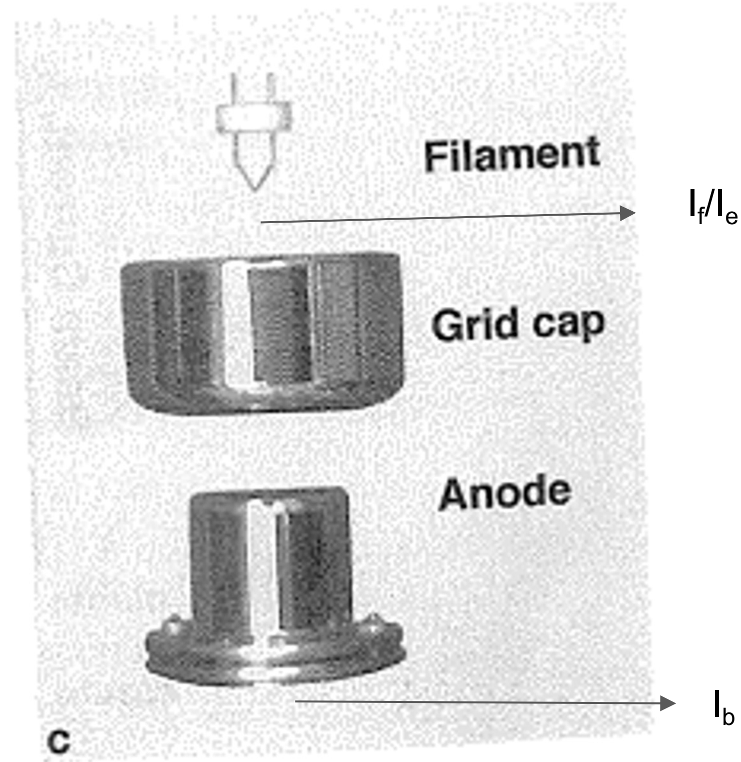
Grid cap - enclosure tied to negative potential. Equipotential lines shown in the image focuses the electron beams to lesser-negative regions



Finally, the Anode plate is grounded. The cathode is set to about -20kV, the large potential difference leads to electron acceleration and a beam current below the anode plate

Currents in electron sources





Electron Lenses

Electron motion in magnetic field is determined by force

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

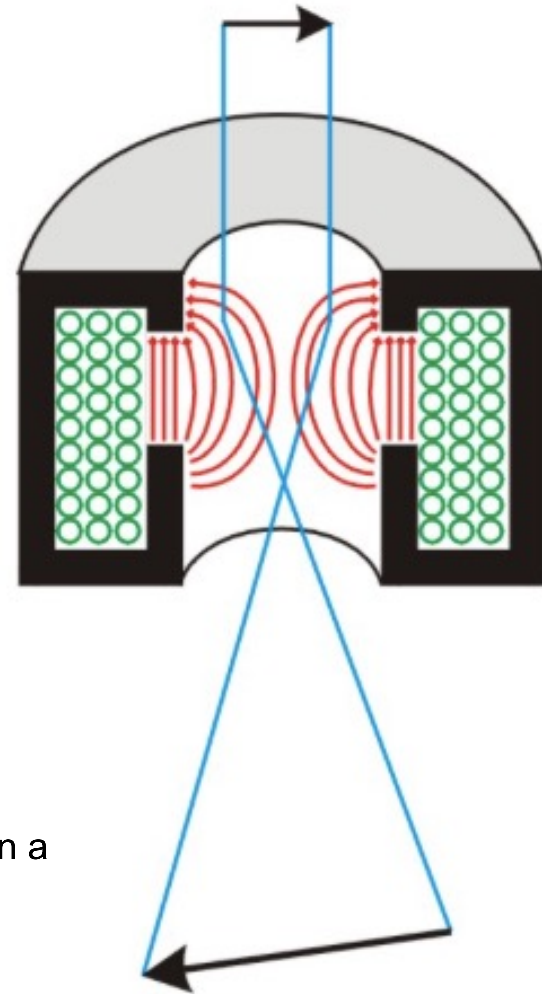
In a purely magnetic field

$$F = evB \sin \theta$$

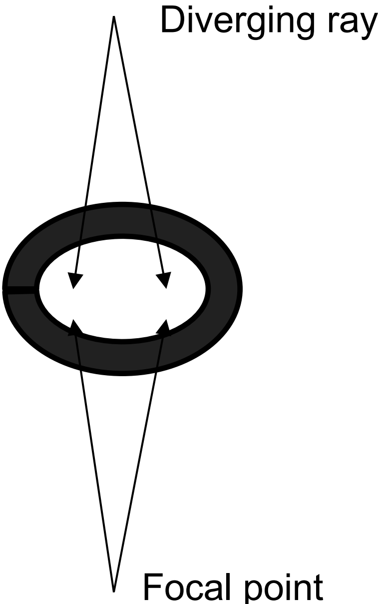
θ angle between v and B

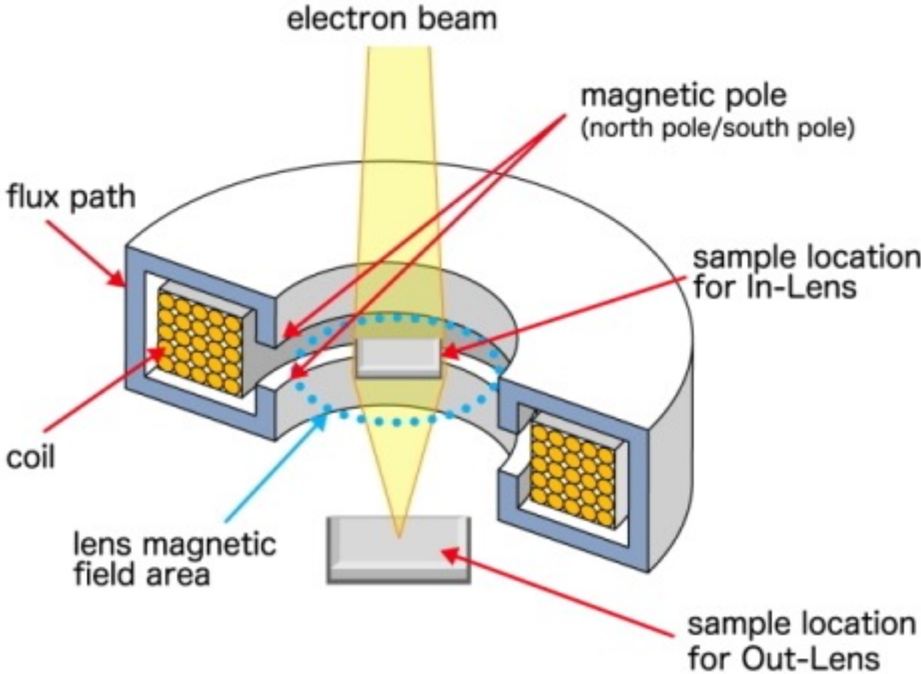
Rotationally symmetric magnetic field is inhomogeneous
- field is stronger near the core and weaker near the center of the bore.

-This makes the electrons near the coils bend towards the center, in a circular fashion
- The extent of bend/deviation depends on the magnetic field (B) which can be controlled by the current through the lens coils.



Electron path inside a lens



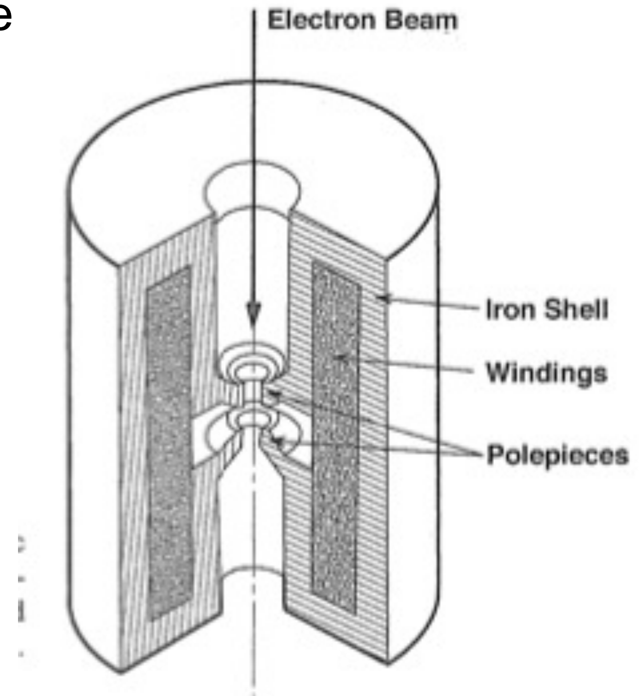


Focusing in electron optics

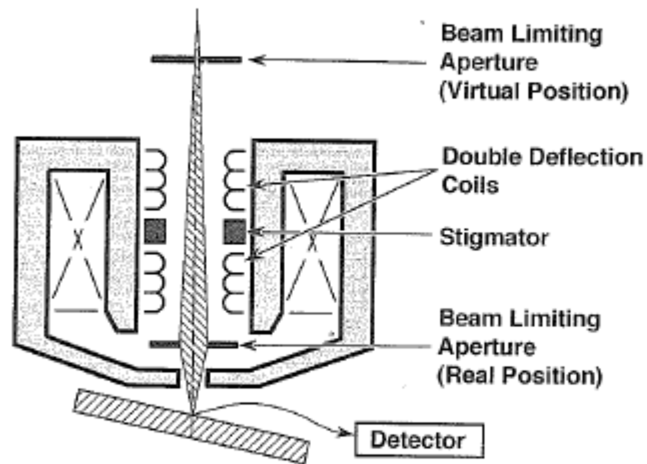
- The focal point is defined as the distance from the point at which the rays begin to bend to the point where it intercepts the axis.
- Typically $f = \frac{V_0}{(NI)^2}$, where V_0 is the accelerating voltage, I current inside the N turn coil.
- Following geometric optics

$$\frac{1}{f} = \frac{1}{p} + \frac{1}{q}$$

Where p is the object distance, q is the image distance and f is the focal length of lens



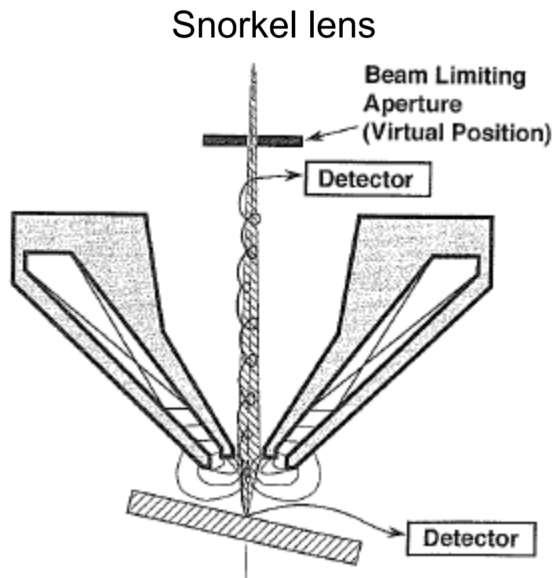
Important point to remember: the focal length of the lens is tunable

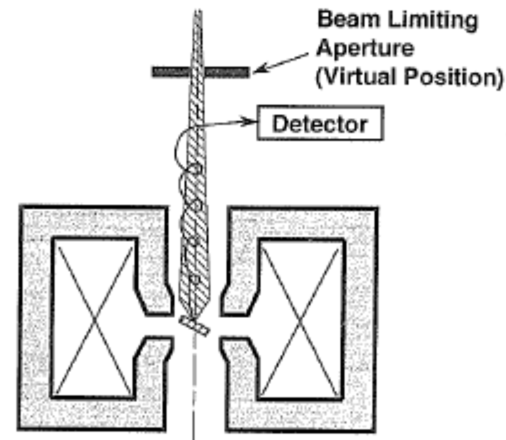


Asymmetric pin hole lens

Specimen outside,
Stronger aberrations

Snorkel lens





Symmetric immersion lens

Specimen inside lens bore

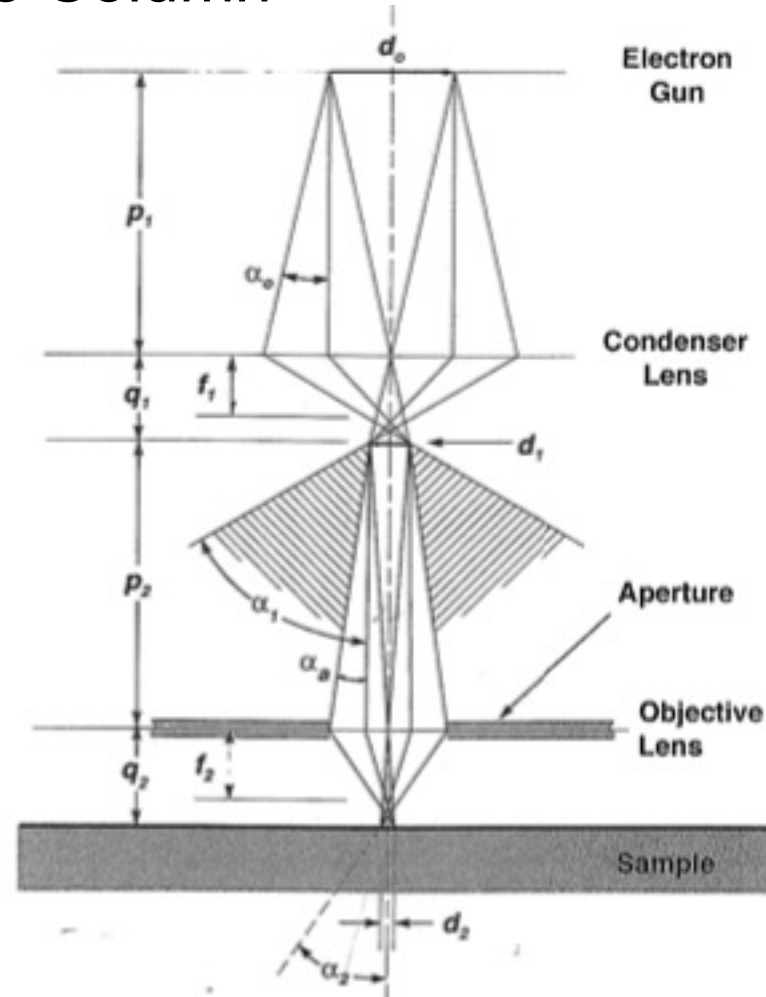
Typical Design of Electron Microscope Column

Object – electron gun typically 10 – 50 μm

Condenser lens accepts rays with an angle α, focusses it down and forms a demagnified image

A objective aperture is used to restrict beam current and limit aberrations

Objective lens converges the beam to form a spot on the sample of disk size d



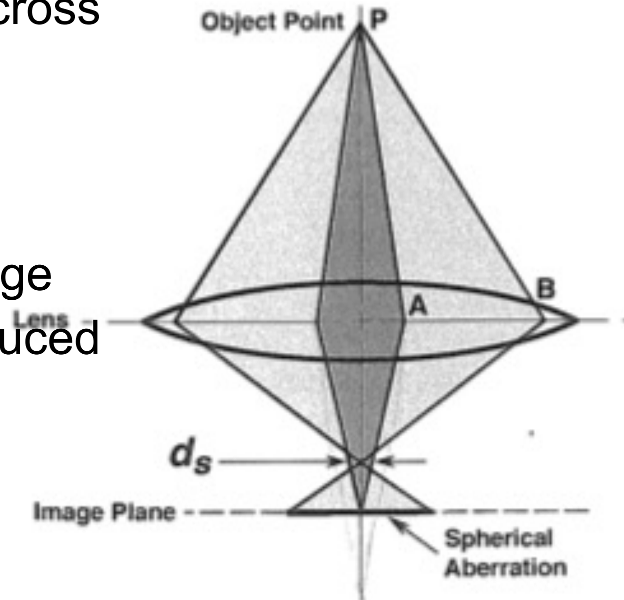
Aberrations in electron lenses

Spherical Aberration

Electrons closer to the coil are bent further than those near the axis.

This causes spread in the image to be spread across along the 'optic axis'

This leads to a disk formation instead of a point. Smallest disk is typically formed just before the right image plane. Disk called disk of least confusion (d_s) can be reduced by reducing the acceptance angle α of the lens.

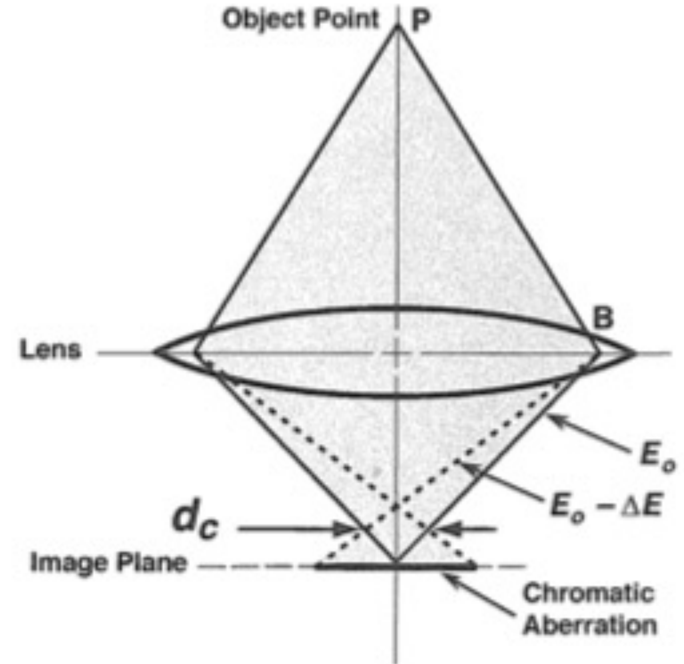


Chromatic Aberration

The electron deflection in lenses also depends on the energy (velocity).

So beams with different energy (E) will be focused differently.

If the incident beam has a central energy E_0 and a spread ΔE, then a disk of least confusion is given by $d_c = C\alpha\left(\frac{\Delta E}{E_0}\right)$

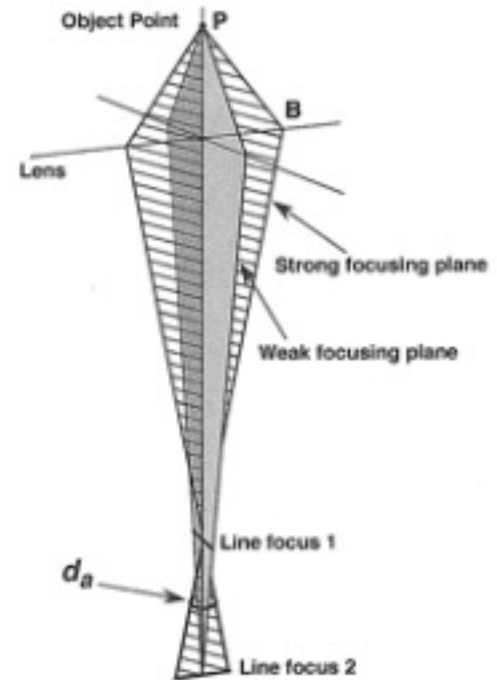


Astigmatism

Machining errors in the lenses, inhomogeneity of pole piece, asymmetery in the lens winding – deviation from perfect cylindrical hole, causes astigmatism.

Image points are stretched either along the x-plane or along the y-plane.

Way to observe:
As you move from out-of-focus to in-focus to out-of-focus, the image will stretch from one direction to its perpendicular



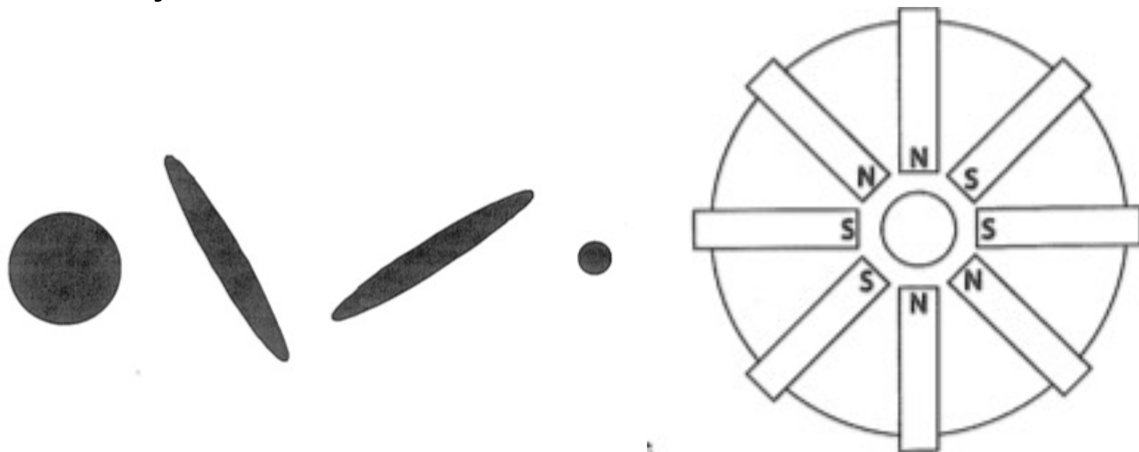
Among the most common of issues with electron lenses

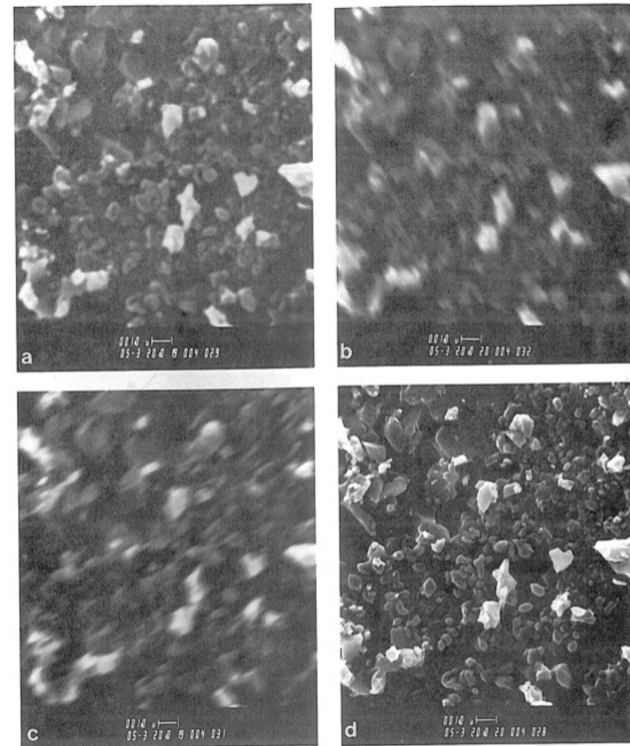
Astigmatism correction:

An octupole magnet is used to correct for astigmatism.

A typical correction cycle (x-stigmator, focus, y-sitgmator, focus) should be employed to correct.

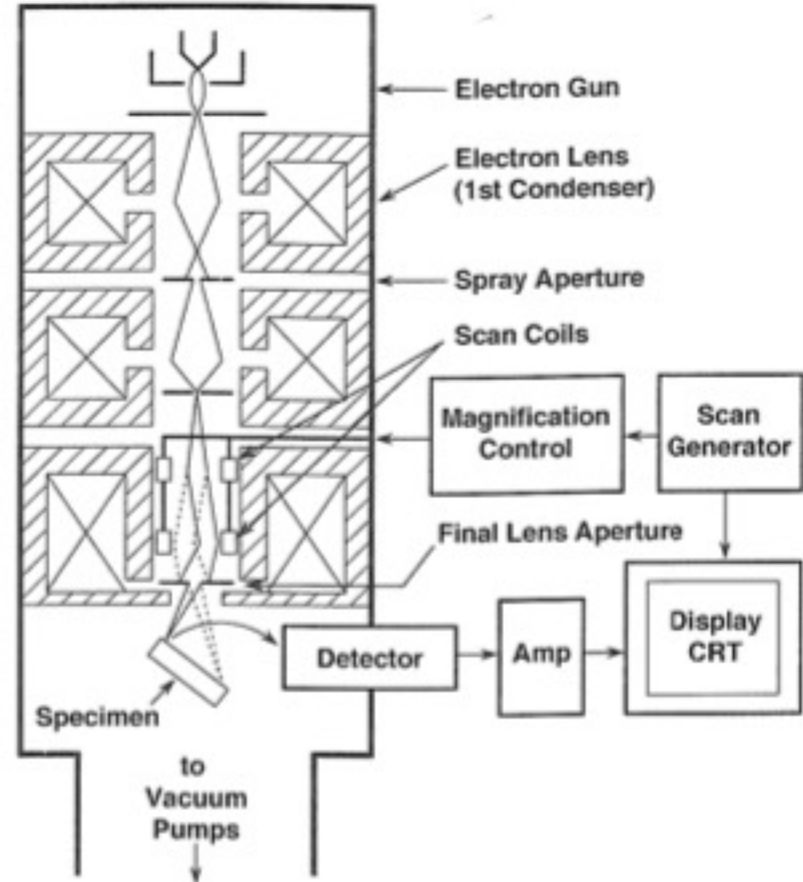
Eventually, the beam cross section will be the smallest



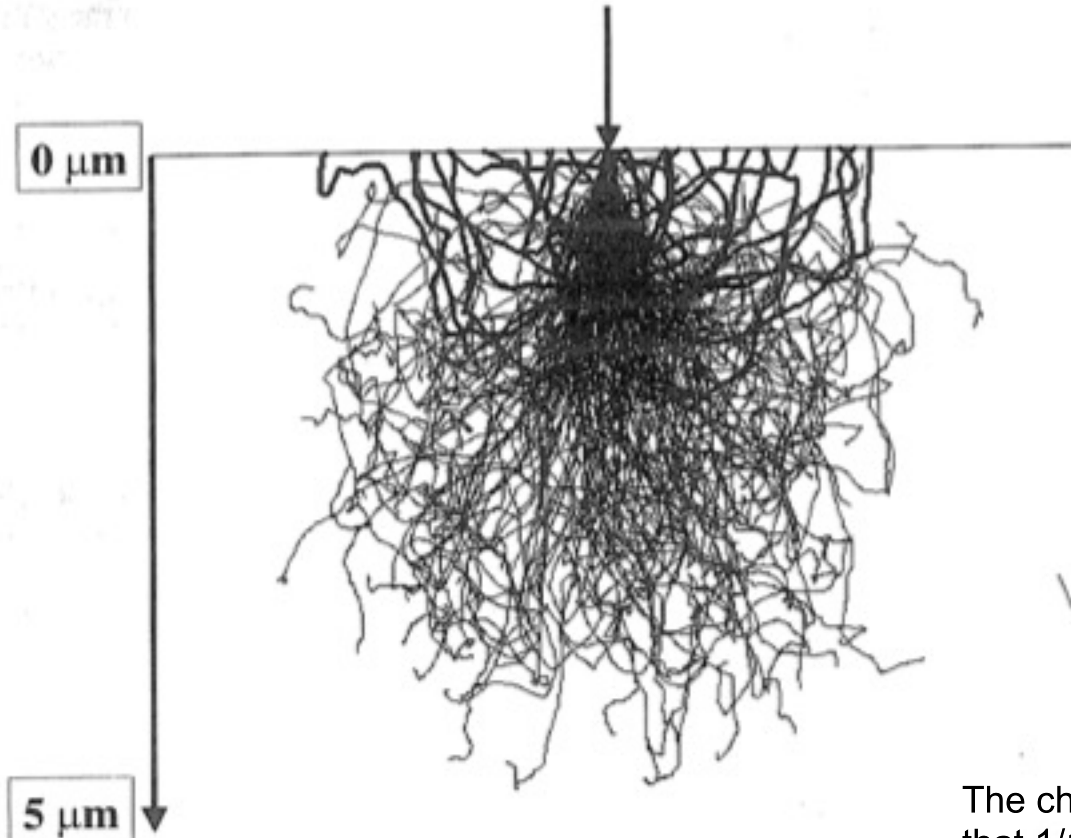


Electron – Sample Interaction

- Electrons are emitted from filament,
- Accelerated by the anode-cathode potential difference
- Diverging rays are converged onto the sample stage using condensing lenses and objective lenses.
- The beams converge on the sample and now we need to understand the e-beam sample interaction.



E-Beam accelerated at 20 kV, striking Si



-Assume the filament has a brightness of 1×10^8 A/cm^2 Sr
-Say the anode is at 20 kV higher than filament.

The electron beam that incidents the sample has a dia of 1nm, divergence 5×10^{-3} sr, and a current of 60 pA.

The chamber is at 10^{-7} Torr, which makes sure that 1/10000 electrons collide with a gas atom

Electron – Material Scattering physics

As accelerated electrons interact with specimen, two kinds of scattering can happen

1. Elastic – (No transfer of energy/mass)
2. In-elastic – (energy transfer from the electron -> atoms)

Probability to scatter elastically $Q(>\phi) = C\frac{Z^2}{E^2}\cot^2\frac{\phi}{2}\ [cm^2]$ –scattering cross section

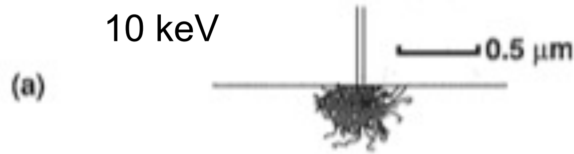
Z – atomic number, E – electron energy, ϕ – scattering angle.

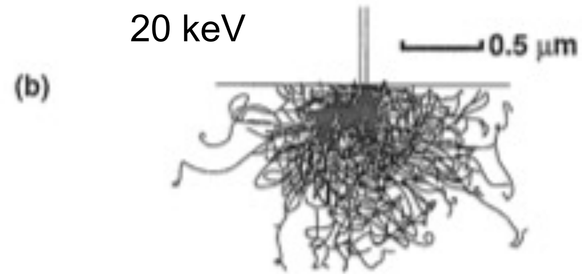
In similar spirit, the rate of energy loss for inelastic scattering is given by Bethe equation

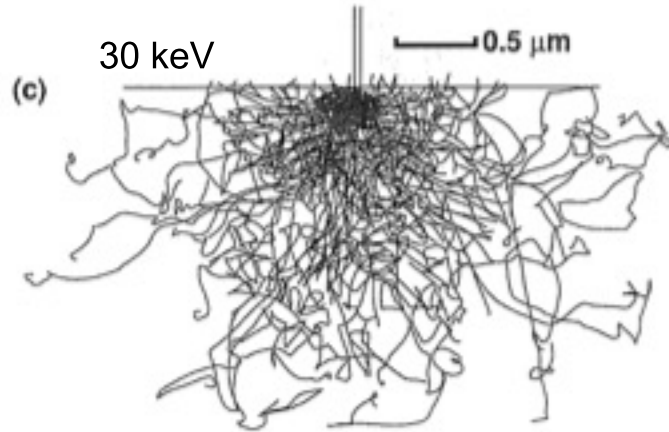
$$\frac{dE}{ds} = C\frac{Z\rho}{AE_i}\ln\frac{1.66E_i}{J}$$

ρ – density, J average loss of energy/event

Variation of Interaction volume with electron acceleration







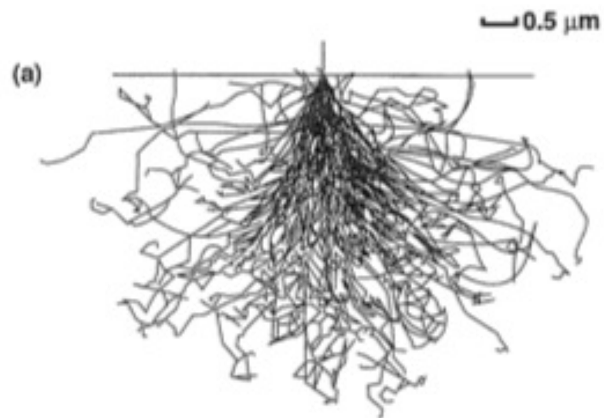
Elastic scattering rate inversely proportional to electron energy

Electron energy loss $\frac{dE}{ds} \, \alpha \, \frac{1}{E}$

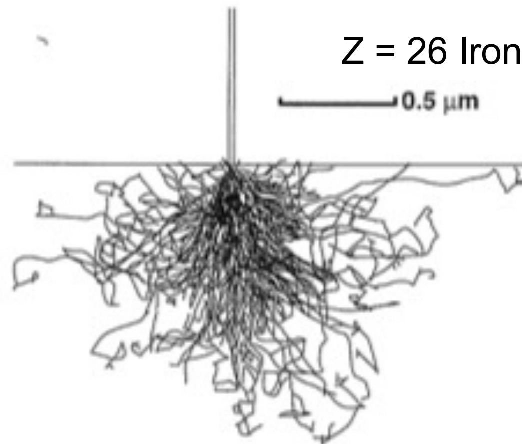
So beam penetrates further inside the material.

Some trajectories exist the material surface – They are called **back scattered electrons** of the beam!

Z = 6 Carbon

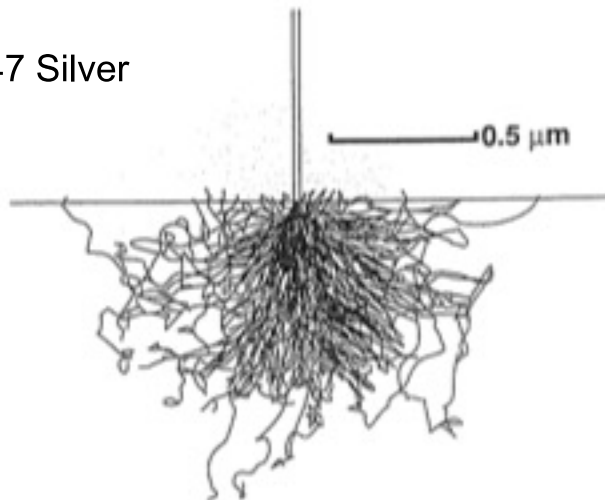


Z = 26 Iron

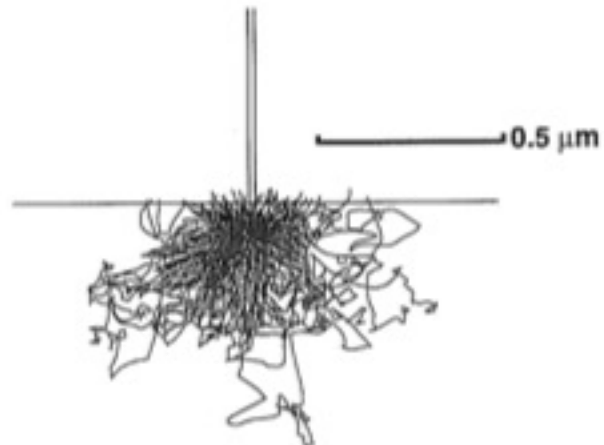


Beam Energy 20 kV

Z = 47 Silver



Z = 92 Uranium

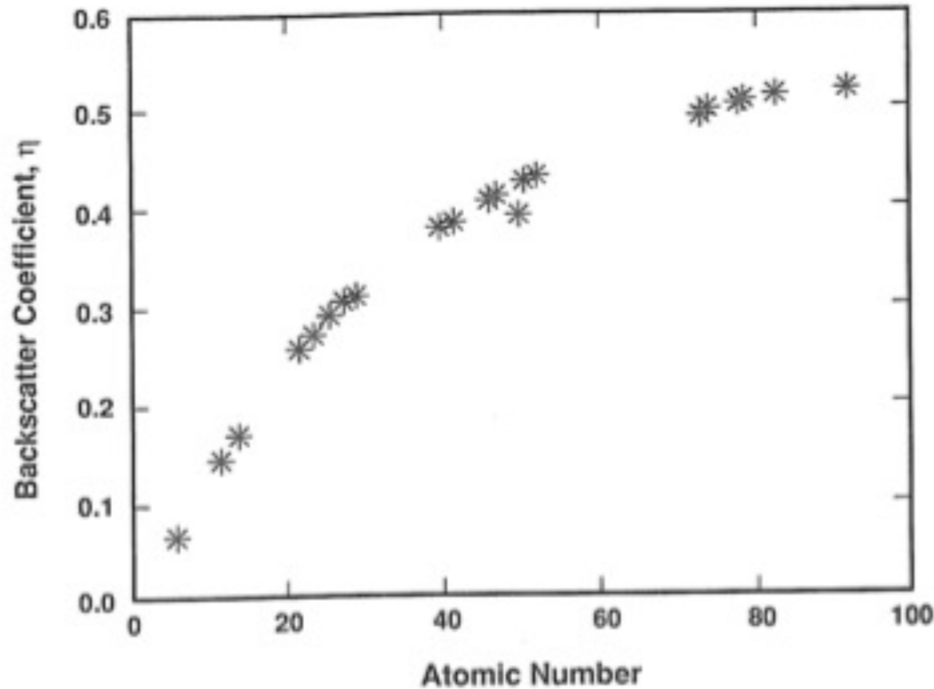


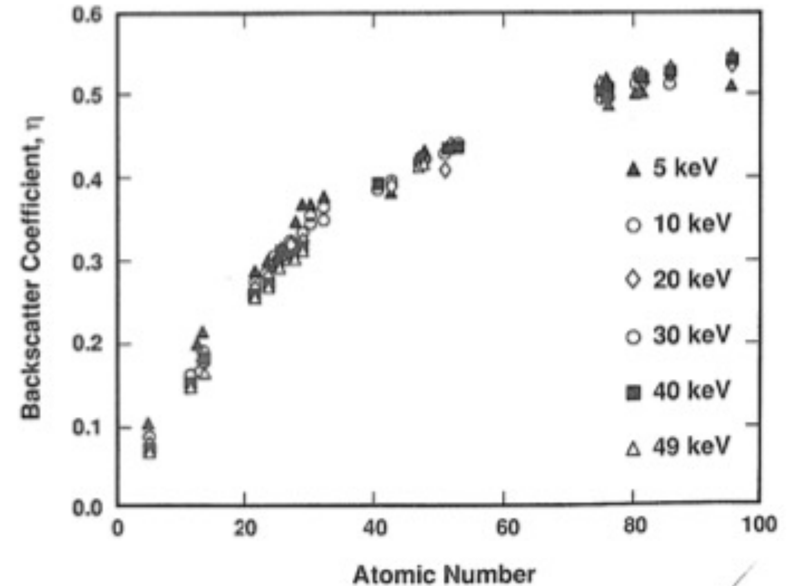
- As Z increases, the cross section (probability) for large angle elastic scattering increases. $Q \sim Z^2$
- For lower atomic number, the scattering angles are not large, the beam penetrates deeper into the sample, decaying slowly. Smaller percentage of electrons exit the material. Interaction volume – pear shaped
- For higher atomic number, the interaction volume is smaller, yield of BSE higher. Interaction volume plane truncated hemisphere.

We define back scattering coefficient $\eta = \frac{n_{BSE}}{n_B}$

Where n_{BSE} is the number of back scattered electrons and n_B the number of electrons in the beam incident on the sample

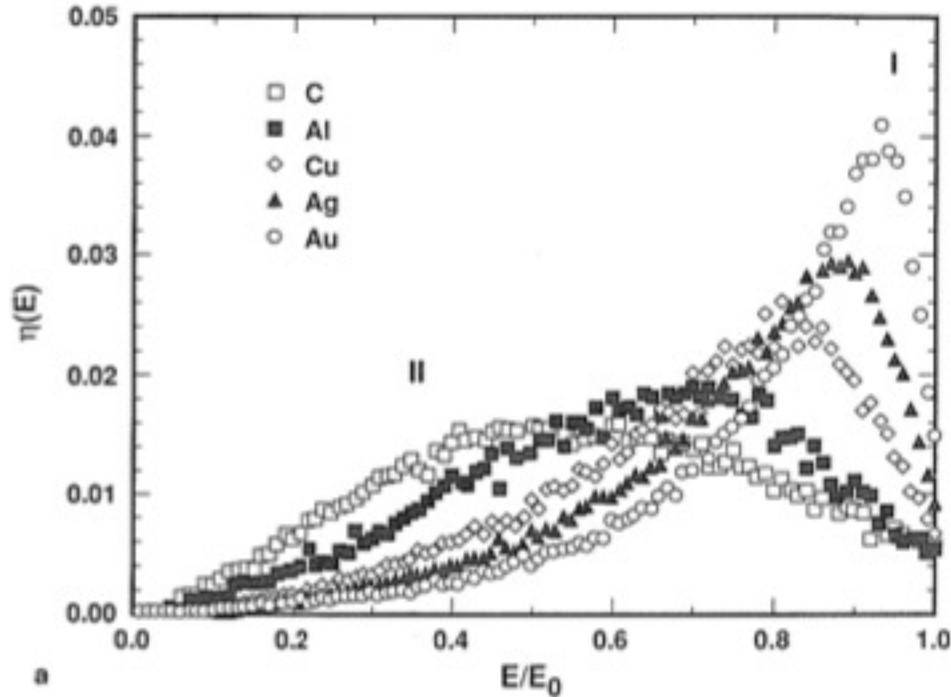
η dependence on energy and sample material





Atomic number is the most dominant contributor to BSE yield. Thus, a great method to obtain material contrast.

Energy of BSE



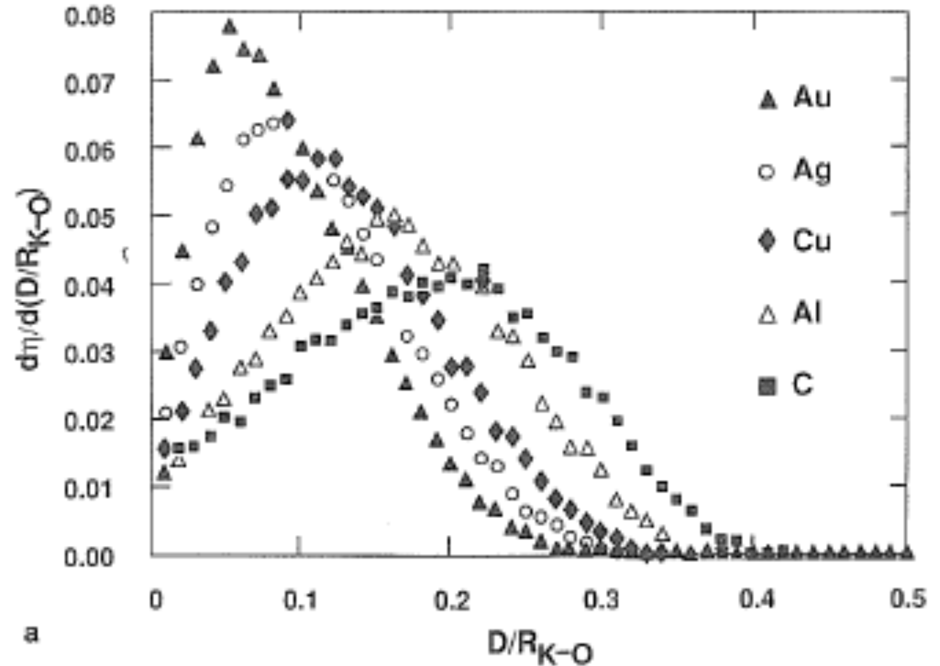
The energy of BSE electrons are clearly separated.

High Z substrates has BSE yield with energy very close to incident beam (Region 1)

Low Z substrates has poor BSE yield and even then their energies are low (region II).

Thickness dependence of BSE yield

1. Heaver atoms give maximum yield
2. The yield is obtained mostly closer to the surface.
3. Lighter elements gives generally a smaller yield
4. Most of the exiting electrons are from certain distance from the surface

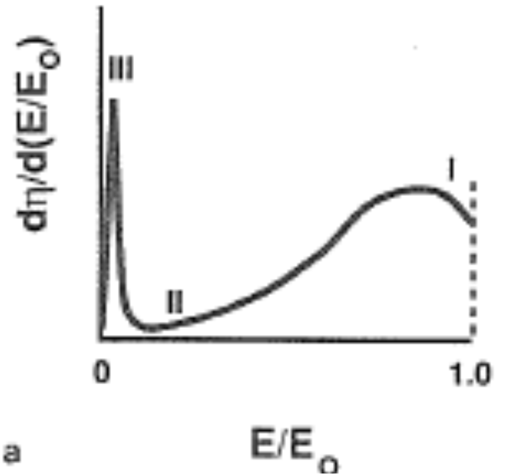


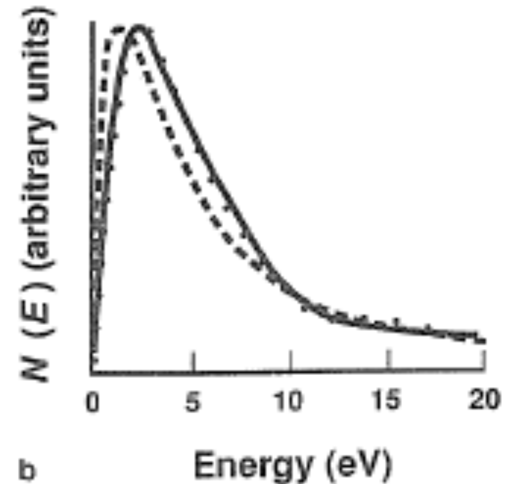
Effect of sample tilt (Given to self-reading)

Very intuitive.

Secondary electron

- The largest (integrated) yield of electrons in SEM are from elastic scattering (similar to the beam energy)
- However, the yield of electrons at very low energy (<50 eV) is higher in numbers than the high energy electrons.
- These are secondary electrons (electrons with energy less than 50 eV (arb. Choice)).
- 90% of the electrons are ejected with energy 0 – 10 eV.





Thickness dependence

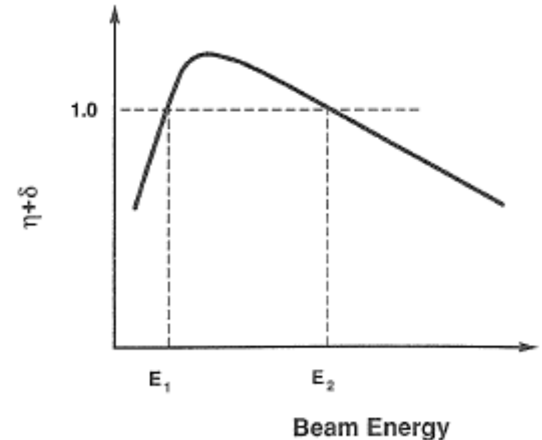
The probability of electron to escape depends on the distance from surface

$$p = e^{-Z/\lambda}$$

Z – distance from surface, λ is mean free length of electron

Typical maximum distance of SE yield 5λ

If we define $\delta = \frac{n_{SE}}{n_{BSE}}$ and $\eta = \frac{n_{BSE}}{n_{beam}}$



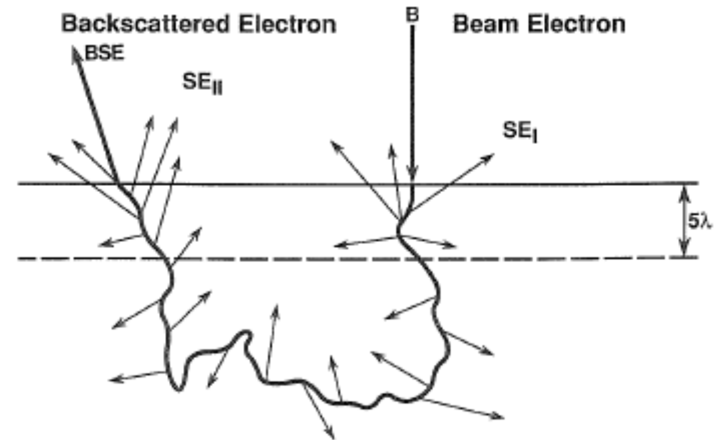
There exists a window of maximum electron yield

Types of secondary electrons

- SE – I emitted electrons at the beam incidence
- SE – II emitter electrons due to the back scattered electrons

SE – 1 which emerges near the beam position depends purely on the surface properties. It has high spatial resolution (electron emerging largely from the beam position.

However, SE- II which has its origin in BSE, has material dependence as well. Can have poor spatial resolution because the electrons exit from very different positions on the surface.

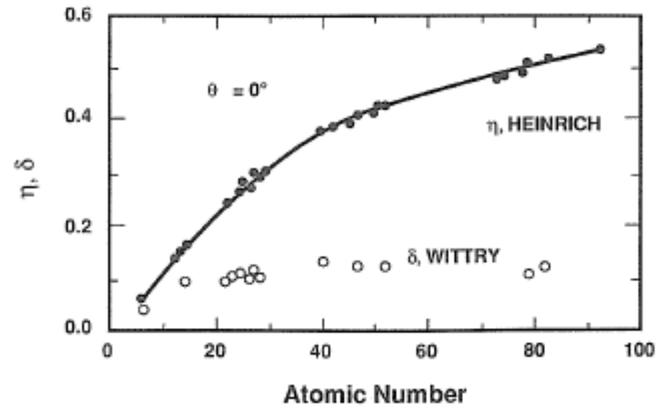


Compositional difference

Total secondary electron count $\delta_T = \delta_1 + \delta_2\eta$

δ_2 has its origin in the BSE and hence is multiplied by η

In typical scenarios, $\delta_2 = (3-4)\delta_1$
ie, BSE is far more efficient in generating SE.



While BSE has almost linear relation with atomic number, the total SE is largely unaffected.

Detectors

- Electron detectors
  - Back-scattered electron detector (Electrons with energy 0.7 – 0.9 E_b)
  - In-lens detector (secondary electron detector (0 – 10 eV)
  - Everhart-Thornley detector
- Photon Detectors
  - X-Ray detectors with EDS/WDS
  - Photomultipliers

Everhart-Thornley detectors

Main components:

Scintillator – capable of luminescence

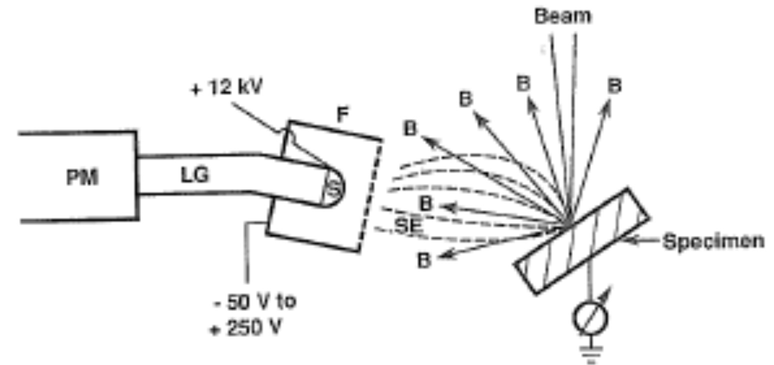
Eg: doped plastic/glass, doped CaF_2

High energy electrons/Xrays incident on scintillator leads to photo emission.

A positive bias is applied to accelerate the electrons when its incident on the material

A faraday cage (metal sheet) can be set to a bias potential to select electron energies.

Photons guided by light guide to photomultiplier

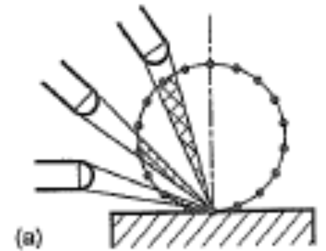


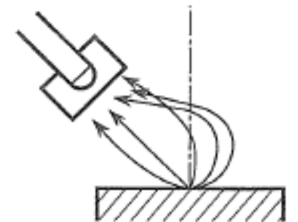
- A large potential is set close to the scintillator material
    - The metal sheet (faraday cage) sheets the electric field from affecting the electron beam)
    - The metal sheet can also be slightly biased to select the electron energy.

When the metal sheet is biased negatively

Electrons with small energies are repelled away (SE electrons).

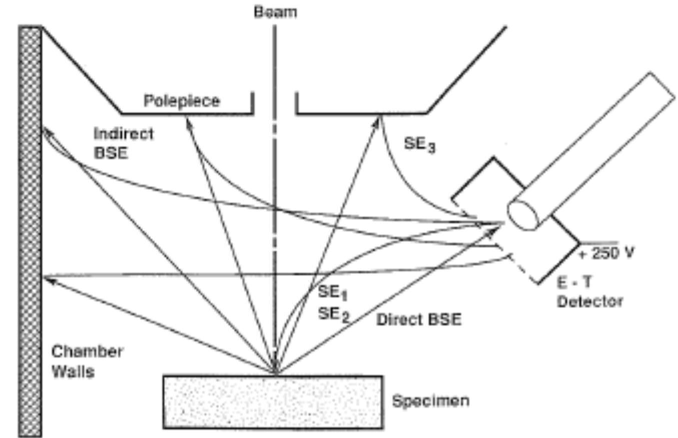
High energy electrons are collected with line-of-sight





When positive potentials are applied, all electrons are collected.
However, BSE count is not changed largely (those with-in the line of sight),

But there is a huge increase in in-directly generated electrons



In short:

In ET detectors with –ve cage bias

- BSEs are largely collected.

In ET detectors with +ve cage bias

- both BSE + SE are collected.

+ve biased detectors are efficient detectors such that they can function even at very low acceleration potentials (~ 1 keV)

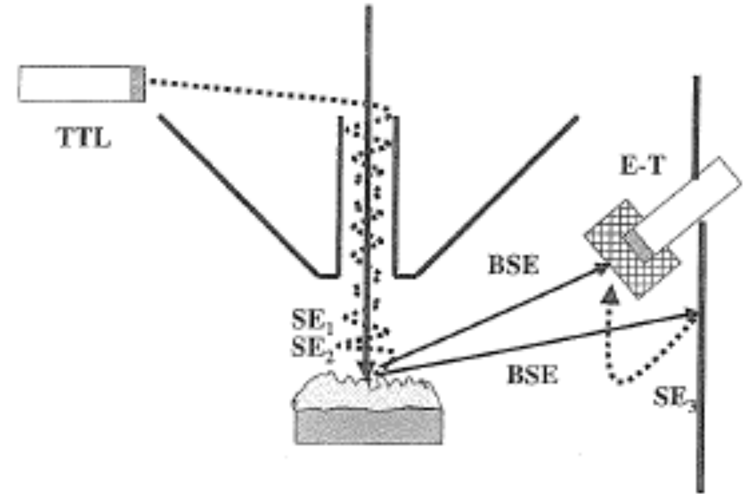
In-lens detectors

- In microscopes with Snorkel lenses

An additional detector can be placed inside the lens.
Magnetic field lines of the lens collects the electrons exiting the surface very near the beam entrance.

Such electrons are collected with high efficiency with positively biased scintillator.

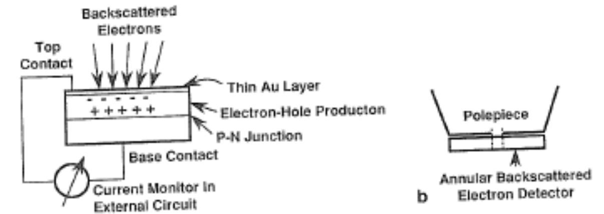
In-lens detector avoids SE3 and BSE efficiently.

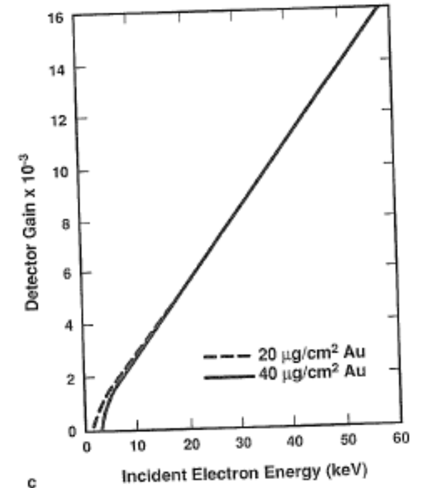


Kinds of BSD

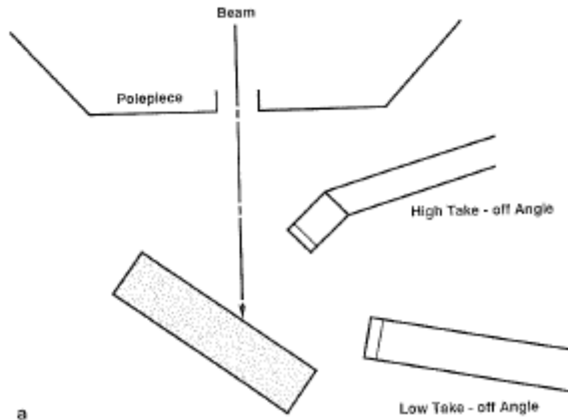
- BSE are easier to detect due to their energy.

Annular BSE detector





Direct Scintillator detector



BSE – SE detector

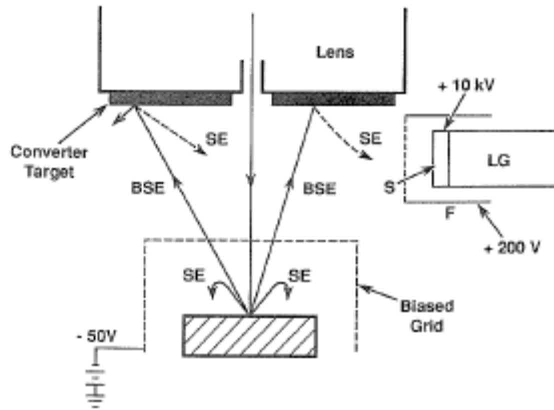
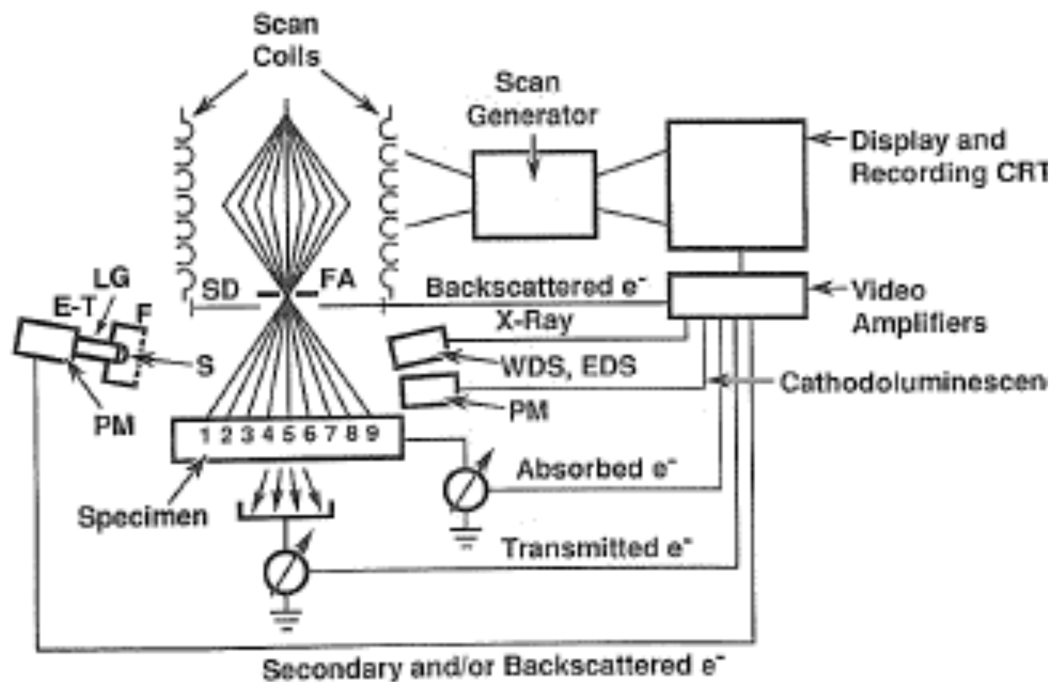


Image Formation



Electron gun

Condensor apertune

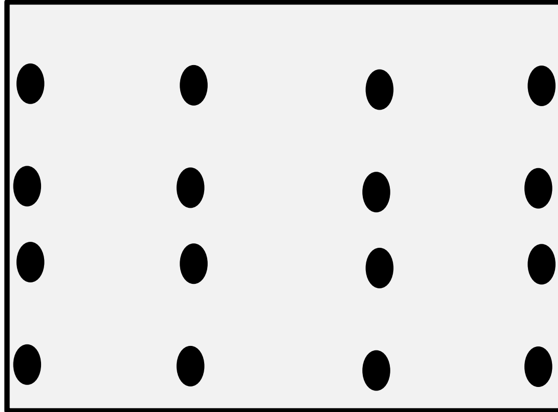
Objective apertune

Objective lens

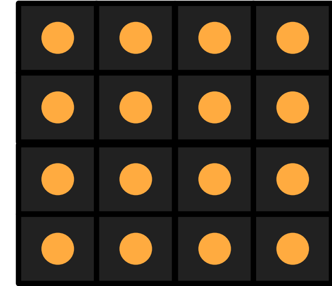
Projector lenses

Fluorescent screen

Image formation in a SEM



Beam scan points



Pixels on monitor

1 – to – 1 correspondence between beam points and pixels on screen

Pixels on monitor is set to gray scale. For instance, a range is set (0 – black and 256 for white).

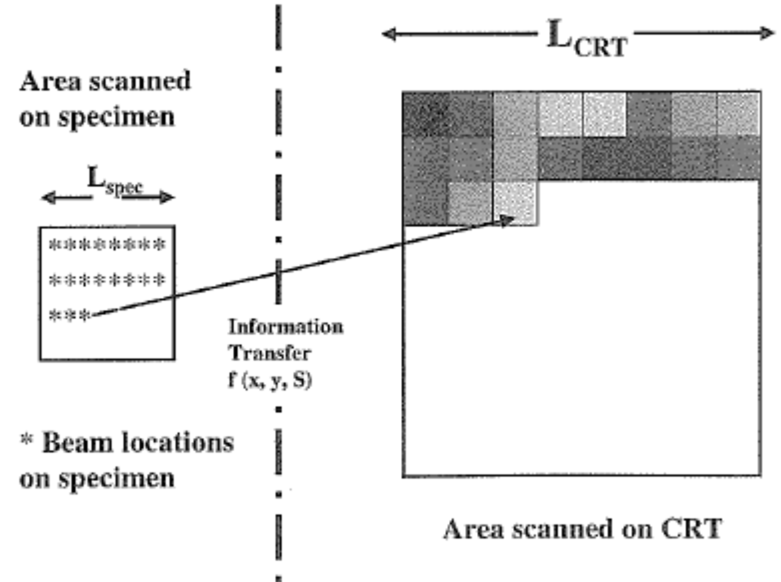
Depending on the intensity obtained in the detector, the gray scale is set.

A scanning electron microscope is mapping operation

- Each point on the sample is converted to a/sequence of points on display device
- Geometric rations need to be retained for faithfu reproduction
- The mapping needs to be synchronous to be relevant

The image – grayscale bitmap can be stored digitall and recovered at need.

Each point on the sample is 'scanned' by rastering the electron beam on the sample sites.



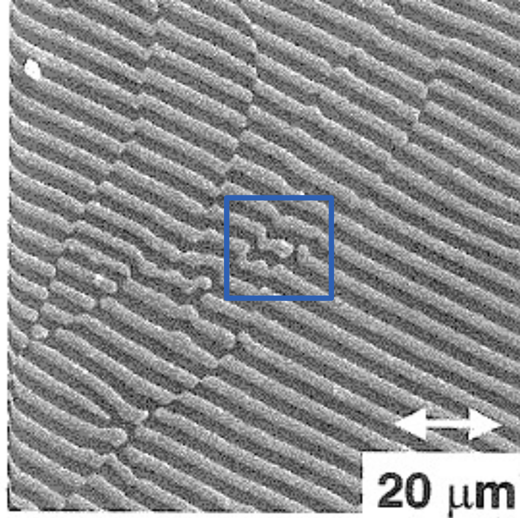
Magnification

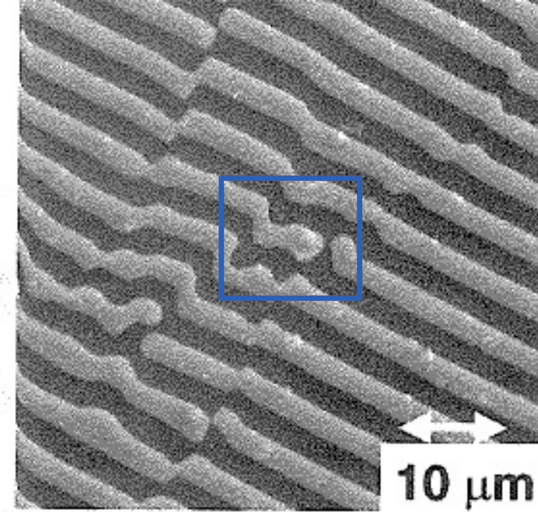
Density of pixels on display device is fixed.

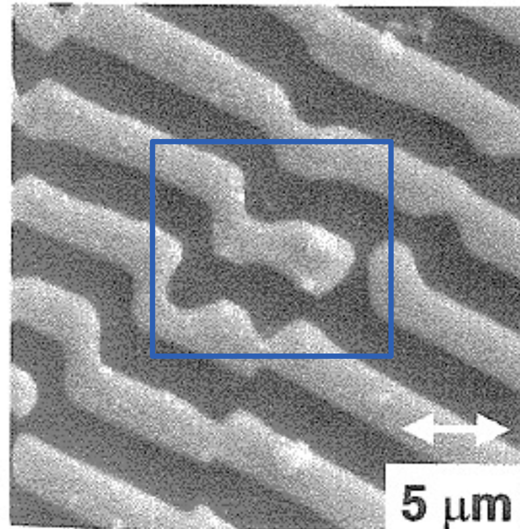
Magnification is fixed by the length of scan region.

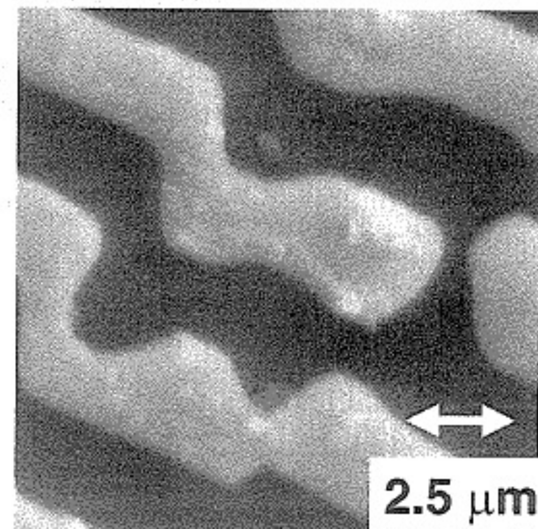
Thus, as the magnification is increased, the spot size needs to reduce.

While changing magnification from 100x – 10000x, the area of the scan reduces 10^4 times !





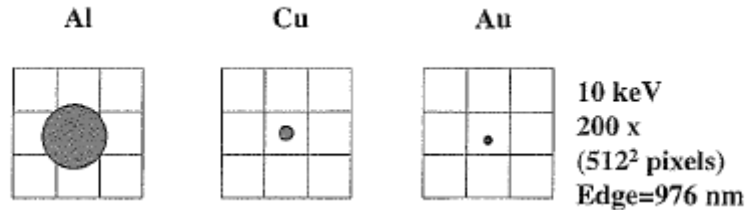




We define a pixel element $D_E = \frac{L_S}{N_P}$ where L_S is the length of scan and N_P is the number of points along the length

Say, we are looking at display resolution of 1024x1024, as the magnification is increased, the pixel element gets narrower.

To have minimal overlap with neighbouring pixels, D_E should be equal to the area from which electron signals are detected



The pixel size (square length) is determined by the magnification. The region from which the signal is obtained in SEM depends on excitation potential and the sample material

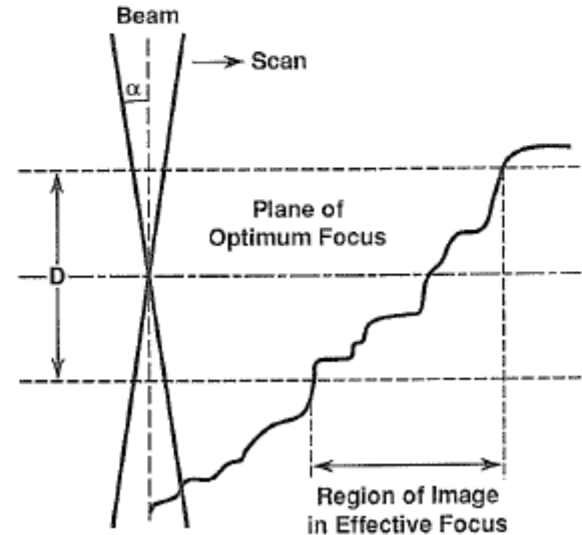
Depth of Field

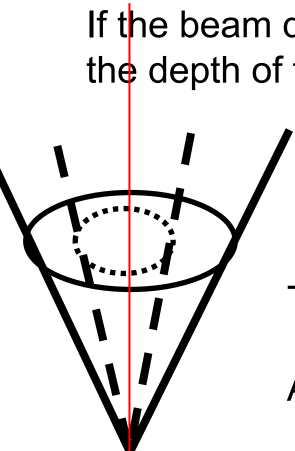
The electron beam is converted to a point with angle α

We bring the sample to focus, when the surface is placed at the smallest beam diameter.

However, if the sample is rough, there can be regions where the beam is not in its sharpest form.

If the beam diameter becomes closer to the pixel size, we define the depth of focus there.





Thus, the deciding factor for depth of field is the convergence angle α

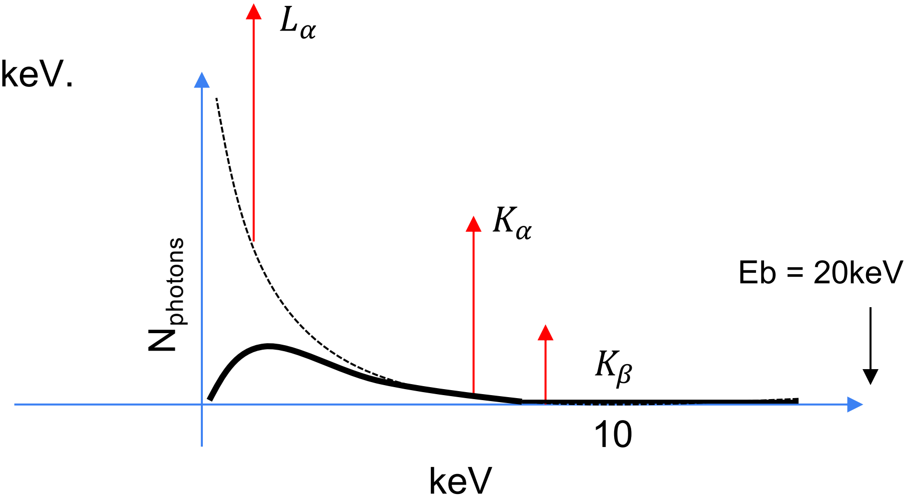
A smaller aperture opening, leads to smaller convergence angle and a higher depth of field!

Remember, the electrons in SEM are accelerated to few keV.

Electrons from beam lose energy by elastic collision.

The lost energy in the sample can be emitted back as photons

$$\Delta E = h\nu$$

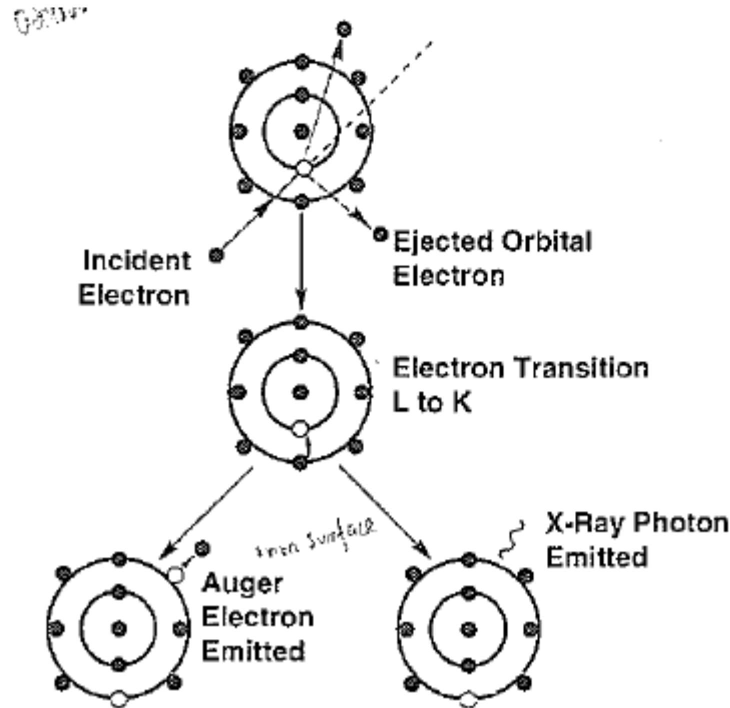


Take the example of Cu, an incident electron beam with energy 20 keV, will give a photon emission spectrum as shown in the dotted black line.

However, a typical measured photon spectrum looks a bit difference. Guess why ?

Allowed transitions in an atom

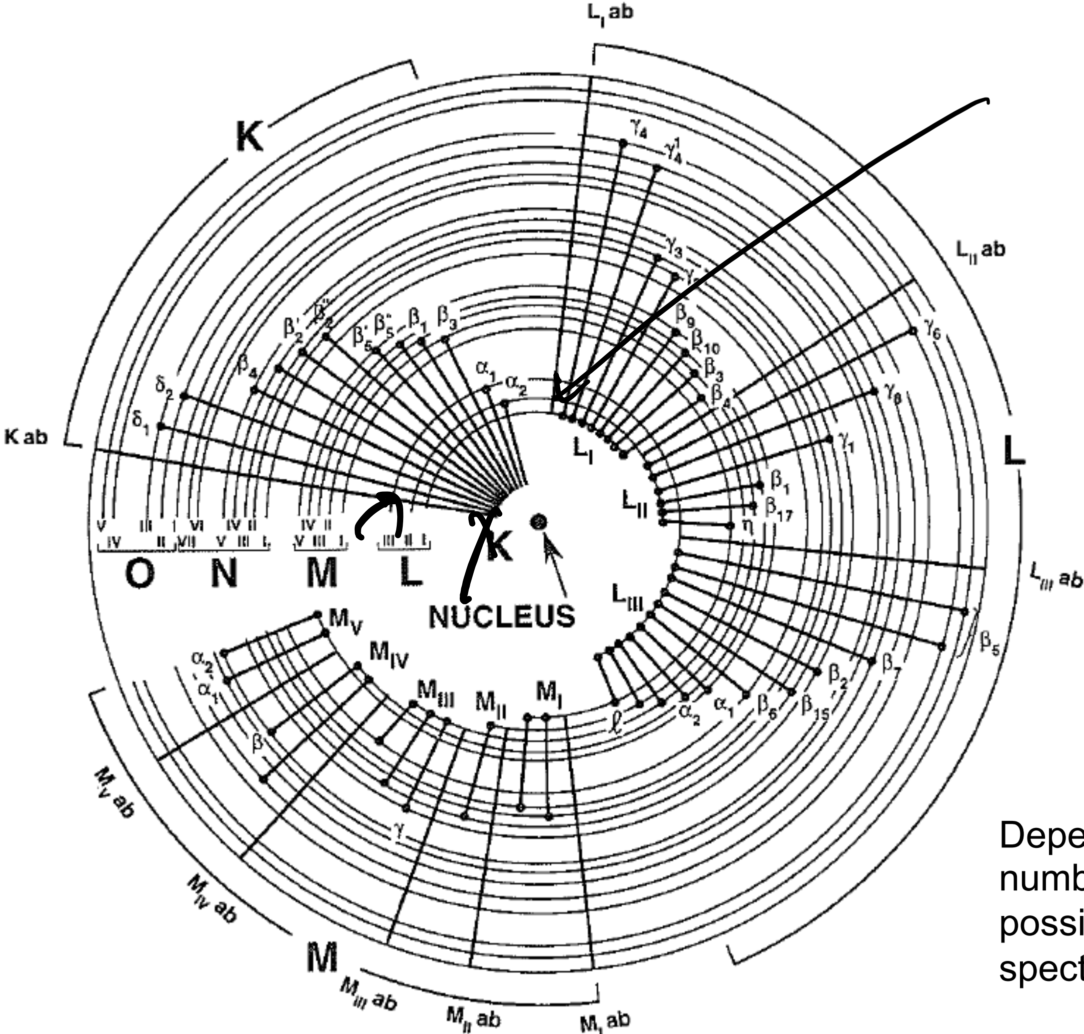
If interested, read about Moseley, Barkla, et. al.,



Auger – when energy is lost to another electron in the atom

Characteristic X-rays can be emitted when the electrons are swapped from outer to the core electrons

Origin of atomic lines



Depending on atomic number, the number of possible lines in the Xray spectrum also increases.

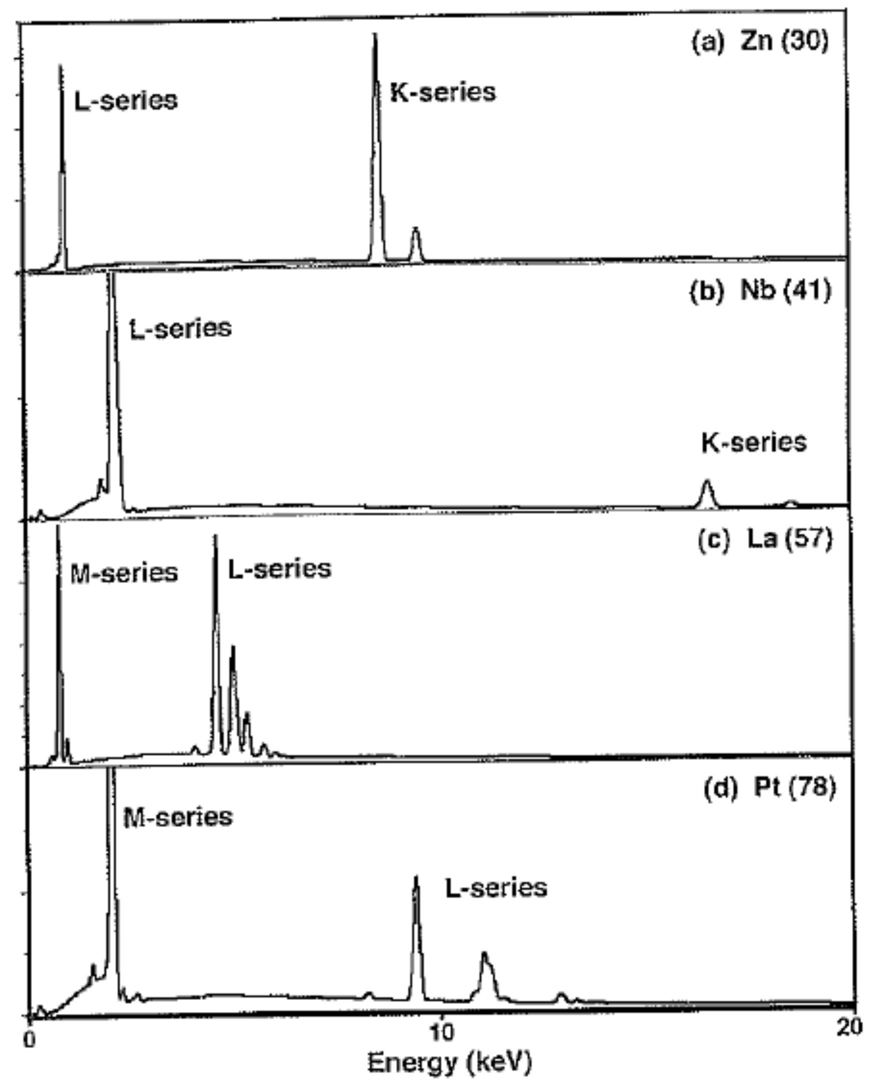
Peaks from different elements

Typical characteristic peaks have width much smaller than the peak energy

$$\Delta E \ll E_{peak}$$

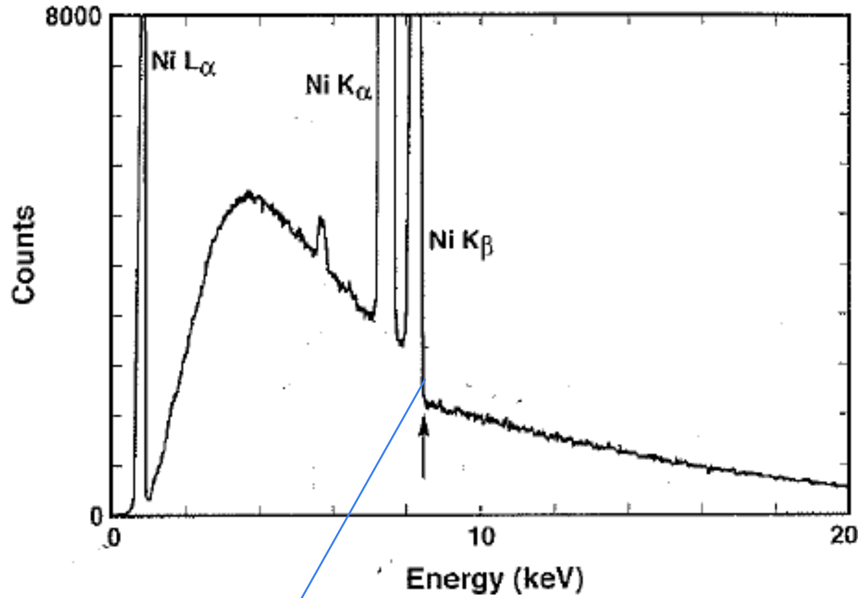
While the L line is singular for smaller element, The lines start to spread out as the Z increases.

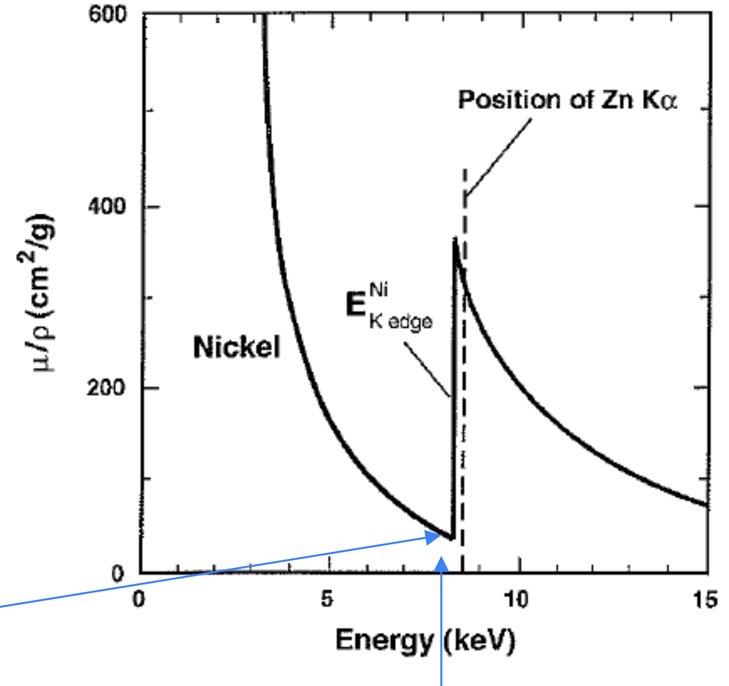
If the energy of the X-ray can be measured along with the number of photons (spectrometer), the atoms under the electron beam can be uniquely identified.



Emission spectra from Ni for excitation at 40 keV

Mass absorption coefficient



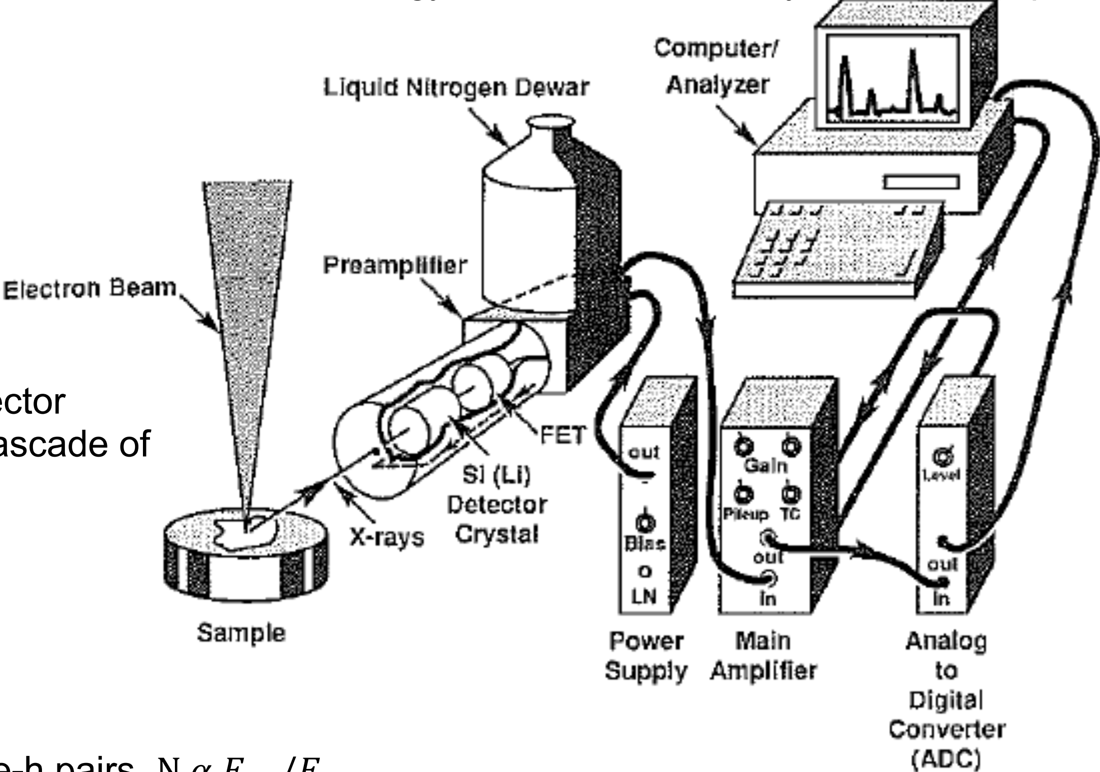


Sudden dip in Bremsstrahlung

This is due to strong increase in absorption at this point.

Typical Energy Dispersive Spectroscope

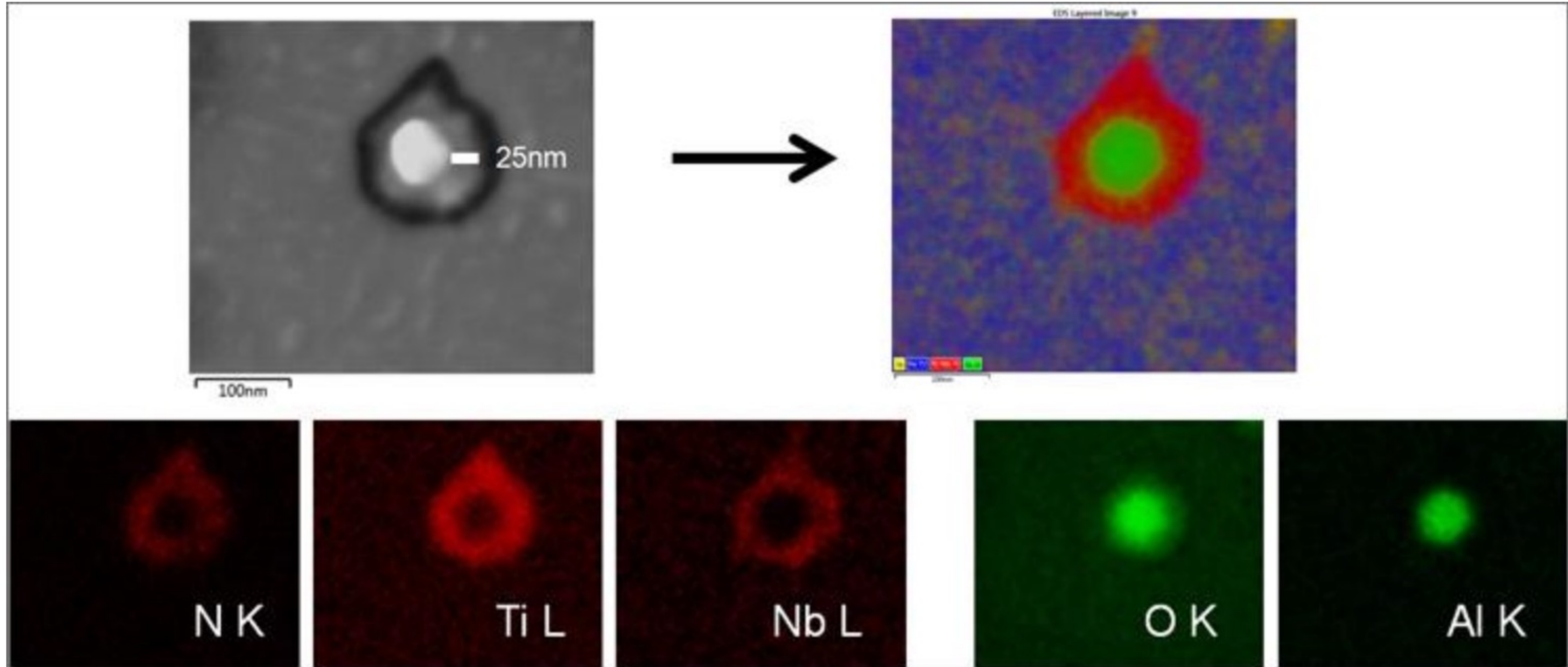
Energy dispersion is used to measure the energy of the incident X-Ray from the sample



A doped Si *p*-*i*-*n* detector
Is used to create a cascade of
e-h pairs

The total number of e-h pairs $\mathrm{N}\ \alpha\ E_{ph}/E_g$

Elemental Mapping in EDS



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